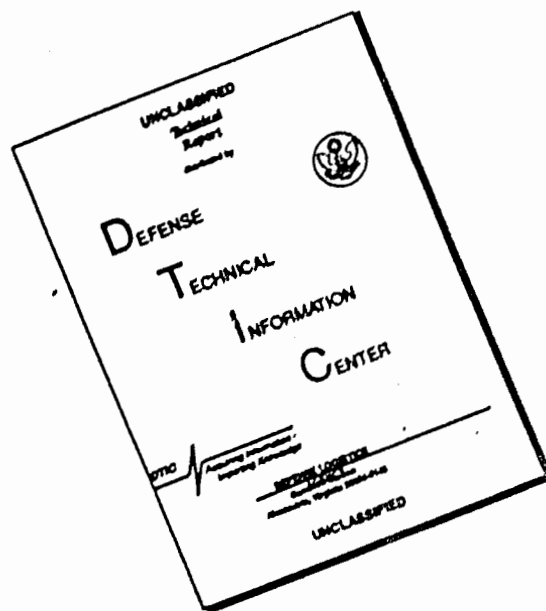


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APPENDIX B--PROJECT QUALITY CONTROL PLAN;

APPENDIX C--COMPUTER PROGRAM FOR REDUCTION OF HPLC SCREEN DATA; and

APPENDIX D--ANALYTICAL METHODS.

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APPENDIX A
LITERATURE SEARCH ABSTRACTS

APPENDIX A
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COMPOUND: PAHs

COMPOUNDS: UDMH
HMX
PETN



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22/5/1

89203658 CA08924203658F

ANALYSIS OF CARBON VERSUS RESIN

AUTHOR: SZACHTA, JAMES M.

LOCATION: CHEM. SYST. LAB., ARMY ARMAMENT RES. DEV. COMMAND, ABERDEEN PROVING GROUND, MD. X

SECTION: CA060002, CA050XXX PUBL CLASS: TECH REP

JOURNAL: U. S. NTIS, AD REP. CODEN: XADRCH PUBL: 78 ISSUE:

AD-A053863, PAGES: 48 PP.

CITATION: GOV. REP. ANNOUNCE. INDEX (U. S.) 1978, 78(16), 336

AVAIL: NTIS

IDENTIFIERS: PINK WATER TREATMENT ADSORBENT, TNT ADSORBENT PINK WATER, RDX ADSORBENT PINK WATER, HMX ADSORBENT PINK WATER, TETRYL ADSORBENT PINK WATER, ACTIVATED CARBON TREATMENT PINK WATER, AMBERLITE XAD4 TREATMENT PINK WATER, AMMUNITION PRODN WASTEWATER TREATMENT

CA08924203658F

DESCRIPTORS: ADSORBENTS; WASTEWATER TREATMENT; ADSORPTION

IDENTIFIERS: REMOVAL PINK ACTIVATED CARBON AMBERLITE XAD4 BIOLOGICAL STUDIES AMMUNITION XAD 4

CAS REGISTRY NUMBERS: 118-96-7 121-82-4 479-45-8 2691-41-0 7440-44-0
37380-42-0 *hmx*

22/5/2

86184123 CA08625184123X

EFFECTS OF POLLUTANTS ON EMBRYOS AND LARVAE OF FROGS: A SYSTEM FOR EVALUATING TERATOGENIC EFFECTS OF COMPOUNDS IN FRESH WATER ENVIRONMENTS X

AUTHOR: GREENHOUSE, GERALD A.

LOCATION: UNIV. CALIFORNIA, IRVINE, CALIF.

SECTION: CA004003 PUBL CLASS: TECH REP

JOURNAL: AEROSP. MED. RES. LAB., (TECH. REP.) AMRL-TR (U. S.) CODEN:

AMRLD3 PUBL: 75 ISSUE: AMRL-TR-125, PAGES: 493-511

IDENTIFIERS: FROG WATER POLLUTION ANALYSIS

CA08625184123X

DESCRIPTORS: TERATOGENESIS; WATER POLLUTION; RANA PIPIENS; XENOPUS LAEVIS

IDENTIFIERS: FROG EMBRYO ANAL BIOLOGICAL STUDIES POLLUTANTS ORG COMPOS EVALUATION POLLUTANT TERATOGENESIS

CAS REGISTRY NUMBERS: 90-30-2 25619-54-9 101-67-7 302-01-2 60-34-4
540-73-8 57-14-2

22/5/3

36126961 CA08618126961H

DETERMINATION AND MONITORING OF SOME ORGANIC EXPLOSIVES IN NATURAL AND EFFLUENT WATER BY SINGLE-SWEEP POLAROGRAPHY

AUTHOR: WHITMACK, GERALD C.

LOCATION: NAV. WEAPONS CENT., CHINA LAKE, CALIF.

SECTION: CA061002, CA050XXX, CA060XXX, CA079XXX PUBL CLASS: CONF PROC

JOURNAL: IDENTIF. ANAL. ORG. POLLUT. WATER, (CHEM. CONGR. NORTH AM. CONT.), 1ST CODEN: 33SXAJ PUBL: 76 PAGES: 265-79 MEETING DATE: 75

PUBLISHER: ANN ARBOR SCI. ADDRESS: ANN ARBOR, MICH

AVAIL: KEITH, LAWRENCE H

IDENTIFIERS: ORG EXPLOSIVE DETN WATER, WASTEWATER EXPLOSIVE DETN, POLAROG DETN ORG EXPLOSIVE WATER

CA08618126961H

IDENTIFIERS: ESTERS DETN NATURAL WATER WASTEWATER SINGLE SWEEP POLAROG ANALYSIS EXPLOSIVE

CAS REGISTRY NUMBERS: 6423-43-4 118-96-7 121-82-4 2691-41-0 7732-18-5

22/5/4

86026591 CA08605026591U

THE EVALUATION OF THE TOXIC EFFECTS OF CHEMICALS IN FRESH WATER BY USING FROG EMBRYOS AND LARVAE

AUTHOR: GREENHOUSE, GERALD

LOCATION: DEP. ANAT., UNIV. CALIFORNIA, IRVINE, CALIF.

SECTION: CA004001 PUBL CLASS: JOURNAL

JOURNAL: ENVIRON. POLLUT. CODEN: ENVPAP PUBL: 76 SERIES: 11 ISSUE: 4 PAGES: 303-15

IDENTIFIERS: POLLUTANT TOXICITY DETN FROG, DEVELOPMENT FROG WATER POLLUTION

CA08605026591U

DESCRIPTORS: WATER POLLUTION; RANA PIPPIENS; XENOPUS LAEVIS; DEVELOPMENT

IDENTIFIERS: DETN TOXICITY POLLUTANT FROG EMBRYOS LARVAE BIOLOGICAL STUDIES DETG INDICATOR

CAS REGISTRY NUMBERS: 90-30-2 25619-54-9 101-67-7 302-01-2 60-34-4

57-14-7P

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93173550 CA09318173550C

CHEMICAL DETECTOR UTILIZING AN ELECTROLYTIC GEL

AUTHOR: LOTZE, THOMAS H.

LOCATION: USA

SECTION: CA061002, CA079XXX PUBL CLASS: PAT

JOURNAL: U.S. CODEN: USXXAM PUBL: 800610 PAGES: 4 PP.

PATENT NO: 4207162 APPLIC NO: 18154 DATE: 790307 CLASS: 204-195R
601N27/46

ASSIGNEE: CAMBRIDGE INSTRUMENT CO., INC.

IDENTIFIERS: HYDRAZINE DETN BOILER WATER APP, ELECTROLYTIC GEL HYDRAZINE
DETN APP, SILVER OXIDE GEL WATER ANALYZER

CA09318173550C P

IDENTIFIERS: ANALYSIS DETN BOILER WATER ELECTROCHEM APP HYDRAZINE ETHERS
ELECTROLYTE GEL CONTG

CAS REGISTRY NUMBERS: 302-01-2 7732-18-5 9004-62-0 20667-12-3

23/5/2

93137581 CA09314137581E

THE OXIDATION STATE DIAGRAM - A POTENTIAL TOOL FOR STUDYING REDOX
CHEMISTRY IN SEA WATER

AUTHOR: WONG, GEORGE T. F.

LOCATION: INST. OCEANOGR., OLD DOMINION UNIV., NORFOLK, VA, 23508, USA

SECTION: CA061001 PUBL CLASS: JOURNAL

JOURNAL: MAR. CHEM. CODEN: MRCHDD PUBL: 80 SERIES: 9 ISSUE: 1

PAGES: 1-12

IDENTIFIERS: OXIDN STATE DIAGRAM REDOX SEAWATER, NITROGEN OXIDN STATE
DIAGRAM SEAWATER, MANGANESE OXIDN STATE DIAGRAM SEAWATER

CA09314137581E

DESCRIPTORS: REDOX REACTION; VALENCE; WATERS, OCEAN

IDENTIFIERS: USES MISCELLANEOUS OXIDN STATE DIAGRAM DETN NITROGEN SYSTEM
CHEM SEAWATER MANGANESE OCCURRENCE STUDY DIAGRAMS SYSTEMS

CAS REGISTRY NUMBERS: 302-01-2 1313-13-9 1332-62-3 1336-21-6 7439-96-5
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93116717 CA09312116717P

ULTRASOUND LEVEL-METER FOR MEASURING PROPELLANT LEVELS IN THE TANKS OF
ARIANE FIRST AND SECOND STAGES X

AUTHOR: DEMARIS, JEAN CLAUDE; DEOM, ALAIN
LOCATION: GROUPE RECH., OFF. NATL. ETUD. RECH. AEROSP., CHATILLON, FR.
SECTION: CA050002 PUBL CLASS: JOURNAL
JOURNAL: RECH. AEROSP. CODEN: REARAU PUBL: 80 SERIES: 194,
PAGES: 9-22 LANGUAGE: FR
IDENTIFIERS: PROPELLANT LEVEL ROCKET TANK

CA09312116717P

DESCRIPTORS: PROPELLANTS; SOUND AND ULTRASOUND; CHEMICAL AND PHYSICAL
EFFECTS.

IDENTIFIERS: DETN LEVEL ROCKET TANKS USES MISCELLANEOUS VALUE
CAS REGISTRY NUMBERS: 57-14-7 10102-44-0

22/5/8

93100878 CA09310100878F

DETECTOR FOR FUMES OF HYDRAZINE AND ITS DERIVATIVES

AUTHOR: CROOMES, EDGAR F.; MURFREE, JAMES A.

LOCATION: USA

SECTION: CA059003, CA079XXX PUBL CLASS: PAT

JOURNAL: U.S. CODEN: USXXAM PUBL: 800429 PAGES: 4 PP.

PATENT NO: 4200608 APPLIC NO: 915706 DATE: 780615 CLASS: 422-97,

601N27/02, 601N27/16, 601N31/10

ASSIGNEE: UNITED STATES DEPT. OF THE ARMY

IDENTIFIERS: HYDRAZINE DETN AIR SENSOR; METHYLHYDRAZINE DETN AIR SENSOR,
DIMETHYLHYDRAZINE DETN AIR SENSOR

CA09310100878F

DESCRIPTORS: AIR ANALYSIS

IDENTIFIERS: DETN APP USES MISCELLANEOUS IRIIDIUM CONTG DETECTORS
HYDRAZINE DERIVS ALUMINA PELLET

CAS REGISTRY NUMBERS: 57-14-7 60-34-4 302-01-2 1344-28-1 7439-88-5

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93097901 CA09310097901A

ANALYSIS OF EXPLOSIVES BY NEGATIVE ION CHEMICAL IONIZATION MASS
SPECTROMETRY X

AUTHOR: YINON, JEHUDA

LOCATION: DEP. ISOT. RES., WEIZMANN INST. SCI., REHOVOT, 76100, ISRAEL

SECTION: CA050003, CA080XXX PUBL CLASS: JOURNAL

JOURNAL: J. FORENSIC SCI. CODEN: JFSCAS PUBL: 80 SERIES: 25

ISSUE: 2 PAGES: 401-7

IDENTIFIERS: EXPLOSIVE ANALYSIS ANION MASS SPECTROMETRY

CA09310097901A

DESCRIPTORS: EXPLOSIVES; MASS SPECTROSCOPY; NEG.-ION; CHEM.-IONIZATION

IDENTIFIERS: ESTERS

CAS REGISTRY NUMBERS: 55-63-0 78-11-5 118-96-7 121-82-4 2691-41-0

ESTN

22/5/4

93188652 CA09320188652M

TESTING PROCEDURE TO DETERMINE THE DEFLAGRATION PROPERTIES OF EXPLOSIVES X

AUTHOR: BIGOURD, J.; MICHOT, C.

LOCATION: CENT. ETUD. RECH. CHARBONAGES, VERNEUIL-EN-HALATTE, F-60550, FR.

SECTION: CA050003 PUBL CLASS: JOURNAL

JOURNAL: PROPELLANTS EXPLOS.

CODEN: PREXD4

PUBL: 80

SERIES: 5

ISSUE: 2-3

PAGES: 34-6

LANGUAGE: FR

IDENTIFIERS: DEFLAGRATION TEST EXPLOSIVE

CA09320188652M

DESCRIPTORS: COMBUSTION; DEFLAGRATION; EXPLOSIVES

IDENTIFIERS: ESTERS DETN USES MISCELLANEOUS COMPOUNDS

CAS REGISTRY NUMBERS: 55-63-0 78-11-5 118-96-7 2691-41-0 6484-52-2 7790-98-9 22113-87-7

DETN

22/5/5

93168765 CA09318168765X

CALIBRATION AND DATA PROCESSING IN HIGH SPEED GEL PERMEATION CHROMATOGRAPHY X

AUTHOR: KOHN, E.; ASHCRAFT, R. W.

LOCATION: DEV. DIV., MASON AND HANGER-SILAS MASON CO., INC., AMARILLO, TX, USA

SECTION: CA035005 PUBL CLASS: JOURNAL

JOURNAL: CHROMATOGR. SCI.

CODEN: CHGSAL

PUBL: 77

SERIES: 8

ISSUE: LIQ. CHROMATOGR. POLYM. RELAT. MATER. PAGES: 105-20

IDENTIFIERS: GEL PERMEATION CHROMATOG INTERNAL STD, MOL WT POLYSTYRENE CHROMATOG STD, DATA PROCESSING GEL CHROMATOG POLYMER, CALCN GEL PERMEATION CHROMATOG POLYMER

CA09318168765X

DESCRIPTORS: AIR; CHROMATOGRAPHY; GEL; DATA; MOLECULAR WEIGHT

IDENTIFIERS: INTERNAL STDS HIGH SPEED PERMEATION WT DETN POLYSTYRENE POLYMERS PROCESSING RELATION

CAS REGISTRY NUMBERS: 95-50-1 2691-41-0 9003-53-6 33086-17-8

22/5/6

93119534 CA09312119534U

DETECTION OF HYDRAZINE

AUTHOR: STETTER, JOSEPH R.

LOCATION: USA

SECTION: CA059001, CA079XXX PUBL CLASS: PAT

JOURNAL: U.S.

CODEN: USXXAM

PUBL: 800506

PAGES: 6 PP.

PATENT NO: 4201634

APPLIC NO: 916296

DATE: 780616

CLASS: 204-1T,

601N27/46

ASSIGNEE: ENERGETICS SCIENCE, INC.

IDENTIFIERS: HYDRAZINE DETECTION AIR ELECTROCHEM APP

CA09312119534U

P

DESCRIPTORS: AIR ANALYSIS

IDENTIFIERS: DETECTION ELECTROCHEM APP HYDRAZINE DERIV

CAS REGISTRY NUMBERS: 57-14-2 60-34-4 ~~60-34-4~~ and ~~60-34-4~~

Air Detn.

22/5/1

94010734 CA09402010734X

ANALYSIS OF VOLATILE AMINES BY GC

AUTHOR: RAULIN, F.; PRICE, P.; PONNAMPERUMA, C.

LOCATION: UNIV. PARIS-VAL, FR.

SECTION: CA080004 PUBL CLASS: JOURNAL

JOURNAL: AM. LAB. (FAIRFIELD, CONN.)

CODEN: ALBYBL PUBL: 80

SERIES: 12 ISSUE: 10 PAGES: 45, 47-8, 50-1

IDENTIFIERS: VOLATILE AMINE ANALYSIS GAS CHROMATOG, HYDROCARBON SEPN
AMINE GAS CHROMATOG, NITRILE SEPN AMINE GAS CHROMATOG

CA09402010734X

DESCRIPTORS: AMINES, ANALYSIS; CHROMATOGRAPHY, GAS; HYDROCARBONS, ANALYSIS;
NITRILES, ANALYSIS

IDENTIFIERS: RELATIVE MOLAR RESPONSE SEPN VOLATILE

CAS REGISTRY NUMBERS: 57-14-7 60-34-4 74-89-5 75-04-7 75-31-0 75-50-3
75-55-8 75-64-9 78-81-9 78-90-0 107-10-8 107-11-9 107-15-3 109-73-9
109-76-2 109-89-7 124-40-3 340-73-8 616-24-0 624-78-2 765-30-0 2372-88-5
2450-71-7

22/5/2

94010729 CA09402010729Z

A LASER PHOTOACOUSTIC DEVICE AND ITS APPLICATIONS IN THE ANALYSIS OF SOME
GASES

AUTHOR: CHEN, CHUAN-WEN; MING, CHANG-JIANG; LIU, YAO-TIAN; WANG, LIAN-JIE
; LI, ZHEN-XIANG; XU, JUN

LOCATION: CHANGCHUN INST. APPL. CHEM., ACAD. SIN., CHANGCHUN, PEOP. R.
CHINA

SECTION: CA080002, CA073XXX PUBL CLASS: JOURNAL

JOURNAL: CHI KUANG

CODEN: CHIKDA

PUBL: 79

SERIES: 6

ISSUE: 10

PAGES: 38-41 LANGUAGE: CH

IDENTIFIERS: LASER PHOTOACOUSTIC SPECTROMETER GAS ANALYSIS, CARBON
DIOXIDE LASER PHOTOACOUSTIC SPECTROMETER

CA09402010729Z

DESCRIPTORS: GAS ANALYSIS; LASER RADIATION, CHEMICAL AND PHYSICAL EFFECTS;
SPECTROCHEMICAL ANALYSIS, OPTOACOUSTIC, LASER-INDUCED; SPECTROMETERS, OPTOACO-
USTIC

IDENTIFIERS: DETN PHOTOACOUSTIC SPECTROMETRY CARBON DIOXIDE SOURCE GASES

CAS REGISTRY NUMBERS: 57-14-7 67-56-1 71-43-2 74-85-1 79-01-6 106-99-0
115-07-1

22/5/3

93222670 CA09324222670C

DYNAMIC MECHANICAL ANALYSIS OF LX-13, AN EXTRUDABLE EXPLOSIVE

AUTHOR: FLOWERS, G. L.

LOCATION: MASON AND HANGER-SILAS MASON CO., INC., AMARILLO, TX, USA

SECTION: CA050003 PUBL CLASS: TECH REP

JOURNAL: REPORT

CODEN: D3REP3

PUBL: 80

ISSUE: MHSMP-80-04,

PAGES: 34 PP.

CITATION: ENERGY RES. ABSTR. 1980, 5 (11), ABSTR. NO. 17983

AVAIL: NTIS

IDENTIFIERS: PETN SILOXANE EXTRUDABLE EXPLOSIVE

CA09324222670C

DESCRIPTORS: EXPLOSIVES; SILOXANES AND SILICONES, DI-ME, USES AND
MISCELLANEOUS

IDENTIFIERS: DYNAMIC MECH ANAL EXTRUDABLE PETN CONTG

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93094587 CA09309094587S

ANALYSIS OF INTRA- AND INTERMOLECULAR INTERACTIONS RELATING TO THE THERMOPHYSICAL BEHAVIOR OF .ALPHA.-, .BETA.-, AND .DELTA.-OCTAHYDRO-1,3,5,7-TETRANITRO-1,3,5,7-TETRAZOCINE

AUTHOR: BRILL, T. B.; REESE, C. D.

LOCATION: DEP. CHEM., UNIV. DELAWARE, NEWARK, DE, 19711, USA

SECTION: CA022008, CA050XXX PUBL CLASS: JOURNAL

JOURNAL: J. PHYS. CHEM. CODEN: JPCHAX PUBL: 80 SERIES: 84

ISSUE: 11 PAGES: 1376-80

IDENTIFIERS: TETRAZOCINE TETRANITRO STABILITY POLYMORPH

CA09309094587S

DESCRIPTORS: CONFORMATION AND CONFORMERS; CRYSTAL STRUCTURE-PROPERTY RELATIONSHIP; POTENTIAL ENERGY AND FUNCTION; THERMAL DECOMPOSITION

IDENTIFIERS: STABILITY FORMS OCTAHYDROTETRANITROTETRAZOCINE POLYMORPHS

CAS REGISTRY NUMBERS: 2691-41-0

22/5/11

93049727 CA09306049727B

NONDESTRUCTIVE CONTROL OF ELECTROEXPLOSIVE INTERFACE OF AN EXPLODED-CORD DETONATOR USING THE THERMAL RESPONSE METHOD

AUTHOR: KASSEL, C.; CHRETIEN, N.

LOCATION: CEA, SEVRAN, 93270, FR.

SECTION: CA050003 PUBL CLASS: TECH REP

JOURNAL: EUR. SPACE AGENCY, (SPEC. PUBL.) ESA SP CODEN: ESPUD4

PUBL: 80 ISSUE: ESA SP-144, EXPLOS. PYROTECH.-APPL. SPAT., PAGES:

163-9 LANGUAGE: FR

IDENTIFIERS: CORD DETONATOR EXPLOSIVE ELECTROTHERMAL ANALYSIS

CA09306049727B

DESCRIPTORS: DETONATORS, CORD; EXPLOSIVES

IDENTIFIERS: ANAL NONDESTRUCTIVE ELECTROTHERMAL

CAS REGISTRY NUMBERS: 78-11-5 PERM

22/5/12

92202642 CA09224202642W

USE OF A GLC CONCENTRATOR TO IMPROVE ANALYSIS OF LOW LEVELS OF AIRBORNE HYDRAZINE AND UNSYMMETRICAL DIMETHYLHYDRAZINE

AUTHOR: MAZUR, J. F.; PODOLAK, G. E.; HEITKE, B. T.

LOCATION: U.S. ARMY ENVIRON. HYG. AGENCY, ABERDEEN PROVING GROUND, MD, 21010, USA

SECTION: CA059001, CA079XXX, CA080XXX PUBL CLASS: JOURNAL

JOURNAL: AM. IND. HYG. ASSOC. J. CODEN: AIHAAP PUBL: 80 SERIES:

41 ISSUE: 1 PAGES: 66-9

IDENTIFIERS: AIRBORNE HYDRAZINE DETN GAS CHROMATOGRAPH, CONCENTRATOR LOW LEVEL DIMETHYLHYDRAZINE DETN, METHYLHYDRAZINE DETN AIR GAS CHROMATOGRAPH

CA09224202642W

DESCRIPTORS: AIR ANALYSIS

IDENTIFIERS: DETN GAS LIQ CHROMATOGRAPH PRECONCENTRATOR COLUMNS HYDRAZINE DIMETHYLHYDRAZINE

CAS REGISTRY NUMBERS: 57-14-7 302-01-2 ...

92183100 CA09222183100Z
ELEMENTARY THEORY OF EXPLOSIONS FOR EJECTIONS AND THEIR SIMULATION USING
ARTIFICIAL GRAVITY
AUTHOR: BARSANAEV, S. B.; GUDOVICH, V. TS.; RASSHIKHIN, K. A.;
STANYUKOVICH, K. P. X
LOCATION: INST. FIZ. MEKH. GORN. POROD, FRUNZE, USSR
SECTION: CA050004 PUBL CLASS: JOURNAL
JOURNAL: DOKL. AKAD. NAUK SSSR CODEN: DANKAS PUBL: 79 SERIES:
249 ISSUE: 1 PAGES: 97-100 (PHYS.) LANGUAGE: RUSS
IDENTIFIERS: BLASTING EXPLOSIVE STRIPPING POWER

CA09222183100Z
DESCRIPTORS: BLASTING; DETONATION
IDENTIFIERS: DETN STRIPPING POWER EXPLOSION EXPLOSIVES
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92165823 CA09220165873Z
DETONATION INITIATION BEHAVIOR OF SOME HMX/AP/A1 PROPELLANTS X
AUTHOR: DICK, J. J.
LOCATION: LOS ALAMOS SCI. LAB., UNIV. CALIFORNIA, LOS ALAMOS, NM, 87545,
USA
SECTION: CA050002 PUBL CLASS: JOURNAL
JOURNAL: COMBUST. FLAME CODEN: CBFMAQ PUBL: 80 SERIES: 37
ISSUE: 1 PAGES: 95-9
IDENTIFIERS: HMX PROPELLANT DETONATION INITIATION

CA09220165873Z
DESCRIPTORS: DETONATION; PROPELLANTS
IDENTIFIERS: DETN INITIATION RELATION CONCN REACTIONS HMX COMPOUNDS CONTG
ALUMINUM AMMONIUM PERCHLORATE
CAS REGISTRY NUMBERS: 2691-41-0 7429-90-5 7790-98-9

22/5/15
92149469 CA09218149469S
DETERMINATION METHOD OF THE POWER OF DETONATOR FUSES X
AUTHOR: PRIOR, J.
LOCATION: DYNAMIT NOBEL A.-G., TROISDORF, FED. REP. GER.
SECTION: CA050003 PUBL CLASS: JOURNAL
JOURNAL: EXPLOSIFS CODEN: EXPLA9 PUBL: 78 SERIES: 31 ISSUE: 2
PAGES: 46-58 LANGUAGE: FR
IDENTIFIERS: DETONATOR PRIMER ENERGY DETN

CA09218149469S
DESCRIPTORS: DETONATORS; PRIMERS; EXPLOSIVE
IDENTIFIERS: CONTG DETN DETONATION ENERGY
CAS REGISTRY NUMBERS: 78-11-5

22/5/16

92127057 CA09215127057D

DETERMINATION OF DAMINOZIDE RESIDUES ON FOODS AND ITS DEGRADATION TO 1,1-DIMETHYLHYDRAZINE BY COOKING

AUTHOR: NEWSOME, WILLIAM H.

LOCATION: FOOD RES. DIV., DEP. NATL. HEALTH WELFARE, OTTAWA, ON, K1A 0L2, CAN.

SECTION: CA017002, CA005XXX PUBL CLASS: JOURNAL

JOURNAL: J. AGRIC. FOOD CHEM. CODEN: JAFCAU PUBL: 80 SERIES: 28
ISSUE: 2 PAGES: 319-21

IDENTIFIERS: DAMINOZIDE GAS CHROMATOG, APPLE DAMINOZIDE DETN, COOKING
APPLE DAMINOZIDE, HYDRAZINE APPLE DAMINOZIDE

CA09215127057D

DESCRIPTORS: APPLE; COOKING; GRAPE; PEACH; PLUM; TOMATO

IDENTIFIERS: FORMATION DAMINOZIDE HYDRAZIDES DETN FOOD GAS CHROMATOG
APPLES DECOMPN DIMETHYLHYDRAZINE

CAS REGISTRY NUMBERS: 57-14-7 1596-84-5

22/5/17

92081488 CA09210081488Q

ELECTROCHEMICAL DETERMINATION OF HYDRAZINE AND METHYL- AND 1,1-DIMETHYLHYDRAZINE IN AIR

AUTHOR: STETTER, J. R.; TELLEFSEN, K. A.; SAUNDERS, R. A.; DECORPO, J. J.

LOCATION: ENERGETICS SCI. DIV., BECTON DICKINSON AND CO., ELMSFORD, NY, 10523, USA

SECTION: CA059002, CA072XXX, CA079XXX, CA080XXX PUBL CLASS: JOURNAL

JOURNAL: TALANTA CODEN: TLNTA2 PUBL: 79 SERIES: 26 ISSUE: 9
PAGES: 799-804

IDENTIFIERS: HYDRAZINE DETN AIR ELECTROCHEM CELL, METHYLHYDRAZINE DETN
AIR ELECTROCHEM CELL

CA09210081488Q

DESCRIPTORS: AIR ANALYSIS; ELECTROLYTIC CELLS

IDENTIFIERS: DETN ELECTROCHEM HYDRAZINE METHYLHYDRAZINE HYDRAZINES

CAS REGISTRY NUMBERS: 57-14-7 60-34-4 302-01-2

22/5/18

92025169 CA09204025169Z

HIGH-RESOLUTION FOURIER TRANSFORM INFRARED SPECTROSCOPY FOR THE INVESTIGATION OF DECOMPOSITION GASES GENERATED BY AGING ORGANIC MATERIALS

AUTHOR: HAALAND, D. M.; RIVORD, G. E.

LOCATION: SANDIA LAB., ALBUQUERQUE, NM, USA

SECTION: CA050003 PUBL CLASS: TECH REP

JOURNAL: REPORT CODEN: D3REP3 PUBL: 79 ISSUE: SAND-79-0935C,
CONF-790632-6, PAGES: 6 PP.

CITATION: ENERGY RES. ABSTR. 1979, 4(17), ABSTR. NO. 45310

AVAIL: NTIS

IDENTIFIERS: AGING EXPLOSIVE LONG TERM, PETN LONG TERM AGING, HNS LONG
TERM AGING

CA09204025169Z

DESCRIPTORS: EXPLOSIVES; INFRARED SPECTRA, FOURIER-TRANSFORM

IDENTIFIERS: AGING DECOMPN GAS PRODUCT DETN LONG TERM FOURIER IR
SPECTROSCOPY ANALYSIS PROPELLANTS TRANSFER GASES

CAS REGISTRY NUMBERS: 78-11-5 124-38-9 630-08-0 7727-37-9 7732-18-5
10024-97-2 10102-43-9 10102-44-0 20062-22-0

COMPOUND: ATNBA

TNBA Literature Search.

1

5/5/2
36026591 CA08605026591U
THE EVALUATION OF THE TOXIC EFFECTS OF CHEMICALS IN FRESH WATER BY USING
FROG EMBRYOS AND LARVAE
AUTHOR: GREENHOUSE, GERALD
LOCATION: DE
? TS/5/1-5

5/5/1
91091604 CA09111091604T
3,6-BIS-SUBSTITUTED S-TETRAZINES
AUTHOR: GUITHER, WILLIAM D.; COBURN, MICHAEL D.; CASTLE, RAYMOND N.
LOCATION: UNIV. WISCONSIN, MENASHA, WI, 54952, USA
SECTION: CA028022 PUBL CLASS: JOURNAL
JOURNAL: HETEROCYCLES CODEN: HTCYAM PUBL: 79 SERIES: 12
ISSUE: 6 PAGES: 745-9
IDENTIFIERS: TETRAZINE DIPHENYL, BENZONITRILE CYCLOCONDENSATION HYDRAZINE
THIOSEMICARBAZIDE PHENYL CYCLOCONDENSATION, ANILINOTETRAZINE,
PHENYLFORMIMIDATE CYCLOCONDENSATION, AMINOTETRAZINE PICRYL

CA09111091604T
DESCRIPTORS: CYCLOCONDENSATION REACTION
IDENTIFIERS: REACTIONS THIOSEMICARBAZIDE METHYLATION PHENYLFORMIMINO
HYDRAZIDE HYDRAZINE TETRAZINE BENZONITRILES TETRAZINES DIAMINOTETRAZINE
OXIMATION DEHYDRATION REDN PREPN NITRATION OXIDATIVE DERIV ACETYLATION
PICRYL CHLORIDE
CAS REGISTRY NUMBERS: 74-88-4 79-19-6 88-88-0 100-47-0 302-01-2 364-44-3
606-34-8 619-24-9 873-62-1 874-90-8 4278-02-8P 5351-69-9 6830-78-0P
14141-66-3P 19617-90-4P 35600-34-1P 37841-25-1P 37932-43-7P 57508-53-9
59995-93-6P 71123-38-1P 71123-39-2P 71123-40-5P 71123-41-6P 71123-42-7P
71123-43-8P 71123-44-9P 71123-45-0P 71123-46-1P 71123-47-2P 71123-48-3P

5/5/2
91059620 CA09108059620J
THE RELATIONSHIP OF IMPACT SENSITIVITY WITH STRUCTURE OF ORGANIC HIGH
EXPLOSIVES. II. POLYNITROAROMATIC EXPLOSIVES
AUTHOR: KAMLET, M. J.; ADDOLPH, H. G.
LOCATION: WHITE OAK LAB., NAV. SURF. WEAPONS CENT., SILVER SPRING, MD,
20910, USA
SECTION: CA050003 PUBL CLASS: JOURNAL
JOURNAL: PROPELLANTS EXPLOS. CODEN: PREXD4 PUBL: 79 SERIES: 4
ISSUE: 2 PAGES: 30-4
IDENTIFIERS: NITROAROM EXPLOSIVE IMPACT SENSITIVITY STRUCTURE

CA09108059620J
DESCRIPTORS: EXPLOSIVES; MOLECULAR STRUCTURE-PROPERTY RELATIONSHIP
IDENTIFIERS: EXPLOSION IMPACT SENSITIVITY POLYNITROAROM COMPS
CAS REGISTRY NUMBERS: 82-71-3 88-89-1 99-35-4 118-96-7P 129-66-8 131-73-7
489-98-5 519-44-8 602-99-3 606-34-8 606-35-9 616-74-0 860-83-3 1150-40-9
1630-08-6 3698-54-2 4328-17-0 4433-16-3 5180-53-0 6093-29-4 6538-39-2
14184-98-6 14185-44-5 14185-47-8 17215-44-0 21985-87-5 22167-47-1
24577-68-2 37841-25-1 42449-44-5 60762-70-1 70862-23-6 70862-24-7
70862-25-8 70862-26-9 70862-27-0 70862-28-1 70862-29-2

T N B A

2

5/5/3

88050419 CA08807050419F

HETEROCYCLES IN ORGANIC SYNTHESIS. VIII. SODIUM 4,6-DIPHENYL-1-OXIDO-2-PYRIDONE: REAGENT FOR THE CONVERSION OF PRIMARY HALIDES INTO ALDEHYDES

AUTHOR: COOK, MICHAEL J.; KATRITZKY, ALAN R.; MILLET, GEORGE H.

LOCATION: SCH. CHEM. SCI., UNIV. EAST ANGLIA, NORWICH, ENGL.

SECTION: CA025015, CA027XXX, CA023XXX PUBL CLASS: JOURNAL

JOURNAL: HETEROCYCLES CODEN: HCYAM PUBL: 77 SERIES: 7 ISSUE:

1 PAGES: 227-30

IDENTIFIERS: ALDEHYDE AROM ALIPH, ALKOXYPYRIDONE PREPN DECOMPN, HYDROXYPYRIDONE ALKYLATION, PYRIDONE ALKOXY DECOMPN

CA08807050419F

DESCRIPTORS: ALDEHYDES, PREPARATION

IDENTIFIERS: REACTION HYDROXYPYRIDONE SODIUM SALT PREPN PRIMARY HALIDES

REACTIONS ALKYL THERMOLYSIS

CAS REGISTRY NUMBERS: 89-98-5P 100-11-8 100-39-0 100-52-7P 106-95-6
111-71-7P 123-72-8P 135-02-4P 591-97-9 606-34-8P 611-19-8 629-04-9
4170-30-3P 7176-28-5 7468-67-9P 22115-41-9 24964-64-5P 26478-91-1
26602-89-1 28188-41-2 52289-93-7 65218-74-8P 65218-75-9P 65218-76-0P
65218-77-1P 65218-78-2P 65218-79-3P 65218-80-6P 65218-81-7P 65218-82-8P
65218-83-9P 65218-84-0P 65218-85-1P 107-02-8P 555-16-8P

5/5/1

92093998 CA09211093998U

HETEROCYCLES IN ORGANIC SYNTHESIS. PART 25. REAGENTS FOR THE CONVERSION OF HALIDES INTO ALDEHYDES AND KETONES

AUTHOR: KATRITZKY, ALAN R.; COOK, MICHAEL J.; BROWN, S. BRUCE; CRUZ, RAYMUNDO; MILLET, GEORGE H.; ANANI, ALI

LOCATION: SCH. CHEM. SCI., UNIV. EAST ANGLIA, NORWICH, NR4 7TJ, ENGL.

SECTION: CA025015, CA023XXX, CA027XXX, CA028XXX PUBL CLASS: JOURNAL

JOURNAL: J. CHEM. SOC., PERKIN TRANS. 1 CODEN: JCPRB4 PUBL: 79
ISSUE: 10 PAGES: 2493-9

IDENTIFIERS: PYRIDINONE BENZYL OXY CONVERSION BENZALDEHYDE, BENZALDEHYDE, ALKANAL, QUINAZOLINONE BENZYL OXY CONVERSION BENZALDEHYDE

CA09211093998U

DESCRIPTORS: ALDEHYDES, PREPARATION; ALKYL HALIDES; ARALKYL BROMIDES; ARALKYL CHLORIDES; KETONES, PREPARATION

IDENTIFIERS: PREPN N ISOPROPOXYPYRIDONE QUINAZOLINONE INTERMEDIATES O ALKYLATION HYDROXYPYRIDONE HYDROXYQUINAZOLINONE DERIVS BENZYL OXYPYRIDONE REACTIONS ALKENYLATION ALLYLOXYPYRIDONE BENZHYDRYLOXYPYRIDONE HYDROXYLATION REACTION SODIUM BENZYL THERMOLYSIS PHOTOLYSIS CONVERSION VIA ALKOXYPYRIDONE ARALKOXYPYRIDONE

CAS REGISTRY NUMBERS: 67-64-1P 75-26-3 89-98-5P 98-86-2P 99-61-6P
100-11-8 100-39-0 100-52-7P 103-63-9 104-82-5 104-83-6 104-87-0P 104-88-1P
105-07-7P 106-95-6 107-02-8P 109-65-9 111-71-7P 119-61-9P 122-78-1P
123-72-8P 124-13-0P 135-02-4P 555-16-8P 585-71-7 591-97-9 606-34-8P
611-19-8 629-04-9 629-27-6 776-74-9 1212-07-3 3958-57-4 4170-30-3P
5162-44-7 5319-72-2 7176-28-5 7319-38-2P 7468-67-9P 17201-43-3 22115-41-9
24964-64-5P 26478-91-1 28188-41-2 52289-93-7 65218-74-8P 65218-75-9P
65218-76-0P 65218-77-1P 65218-78-2P 65218-79-3P 65218-80-6P 65218-81-7P
65218-82-8P 65218-83-9P 65218-84-0P 65218-85-1P 67927-04-2P 67927-05-3P
67927-06-4P 67927-07-5P 67927-08-6P 67927-09-7P 67927-10-0P 72158-35-1P
72158-36-2P 72158-37-3P 72805-13-1P 72805-14-2P 72805-15-3P 72805-16-4P
72805-17-5P 72805-18-6P 72805-19-7P 72805-20-0P 72805-21-1P 72805-22-2P
72805-23-3P 72805-24-4P 72805-25-5P 72805-26-6P 72805-27-7P 72805-28-8P
72812-98-7P 72812-99-8P

TNBA

3

5/5/4

37161145 CA08720161145N

USE OF MICELLAR SYSTEMS IN ANALYTICAL CHEMISTRY - THEIR APPLICATION TO THE SPECTROPHOTOMETRIC DETERMINATION OF SULFITE ION WITH ACTIVATED AROMATIC COMPOUNDS

AUTHOR: MINZE, WILLIE L.

LOCATION: CHEM. DEP., WAKE FOREST UNIV., WINSTON-SALEM, N. C.

SECTION: CA079006 PUBL CLASS: CONF PROC

JOURNAL: COLLOID INTERFACE SCI., (PROC. INT. CONF.), 50TH CODEN:

3SYRAB PUBL: 76 SERIES: 5, PAGES: 425-36

PUBLISHER: ACADEMIC ADDRESS: NEW YORK, N. Y

AVAIL: KERKER, MILTON

IDENTIFIERS: SULFITE DETN PHOTOMETRY, NITROAROM REAGENT SULFITE DETN, AROM NITRO REAGENT SULFITE DETN, MICELLE MEDIUM SULFITE DETN, NITROBENZENE REAGENT SULFITE DETN, NITROBENZALDEHYDE REAGENT SULFITE DETN, NITRONAPHTHALENE REAGENT SULFITE DETN

CA08720161145N

DESCRIPTORS: SULFITES; MICELLES; SURFACTANTS; CATIONIC

IDENTIFIERS: DETN SPECTROPHOTOMETRY SPECTRUM SPECTROPHOTOMETRIC

POLYNITROAROM COMPODS

CAS REGISTRY NUMBERS: 99-35-4 606-34-8 606-37-1 28995-89-3 29535-21-5 64385-47-3 64426-50-2 64426-51-3 57-09-0

5/5/5

86005295 CA08601005295K

REDUCTION OF 2,4,6-TRINITROBENZALDEHYDE BY SODIUM BOROHYDRIDE

AUTHOR: SOKOLOVA, V. A.; BOLDYREV, M. D.; GUDASPOV, B. V.

LOCATION: LENINGR. TEKHNOL. INST., LENINGRAD, USSR

SECTION: CA027022, CA024XXX PUBL CLASS: JOURNAL

JOURNAL: ZH. ORG. KHIM. CODEN: ZORKAE PUBL: 76 SERIES: 12

ISSUE: 7 PAGES: 1525-7 LANGUAGE: RUSS

IDENTIFIERS: REDN TRINITROBENZALDEHYDE, BENZALDEHYDE TRINITRO REDN, HEXANEMETHANOL TRINITRO, AZADAMANTANE METHANOL TRINITRO

CA08601005295K

DESCRIPTORS: REDUCTION

IDENTIFIERS: REDN SODIUM BOROHYDRIDE DTD TRINITROBENZALDEHYDE REACTIONS

DIFLUOROSTYRENE CHAIN LENGTHENING DIFLUOROSTYLLITHIUMS

CAS REGISTRY NUMBERS: 606-34-8 61103-61-5P 61103-62-6P 61103-63-7 61103-64-8P 16940-66-2 109-72-8 127-18-4 116-15-4

COMPOUNDS: IMPA
35DNP

phosphorus flame photometric detector response

- 94014907 CA094030149079
Structure and isotopic fractionation factors of complexes,
ANAL-
Author: Kreevoy, Maurice M.; Liang, Tai Ming
Location: Dep. Chem., Univ. Minnesota, Minneapolis, MN,
55456, USA
Section: CA023008 Pub Class: JOURNAL
Journal: J. Am. Chem. Soc. Coden: JACSAT Publ: 90
Series: 102 Issue: 10 Pages: 3315-22
Identifiers: isotopic exchange homoconjugate phenoxide,
fractionation isotopic heteroconjugate carboxylate, hydrogen
bond heteroconjugate carboxylate, potential function
heteroconjugate carboxylate, isotope effect deuterium
heteroconjugate carboxylate
- 93149529 CA093181495296
Thermal stability of 1,3-dinitrobenzene derivatives
Author: Kaderabek, Vladimir; Koudelkova, Vajsa; Myskova,
Vlasta
Location: Vys. Sk. Chemotechnol., Pardubice, Czech.
Section: CA023007 Pub Class: JOURNAL
Journal: Chem. Prum. Coden: CHEM44 Publ: 90
Series: 30 Issue: 5 Pages: 236-42 Language: Czech
Identifiers: LFER BTA nitrobenzene, thermal stability
dinitrobenzene
- 93108146 CA093111081468
Chemiluminescence method for the determination of nanogram
amounts of highly toxic alkylphosphates
Author: Fritsche, U.
Location: Fraunhofer-Inst., Toxikol., Aerosolforsch.,
Seibenberg-Graschoff, D-9046, Fed. Rep. Ger.
Section: CA004001, CA090111 Pub Class: JOURNAL
Journal: Anal. Chim. Acta Coden: AACAM Publ: 90
Series: 118 Issue: 1 Pages: 179-83
Identifiers: insecticide alkyl phosphate data
chemiluminescence
- 92236119 CA092362361196
Structure-response relationship of gas chromatography-flame
photometric detection to some organophosphorus compounds
Author: Szes, Samuel; Parker, George A.
Location: Res. Div., Chem. Syst. Lab., Aberdeen Proving
Ground, MD, 21010, USA
Section: CA090004 Pub Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 90
Series: 199 Issue: 2 Pages: 231-49
Identifiers: gas chromatog. organophosphorus detector
response, response flame photometric detector organophosphorus

92203316 CA092242033182

Analysis of phosphonic acids by ion chromatography

Author: Schiff, Leon J.; Pleva, Stephen G.; Server, Emory W.

Location: Chem. Syst. Lab., Dep. of the Army, Aberdeen

Proving Ground, MD, USA

Section: CA091003, CA0991XX, CA0991XX Pub Class: COW

PBOC

Journal: Ion Chromatogr. Anal. Environ. Pollut. Coden:

373MAD Publ: 79 Series: 2, Pages: 329-44

Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich

Avail: Swick, Eugene; Kull, James D

Identifiers: phosphonic acid analysis ion chromatog.

isopropyl methylphosphonate ion chromatog. ethyl

methylphosphonate ion chromatog. methylphosphonic acid ion

chromatog. groundwater phosphonic acid ion chromatog. urine

phosphonic acid ion chromatog

92069022 CA09207090222

Acid-base properties of substituted phenols and carboxylic

acids in nitromethane and water

Author: Korolev, B. A.; Kashkovskaya, E. I.

Location: Neuchno-Issled. Inst. Org. Poluprod. Krasitel, Moscow, USSR

Section: CA023008 Pub Class: JOURNAL

Journal: Zh. Obshch. Khim. Coden: ZOBKHA4 Publ: 79

Series: 49 Issue: 10 Pages: 2360-5 Language: Russ

Identifiers: phenol acidity nitromethane LFER, carboxylic

acid acidity solvent effect, solvent effect acidity LFER

92038734 CA09205038734F

Anticholinesterase properties of toxic phospho-organic

compounds. I. Determination of kinetic parameters of

anticholinesterase

Author: Choss, Jerzy; Glosak, Stanislaw

Location: Wojciech Akad. Tech., Warsaw, Pol.

Section: CA097003 Pub Class: JOURNAL

Journal: Biol. Wojek. Akad. Tech. Coden: BWATK Publ:

79 Series: 28 Issue: 8 Pages: 143-51 Language: Pol

Identifiers: cholinesterase serum inhibition kinetics,

organophosphate inhibition cholinesterase serum

9112563 CA09119152563
Gas chromatographic determination of phosphorus-containing pesticide metabolites via benzoylation
Author: Daughton, Christian G.; Cook, Alasdair M.; Alexander, Martin
Location: Dep. Agron., Cornell Univ., Ithaca, NY, 14853, USA
Section: CA095001 Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 79 Series: 51 Issue: 12 Pages: 1949-53
Identifiers: pesticide phosphorus gas chromatog

9112574 CA09119152574
Analysis of toxic alkyl phosphates
Author: Fritzsche, Ulrich
Location: Fed. Rep. Ger.
Section: CA004001 Publ Class: PAT
Journal: Ger. Offen. Coden: GUXJGX Publ: 780816
Pages: 10 pp. Language: Ger
Patent No: 2806046 Applic No: 2806046 Date: 780214
Class: G01N3/72
Assignee: Fraunhofer-Gesellschaft zur Foerderung der Angewandten Forschung e.V.
Identifiers: chemiluminescence nerve gas insecticide acaricide, organophosphate detn chemiluminescence EDTA chloride

91001345 CA09101001345
Phosphate and soil binding: factors limiting bacterial degradation of toxic phosphorus-containing pesticide metabolites
Author: Daughton, Christian G.; Cook, Alasdair M.; Alexander, Martin
Location: Dep. Agron., Cornell Univ., Ithaca, NY, 14853, USA
Section: CA095015 Publ Class: JOURNAL
Journal: Appl. Environ. Microbiol. Coden: AEMIDF Publ: 79 Series: 37 Issue: 3 Pages: 805-9
Identifiers: phosphate sorption phosphonate soil, bacteria phosphonate degradn phosphate, Pseudomonas phosphonate degradn phosphate

9014655 CA0901914655A
Biotransformation of phosphonate toxicants yields methans or ethans on cleavage of the carbon-phosphorus bond
Author: Daughton, C. G.; Cook, A. M.; Alexander, M.
Location: Dep. Agron., Cornell Univ., Ithaca, N. Y.
Section: CA004004 Publ Class: JOURNAL
Journal: FEMS Microbiol. Lett. Coden: FMELE7 Publ: 79 Series: 5 Issue: 2 Pages: 91-3
Identifiers: alkyl phosphonate metab Pseudomonas

9005924 CA09000405924T
Phosphorus-containing pesticide breakdown products: quantitative utilization as phosphorus sources by bacteria
Author: Cook, Alasdair M.; Daughton, Christian G.; Alexander, Martin
Location: Dep. Agron., Cornell Univ., Ithaca, N. Y.
Section: CA010002, CA0040XX Publ Class: JOURNAL
Journal: Appl. Environ. Microbiol. Coden: AEMIDF Publ: 79 Series: 36 Issue: 5 Pages: 868-72
Identifiers: bacteria organophosphate metab, phosphate org metab bacteria

90054301 CA090007054301A
Resolution of acid strength in tert-butyl alcohol and isopropyl alcohol of substituted benzoic acids, phenols, and aliphatic carboxylic acids
Author: Chantooni, M. K., Jr.; Kolthoff, I. M.
Location: Dep. Chem., Univ. Minnesota, Minneapolis, Minn.
Section: CA022008 Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 79 Series: 51 Issue: 1 Pages: 133-40
Identifiers: soly product carboxylate, acidity benzoic acid phenol, acetic acid acidity, disocn const acid alc, activity coeff acid anton

90024517 CA090004024517H
Determination of a polymer-serbate interaction parameter by a sorption method
Author: Michalek, Stefan
Location: Pol.
Section: CA038012 Publ Class: JOURNAL
Journal: Zesz. Nauk. Politech. Krakow., Chem. Coden: ZNPKZ8 Publ: 77 Series: 9, Pages: 37-43
Language: Pol
Identifiers: solvent rubber interaction parameter

9001757 CA00003017157N GB = SARIM
Gas chromatographic methods for the analysis of trace
quantities of oil and associated compounds (demilitarization
effluent, brine, dried salts, munition exudates)
Author: Fisher, Timothy L.; Steger, Ralph J.; Parker, George
A.; Sess, Samuel
Location: Edgewood Arsenal, Aberdeen Proving Ground, Md.
Section: CA004003 Pub Class: TECH REP
Journal: Report Coden: DEREPA Publ: 76 Issue:
EC-18-76080; Order No. AD-8018464, Pages: 25 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1978, 78(19),
140
Avail: NTIS
Identifiers: gas data gas chromatog

99203145 CA08924203145N
Monitoring the disposal of hazardous materials
Author: Colburn, Edward F.
Location: Chem. Syst. Lab., Aberdeen Proving Ground, Md.
Section: CA089002 Pub Class: CONF PROC
Journal: Jt. Conf. Sess. Environ. Pollut., (Conf. Proc.),
4th Coden: 38A940 Publ: 78 Pages: 489-92 Meeting
Date: 77
Publisher: ACS, Address: Washington, D. C.
Identifiers: chem warfare agent disposal monitoring, waste
gas toxic chem disposal, incineration toxic chem flue gas

9619665 CA0892619665N
A field portable mass spectrometer for monitoring organic
vapors
Author: Meier, Robert V.
Location: U. S. Army Environ. Hyg. Agency, Edgewood Arsenal,
Md.
Section: CA089002, CA07311X, CA04711X Pub Class: JOURNAL
Journal: J. Am. Ind. Hyg. Assoc. Coden: AIHMAP Publ:
78 Series: 29 Issue: 3 Pages: 223-9
Identifiers: mass spectrometer air pollution monitoring, org
compd monitor mass spectrometer, computer mass spectrometer
pollution monitor

97140318 CA08718140318F
Plasma chromatography of phosphorus esters
Author: Preston, J. N.; Karasek, F. W.; Kim, S. H.
Location: Natl. Def. Headquarters, Def. Res. Establ. Ottawa,
Ottawa, Ont.
Section: CA089001, CA08921X, CA08911X Pub Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 77 Series:
49 Issue: 12 Pages: 1746-50
Identifiers: phosphorus ester data air, insecticide data air
warfare agent data air

97113820 CA08718113820G
Spontaneous reactivation of acetylcholinesterase following
organophosphate inhibition. 1. An analysis of anomalous
reactivation kinetics
Author: Hovanes, Joseph W.; Broomfield, Clarence A.;
Steinberg, George M.; Lanks, Karl W.; Lieske, Claire M.
Location: Biomed. Lab., Edgewood Arsenal, Aberdeen Proving
Ground, Md.
Section: CA007004 Pub Class: JOURNAL
Journal: Biochim. Biophys. Acta Coden: BBACAO Publ: 77
Series: 483 Issue: 2 Pages: 312-19
Identifiers: acetylcholinesterase reactivation kinetics

9707648 CA0870907648G
Enthalpies and entropies of ionization of 2- and
3-substituted phenols in methanol + water mixtures
Author: Rochester, Colin H.; Wilson, David M.
Location: Chem. Dep., Univ. Nottingham, Nottingham, Engl.
Section: CA022008, CA08911X Pub Class: JOURNAL
Journal: J. Chem. Soc., Faraday Trans. 1 Coden: JCFIAR
Publ: 77 Series: 73 Issue: 4 Pages: 569-81
Identifiers: phenol thermodyn aq methanol, methylphenol
thermodyn aq methanol, chlorophenol thermodyn aq methanol,
nitrophenol thermodyn aq methanol, soln heat phenol aq methanol,
ionization heat phenol aq methanol, entropy ionization
phenol aq methanol, energy ionization phenol aq methanol,
substituent phenol thermodyn aq methanol, transfer heat phenol,
aq methanol, enthalpy phenol aq methanol, free energy transfer
nitrophenol methanol

97022115 CA08703022115G
Factors governing the influence of a first hydrogen bond on
the formation of a second one by the same molecule or ion
Author: Hystkens, Pierre L.
Location: Dep. Chem., Univ. Leuven, Heverlee, Belg.
Section: CA032008 Pub Class: JOURNAL
Journal: J. Am. Chem. Soc. Coden: JACSAT Publ: 77
Series: 99 Issue: 8 Pages: 2878-82
Identifiers: hydrogen bond formation effect, phenol hydrogen
bond equil, IR hydrogen bond phenol, amine hydrogen bond

84106662 CA096191056624
 Ionization constants of phenols in methanol + water mixtures
 Author: Rochester, Colin H.; Wilson, David M.
 Location: Chem. Dep., Univ. Nottingham, Nottingham, Engl.
 Section: CA022008 Publ. Class: JOURNAL
 Journal: J. Chem. Soc., Faraday Trans. 1 Coden: JCFYAR
 Publ: 76 Series: 72 Issue: 12 Pages: 2930-8
 Identifiers: phenol ionization substituent effect, reaction
 const phenol ionization

84098399 CA09614098399Z
 Effect of nitroderivatives of benzene, toluene, aniline, and
 phenol on anaerobic sludge digestion
 Author: Horakova, Marta
 Location: Vys. Sh. Chem. Technol., Prague, Czech.
 Section: CA060002 Publ. Class: JOURNAL
 Journal: Vodni Hospod., 8 Coden: VODMAF Publ: 76
 Series: 26 Issue: 4 Pages: 97-9 Language: Czech
 Identifiers: nitrobenzene inhibition wastewater treatment,
 nitrotoluene inhibition wastewater treatment, nitroaniline
 inhibition wastewater treatment, nitrophenol inhibition
 wastewater treatment, biol wastewater treatment inhibition

01070000 CA00700000
Solvent effect in radical copolymerization of
p-vinylbenzoate and methyl methacrylate
Author: Lohm, Anthony; Galli, Giancarlo; Chieffini, Edo;
Solera, Roberto
Location: Dep. Inorg., Phys. Ind. Chem., Univ. Liverpool,
Liverpool, L69 3BX, Eng.
Section: CA00700000 Publ Class: JOURNAL
Journal: Polym. Bull. (Berlin) Coden: POLUDN Pub: 79
Series: 1 Issue: 7 Pages: 491-9
Identifiers: vinylbenzoate methacrylate copolymer reactivity
solvent effect radical copolymer

00107045 CA00700045
Interpretation of polarized absorption and emission spectra
of solutions incorporated in stretched polymer films
Author: Aviv, Abraham; Margulies, Leon; Sagiv, Jacob; Yegor,
Aron; Masur, Yehuda
Location: Inst. Rep., Weizmann Inst. Sci., Rehovot, Israel
Section: CA00700045 Publ Class: CONF PABC
Journal: Linear Dichroism Spectrosc., Proc. Nobel Workshop
No. 8, Opt. Dichroism Chem. Appl. Polariz. Spectrosc. Coden:
201945 Pub: 77 Pages: 54-60 Meeting Date: 76
Publisher: Lund Univ. Press Address: Lund, Sweden
Avail: Morden, Bengt
Identifiers: dichroic ratio cholesterol spectra,
polyethylene cholesterol distribution, aram hydrocarbon
distribution polyethylene, fluorescence spectra polarization
hydrocarbon, UV spectra polarization hydrocarbon

07100001 CA00710001
Correction of instrumental time response variation with
wavelength in fluorescence lifetime determinations in the
ultraviolet region
Author: Sawyer, D. M.; McKinnon, A. E.; Szabo, A. G.
Location: Div. Biol. Sci., Natl. Res. Coun. Canada, Ottawa,
Ont.
Section: CA00700000 Publ Class: JOURNAL
Journal: Rev. Sci. Instrum. Coden: RSIMAX Pub: 77
Series: 48 Issue: 8 Pages: 1090-4
Identifiers: fluorescence lifetime time response variation,
ratio correction fluorescence UV

85128119 CA08519138119Q
Improved automated analysis for nanogram quantities of organophosphorus agents GB and VX

Author: Tomlinson, Dale H.; Feller, Harold L.
Location: Edgewood Arsenal, Aberdeen Proving Ground, Md.
Section: CA004001 Publ Class: Y
Journal: U. S. M115, AD Rep.
Issue: AD-A025798, Pages: 23 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1976, 76(16), 186

Avail: NTIS

Identifiers: organophosphorus compd detn autoanalyzer, GB
VX detn autoanalyzer

85122956 CA08517122956S

The role of heterogeneity in the kinetics of a surface reaction. I. Infrared characterization of the adsorption structures of organophosphates and their decomposition
Author: Kuiper, A. E. T.; Van Bokhoven, J. J. G. M.; Nedasa, J.
Location: Chem. Lab., TNO, Rijswijk, Neth.
Section: CA023003, CA004000 Publ Class: J
Journal: J. Catal. Coden: JCTLAS Publ: 76 Series: 43 Issue: 1-3 Pages: 154-67

Identifiers: sarin adsorption alumina IR, decoupled methylphosphonofluoridate alumina, phosphonofluoridate methyl adsorption, toxic organophosphorus adsorption

85093604 CA08513083604T

Comparison of substituent effects on dissociation and conjugation of phenols with those of carbonyl compounds in acetonitrile, N,N-dimethylformamide, and dimethyl sulfoxide
Author: Chentooni, M. K., Jr.; Kalthoff, I. M.
Location: Sch. Chem., Univ. Minnesota, Minneapolis, Minn.
Section: CA022008 Publ Class: J

Journal: J. Phys. Chem. Coden: JPCMAJ Publ: 76 Series: 80 Issue: 12 Pages: 1908-10
Identifiers: ionization benzoate phenol substituent solvent, substituent effect ionization benzoate phenol, solvent effect ionization benzoate phenol, conjugation benzoate phenol substituent solvent

84111028 CA09416111028X

Selecting organophosphorus agents using 1-phenyl-1,2,3-butatriene-2-oxime and cyanide indicating composition
Author: Postonah, Edward J.; Crabtree, Eleanor V.; Kramer, David M.
Location: USA
Section: CA086002, CA086000 Publ Class: P

Journal: U. S. Coden: USXXAM Publ: 751007 Pages: 4 pp.

Patent No: 3910763 Applic No: 888,663 Date: 691003
Class: 23-2328, GOIN
Assignee: United States Dept. of the Army
Identifiers: Chem warfare agent detector, phosphonofluoridate detector, phenylbutanetrioxime detector, dinitrobenzene phosphonofluoridate detector, nitrobenzaldehyde phosphonofluoridate detector

84086097 CA08413086097F

Spontaneous reactivation of acetylcholinesterase following organophosphate inhibition. I. An analysis of anomalous reactivation kinetics

Author: Hovanec, Joseph V.; Broomefield, Clarence A.; Steinberg, George M.; Lanks, Karl W.; Lieske, Claire M.
Location: Edgewood Arsenal, Aberdeen Proving Ground, Md.
Section: CA007004 Publ Class: Y
Journal: U. S. M115, AD Rep.
Issue: AD-A015562, Pages: 15 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1975, 75(24), 28
Avail: NTIS

Identifiers: acetylcholinesterase phosphoryl deriv spontaneous reactivation

83147805 CA08317147805Y

Cyclic phosphinic acid esters

Author: Finkle, Manfred; Kleiner, Hans J.

Location: Ger.

Section: CA028007 Publ Class: P
Journal: Ger. Offen. Coden: GXXBXX Publ: 750710
Pages: 11 pp.

Patent No: 2363852 Applic No: P 23 63 852.0 Date: 731221 Class: C07F

Assignee: Hoechst A.-G.

Identifiers: oxaphosphorane, cyclization phosphonate allyl alc

82144808 CA08222144806J
Toxic agent leak detector
Author: Fromm, Bernard W.; Silvestri, Achille; Jones, Arthur R., Jr.
Section: CA089002, CA047000 Publ Class: P
Journal: U.S. Coden: USXXAM Publ: 741217 Pages: 5
Patent No: 3854885 Applic No: 76,316 Date: 700903
Class: 23-264, 8 Oin
Assignee: United States Dept. of the Army
Identifiers: safety toxic agent leak detector, nerve toxicant leak detector container

81022117 CA081060221117J
fluoride selective electrode procedure for fluorated organophosphate cholinesterase inhibitors
Author: De Clercq, H.; Mertens, J.; Massart, D. L.
Location: Farm Inst., Vrije Univ., Brussels, Belg.
Section: CA005001 Publ Class: J
Journal: Arch. Toxicol. Coden: ATXKAS Publ: 73
Series: 31 Issue: 2 Pages: 185-91
Identifiers: sarin analysis fluoride selective electrode, acetylcholinesterase sarin analysis

82097483 CA08215097483P
Thermodynamics of hydration of 3-substituted and 3,5-disubstituted phenols
Author: Parsons, Gerald M.; Rochester, Colin H.
Location: Chem. Dep., Univ. Nottingham, Nottingham, Engl.
Section: CA022008 Publ Class: J
Journal: J. Chem. Soc., Perkin Trans. 2 Coden: JCPKSH
Publ: 74 Issue: 11 Pages: 1312-18
Identifiers: phenol hydration thermodyn substituent

80078177 CA08014078177J
Fluorescent determination of some fluorine-containing phosphoorganic substances
Author: Vrabashev, M. I.
Location: Madara Fac. Lab., Shumen, Bulg.
Section: CA080008, CA040000 Publ Class: J
Journal: Dokl. Bulg. Akad. Nauk Coden: DBANAD Publ: 73
Series: 26 Issue: 11 Pages: 1497-9
Identifiers: fluorine phosphorus org compd detn, fluorometry fluorine phosphorus compd, samn detn, sarin detn

82086341 CA08213086341Q
Reactions of tert-butyl per esters. XII. Synthesis and reactions of alkyl tert-butylperoxy alkylphosphonates
Author: Sosnovsky, G.; Konieczny, M.
Location: Dep. Chem., Univ. Wisconsin, Milwaukee, Wis.
Section: CA029007 Publ Class: J
Journal: Phosphorus Coden: PHOSBV Publ: 74 Series: 4 Issue: 4 Pages: 289-94
Identifiers: peroxy alkylphosphonate, phosphonate peroxy, phosphine triphenyl peroxyphosphonate reaction, dicyclohexylamine peroxyphosphonate reaction, pyrophosphonate

80030608 CA08006030608R
Detection and estimation of isopropyl methylphosphonofluoridate and O-ethyl S-diisopropylmethylphosphonothioate in sea water in parts-per-trillion level
Author: Michel, Harry D.; Gordon, Eric C.; Epstein, Joseph
Location: Edgewood Arsenal, Aberdeen Proving Ground, Md.
Section: CA081002, CA080000 Publ Class: J
Journal: Environ. Sci. Technol. Coden: ESTHAG Publ: 73
Series: 7 Issue: 11 Pages: 1048-9
Identifiers: anticholinesterase detn sea water, phosphonofluoridate detn sea water, phosphonothioate detn sea water, sea water analysis anticholinesterase

82023485 CA08204023485U
Electroreduction of nitro compounds in diethylene glycol in a wide temperature range
Author: Khitrova, L. M.; Gorbachev, S. V.
Location: USSR
Section: CA072007, CA087000, CA022000, CA025000 Publ Class: J
Journal: Tr. Mosk. Khim.-Tekhnol. Inst. Coden: TEK1AT
Publ: 73 Series: 79 Pages: 102-4 Language: Russ
Identifiers: nitro compd electroredn platinum, redn electrochem kinetics nitro compd, benzoic acid nitro electroredn, phenol acid nitro electroredn, phenol nitro electroredn, polarization electrolytic concn nitro compd

79114967 CA07919114967Q
Relation between the molecular structure of phenols and their chromatographic properties
Author: Degenhardt, M. K.; Gogele, V. G.
Location: Inst. Neorg. Khim. Elektrokhim., Tbilisi, USSR
Section: CA022008 Publ Class: J
Journal: Soobshch. Akad. Nauk Gruz. SSR Coden: SAKMAH
Publ: 73 Series: 71 Issue: 1 Pages: 121-4
Language: Russ
Identifiers: thin layer chromatog phenol, structure effect chromatog phenol

DIALOG File3: CA Search - 1972 thru 1976 (Copr. Am. Chem. Soc.) (Item 15 of 15) User 3631 2apr81

1002

77078275 CA077110782755

Reactions of tert-butyl peresters. XI. Reactions of alkyl
tert-butyloxy alkyloxyphosphates, dialkyl tert-butyloxy
phosphates, and other phosphorus esters with benzene and
aluminum chloride, and reactions of dialkyl tert-butyloxy
phosphates with phenylmagnesium bromide

Author: Sosnovsky, G.; Zaret, E. H.; Konieczny, M.

Location: Dep. Chem., Univ. Wisconsin, Milwaukee, Wis.

Section: CA029007 Publ Class: J

Journal: J. Org. Chem.

Series: 37 Issue: 14 Pages: 2267-72

Identifiers: phosphonate benzene reactions

Coden: JOCCEAH

Publ: 72

Print 21/2/1-2 1003
DIALOG File3: CA Search - 1972 thru 1976 (Copr. Am. Chem. Soc.) (Item 1 of 2) User 3831 2apr81

83154921 CA083181549218
Intermolecular interactions and spectra of molecules in
multicomponent solutions. VI. Disturbance of the universal
ratio between absorption and fluorescence spectra in liquid
three-component systems
Author: Gorodyskii, V. A.; Tikhonolov, A. A.; Bekhtiev, N.
G.

Location: USSR
Section: CA073003 Publ Class: J
Journal: Opt. Spektrosk. Publ: 75
Series: 38 Issue: 5 Pages: 875-81 Language: Russ
Identifier: absorption fluorescence ternary soln

80081719 CA080180817191
Electronic states of imino radicals formed from the
vacuum-ultraviolet photolysis of ethylenimine
Author: Kawasaki, Masahiro; Iwasaki, Masahiro; Tanaka, Ikuzo
Location: Dep. Chem., Tokyo Inst. Technol., Tokyo, Japan
Section: CA022004 Publ Class: J
Journal: J. Chem. Phys. Coden: JCPSA6 Publ: 73
Series: 59 Issue: 12 Pages: 6238-33
Identifier: photolysis ethylenimine imino radical, energy
level imino radical, electronic state imino radical

COMPOUND: PAHs

94083269 CA09412089258K

Quantitative analysis of polynuclear aromatics from diesel exhaust by high performance liquid chromatography using UV photometric detection

Author: Roumeliotis, P.; Unger, K. K.
Location: Inst. Anorg. Chem., Anal. Chem., Johannes Gutenberg-Universität Mainz, 5500, Fed. Rep. Ger.
Section: CA089002, CA089011X Pub) Class: JOURNAL
Journal: Anal. Chem. Symp. Ser. Coden: ACS50R Pub) 80
Series: 3 Issue: Recent Dev. Chromatogr. Electrophor.
Pages: 229-45

Identifiers: diesel exhaust gas sepn, polynuclear arom detn diesel exhaust

CA09412089258K

Descriptors: Exhaust gases, diesel; Gas analysis
Identifiers: detn liq chromatog UV spectrometry polynuclear aroms
CAS Registry Numbers: 50-32-8 85-01-8 129-00-0 191-24-2 192-97-2 193-26-5 198-55-0 206-82-3 206-99-2 206-44-0 207-06-9 217-59-4 218-01-9

94083207 CA09410088207U

Microanalysis of polynuclear aromatic hydrocarbons in petroleum

Author: Matsushita, Hidetaru
Location: Dep. Community Environ. Sci., Natl. Inst. Public Health, Tokyo, 108, Japan
Section: CA081001, CA08911X, CA08911X Pub) Class: JOURNAL
Journal: Progr. Rep. - Am. Chem. Soc., Div. Fuel Chem. Coden: ACPA1 Pub) 78 Series: 24 Issue: 1 Pages: 292-8

Identifiers: arom hydrocarbon chromatog petroleum, polycyclic arom hydrocarbon chromatog, dual band thin layer chromatog

CA09410088207U

Descriptors: Aromatic hydrocarbons, polycyclic, analysis; Chromatography, thin-layer; Gasoline; Petroleum products
Identifiers: detn dual band
CAS Registry Numbers: 50-32-8 85-01-8 129-00-0 191-24-2 191-26-4 198-55-0 206-44-0 207-06-9 218-01-9

94083459 CA09408036459Z

Trace analysis of polynuclear aromatic hydrocarbons in automobile emissions

Author: Nagasawa, S.
Location: Lab. Spectrom. Masse, CEA-Saclay, Saclay, Fr.
Section: CA089000, CA08911X, CA08911X Pub) Class: JOURNAL
Journal: Pollut. Atmos. Coden: POATDH Pub) 80
Series: 85, Pages: 21-8 Language: Fr

Identifiers: review polycyclic arom hydrocarbon detn, exhaust polycyclic arom hydrocarbon review

CA09408036459Z

Descriptors: Aromatic hydrocarbons, polycyclic, analysis; Exhaust gases
Identifiers: detn

94020132 CA09404030132P

Determination of polynuclear aromatic hydrocarbons in water
Author: Crosby, M. F.; Hunt, D. C.
Location: Lab. Gov. Chem., Dep. Ind., London, SE1 8NO, Engl.
Section: CA081002, CA07911X Pub) Class: JOURNAL
Journal: Anal. Proc. (London) Coden: ANPRDI Pub) 80
Series: 17 Issue: 9 Pages: 381-4

Identifiers: polynuclear arom hydrocarbon detn water

CA09404030132P

Descriptors: Aromatic hydrocarbons, polycyclic, analysis
Identifiers: detn river water extn high performance liq chromatog
CAS Registry Numbers: 50-32-8 191-24-2 193-26-5 205-99-2 206-44-0 207-06-9 7732-18-5

94018017 CA09404018017M

Quantitative analysis of polynuclear aromatic hydrocarbons in liquid fuels
Author: Parr, Jerry L.
Location: Radian Corp., Austin, TX, USA
Section: CA081006, CA08911X Pub) Class: TECH REP
Journal: Report Coden: D8REP4 Pub) 80 Issue: EPA-600/2-80-089; Order No. PB80-187389. Pages: 44 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1980, 80(19), 4200

Avail: NTIS

Identifiers: polycyclic arom detn fuel, hydrocarbon polycyclic arom fuel

CA09404018017M

Descriptors: Fuels, diesel; Fuels, jet aircraft; Gasoline
Identifiers: occurrence liq hydrocarbon polycyclic arom hydrocarbons
CAS Registry Numbers: 50-32-8 85-01-8 120-12-7 129-00-0 191-24-2 192-97-2 206-44-0 217-59-4 218-01-9

93207241 CA09322207241V
Identification and quantification of polynuclear aromatic compounds in Synthoil by room-temperature phosphorimetry
Author: Vo-Dinh, Tuan; Gammage, R. S.; Martinez, P. R.
Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak Ridge, TN, 37830, USA
Section: CA09322207241V Pubi Class: JOURNAL
Journal: Anal. Chm. Acta Coden: ACHAM Pubi: 80
Series: 118 Issue: 2 Pages: 313-33
Identifiers: coal liq arom phosphorimetry

CA09322207241V
Descriptors: Aromatic compounds; Aromatic hydrocarbons, analysis; Coal, liquefied
Identifiers: detn Synthoil phosphorimetry compds liq polynuclear Synthoil process
CAS Registry Numbers: 80-32-8 85-01-8 86-73-7 129-00-0 208-44-0 218-01-9 301-04-2 7681-82-8 7789-17-5

93179100 CA09318179100C
Selectivity properties in Spil'skii fluorescence of polynuclear aromatic hydrocarbons
Author: Calais-Joe, Anders L.; Gestman, Conny E.
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, S-106 91, Sweden
Section: CA09318179100C Pubi Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Pubi: 80
Series: 52 Issue: 13 Pages: 2083-5

Identifiers: polynuclear arom hydrocarbon analysis fluorescence, Spil'skii fluorescence polynuclear arom hydrocarbon, selectivity fluorescence polynuclear arom hydrocarbon, chromatog polynuclear arom hydrocarbon, liq chromatog polynuclear arom hydrocarbon, high performance liq chromatog hydrocarbon

CA09318179100C
Descriptors: Aromatic hydrocarbons, polycyclic, analysis; Chromatography, column and liquid, high-performance; Chromatography, gas; Fluorescence; Petroleum; Spectrochemical analysis, fluorometric
Identifiers: identification pyrolysis products Spil'skii polynuclear selectivity detection effect
CAS Registry Numbers: 80-32-8 83-70-3 85-55-3 129-00-0 191-07-1 191-24-2 191-26-4 192-87-2 207-08-9 2381-21-7 2383-12-8

93155566 CA09316155566H
Studies on the polynuclear aromatic hydrocarbons in crude oils and heavy fuel oils by gas chromatography
Author: Uehiro, Shirohiko; Takada, Tadashi
Location: Japan Marit. Saf. Acad., Kure, Japan

Section: CA09316155566H Pubi Class: JOURNAL
Journal: Kankyo Tokoku - Kankyo Hoan Daigakko, Dai-2-bu Coden: KKHJAH Pubi: 80 Series: 25 Issue: 2 Pages: 1-9
Language: Japanese Meeting Date: 79
Identifiers: fingerprinting petroleum pollutant water chromatog

CA09316155566H
Descriptors: Aromatic hydrocarbons, polycyclic, biological studies; Chromatography, gas; Fuel oil; Petroleum products; Petroleum; Water pollution
Identifiers: oils contg fingerprinting polynuclear crude heavy pollutant spills

93143926 CA09315143926M
Methods of analysis for polynuclear aromatic hydrocarbons in environmental samples
Author: Pancirov, R. J.; Seerl, T. D.; Brown, R. A.
Location: Exxon Res. and Eng. Co., Linden, NJ, 07036, USA
Section: CA09315143926M Pubi Class: JOURNAL
Journal: Prepr., Div. Pet. Chem., Am. Chem. Soc. Coden: APCPAT Pubi: 78 Series: 23 Issue: 3 Pages: 855-89
Identifiers: review environment arom hydrocarbon analysis

CA09315143926M
Descriptors: Aromatic hydrocarbons, polynuclear, analysis; Environment
Identifiers: detn environmental samples methods

93124744 CA09314124744Z
Analysis of polynuclear aromatic hydrocarbons at trace level in white oils by liquid chromatography and UV spectrofluorimetry
Author: Collin, J. M.; Vion, G.
Location: Cent. Rech., Co. Fr. Raffinage, Harfleur, 76700, France
Section: CA09314124744Z Pubi Class: JOURNAL
Journal: Analusis Coden: ANALSV Pubi: 80 Series: 8
Issue: 6 Pages: 224-5
Language: French
Identifiers: white oil polynuclear arom. chromatog spectrofluorimetry white oil

CA09314124744Z
Descriptors: Aromatic hydrocarbons, polycyclic, analysis; Hydrocarbon oils, white oils
Identifiers: detn polynuclear
CAS Registry Numbers: 80-32-8 85-55-3 85-01-8 129-00-0 191-28-4 198-55-0 208-44-0 218-01-9

- 93100700 CA093101007005
Improved resolution in high performance liquid chromatography analysis of polynuclear aromatic hydrocarbons using ternary solvent systems
Author: Salinsky, Barry B.
Location: Natl. Inst. Occup. Saf. Health, Cincinnati, OH, 45226, USA
Section: CA0930002, CA0930003 Pub. Class: JOURNAL
Journal: ACS Symp. Ser. Coden: ACSMCH Pub. 80
Series: 120 Issue: Anal. Tech. Occup. Health Chem. Pages: 149-66
Identifiers: polynuclear aromatic hydrocarbon detn chromatog
- 93100885 CA09310100885F
Correlation between the concentrations of polynuclear aromatic hydrocarbons and those of particulates in an urban atmosphere
Author: Honda, Takashi; Kato, Yoshihiro; Yamamura, Takaki; Ishii, Tadashi; Sudo, Kyo
Location: Fac. Sci., Sci. Univ. Tokyo, Tokyo, 102, Japan
Section: CA0930002, CA0930003 Pub. Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTH60 Pub. 80
Series: 14 Issue: 4 Pages: 416-22
Identifiers: polynuclear aromatic hydrocarbon air particulate
- CA09310100885F
Descriptores: Aromatic hydrocarbons, polynuclear, analysis; Particles
Identifiers: data air correlation concn particulates urban atm polycyclic aromatic hydrocarbons via HPLC
CAS Registry Numbers: 80-32-8 86-85-3 129-00-0 191-24-2 196-56-0 210-01-9
- 9308175 CA0930008175Q
Use of an aqueous micellar mobile phase for separation of phenols and polynuclear aromatic hydrocarbons via HPLC
Author: Armstrong, Daniel V.; Henry, Susan J.
Location: Dep. Chem., Georgetown Univ., Washington, DC, 20057, USA
Section: CA0930004 Pub. Class: JOURNAL
Journal: J. Liq. Chromatogr. Coden: JLCHE6 Pub. 80
Series: 3 Issue: 5 Pages: 657-62
Identifiers: micellar mobile phase liq chromatog, sodium dodecyl sulfate mobile phase, phenol liq chromatog, micellar eluent, hydrocarbon liq chromatog micellar eluent, polynuclear aromatic hydrocarbon liq chromatog
- 9308174 CA0930008174P
Column-induced selectivity in separation of polynuclear aromatic hydrocarbons by reversed-phase, high-performance liquid chromatography
Author: Colasfio, A. L.; MacDonald, J. C.
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, S-106 81, Swed.
Section: CA0930004 Pub. Class: JOURNAL
Journal: Chromatographia Coden: CHRO87 Pub. 80
Series: 13 Issue: 6 Pages: 350-2
Identifiers: liq chromatog, polynuclear aromatic hydrocarbon, reversed-phase liq chromatog column, high performance liq chromatog column, column induced selectivity liq chromatog, manufacturer variation column selectivity chromatog
- CA0930008174P
Descriptores: Aromatic hydrocarbons, polynuclear, analysis; Chromatography, column and liquid, high-performance, reversed-phase
Identifiers: seven variations columns different manufacturers relation selectivity
CAS Registry Numbers: 80-32-8 82-70-3 86-85-3 191-07-1 191-24-2 193-39-8 205-82-3 207-08-9 215-88-7 217-89-4 218-01-9
- 9308249 CA0930008249X
Methods of analysis for polynuclear aromatic hydrocarbons in environmental samples
Author: Pencirov, R. J.; Seerl, T. D.; Brown, R. A.
Location: Exxon Res. and Eng. Co., Linden, NJ, 07036, USA
Section: CA0930000 Pub. Class: JOURNAL
Journal: Adv. Chem. Ser. Coden: ADCSAJ Pub. 80
Series: 185 Issue: Pat. Mar. Environ. Pages: 123-42
Identifiers: review aromatic hydrocarbon analysis environment
- CA0930008249X
Descriptores: Aromatic hydrocarbons, polycyclic, analysis; Environment
Identifiers: detn environmental samples polynuclear

93012472 CA08302012472P
Portable fluorometric monitor to detect polynuclear aromatic
hydrocarbon contamination of work area surfaces
Author: Schurek, D. D.
Location: Oak Ridge Natl. Lab., Oak Ridge, TN, USA
Section: CA083001, CA0821XX, CA0791XX, Publ Class: TECH
REP
Journal: Report Coden: D3REP3 Publ: 79 Issue:
CONF-790885-2, Pages: 18 pp.
Citation: Energy Res. Abstr. 1979, 4(23), Abstr. No. 84101
Avail: NTIS
Identifiers: polynuclear aromatic hydrocarbon dain fluorometer,
coal conversion surface contamination fluorometer

CA08302012472P
Descriptores: Aromatic hydrocarbons, polynuclear, analysis; Coal
Identifiers: dain surface clean areas conversion portable
fluorometer surfaces

92208575 CA08225208575K
Methodology for the isolation of polynuclear aromatic
hydrocarbons for qualitative, quantitative, and bioassay
studies
Author: Severson, R. F.; Snook, M. E.; Arrandale, R. F.;
Chortyk, D. I.
Location: Job. Lab., Sci. Educ. Adm., Athens, GA, 30604, USA
Section: CA084000, Publ Class: JOURNAL
Journal: Environ. Sci. Res. Coden: EVS88T Publ: 80
Series: 16 Issue: Hydrocarbons Halogenated Hydrocarbons
Aquat. Environ. Pages: 91-108 Meeting Date: 78
Identifiers: review aromatic hydrocarbon isolation dain

CA08225208575K
Descriptores: Aromatic hydrocarbons, polynuclear, analysis
Identifiers: isolation relation

92208580 CA08224208580M
Evaluation of sensitized fluorescence for polynuclear
aromatic hydrocarbon detection
Author: Smith, T. R.
Location: Def. Space Syst. Group, TRW, Redondo Beach, CA,
USA
Section: CA084006, CA0811XX, CA0891XX, Publ Class: TECH
REP
Journal: Report Coden: D8REP4 Publ: 79 Issue:
EPA/600/77-79/207; Order No. PB80-108476, Pages: 47 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1980, 80(5), 770
Avail: NTIS
Identifiers: fluorescence detection polynuclear aromatic
hydrocarbon, spot test polynuclear aromatic hydrocarbon,
combustion effluent screening aromatic hydrocarbon, gas chromatog
polynuclear aromatic hydrocarbon, mass spectrometry polynuclear

arom hydrocarbon

CA08224208580M
Descriptores: Aromatic hydrocarbons, polynuclear, analysis;
Chromatography, gas; Combustion gases; Mass spectroscopy
Identifiers: dain effluents fluorescent spot test screening
samples combined fluorescence sample prior

92202682 CA08224202682J
A comparison of two techniques for the collection and
analysis of polynuclear aromatic compounds in ambient air
Author: Lindgren, James L.; Kraus, Henry J.; Fox, Marye
Anne

Location: Texas Air Control Board, Austin, TX, USA
Section: CA089002, CA0791XX, Publ Class: JOURNAL
Journal: Proc., Annu. Meet. - Air Pollut. Control Assoc.
Codan: PRAPAP Publ: 78 Series: 71st. Vol. 2,
78-25.4, 14 pp.

Identifiers: Bondapak polynuclear arom hydrocarbon sampling,
polyurethane polynuclear arom hydrocarbon sampling, atm
polynuclear arom hydrocarbon sampling

CA08224202682J
Descriptores: Air analysis; Aromatic hydrocarbons, polycyclic,
analysis; Urethane polymers, uses and miscellaneous
Identifiers: sample app collection dain sampling absorbents
au Bondapak C18 polyurethane
CAS Registry Numbers: 99900-31-6

92148926 CA08220168926M
Analysis of polynuclear aromatic hydrocarbons in
environmental waters by high-pressure liquid chromatography
Author: Sorrell, R. K.; Reding, R.
Location: Off. Drinking Water, EPA, Cincinnati, OH, 45268,
USA

Section: CA081002, CA0791XX, Publ Class: JOURNAL
Journal: J. Chromatogr. Codan: JOCRAM Publ: 79
Series: 185 Issue: 1 Pages: 655-70
Identifiers: polynuclear arom hydrocarbon dain water

CA08220168926M
Descriptores: Aromatic hydrocarbons, polynuclear, analysis
Identifiers: dain drinking natural water high pressure liq
chromatog UV
CAS Registry Numbers: 50-32-8 52-70-2 56-55-2 85-01-8
120-12-7 129-00-0 191-24-2 192-97-2 193-38-5 198-58-0 205-99-2
208-44-0 207-08-9 218-01-9 2381-21-7 7732-18-5

92134941 CA09214116127A
Determination of polynuclear aromatic hydrocarbons in water and wastewater by a gas chromatographic-ultraviolet spectrophotometric method
Author: Searl, T. B.; Robbins, W. K.; Brown, B. A.
Location: Exxon Res. Eng. Co., Linden, NJ, 07036, USA
Section: CA081002, CA0801XX, CA0801XX Publ Class: TECH REP
Journal: ASTM Spec. Tech. Publ. Coden: ASTTAS Publ: 78
Issue: STP 686, Mass. Org. Pollut. Water Wastewater, Pages: 184-80 Meeting Date: 78
Identifiers: arom polycyclic hydrocarbon detn wastewater, gas chromatog UV spectrometry

CA092141249418
Descriptors: Aromatic hydrocarbons polynuclear, analysis
Identifiers: detn gas chromatog UV spectrophotometric water
CAS Registry Numbers: 7732-18-5

92116133 CA09214116132Z
Determination of polynuclear aromatic hydrocarbons in refinery water streams
Author: Preston, H. G.; Macaluso, A.
Location: Texaco Inc., Port Arthur, TX, 77640, USA
Section: CA081002, CA0811XX, CA0801XX Publ Class: TECH REP
Journal: ASTM Spec. Tech. Publ. Coden: ASTTAS Publ: 79
Issue: STP 686, Mass. Org. Pollut. Water Wastewater, Pages: 182-83 Meeting Date: 78
Identifiers: polynuclear arom hydrocarbon wastewater refinery, liq chromatog polynuclear arom hydrocarbon, reverse concn polynuclear arom hydrocarbon

CA09214116132Z
Descriptors: Aromatic hydrocarbons, analysis
Identifiers: polynuclear detn petroleum refining wastewater
CAS Registry Numbers: 7732-18-5

92116127 CA09214116127A
Development of an aqueous polynuclear aromatic hydrocarbon standard reference material
Author: May, W. E.; Brown, J. M.; Chesler, S. M.; Guenther, P.; Milper, L. R.; Heriz, H. S.; Wise, S. A.
Location: Natl. Bur. Stand., Washington, DC, 20234, USA
Section: CA081002, CA0811XX, CA0801XX Publ Class: COMF PROC
Journal: Polynuclear Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 78
Pages: 411-18 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avail: Jones, Peter M.; Leber, Philip
Identifiers: arom hydrocarbon std ref material

CA09214116127A
Descriptors: Aromatic hydrocarbons, polynuclear, preparation
Identifiers: analysis std ref material environmental assessment prepn certification
CAS Registry Numbers: 7732-18-5

92115928 CA09214115928A
Quantitative analysis of selected PAH (polynuclear aromatic hydrocarbons) in aqueous effluent by high-performance liquid chromatography
Author: Wilkinson, John E.; Strup, Paul E.; Jones, Peter M.
Location: Battelle-Columbus Lab., Columbus, OH, 43201, USA
Section: CA060003, CA0791XX Publ Class: COMF PROC
Journal: Polynuclear Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 79
Pages: 217-26 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avail: Jones, Peter M.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon detn wastewater

CA09214115928A
Descriptors: Aromatic hydrocarbons, polynuclear, analysis
Identifiers: detn wastewater extn liq chromatog fluorescence waste
CAS Registry Numbers: 53-70-3 91-20-3 120-12-7 206-44-0
218-01-8 7732-18-5

92118549 CA09214118549C
PAH (polynuclear aromatic hydrocarbons) emissions from a stratified-charge vehicle with and without oxidation catalyst: sampling and analysis evaluation
Author: Lee, Frank S. C.; Prater, T. J.; Ferris, F.
Location: Sci. Res. Lab., Ford Mot. Co., Dearborn, MI. 48121, USA

Section: CA090002, CA0900XX Publ Class: CNF PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41MSAL Publ: 79
Pages: 83-110 Meeting Date: 78 Address: Ann Arbor, Mich
Publisher: Ann Arbor Sci. Leber, Philip
Avail: Jones, Peter V.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon exhaust gas

CA09214118549C
Descriptors: Aromatic hydrocarbons, polynuclear, uses and miscellaneous, exhaust gases
Identifiers: emission stratified charge vehicle without oxid catalyst charged
CAS Registry Numbers: 80-32-8 86-88-3 88-01-8 120-12-7 128-00-0 191-07-1 191-24-2 192-97-2 193-43-1 198-58-0 208-44-0 213-46-7 217-59-4 218-01-8 414-29-9 613-12-7 26140-80-3 27877-80-8 28083-98-4 28083-00-1 30232-36-9 30881-10-1 31711-83-2 34488-71-5 34820-92-7 41893-24-2 58532-75-6 58515-36-4 81089-87-0 73020-30-1

92118504 CA09214118504J
Gas chromatographic separation of high-molecular polynuclear aromatic hydrocarbons in samples from different sources, using temperature-stable glass capillary columns
Author: Stenberg, Ulf; Alberg, Tomas; Stenberg, Lars; Wennman, Thomas
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, Swed.

Section: CA090001, CA0733XX, CA0900XX Publ Class: CNF PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41MSAL Publ: 79
Pages: 313-36 Meeting Date: 78 Address: Ann Arbor, Mich
Publisher: Ann Arbor Sci. Leber, Philip
Avail: Jones, Peter V.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon, combustion gas, polynuclear arom hydrocarbon

CA09214118504J
Descriptors: Air analysis, aromatic hydrocarbons, polynuclear, analysis
Identifiers: clean exhaust gases combustion emissions gas chromatog
CAS Registry Numbers: 80-32-8 83-70-3 86-88-3 88-01-8 120-12-7 128-00-0 191-07-1 191-24-2 191-26-4 192-97-2 193-43-1 198-58-0 203-12-3 208-99-2 208-44-0 207-08-9

218-01-9 238-84-6 243-17-4 27208-37-3 58615-36-4 72957-39-2

92087601 CA09210087601I
Separation and identification of sulfur-containing polycyclic aromatic hydrocarbons (thiophene derivatives) from some PAH (polynuclear aromatic hydrocarbons)
Author: Karcher, W.; Depaus, R.; Van Eijk, J.; Jacob, J.
Location: Jt. Res. Cent., Comm. Eur. Communities, Petten, Neth.

Section: CA090006 Publ Class: CNF PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41MSAL Publ: 79
Pages: 341-56 Meeting Date: 78 Address: Ann Arbor, Mich
Publisher: Ann Arbor Sci. Leber, Philip
Avail: Jones, Peter V.; Leber, Philip
Identifiers: thiophene identification polycyclic arom hydrocarbon, polycyclic arom hydrocarbon analysis thiophene, pyrene analysis thiophene, benzopyrene analysis thiophene, benzopyrene analysis thiophene, benzofluorothene analysis thiophene

CA09210087601I
Descriptors: Aromatic hydrocarbons, polycyclic, analysis
Identifiers: thiophene deriv identification deriv benzofluoranthene pyrene benzopyrene benzopyrene
CAS Registry Numbers: 80-32-8 110-02-10 128-00-0 191-24-2 194-88-0 208-82-3 242-83-5 30786-82-0 31473-78-3 72072-30-9 72076-88-3

92081501 CA09210081501P
Development of a prototype instrument for field monitoring
of polynuclear aromatic hydrocarbons (PAH) vapors
Author: Heathorne, A. R.; Thorngate, J. H.; Gansage, R. B.;
Vo-Glinh, T.
Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak
Ridge, TN, 37830, USA
Section: CA089002, CA072XXX, CA080LXX Publ Class: CONF
PROC
Journal: Polynuci. Aromat. Hydrocarbons, Int. Symp. Chem.
Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 79
Pages: 299-311 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avall: Jones, Peter W.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon air spectrometer,
UV spectrometer air analysis

CA09210081501P
Descriptors: Air analysis; Aromatic hydrocarbons, polynuclear-
analysis; Spectrochemical analysis, UV; Spectrometers, UV
Identifiers: biological studies second deriv UV spectra detn
monitoring
CAS Registry Numbers: 88-01-8 91-20-3 120-12-7 129-00-0

92081500 CA09210081500M
Some analytical aspects of the quantitative determination of
polynuclear aromatic hydrocarbons in fugitive emissions from
coal liquefaction processes
Author: White, Curt M.; Sharkey, A. G., Jr.; Lee, Milton L.;
Vassilatos, Daniel L.
Location: Pittsburgh Energy Technol. Cent., Dep. Energy,
Pittsburgh, PA, 15213, USA
Section: CA089002, CA081XXX, CA080LXX Publ Class: CONF
PROC
Journal: Polynuci. Aromat. Hydrocarbons, Int. Symp. Chem.
Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 79
Pages: 281-75 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avall: Jones, Peter W.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon coal liquefaction,
gas chromatog polynuclear arom hydrocarbon, Tenax sampling
polynuclear hydrocarbon air

CA09210081500M
Descriptors: Air analysis; Aromatic hydrocarbons, polynuclear-
analysis; Coal
Identifiers: detn liquefaction fraction gas chromatog mass
spectroscopy sampling ambient spectrometry emissions
CAS Registry Numbers: 83-32-9 88-01-8 88-73-7 90-12-0
91-20-3 91-57-6 92-52-4 119-64-2 120-12-7 129-00-0 132-64-9
206-44-0 480-72-6 571-61-9 573-96-8 578-37-1 578-41-7 578-43-8
581-42-0 582-16-1 776-35-2 939-27-5 1013-08-7 1079-71-8
24930-84-9 27123-93-3 31281-71-1

92081371 CA09210081371W
Determination of polynuclear aromatic hydrocarbons in the
working environment
Author: Sjoereth, Alf
Location: Cent. Inst. Ind. Res., Oslo, Norway
Section: CA089001, CA081XXX, CA086XXX, CA080LXX Publ
Class: CONF PROC
Journal: Polynuci. Aromat. Hydrocarbons, Int. Symp. Chem.
Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 79
Pages: 371-81 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avall: Jones, Peter W.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon air pollution,
coke plant polynuclear arom hydrocarbon, aluminum plant
polynuclear arom hydrocarbon, urine plant polynuclear arom
hydrocarbon

CA09210081371W
Descriptors: Aromatic hydrocarbons, polynuclear, biological
studies; Carbonization and Coking; Health hazard; Particle size;
Urine
Identifiers: air pollution aluminum coke plants relation
working environments environment plant workers mutagenic
activity
CAS Registry Numbers: 50-32-8 56-55-3 85-01-8 120-12-7
129-00-0 191-07-1 191-24-2 191-26-4 192-97-2 193-39-8 196-19-7
198-85-0 206-82-3 208-98-2 208-44-0 207-08-9 217-58-4
218-01-9 7429-90-5 26911-18-1 30777-18-5 30777-19-6 31711-83-2
41593-25-3 41593-26-4 58615-36-4

92081370 CA09210081370V
Determination of selected polynuclear aromatic hydrocarbons in settled dust by high-performance liquid chromatography with multi-length detection
Author: Fechner, Detlef; Seifert, Bernd
Location: Inst. Wasser-, Boden- Lufthyg. Bundesgesundheits-
ates, Berlin, Fed. Rep. Ger.
Section: CA095001, CA0901XX Pub Class: CNMF PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem.
Biol. - Carcinog. Mutagen., 3rd Coden: 41NSAL Publi: 79
Pages: 191-9 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avail: Jones, Peter W.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon dust, fluorescence
polynuclear arom hydrocarbon dust

CA09210081370V
Descriptores: Air analysis; Air pollution; Aromatic
hydrocarbons, polynuclear analysis; Chromatography, column and
liquid, high-performance; Dust; Spectrochemical analysis, fluoro-
metric
Identifiers: dehn fluorescence detector detection conig
monitoring settled
CAS Registry Numbers: 50-22-8 86-01-8 86-73-7 120-12-7
129-00-0 191-07-1 191-24-2 192-97-2 198-88-0 208-99-2 208-44-0
207-08-9 30777-19-6

9207369 CA09210081369B
Identification of polynuclear aromatic hydrocarbon
mixtures in high-performance liquid chromatography fractions
utilizing the Supel'ok effect
Author: Galesloot, Anders; Stenberg, Ulf
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm,
Swed.
Section: CA095001, CA0731XX, CA0901XX Pub Class: CNMF
PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem.
Biol. - Carcinog. Mutagen., 3rd Coden: 41NSAL Publi: 79
Pages: 121-9 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avail: Jones, Peter W.; Leber, Philip
Identifiers: polynuclear arom hydrocarbon exhaust gas,
quasilinear fluorescence polynuclear arom hydrocarbon

CA09210081369B
Descriptores: Aromatic hydrocarbons, polynuclear analysis;
Spectrochemical analysis, fluorometric
Identifiers: dehn exhaust gases high performance liq
chromatog quasilinear fluorescence
CAS Registry Numbers: 50-22-8 86-88-2 120-00-0 191-07-1
191-24-2 191-26-4 192-97-2 207-08-9 208-1-21-7 208-12-6

92081365 CA09210081365X
Optimization of the phase system in the analysis of
polynuclear aromatics (PNA) from diesel engine exhaust by high
performance liquid chromatography (HPLC)
Author: Roumeliotis, P.; Unger, K. K.; Tesarik, G.;
Muehlberg, E.
Location: Inst. Anorg. Chem. Anal. Chem., Johannes
Gutenberg Univ., Mainz, D-6500, Fed. Rep. Ger.
Section: CA095001 Pub Class: JOURNAL
Journal: Fresenius' Z. Anal. Chem. Coden: ZACFAU Publi:
79 Series: 298 Issue: 4 Pages: 241-9
Identifiers: diesel exhaust polynuclear arom hydrocarbon

CA09210081365X
Descriptores: Aromatic hydrocarbons, polynuclear analysis
Identifiers: dehn diesel exhaust phase system optimization

92079253 CA09210079253B
Portable fluorometric monitor for detection of surface
contamination by polynuclear aromatic compounds
Author: Schurekko, Daniel D.
Location: Chem. Technol. Div., Oak Ridge Natl. Lab., Oak
Ridge, TN, 37830, USA
Section: CA091029, CA0901XX, CA0901XX Pub Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publi: 80 Series:
52 Issue: 2 Pages: 371-3
Identifiers: safety arom hydrocarbon fluorometry, industrial
hygiene arom hydrocarbon, coal liquefaction hydrocarbon
fluorometry, waste coal liquefaction fluorometry,
contamination detector arom hydrocarbon

CA09210079253B
Descriptores: Aromatic hydrocarbons analysis; Coal; Environment
; Fuel oil; Petroleum, synthetic; Shale oils; Solvents; Spectrochem-
ical analysis, fluorometric; Waste
Identifiers: dehn industrial hygiene monitor liquefaction
relation pollution monitoring fluorescence polynuclear arom
portable app

92063683 CA09206063683V
Methods for characterization of complex mixtures of polynuclear aromatic hydrocarbons
Author: Snook, M. E.; Severson, R. F.; Higman, H. C.; Arrandale, R. F.; Chortyk, O. T.
Location: Tob. Health Res. Lab., Sci. Educ. Adm., Athens, GA 30604, USA

Section: CA09206000, CA092060XX Pub Class: COW PROC
Journal: Polynuc. Aromat. Hydrocarbons, Int. Symp. Chem. Biol. - Carcinog. Mutagen., 3rd Coden: 41WSAL Publ: 79
Pages: 231-60 Meeting Date: 78

Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avail: Jones, Peter M.; Leber, Philip
Identifiers: review cigaret smoke condensate analysis, polycyclic aro hydrocarbon analysis review

CA09206063683V
Descriptores: Air analysis;Aromatic hydrocarbons,polycyclic,- analysis;tobacco smoke and smoking,condensates
Identifiers: characterization cigaret relation polynuclear

92061475 CA09206061475R
Characterization of environmental samples for polynuclear aromatic hydrocarbons by an x-ray excited optical luminescence technique
Author: Moo, C. S.; D'Silva, A. P.; Fassel, V. A.

Location: Dep. Chem., Iowa State Univ., Ames, IA, 50011, USA
Section: CA09206000, CA092060XX Pub Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 80 Series: 52 Issue: 1. Pages: 159-64

Identifiers: polynuclear aro hydrocarbon identification environment, x ray excited luminescence hydrocarbon, luminescence analysis polynuclear aro hydrocarbon, coal combustion analysis aro hydrocarbon, conversion coal analysis aro hydrocarbon, shale oil analysis aro hydrocarbon, fuel oil analysis aro hydrocarbon

CA09206061475R
Descriptores: Air analysis;Aromatic hydrocarbons,polynuclear,- analysis;Ashes(residues),fly;Coal;Environment;Fuel gas manuf- acturing,gasification;fuel oil;Shale oils;Solvents;Spectroche- mical analysis,luminescence, x-ray excited
Identifiers: identification environmental samples optical refining excited products exts processing
CAS Registry Numbers: 50-32-8 53-70-3 54-49-5 54-55-3 56-01-8 61-57-8 120-12-7 129-00-0 122-65-0 189-55-9 189-64-0 191-07-1 191-24-2 192-97-2 194-59-2 198-55-0 206-44-0 217-59-4 218-01-9 239-64-5

92044423 CA09206044423A
Characterization of polynuclear aromatic and aliphatic hydrocarbon fractions of solvent-refined coal by glass

capillary gas chromatography/mass spectrometry
Author: Schultz, Rosemary V.; Jorgenson, James M.; Mastaric, Michael P.; Novotny, Milos; Todd, Lee J.
Location: Dep. Chem., Indiana Univ., Bloomington, IN, 47405, USA

Section: CA092060XX Pub Class: JOURNAL
Journal: Fuel Coden: FUELAC Publ: 79 Series: 58 Issue: 11 Pages: 783-9
Identifiers: polynuclear aro aliph coal, gas capillary chromatog coal, mass spectrometry hydrocarbon coal, solvent refined coal assay, partition hydrocarbon coal solvent, cycle oil coal liquefaction

CA09206044423A
Descriptores: Alkanes,analysis;Aromatic hydrocarbons,polycyc- lic,analysis;Chromatography,gas;Coal,solvent-refined;Mass spectroscopy
Identifiers: detn capillary combined aliph

92016451 CA09203016451H
Determination of polynuclear aromatic hydrocarbons in sediment by mass fragmentography
Author: Matsushita, Hajime; Nanya, Takahisa

Location: Fac. Sci., Tokyo Metropol. Univ., Tokyo, 158, Japan

Section: CA092030XX Pub Class: JOURNAL
Journal: Agric. Biol. Chem. Coden: ABCHAG Publ: 79 Series: 43 Issue: 8 Pages: 1633-9
Identifiers: aro hydrocarbon detn sediment Japan, mass fragmentog aro hydrocarbon sediment, polynuclear aro hydrocarbon sediment fragmentog

CA09203016451H
Descriptores: Aromatic hydrocarbons,polynuclear,analysis; Chromatography,gas;Geological sediments;Mass spectroscopy
Identifiers: detn Japan fragmentog combined
CAS Registry Numbers: 50-32-8 53-70-3 56-49-5 56-55-3 58-01-8 120-12-7 129-00-0 191-24-2 192-97-2 198-55-0 206-44-0 218-01-9

DIALOG Filed: CA Search 80-81/Vol 94(12) (See file 104) (Copr. Am. Chem. Soc.) (Item 44 of 47) User 3631 2Apr81 13

92014900 CA09202014900E
Use of micelles in the TLC separation of polynuclear aromatic compounds and amino acids
Author: Armstrong, Daniel W.; McNeely, Marguerite
Location: Dep. Chem., Soudan Coll., Brunswick, ME. 04011.
USA
Section: CA090004 Pub Class: JOURNAL
Journal: Anal. Lett. Coden: ANALBP Pub: 79 Series: 12 Issue: A12 Pages: 1285-91
Identifiers: thin layer chromatog micelle reagent, amino acid thin layer chromatog, arom hydrocarbon thin layer chromatog, polynuclear arom thin layer chromatog, dodecylsulfate solvent thin layer chromatog, octylsulfosuccinate solvent thin layer chromatog

CA09202014900E
Descriptors: Amino acids, analysis; Aromatic hydrocarbons, polynuclear, analysis; Chromatography, thin-layer; Micelles
Identifiers: micellar sodium dodecylsulfate solns eluent; octylsulfosuccinate reverse phase eluents
CAS Registry Numbers: 50-32-8 51-38-4 52-90-4 56-40-6 56-45-1 56-84-6 56-85-9 56-86-0 56-87-1 60-18-4 61-90-5 63-48-2 63-51-2 70-47-3 71-00-1 72-18-4 72-19-5 73-22-3 73-32-5 74-79-3 85-01-8 86-73-7 91-20-3 120-12-7 129-00-0 147-85-3 151-21-3 192-97-2 198-55-0 577-11-7 88867-09-9
Section: CA09202014900E
Journal: Anal. Lett. Coden: ANALBP Pub: 79 Series: 12 Issue: A12 Pages: 1285-91
Identifiers: thin layer chromatog micelle reagent, amino acid thin layer chromatog, arom hydrocarbon thin layer chromatog, polynuclear arom thin layer chromatog, dodecylsulfate solvent thin layer chromatog, octylsulfosuccinate solvent thin layer chromatog

92010606 CA09202010606E
Factor analysis and derivation of an experimental equation on polynuclear aromatic hydrocarbon emissions from automobiles
Author: Ikeda, Takashi; Yamaura, Takaki; Kato, Yoshihiro; Saito, Shoichi; Ishii, Tadashi
Location: Fac. Sci., Sci. Univ. Tokyo, Tokyo, 162, Japan
Section: CA090002 Pub Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTHAG Pub: 79 Series: 13 Issue: 9 Pages: 1077-81
Identifiers: automobile emission polycyclic arom hydrocarbon, exhaust polycyclic arom hydrocarbon equation
CA09202010606E
Descriptors: Aromatic hydrocarbons, polycyclic, uses and miscellaneous; Exhaust gases
Identifiers: automobile calcn equation mileage engine oil relation emission calcn
CAS Registry Numbers: 50-32-8

92010470 CA09202010470E
Determination of average emission rates of polynuclear aromatic hydrocarbons from an automobile
Author: Ikeda, Takashi; Yamaura, Takaki; Kato, Yoshihiro; Saito, Shoichi; Ishii, Tadashi
Location: Fac. Sci., Sci. Univ. Tokyo, Tokyo, Japan
Section: CA090001 Pub Class: JOURNAL

Journal: Talki Osen Gakkaishi Coden: TOSGDC Pub: 79 Series: 14 Issue: 3 Pages: 98-105 Language: Japan
Identifiers: polynuclear arom hydrocarbon exhaust sampling
CA09202010470E
Descriptors: Aromatic hydrocarbons, polycyclic, analysis; Exhaust gases; Sampling
Identifiers: detn comparison methods method

92006293 CA09201006293U
Part I: The synthesis and characterization of some monocarbon carbonates and derivatives. Part II: Characterization of the polynuclear aromatic hydrocarbon and aliphatic fractions of solvent refined coal by capillary-column gas chromatography-mass spectroscopy
Author: Schultz, Rosemary V.
Location: Indiana Univ., Bloomington, IN, USA
Section: CA026001, CA023XXX, CA028XXX, CA081XXX Pub Class: DISS
Codon: D4888A Pub: 79 Pages: 118 pp.
Citation: Diss. Abstr. Int. 8 1978, 40(2), 744
Avail: Univ. Microfilms Int., Order No. 7916925
Identifiers: carbonane monocarbon, hydrocarbon characterization solvent refined coal, arom hydrocarbon polynuclear coal

CA09201006293U
Descriptors: Aromatic hydrocarbons, polynuclear, properties; Carbonanes, monocarbon; Coal, solvent-refined; Hydrocarbons, aliph., properties
Identifiers: characterization prepn

fluorescence spectroscopy polynuclear arom hydrocarbon,
chromatog liq polynuclear arom hydrocarbon

91232065 CA09126322066U
Rapid analysis for polynuclear aromatic hydrocarbons by
linear-sweep differential pulse voltammetry
Author: Burrows, Kerilyn C.; Hughes, Michael C.
Location: Dep. Chem., Lehigh Univ., Bethlehem, PA, 18015,
USA
Section: CA090006 Publ Class: JOURNAL
Journal: Anal. Chim. Acta Coden: ACACAM Publ: 79
Series: 110 Issue: 2 Pages: 255-60
Identifiers: polynuclear arom hydrocarbon detn voltammetry

91205192 CA09126305192B
Rapid sample preparation technique for analyzing
polynuclear aromatic hydrocarbons in sediments by gas
chromatography-mass spectrometry
Author: Tan, Yulin L.
Location: Environ. Meas. Lab., Dep. Energy, New York, NY,
10014, USA
Section: CA0904001 Publ Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 79
Series: 176 Issue: 3 Pages: 319-27
Identifiers: arom hydrocarbon sediment chromatog
spectrometry, polynuclear arom hydrocarbon detn sediment, gas
chromatog arom hydrocarbon sediment, mass spectrometry arom
hydrocarbon sediment

91203998 CA09124203998A
X-ray excited optical luminescence of polynuclear aromatic
hydrocarbons
Author: Destelch, G. J.
Location: Ames Lab., Ames, IA, USA
Section: CA090006 Publ Class: TECH REP
Journal: Report Coden: D3REP3 Publ: 79 Issue:
15-T-856, Pages: 164 pp.
Citation: Energy Res. Abstr. 1979, 4(16), Abstr. No. 43u03
Avail: NTIS
Identifiers: polynuclear arom hydrocarbon analysis
luminescence, x ray excited luminescence analysis

91198417 CA09124198417M
Analysis of Raritan Bay bottom waters for polynuclear
aromatic hydrocarbons
Author: Stalman, Dennis; Frank, Uwe
Location: Ind. Environ. Res. Lab., EPA, Edison, NJ, 08817,
USA
Section: CA091001 Publ Class: JOURNAL
Journal: Bull. Environ. Contam. Toxicol. Coden: BECTAS
Publ: 79 Series: 22 Issue: 4-5 Pages: 480-7
Identifiers: polynuclear arom hydrocarbon Raritan Bay,

91177827 CA09122177827E
Rapid analysis of PNA (polynuclear aromatic) compounds in
complex samples by room temperature phosphorimetry
Author: Vo-Dinh, T.
Location: Oak Ridge Natl. Lab., Oak Ridge, TN, USA
Section: CA051025, CA090XXX Publ Class: TECH REP
Journal: Report Coden: D3REP3 Publ: 78 Issue:
CONF-781150-1, Pages: 11 pp.
Citation: Energy Res. Abstr. 1979, 4(9), Abstr. No. 24068
Avail: NTIS
Identifiers: phosphorimetry polynuclear arom detn, fossil
fuel polynuclear arom, coal liquefied phosphorimetry arom,
synthetic petroleum phosphorimetry arom

91177782 CA09122177782M
Separation and identification of polynuclear aromatic
compounds in coal tar by using glass capillary chromatography
including combined gas chromatography-mass spectrometry
Author: Borwitsky, M.; Schomburg, G.
Location: Max-Planck-Inst. Kohlenforsch., Muelheim/Ruhr,
D-4330, Fed. Rep. Ger.
Section: CA051025, CA090XXX Publ Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 79
Series: 170 Issue: 1 Pages: 99-124
Identifiers: coal tar polynuclear arom, chromatog
hydrocarbon coal tar, gas chromatog coal tar, mass
spectroscopy coal tar

91177871 CA09122177871S
Analysis of polynuclear aromatic hydrocarbons
Author: Brown, Ralph A.; Searl, Thomas D.
Location: Anal. Inf. Div., Exxon Res. and Eng. Co., Linden,
NJ, USA
Section: CA051000, CA090XXX Publ Class: JOURNAL
Journal: Chromatogr. Sci. Coden: CHGSAI Publ: 79
Series: 11 Issue: Chromatogr. Pet. Anal. Pages: 367-94
Identifiers: review polynuclear arom hydrocarbon analysis,
gas chromatog arom hydrocarbon review, UV spectroscopy arom
hydrocarbon review

91167966 CA09120167966X
X-ray excited optical luminescence of polynuclear aromatic hydrocarbons

Author: Gestrreich, Gregory Joseph
Location: Iowa State Univ., Ames, IA, USA
Section: CA080008 Pub Class: DISS
Coden: DUBBMA Publ: 79 Pages: 161 pp.
Citation: Dias. Abstr. Int. 8 1979, 40(1), 203
Avail: Univ. Microfilms Int., Order No. 7916208
Identifiers: polynuclear arom hydrocarbon analysis luminescence, x ray excited luminescence hydrocarbon

91167361 CA09120167361Z
Polynuclear aromatic hydrocarbons associated with coal combustion

Author: Sucre, L.; Jennings, W.; Fisher, G. L.; Rasbe, D. G.; Diachno, J.
Location: Dep. Food Sci. Technol., Univ. California, Davis, CA 95616, USA
Section: CA080001, CA081XXX Pub Class: JOURNAL
Journal: NBS Spec. Publ. (U. S.) Coden: XNBSAV Publ: 79 Series: 819 Issue: Trace Org. Anal.: New Front. Anal. Chem. Pages: 109-20
Identifiers: coal combustion polynuclear arom hydrocarbon

91145344 CA081181453440
Development of a prototype instrument for field monitoring of PMA (polynuclear aromatic compound) vapors

Author: Heathorne, A. R.; Thorngate, J. H.; Gamage, R. B.; Vo-Dinh, T.
Location: Oak Ridge Natl. Lab., Oak Ridge, TN, USA
Section: CA080003, CA081XXX, CA079XXX Pub Class: TECH REP
Journal: Report Coden: D3REP3 Publ: 78 Issue: COM-781038-1, Pages: 21 pp.
Citation: Energy Res. Abstr. 1979, 4(6), Abstr. No. 14147
Avail: NTIS
Identifiers: polynuclear arom compd detn spectrometer

91145267 CA08118145267N
Preliminary thoughts on grey PMA (polynuclear aromatic) compounds in the vapor and solid phase

Author: Gamage, R. B.
Location: Oak Ridge Natl. Lab., Oak Ridge, TN, USA
Section: CA080002, CA081XXX, CA081XXX, CA081XXX Pub Class: TECH REP
Journal: Report Coden: D3REP3 Publ: 78 Issue: COM-781150-5, Pages: 17 pp.
Citation: Energy Res. Abstr. 1979, 4(6), Abstr. No. 31268
Avail: NTIS

Identifiers: polynuclear arom hydrocarbon proxy detn. environment polynuclear arom hydrocarbon proxy

91145161 CA08118145161S
On the analytical potential of micro-Raman spectroscopy in the trace characterization of polynuclear aromatic hydrocarbons

Author: Etz, Edgar S.; Wise, Stephen A.; Heinrich, Kurt F.
J. Location: Cent. Anal. Chem., Natl. Bur. Stand., Washington, DC, 20234, USA
Section: CA080001, CA081XXX, CA080XXX Pub Class: JOURNAL
Journal: NBS Spec. Publ. (U. S.) Coden: XNBSAV Publ: 79 Series: 819 Issue: Trace Org. Anal.: New Front. Anal. Chem. Pages: 723-9
Identifiers: arom hydrocarbon detn environment spectroscopy

9111738 CA08114111738V
A comparison of some chromatographic methods for the estimation of polynuclear aromatic hydrocarbons in pollutants

Author: Burchill, P.; Herod, A. A.; James, R. G.
Location: Coal Res. Establ., Natl. Coal Board, Chatterham/Blox., Engl.
Section: CA080001, CA080XXX Pub Class: TECH REP
Journal: Comm. Eur. Communities, (Rep.) EUR Coden: CECE09 Publ: 78 Issue: EUR 8075, Round Table Meet. Chem. Phys. Valorization Coal, Pages: 206-23 Meeting Date: 77
Identifiers: polynuclear arom hydrocarbons detn, chromatog benzopyrene coke oven

91101942 CA08112101942N
Contamination of some polynuclear aromatic standards

Author: Frycka, Josef
Location: Res. Inst. Coal Tar Chem., Urzov zavody N. P., Valasske Mezirici, 757 27, Czech.
Section: CA080001, CA081XXX Pub Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 79 Series: 174 Issue: 2 Pages: 488-9
Identifiers: polynuclear arom hydrocarbon std contamination, gas chromatog polynuclear arom std, phenanthrene std contamination, anthracene std contamination

- 91096407 CA09112064078
 Routine liquid chromatographic method for assessing polynuclear aromatic hydrocarbon pollution in fresh water environments
 Author: Black, J. J.; Dymerski, P. P.; Zapisek, W. F.
 Location: Roswell Park Res. Inst., Div. New York State Dep. Health, Buffalo, NY 14263, USA
 Section: CA091003, CA0791XX Pub Class: JOURNAL
 Journal: Bull. Environ. Contam. Toxicol. Coden: BECTAG
 Pub: 79 Series: 22 Issue: 1-2 Pages: 278-84
 Identifiers: polynuclear arom hydrocarbon estn river
- 9102566 CA09110082646
 Development of an aqueous polynuclear aromatic hydrocarbon standard reference material
 Author: May, W. E.; Brown, J. M.; Chesler, S. M.; Guenther, F.; Hilpert, L. B.; Hertz, H. S.; Wise, S. A.
 Location: Natl. Bur. Stand., Washington, DC 20234, USA
 Section: CA090003, CA0791XX Pub Class: JOURNAL
 Journal: NBS Spec. Pub. (U. S.) Coden: NBSAV Pub: 79 Series: 819 Issue: Trace Org. Anal.: New Front. Anal. Chem. Pages: 219-24
 Identifiers: std ref polynuclear arom hydrocarbon, liq chromatog polynuclear arom hydrocarbon
- 91081729 CA09100081729Y
 Chromatographic determination of polynuclear aromatic hydrocarbons in the environment
 Author: Korol, A. M.; Lysyuk, L. S.
 Location: Inst. Phys. Chem., Kiev, USSR
 Section: CA090000, CA0791XX Pub Class: JOURNAL
 Journal: Zh. Anal. Khim. Coden: ZAKJAB Pub: 79 Series: 34 Issue: 3 Pages: 877-80 Language: Russ
 Identifiers: review polynuclear arom hydrocarbon detn, gas chromatog arom hydrocarbon review
- 91032434 CA09104022434Q
 Application of second-derivative UV absorption spectrometry to polynuclear aromatic compound analysis
 Author: Iewthorne, Alan B.; Thorngate, John H.
 Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak Ridge, TN 37830, USA
 Section: CA090006, CA0901XX Pub Class: JOURNAL
 Journal: Appl. Spectrosc. Coden: APSPAA Pub: 79 Series: 33 Issue: 3 Pages: 301-6
 Identifiers: UV second deriv polynuclear arom, polynuclear arom detn UV photometry, health protection polynuclear arom detn
- 91013188 CA09102013188U
 Entropy dominated high performance liquid chromatographic separations of polynuclear aromatic hydrocarbons. Temperature as a separation parameter
 Author: Chelkowski, J.; Sawatzky, H.
 Location: Canada Cent. Miner. Energy Technol., Ottawa, ON, KIA 0G1, Can.
 Section: CA090004, CA091XX Pub Class: JOURNAL
 Journal: J. Chromatogr. Sci. Coden: JCHS82 Pub: 79 Series: 17 Issue: 8 Pages: 248-52
 Identifiers: entropy high performance liq chromatog, polynuclear arom hydrocarbon liq chromatog, temp effect liq chromatog thermodyn
- 91009279 CA09102009279J
 On methodology of polynuclear aromatic hydrocarbon determination in water
 Author: Dudkiewicz, T.; Rybors, S.; Mastowski, J.
 Location: Cent. Environ. Dev., Res. Inst. Environ. Dev., Katowice, Pol.
 Section: CA091002, CA0191XX, CA0909XX, CA090XXX Pub Class: JOURNAL
 Journal: Environ. Prot. Eng. Coden: EPEND9 Pub: 78 Series: 4 Issue: 3 Pages: 283-6
 Identifiers: polynuclear arom hydrocarbon detn water, air polynuclear arom hydrocarbon detn, soil polynuclear arom hydrocarbon detn
- 91008964 CA09102008964S
 Analysis for polynuclear aromatic hydrocarbons in working atmospheres by computerized gas chromatography-mass spectrometry
 Author: Bjoerseth, Alf; Eklund, Goran
 Location: Cent. Inst. Ind. Res., Oslo, 3, Norway
 Section: CA090002, CA0901XX Pub Class: JOURNAL
 Journal: Anal. Chim. Acta Coden: ACACAM Pub: 79 Series: 106 Issue: 1 Pages: 119-28
 Identifiers: polynuclear arom hydrocarbon detn air

9100875 CA09102004875P
The separation and determination of polynuclear aromatic hydrocarbons by high-performance liquid chromatography: Part I

British Carbonization Research Assoc.
Location: Chesterfield/Derbyshire, S42 6J5, Engl.
Section: CA080002, CA0811XX Publ Class: JOURNAL
Journal: Carbonization Res. Rep. Coden: CRREDS
76 Series: 28, Pages: 19 pp.
Identifiers: arom hydrocarbon detn fuel gas, benzopyrene detn chromatog fuel gas

90202268 CA09025202268X
Determination of polynuclear aromatics in shellfish by high-pressure liquid chromatography
Author: Harue, James P.
Location: Food Drug Adm., Dallas, Tex.
Section: CA017001, CA0041XX Publ Class: JOURNAL
Journal: EDR0 SARAP Res. Tech. Rep. Coden: ESRRDS
Publ: 78 Series: 3, Pages: 51-67
Identifiers: polycyclic hydrocarbon detn oyster, chromatog polycyclic hydrocarbon

90197162 CA09024197162S
Analysis of polynuclear aromatic hydrocarbons using high-speed liquid chromatography
Author: Kanata, Kunihiko; Kan, Teruo; Yamazoe, Ritsuko; Harada, Hirofumi
Location: Tokyo Metrop. Res. Lab. Public Health, Tokyo, Japan
Section: CA080004 Publ Class: JOURNAL
Journal: Tokyo-toritsu Eisai Kenkyusho Kenkyu Memo
Codon: TREMAF Publ: 77 Series: 28-1, Pages: 95-9
Language: Japan
Identifiers: high speed liq chromatog, polynuclear arom hydrocarbon liq chromatog, benzopyrene liq chromatog

90195011 CA09023185011Q
Polynuclear aromatic hydrocarbons in food additives. (III). Analysis of benzo(a)pyrene in caramel
Author: Hirokado, Masako; Nakajima, Iwao; Usami, Hiroyuki; Endo, Fuyuyoshi
Location: Tokyo Metrop. Res. Lab. Public Health, Tokyo, Japan
Section: CA017001 Publ Class: JOURNAL
Journal: Tokyo-toritsu Eisai Kenkyusho Kenkyu Memo
Codon: TREMAF Publ: 77 Series: 28-1, Pages: 184-8
Language: Japan
Identifiers: benzopyrene caramel detn

90185009 CA09023185009J
Polynuclear aromatic hydrocarbon in food additives. (IV). Analysis of benzo(a)pyrene in activated carbon and carbon black
Author: Nakajima, Iwao; Hirokado, Masako; Usami, Hiroyuki; Mizoiri, Shigeru; Endo, Fuyuyoshi
Location: Tokyo Metrop. Res. Lab. Public Health, Tokyo, Japan
Section: CA017001 Publ Class: JOURNAL
Journal: Tokyo-toritsu Eisai Kenkyusho Kenkyu Memo
Codon: TREMAF Publ: 78 Series: 29-1, Pages: 203-5
Language: Japan
Identifiers: benzopyrene detn carbon black, activated carbon benzopyrene detn

90173795 CA09022173795Z
Determination of benzo(a)pyrene and other polynuclear aromatic hydrocarbons in airborne particulate material by ultrasonic extraction and reverse phase high pressure liquid chromatography
Author: Golden, C.; Seivick, E.
Location: Off. Res. Dev., EPA, Research Triangle Park, N. C.
Section: CA080002, CA0801XX
Journal: Anal. Lett. Coden: ANALBP Publ: 78 Series: All Issue: 12 Pages: 1051-62
Identifiers: benzopyrene detn air particle, polynuclear arom hydrocarbon detn air

90145358 CA09018145358R
Vacuum sublimation of polynuclear aromatic hydrocarbons separated by thin-layer chromatography for detection with Shpol'skii low-temperature fluorescence
Author: Colasjo, Anders; Stenberg, Ulf
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, Sued.
Section: CA080004 Publ Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 79 Series: 168, Pages: 206-12
Identifiers: vacuum sublimation polynuclear arom hydrocarbon, thin layer chromatog arom hydrocarbon, fluorescence detection polynuclear arom hydrocarbon, benzopyrene chromatog sublimation fluorescence detection, pyrene chromatog sublimation fluorescence detection, benzopyrene chromatog sublimation fluorescence detection

- 90148257 CA090181453670
Analysis of polynuclear aromatic hydrocarbons by glass capillary gas chromatography using simultaneous flame ionization and electron capture detection
Author: Bjorseth, Alf; Eklund, Goran
Location: Cent. Inst. Ind. Res., Oslo, Norway
Section: CA090004, CA090004X Pub Class: JOURNAL
Journal: HEC CC, J. High Resolut. Chromatogr. Chromatogr. Commun. Coden: HECJCH Publi: 79 Series: 3 Issue: 1 Pages: 23-6
Identifiers: polynuclear aromatic hydrocarbon detn environment, gas chromatog polynuclear aromatic hydrocarbon, environment, analysis polynuclear aromatic hydrocarbon, flame ionization detection polynuclear aromatic, electron, capture detection polynuclear aromatic, atm particulate analysis polynuclear aromatic
- 90141591 CA09018141591P
Synchronous spectroscopy for analysis of polynuclear aromatic compounds
Author: Vo-Binh, Tuan; Gammage, Richard B.; Hawthorne, Alan R.; Thorngate, John M.
Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak Ridge, Tenn.
Section: CA090003 Publi Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTHAG Publi: 78 Series: 12 Issue: 12 Pages: 1297-302
Identifiers: polynuclear aromatic compd detn water
- 90133777 CA09017133777P
Nitrogen analogs of polynuclear aromatic hydrocarbons in tobacco smoke
Author: Snook, M. E.
Location: Tob. Lab., ARS, Athens, Ga.
Section: CA090013 Publi Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publi: 78 Series: 3 Issue: Polynuc. Aromat. Hydrocarbons Pages: 203-18
Identifiers: smoke, hetero analog polynuclear aromatic hydrocarbon, nitrogen analog polycyclic aromatic hydrocarbon smoke
- 90141579 CA09014141579J
Room temperature phosphorescence of several pharmaceutical preparations and polynuclear aromatic hydrocarbons
Author: Bover, Esther Almeida
Location: Univ. Florida, Gainesville, Fla.
Section: CA090008, CA07311X Publi Class: DISS
Codon: 042884 Publi: 78 Pages: 186 pp.
Citation: Diss. Abstr. Int. B 1979, 38(7), 3346
Avail: Univ. Microfilms Int., Order No. 7900036
Identifiers: phosphorimetry pharmaceutical aromatic hydrocarbon
- analyses, pharmaceutical analysis room temp phosphorimetry, polynuclear aromatic hydrocarbon analysis phosphorimetry
- 90086084 CA090130860840
The effect of simple environment on the room-temperature phosphorescence of several polynuclear aromatic hydrocarbons
Author: Bover, Esther Almeida; Winefordner, J. D.
Location: Dep. Chem., Univ. Florida, Gainesville, Fla.
Section: CA090001 Publi Class: JOURNAL
Journal: Anal. Chim. Acta Coden: ACACAM Publi: 78 Series: 102 Pages: 1-13
Identifiers: aromatic hydrocarbon phosphorescence, tellurium aromatic hydrocarbon phosphorescence
- 90085380 CA09011085380E
High pressure liquid chromatographic determination of polynuclear aromatic hydrocarbons in oysters
Author: Harnes, James P.; Guerrero, Humberto; Biehl, Edward R.; Kerner, Charles T.
Location: Food Drug Adm., Dallas, Tex.
Section: CA017001, CA0900XX Publi Class: JOURNAL
Journal: J. Assoc. Off. Anal. Chem. Coden: JANCA2 Publi: 79 Series: 62 Issue: 1 Pages: 29-35
Identifiers: polynuclear aromatic hydrocarbon oyster, liq chromatog hydrocarbon oyster
- 90075817 CA09010075817P
Gel elution of heterocyclic analogs of polynuclear aromatic hydrocarbons from bio-beads
Author: Snook, M. E.
Location: Tob. Lab., ARS, Athens, Ga.
Section: CA090002, CA0900XX Publi Class: JOURNAL
Journal: Anal. Chim. Acta Coden: ACACAM Publi: 78 Series: 99 Issue: 2 Pages: 299-304
Identifiers: aromatic hydrocarbon detn air chromatog, bio bead gel elution

- 90043309 CA0900043309H
Identification of polynuclear aromatic hydrocarbons by
Spectral low temperature fluorescence
Author: Colasjo, Anders; Sterberg, Ulf
Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm,
Sweden.
Section: CA090002, CA0791XX Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 79 Series:
51 Issue: 1 Pages: 145-50
Identifiers: polynuclear aromatic hydrocarbon detn. fluorescence,
exhaust polynuclear aromatic hydrocarbon detn. waste gas
polynuclear aromatic hydrocarbon
- 90039116 CA0900039116A
Determination of bifunctional compounds, part III.
Polynuclear aromatic benzoic acids as selective fluorescent
reagents for HPLC and HPLC
Author: Poole, C. F.; Singhavangcha, S.; Zlatkic, A.;
Morgan, E. D.
Location: Dep. Chem., Univ. Houston, Houston, Tex.
Section: CA032006, CA023XXX, CA024XXX, CA080XXX
Publ Class: JOURNAL
Journal: HRC CC, J. High Resolut. Chromatogr. Chromatogr.
Commun. Coden: HRCJCB Publ: 79 Series: 1 Issue: 2
Pages: 96-7
Identifiers: high pressure liq chromatog., thin layer
chromatog high pressure, naphthaleneboronate pinacol chromato
g., pinacol naphthaleneboronate chromatog., phenanthreneboronate
chromatog sepn, ecdysterone phenanthreneboronate
chromatog sepn
- 90036496 CA0900036496A
Multi-alkylated polynuclear aromatic hydrocarbons of tobacco
smoke: separation and identification
Author: Snook, M. E.; Severson, R. F.; Arrandale, R. F.;
Higman, M. C.; Chortyk, O. T.
Location: Tob. Lab., Sci. Educ. Adm., Athens, Ga.
Section: CA010007, CA0091XX Publ Class: JOURNAL
Journal: Beitr. Tabakforsch. Coden: BETAAV Publ: 78
Series: 9 Issue: 4 Pages: 222-47
Identifiers: polynuclear aromatic hydrocarbon cigaret smoke
- 90012112 CA0900012112K
Separation by high performance liquid chromatography of
polynuclear aromatic hydrocarbons listed by the World Health
Organization as pollution indicators in drinking water
Author: Hunt, B. C.; Wild, P. J.; Crosby, M. T.
Location: Dep. Ind., Lab. Gov. Chem., London, Engl.
Section: CA051008 Publ Class: JOURNAL
Journal: Water Res. Coden: WATERB Publ: 78 Series:
- 12 Issue: 6 Pages: 643-4
Identifiers: phthalisodipropyltrichlorosilane chromatog
packing, polynuclear aromatic hydrocarbon sepn
- 89230046 CA089230046X
Chromatographic and spectral analysis of polynuclear
aromatic hydrocarbons - quantitative distribution in air of
Ontario cities
Author: Katz, Morris; Sakuma, Takao; Ho, Andrew
Location: Cent. Res. Environ. Qual., York Univ., Toronto,
Ont.
Section: CA089002, CA0791XX Publ Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTHAG Publ: 78
Series: 12 Issue: 8 Pages: 909-15
Identifiers: polycyclic aromatic hydrocarbon detn particulate,
thin layer chromatog aromatic hydrocarbon, fluorescence
spectroscopy polycyclic aromatic hydrocarbon
- 89203126 CA08924203126J
A comparison of some chromatographic methods for estimation
of polynuclear aromatic hydrocarbons in pollutants
Author: Burchill, P.; Herod, A. A.; James, R. G.
Location: Coal Res. Estab., Natl. Coal Board,
Chatterhouse/Glos., Engl.
Section: CA089001, CA080XXX Publ Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ:
78 Series: 3 Issue: Polynuc. Aromat. Hydrocarbons
Pages: 35-45
Identifiers: polynuclear aromatic hydrocarbon analysis, gas
chromatog polynuclear aromatic hydrocarbon, mass spectroscopy
polynuclear aromatic hydrocarbon
- 89203112 CA08924203112Z
Analytical methods for polynuclear aromatic hydrocarbons
Author: Schmitt, Irwin; Brunemann, Klaus D.; Hoffmann,
Dietrich
Location: Naylor Dana Inst. Dis. Prev., Am. Health Found.,
Valhalla, N. Y.
Section: CA089000, CA080XXX Publ Class: CONF PRPC
Journal: Prev. Detect. Cancer, (Proc. Int. Symp.), 3rd
Codan: 27ATAD Publ: 78 Series: 1 Issue: 2 Pages:
1973-92 Meeting Date: 76
Publisher: Dekker Address: New York, N. Y
Avail: Nisbarga, Herbert E
Identifiers: review polynuclear aromatic hydrocarbon detn,
tobacco smoke aromatic hydrocarbon review

89100912 CA080241999120
A review of methods for the estimation of polynuclear aromatic hydrocarbons with particular reference to coke oven emissions

Author: Herod, A. A.; James, R. G.
Location: Coal Res. Establ., Natl. Coal Board, Stoke Orchard/Cheltenham, Engl.
Section: CA081000, CA0801XX, CA0801XX Publ Class: JOURNAL
Journal: J. Inst. Fuel Coden: JIFUA4 Publ: 78
Series: 51 Issue: 408 Pages: 164-77
Identifiers: review polynuclear arom hydrocarbon detn, coke oven emission hydrocarbon review, gas chromatog arom hydrocarbon review, liq chromatog arom hydrocarbon review, mass spectrometry arom hydrocarbon review, thin layer chromatog hydrocarbon review

89100916 CA08020169163V
Determination of polynuclear aromatic hydrocarbons in poly(vinyl chloride) smoke particulates by high pressure liquid chromatography and gas chromatography-mass spectrometry
Author: Liao, John C.; Browner, Richard R.
Location: Sch. Aeronaut. Eng., Georgia Inst. Technol., Atlanta, Ga.

Section: CA080002, CA0801XX, CA0801XX Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 50 Issue: 12 Pages: 1683-6
Identifiers: polynuclear arom hydrocarbon PVC smoke, chromatog PVC smoke analysis, gas chromatog PVC smoke analysis

89150222 CA08019150222V
Analysis of polynuclear aromatic hydrocarbons and organochlorine pollutants in Great Lakes herring gulls by high resolution gas chromatography
Author: Hallett, Douglas J.; Norstrom, Ross J.; Orntaka, Francis J.; Coombe, Michael E.
Location: Can. Wildl. Serv., Dep. Fish. Environ., Ottawa, Ont.

Section: CA004001 Publ Class: CONF PHOC
Journal: Glass Capillary Chromatogr., Int. Symp., 2nd Coden: 36CKAA Publ: 77 Pages: 115-25
Publisher: Inst. Chromatogr. Address: End Duerkheim, Ger
Avail: Kaiser, Rudolf E
Identifiers: hydrocarbon organochlorine gull chromatog, herring gull pollutant chromatog

89152408 CA08019152408Q
Monitoring of polynuclear aromatic hydrocarbons in water. II. Extraction and recovery of six representative compounds with polyurethane foams

Author: Basu, Dipak K.; Saxena, Jitendra
Location: Cent. Chem. Hazard Assessment, Syracuse Res. Corp., Syracuse, N. Y.
Section: CA081002, CA0801XX Publ Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTIMQ Publ: 78
Series: 12 Issue: 7 Pages: 791-5
Identifiers: arom hydrocarbon polynuclear detn water, polychlorinated arom hydrocarbon polynuclear concn, hydrocarbon arom polynuclear detn water

89152407 CA08019152407P
Methods for polynuclear aromatic hydrocarbon analysis in the marine environment
Author: Wise, S. A.; Chesler, S. N.; Heriz, H. S.; Hilpert, L. B.; May, V. E.
Location: Inst. Mater. Res., Natl. Bur. Stand., Washington, D. C.

Section: CA081002, CA0801XX Publ Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: 76 Series: 3 Issue: Polynuci. Aromat. Hydrocarbons Pages: 175-82
Identifiers: hydrocarbon arom polynuclear detn seawater, sediment polynuclear arom hydrocarbon detn

89151729 CA08019151729H
Measurement of polynuclear aromatic hydrocarbons in diesel exhaust gases
Author: Brickleyer, B. A.; Spindt, R. S.
Location: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CA080002 Publ Class: JOURNAL
Journal: SAE (Tech. Pap.) Coden: SAEPCY Publ: 78 Series: 780115, Pages: 12 pp.
Identifiers: polynuclear arom hydrocarbon exhaust, exhaust gas hydrocarbon measurement, diesel engine exhaust hydrocarbon, benzopyrene diesel engine exhaust, benzoanthracene diesel engine exhaust, pollution air diesel exhaust

89152456 CA08019152456P
Polynuclear aromatic hydrocarbons in selected U.S. drinking waters and their raw water sources
Author: Basu, Dipak K.; Saxena, Jitendra
Location: Cent. Chem. Hazard Assessment, Syracuse Res. Corp., Syracuse, N. Y.
Section: CA081001 Publ Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTIMQ Publ: 78
Series: 12 Issue: 7 Pages: 795-9
Identifiers: arom hydrocarbon polynuclear water, drinking water polynuclear arom hydrocarbon

8917428 CA089141174282
Method development and monitoring of polynuclear aromatic hydrocarbons in selected US waters
Author: Saxena, J.; Basu, D. K.; Kozuchowski, J.
Location: Syracuse Res. Corp., Syracuse, N. Y.
Section: CA081002 Publ Class: TECH REP
Journal: U. S. NTIS, PB Rep. Coden: XPBRCA Publ: 77
Issues: PB-276435, Pages: 97 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1978, 78(9), 181
Avail: NTIS
Identifiers: polynuclear arom hydrocarbon water

89101057 CA089121010573
Determination of polynuclear aromatic hydrocarbons contaminated with chlorinated hydrocarbon pesticides
Author: Negishi, Takashi
Location: Dep. Agro-Environ. Sci., Obihiro Univ. Agric. Vet. Med., Obihiro, Japan
Section: CA004001 Publ Class: JOURNAL
Journal: Bull. Environ. Contam. Toxicol. Coden: BECTAB
Publ: 78 Series: 19 Issue: 5 Pages: 545-8
Identifiers: hydrocarbon gas chromatog mass spectrometry

89084346 CA08912084346E
New generation of monitors for PAH's (polynuclear aromatic hydrocarbons) from synthetic fuel production
Author: Gamage, R. S.; Vo Dinh Tuan; Hawthorne, A. R.; Thorngate, J. H.; Parkinson, W. W.
Location: Oak Ridge Natl. Lab., Oak Ridge, Tenn.
Section: CA089003, CA081XXX, CA080XXX Publ Class: TECH REP
Journal: Report Coden: D3REP3 Publ: 77 Issue: CBE-770863-3, Pages: 40 pp.
Citation: Energy Res. Abstr. 1978, 3(8), Abstr. No. 16870
Avail: NTIS
Identifiers: polynuclear arom hydrocarbon detn air

89079470 CA08910079470X
Field evaluation and comparison of sampling matrices for polynuclear aromatic hydrocarbons in occupational atmospheres
Author: Jackson, James O.; Cuppe, James A.
Location: Mod. Health Resour. Div., Gulf Sci. and Technol. Co., Pittsburgh, Pa.
Section: CA089002 Publ Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSIDL Publ: 78 Series: 3 Issue: Polynuc. Aromat. Hydrocarbons
Pages: 183-91
Identifiers: polynuclear arom hydrocarbon air sampler

89079469 CA089100794690
A new generation of monitors for polynuclear aromatic hydrocarbons from synthetic fuel production
Author: Gamage, R. S.; Vo-Dinh, Tuan; Hawthorne, A. R.; Thorngate, J. H.; Parkinson, W. W.
Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak Ridge, Tenn.
Section: CA089002, CA081XXX, CA073XXX Publ Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSIDL Publ: 78 Series: 3 Issue: Polynuc. Aromat. Hydrocarbons
Pages: 155-74
Identifiers: polynuclear arom hydrocarbon air monitoring, fuel processing polynuclear hydrocarbon monitoring

89071880 CA08909071880X
Carcinogenesis - A Comprehensive Survey, Vol. 3: Polynuclear Aromatic Hydrocarbons: 2nd International Symposium on Analysis, Chemistry, and Biology
Author: Jones, Peter M.; Freudenthal, Ralph I.; Editors
Location: USA
Section: CA004007 Publ Class: BOOK
Codex: 800KA7 Publ: 78 Pages: 487 pp.
Publisher: (Raven Press Address: New York, N. Y.)
Identifiers: book polynuclear arom hydrocarbon carcinogen

89070474 CA08908070474U
Detectors for liquid chromatographic analysis for polynuclear aromatic hydrocarbons
Author: Christensen, R. G.; May, W. E.
Location: Anal. Chem. Div., Natl. Bur. Stand., Washington, D. C.
Section: CA089002 Publ Class: JOURNAL
Journal: J. Liq. Chromatogr. Coden: JLCHE8 Publ: 78 Series: 1 Issue: 3 Pages: 385-99
Identifiers: liq chromatog detector arom hydrocarbon, environment analysis polynuclear arom hydrocarbon, high performance liq chromatog

89048972 CA08904048972X
Semi-quantitative and quantitative methods for the determination of polynuclear aromatic hydrocarbons in bituminoids and petroleum
Author: Sabina, M. N.
Location: USSR
Section: CA081001, CA0801XX Publ Class: JOURNAL
Journal: Tr. Sib. Nauchno-Issled. Inst. Geol. Geofiz. Miner. Syr'ya Coden: TSIGAL Publ: 76 Series: 231, Pages: 144-8 Language: Russ
Identifiers: petroleum arom luminescence detn, bituminoid arom luminescence detn, polynuclear arom luminescence detn

89011851 CA089020118515
Determination of aqueous chlorination reaction products of polynuclear aromatic hydrocarbons by reversed phase high performance liquid chromatography-gas chromatography
Author: Gylar, A. R.; Bodamer, D. L.; Welch, K. J.; Lukonen, P. D.; Arison, B. R.; Kappelman, H. L.; Caple, R.
Location: Dep. Chem., Univ. Minnesota, Duluth, Minn.
Section: CA081002, CA0801XX Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 78 Series: 50 Issue: 7 Pages: 837-42
Identifiers: arom hydrocarbon detn water, anthraquinone detn water, phenanthrene detn water, methylphenanthrene detn water, methylnaphthalene detn water

88194676 CA08826194676N
The use of a combination of ultraviolet and fluorescence detectors for the selective detection and quantitation of polynuclear aromatic hydrocarbons by high pressure liquid chromatography
Author: Sallie, R. D.; Meng, D. T.; Horecz, D.
Location: Lab. Serv. Branch, Ontario Minist. Environ., Reville, Ont.
Section: CA089002, CA0801XX Publ Class: JOURNAL
Journal: J. Environ. Sci. Health, Part A Coden: JESEBU Publ: 78 Series: A13 Issue: 1 Pages: 47-59
Identifiers: arom hydrocarbon detn air chromatog

88182058 CA08824182058X
Separation of polynuclear aromatic hydrocarbons by reversed phase thin-layer chromatography
Author: Shirahishi, Yoshiko; Yamashita, Tadao; Shiratori, Tsuyako
Location: Natl. Inst. Public Health, Tokyo, Japan
Section: CA080004 Publ Class: JOURNAL
Journal: Eisei Kagaku Coden: ESKGJ2 Publ: 77 Series: 23 Issue: 5 Pages: 210-12 Language: Japan
Identifiers: polynuclear arom hydrocarbon chromatog, thin

layer chromatog arom hydrocarbon, reversed phase thin layer chromatog

88131516 CA08819131516Q
Identification of environmental polynuclear aromatic hydrocarbons by pulse Fourier-transforma proton nuclear magnetic resonance spectroscopy
Author: Bartle, K. D.; Lee, M. L.; Novotny, M.
Location: Dep. Phys. Chem., Univ. Leeds, Leeds, Engl.
Section: CA004001 Publ Class: JOURNAL
Journal: Analyst (London) Coden: ANALAO Publ: 77 Series: 102 Issue: 1219 Pages: 721-8
Identifiers: polynuclear arom hydrocarbon identification, NMR Fourier transforma hydrocarbon, air polynuclear arom hydrocarbon, tobacco smoke polynuclear hydrocarbon, barbiturates polynuclear hydrocarbon

88123566 CA08818123566P
Polynuclear aromatic hydrocarbons in coal - identification by their x-ray excited optical luminescence
Author: Woo, Ching S.; D'Silva, Arthur P.; Fassel, Valter A.; Oestreich, Gregory J.
Location: Ames Lab., Iowa State Univ., Ames, Iowa
Section: CA081018, CA0801XX Publ Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTHAG Publ: 78 Series: 12 Issue: 2 Pages: 173-4
Identifiers: arom hydrocarbon coal x ray, luminescence arom hydrocarbon coal, safety carcinogen coal detn

88083781 CA088100457815
Open-pore polyurethane columns for collection and preconcentration of polynuclear aromatic hydrocarbons from water
Author: Navratil, James D.; Siewers, Robert E.; Walton, Harold F.
Location: Rocky Flats Plant, Rockwell Int., Golden, Colo.
Section: CA081002, CA0371XX Publ Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 77 Series: 49 Issue: 14 Pages: 2360-3
Identifiers: polyurethane pyrene removal water, urethane polymer pyrene removal water, hydrocarbon removal water

89038415 CA08004028415E
Determination of the aqueous solubility of polynuclear aromatic hydrocarbons by a coupled column liquid chromatographic technique
Author: May, Willie E.; Vesik, Stanley P.; Freeman, David H.
Location: Inst. Mater. Res., Natl. Bur. Stand., Washington, D. C.
Section: CA080001, CA080011, CA080012 Pub. Class: JOURNAL
Journal: Anal. Chem. Coden: ANCHAM Publ: 78 Series: 50
Issue: 1 Pages: 175-8
Identifiers: only detn arom liq chromatog. polynuclear arom
only detn chromatog

89011156 CA08002011156Y
Polynuclear aromatic content of heavy duty diesel engine exhaust gases
Author: Spindt, R. S.
Location: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CA080001, CA080011, CA080012 Pub. Class: JOURNAL
Journal: U. S. NTIS, PB Rep. Coden: XPERCA Publ: 77
Issue: PB-267774, Pages: 80 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1977, 77(16), 100
Avail: NTIS
Identifiers: polynuclear arom detn exhaust gas, hydrocarbon
arom detn exhaust gas

87210497 CA08726210497B
Advances in the analysis of polynuclear aromatics
Author: Sauerland, H. D.; Stadthofer, J.; Thoms, R.; Zander, M.
Location: Ruetgerwerke A.-G., Castrop-Rauxel, Ger.
Section: CA080001, CA080011, CA080012 Pub. Class: JOURNAL
Journal: Compend. - Tech. Ges. Mineralogwiss. Kohlechem.
Codon: C80006 Publ: 76 Series: 76-77 Issue: 2
Pages: 961-7 Language: Ger
Identifiers: polynuclear arom hydrocarbon anal, gas chromatog polynuclear arom hydrocarbon, liq chromatog polynuclear arom hydrocarbon, fluorescence polynuclear arom hydrocarbon, NMR polynuclear arom hydrocarbon

87192286 CA08724192286A
Chemically-bonded aminostilbene stationary phase for the high-performance liquid chromatographic separation of polynuclear aromatic compounds
Author: Wise, S. A.; Cheeler, S. M.; Hertz, H. S.; Hilpert, L. R.; May, W. E.
Location: Inst. Mater. Res., Natl. Bur. Stand., Washington, D. C.
Section: CA080004 Pub. Class: JOURNAL

Journal: Anal. Chem. Coden: ANCHAM Publ: 77 Series: 49
Issue: 14 Pages: 2306-10
Identifiers: aminostilbene bonded phase liq chromatog. stationary phase aminostilbene liq chromatog. high performance liq chromatog. polynuclear arom liq chromatog. hydrocarbon aliph liq chromatog

87184378 CA08720184378N
News in the analysis of polynuclear aromatics
Author: Sauerland, H. D.; Stadthofer, J.; Thoms, R.; Zander, M.
Location: Ruetgerwerke A.-G., Castrop-Rauxel, Ger.
Section: CA080001, CA080011, CA080012 Pub. Class: JOURNAL
Journal: Erdöl Kohle, Erdgas, Petrochem. Coden: EKEPAB
Publ: 77 Series: 30 Issue: 5 Pages: 215-18
Language: Ger
Identifiers: polycyclic arom hydrocarbon analysis, chromatog polycyclic arom hydrocarbon, column chromatog arom hydrocarbon, gas chromatog arom hydrocarbon, fluorescence spectroscopy fluoranthene deriv, NMR carbon 13 arom hydrocarbon

87184137 CA08720184137N
Adsorbent for polynuclear aromatic compounds
Author: Stalling, David L.; Muckins, James M.; Seith, William Allen
Location: USA
Section: CA080001, CA080011, CA080012 Pub. Class: PAT
Journal: U. S. Pat. Appl. Coden: KAKXAV Publ: 761018
Pages: 7 pp. Avail: NTIS
Patent No: 733500 Applic No: 733500 Date: 761018
Assignee: United States Dept. of the Interior
Identifiers: charcoal polyurethane adsorbent, polynuclear arom comp adsorbent, chlorobenzodioxin detn, dioxin deriv detn

87106807 CA08714106807O
Gas chromatographic method for the analysis of major polynuclear aromatics in particulate matter
Author: John, E. D.; Nickless, G.
Location: Dep. Inorg. Chem., Univ. Bristol, Bristol, Engl.
Section: CA080002, CA080011, CA080012 Pub. Class: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 77
Series: 138 Issue: 2 Pages: 399-412
Identifiers: polynuclear arom hydrocarbon sediment, gas chromatog sediment

87089825 CA0871089825
The high pressure liquid chromatography and its application
to the separation of polynuclear aromatic hydrocarbons in
atmospheric dust and burning residues
Author: Lopez, M. C.
Location: GEN. Comis. Enorg. Al., Grenoble, Fr.
Section: CA089001, CA0801XX Publ Class: TECH REP
Journal: Report Coden: D2REPU Publ: 75 Issue:
CEA-R-4678, Pages: 44 pp. Language: fr
Citation: IMIS Atomindex 1978, 7(7), Abstr. No. 232867
Avail: IMIS

Identifiers: sep polycyclic hydrocarbon chromatog. dust
polycyclic hydrocarbon chromatog. combustion residue
hydrocarbon chromatog

87079184 CA0871079184
Monitoring of polynuclear aromatic hydrocarbons in water.
I. Extraction and recovery of benzo(a)pyrene with porous
polyurethane foam
Author: Szere, Jitendra; Kozuchowski, Jack; Bau, Dipak K.
Location: Cent. Chem. Hazard Assessment, Syracuse Res. Corp.
Syracuse, N. Y.
Section: CA004001 Publ Class: JOURNAL
Journal: Environ. Sci. Technol. Coden: ESTHAG Publ: 77
Series: 11 Issue: 7 Pages: 682-8
Identifiers: benzopyrene removal water polyurethane

87085517 CA0870805517A
Fluorescence characterization and identification of
polynuclear aromatic hydrocarbons in shale oil
Author: Thutubise, R. J.; Schubert, J. F.; Feaster, J. D.;
Therkildsen, D. H.; Poulsen, R. E.
Location: Dep. Chem. Univ. Wyoming, Laramie, Wyo.
Section: CA081014, CA0801XX Publ Class: JOURNAL
Journal: Anal. Chim. Acta Coden: ACACAM Publ: 77
Series: 89 Issue: 2 Pages: 377-82
Identifiers: fluorescence spectrometry arom hydrocarbon,
chromatog arom hydrocarbon shale oil, pyrene dain shale oil,
benzopyrene dain shale oil

87027950 CA08704027950T
Determination of polynuclear aromatic hydrocarbons by
thin-layer chromatography
Author: Nikolaeva, M. N.; Koroleva, K. I.; Korunov, A. A.
Location: USSR
Section: CA089002, CA0811XX, CA0801XX Publ Class: JOURNAL
Journal: Tr. Vses. Nauchno-Issled. Proektiro-Tekhnol. Inst.
Elektroorg/nykh Izdelii Coden: TNIZDC Publ: 75
Series: 3, Pages: 128-30 Language: Russ
Identifiers: phenanthrene emission binder, fluoranthene

emission binder, polycyclic hydrocarbon emission binder,
benzopyrene emission binder, methylanthrene emission
binder, cholanthrene methyl emission binder, tar binder,
polycyclic hydrocarbon, pitch binder polycyclic hydrocarbon,
anthracene oil binder emission, carbon graphite article
emission, chromatog polycyclic arom hydrocarbon, thin layer
chromatog hydrocarbon

87020523 CA08702020523X
Determination of polynuclear aromatics in yeast produced by
paraffin fermentation and n-hydrocarbon feedstocks
Author: McGinnis, E. L.; Norris, M. S.
Location: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CA016001 Publ Class: JOURNAL
Journal: Prepr., Div. Pet. Chem., Am. Chem. Soc. Coden:
ACPCAT Publ: 75 Series: 20 Issue: 4 Pages: 828-37
Identifiers: polynuclear arom single cell protein

86194119 CA08626194119Q
Tentative method of analysis for polynuclear aromatic
hydrocarbons in automobile exhaust
Author: Sawicki, E.
Location: USA
Section: CA089001, CA0801XX Publ Class: JOURNAL
Journal: Health Lab. Sci. Coden: HLSAE Publ: 74
Series: 11 Issue: 3 Pages: 228-39
Identifiers: arom hydrocarbon dain exhaust

86194118 CA08626194118P
Tentative method of analysis for polynuclear aromatic
hydrocarbons in coke oven effluents
Author: Sawicki, E.
Location: USA
Section: CA089001, CA0811XX, CA0801XX Publ Class: JOURNAL
Journal: Health Lab. Sci. Coden: HLSAE Publ: 74
Series: 11 Issue: 3 Pages: 218-27
Identifiers: arom hydrocarbon dain coke oven

86164856 CA08622164856X
Phthalimidepropylsilane - a new chemically bonded stationary phase for the determination of polynuclear aromatic hydrocarbons by high-pressure liquid chromatography
Author: Hunt, D. C.; Wild, P. J.; Crosby, N. I.
Location: Nat. Gov. Chem., London, Engl.
Journal: J. Chromatogr.
Series: 130. Pages: 320-3

Section: CA0860004, CA086011X, CA01711X Pub Class: JOURNAL
Journal: J. Chromatogr. Coden: JCHROM Publ: 77
Identifiers: high pressure liq chromatog, phthalimidepropylsilane stationary phase liq chromatog, propylsilane phthalimide phase liq chromatog, silane phthalimidepropyl phase liq chromatog, polynuclear arom hydrocarbon liq chromatog, mussel analysis polynuclear arom hydrocarbon, benzofluoranthene detection mussel, perylene detection mussel

86160217 CA08622160217E
Investigation on a long-term collection of polynuclear aromatic hydrocarbons in environmental air
Author: Matsushita, Hidetaru; Arashidani, Keiichi; Honda, Takashi
Location: Natl. Inst. Ind. Health, Kawasaki, Japan
Section: CA0860001, CA086011X Pub Class: JOURNAL
Journal: Bunseki Kagaku Coden: BNSKAK Publ: 76
Series: 25 Issue: 6 Pages: 415-17 Language: Japan
Identifiers: polycyclic arom hydrocarbon sampling air, glass filter arom hydrocarbon retention

86160216 CA08622160216D
Vacuum sublimation method for extraction of polynuclear aromatic hydrocarbons from airborne particulates
Author: Matsushita, Hidetaru; Arashidani, Keiichi; Hayashi, Miso
Location: Natl. Inst. Ind. Health, Kawasaki, Japan
Section: CA0860001, CA086011X Pub Class: JOURNAL
Journal: Bunseki Kagaku Coden: BNSKAK Publ: 76
Series: 25 Issue: 6 Pages: 412-15 Language: Japan
Identifiers: arom hydrocarbon vacuum sublimation app, polycyclic arom hydrocarbon extn particulate

86157966 CA08622157966V
A simple rapid analysis of polynuclear aromatic hydrocarbons in gasoline
Author: Matsushita, Hidetaru; Arashidani, Keiichi; Koyano, Michiko; Honda, Takashi
Location: Natl. Inst. Ind. Health, Kawasaki, Japan
Section: CA0860001, CA086011X Pub Class: JOURNAL
Journal: Taihei Oen Kenkyu Coden: TOREAV Publ: 76
Series: 11 Issue: 1 Pages: 44-53 Language: Japan
Identifiers: gasoline benzopyrene detn, arom detn petroleum

oil, arom polycyclic hydrocarbon detn

86126256 CA08618126256G
Analysis of polynuclear aromatic hydrocarbons in the respiratory environment
Author: Brunnebaum, Klaus D.; Hoffmann, Dietrich
Location: Maylor Dana Inst. Dis. Prev., Am. Health Found., Valhalla, N. Y.
Section: CA0860002, CA0111X Pub Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: 76 Series: 1. Pages: 283-97
Identifiers: tobacco smoke carcinogen, polycyclic arom hydrocarbon smoke, chrysene cigaret smoke, fluoranthene cigaret smoke

86100551 CA08614100551Z
An integrated approach to the analysis of air-pollutant polynuclear aromatic hydrocarbons
Author: Bartle, K. D.; Lee, M. L.; Novotny, M.
Location: Sch. Chem., Univ. Leeds, Leeds, Engl.
Section: CA0860006, CA00411X, CA086011X Pub Class: JOURNAL
Journal: Proc. Anal. Div. Chem. Soc. Coden: PADSDZ Publ: 76 Series: 13 Issue: 10 Pages: 304-7
Identifiers: carcinogen arom fingerprinting, air pollutant polynuclear arom

86098224 CA08614098224P
Quantitative distribution of polynuclear aromatic hydrocarbons in relation to particle size of urban particulates
Author: Katz, Morris; Pierce, Ronald C.
Location: Cent. Res. Environ. Qual., York Univ., Toronto, Ont.
Section: CA0860002 Pub Class: JOURNAL
Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: 76 Series: 1. Pages: 413-28
Identifiers: polycyclic arom hydrocarbon distribution, particle size polycyclic hydrocarbon

84059745 CA08610059745A
 Determination of polynuclear aromatic hydrocarbons (PAH)
 Author: Stepanova, M. I.
 Location: Ozerzhinsk, Filial, Gos. Nauchno-Issled. Inst.
 Prom. Sanit. Ozhistke Gazov, Dzerzhinsk, USSR
 Section: CA086001, CA086033 Publ Class: JOURNAL
 Journal: Prom. Sanit. Ozhistke Gazov Coden: PSQADK
 Publi: 76 Issue: 2 Pages: 30-1 Language: Russ
 Identifiers: polynuclear arom hydrocarbon waste gas

84059708 CA08610059708E
 Microanalytical methods for polynuclear aromatic
 hydrocarbons in environmental air
 Author: Matsushita, Hidetsuru
 Location: Natl. Inst. Ind. Health, Kawasaki, Japan
 Section: CA086000, CA086033 Publ Class: JOURNAL
 Journal: Taiki Gosen Kenkyu Coden: TOKKAV Publ: 76
 Series: 10 Issue: 6 Pages: 723-31 Language: Japan
 Identifiers: review polynuclear arom hydrocarbon air

84026585 CA08605026585V
 Determination of carcinogens in tobacco smoke and
 coal-derived samples - trace polynuclear aromatic hydrocarbons
 Author: Kubota, H.; Griest, M. H.; Guerin, M. R.
 Location: Anal. Chem. Div., Oak Ridge Natl. Lab., Oak Ridge,
 Tenn.
 Section: CA086001 Publ Class: JOURNAL
 Journal: Trace Subst. Environ. Health Coden: PUMTAG
 Publi: 76 Series: 9 Pages: 281-9
 Identifiers: tobacco smoke arom hydrocarbon detn, coal
 product arom hydrocarbon detn

8519472 CA085261949737
 Analytical methods for polynuclear aromatic hydrocarbons in crude oils, heating oils, and marine tissues
 Author: Pencirov, R. J.; Brown, R. A.
 Location: Anal. Inf. Div., Exxon Res. and Eng. Co., Linden, N. J.
 Section: CA081001, CA080000 Publ. Class: C
 Journal: Proc. Conf. Prev. Control Oil Pollut. Coden: 33PRA1 Publ: 75 Pages: 103-13
 Publisher: Am. Pet. Inst. Address: Washington, D. C.
 Identifiers: polynuclear arom hydrocarbon petroleum

8519419 CA08525189419W
 Isolation, identification, and quantitation of the polynuclear aromatic hydrocarbons in tobacco smoke
 Author: Severson, Roy F.; Snook, Maurice E.; Higman, Howard C.; Chortyk, Gresten T.; Akin, Frank J.
 Location: Tob. Health Res. Lab., Agric. Res. Cent., Athens, Ga.
 Section: CA011007 Publ. Class: J
 Journal: Carcinog. - Compr. Surv. Coden: GCSUDL Publ: 76 Series: 1, Pages: 283-70
 Identifiers: tobacco smoke aromatic hydrocarbon

85191642 CA08524181642J
 Determination of polynuclear aromatic hydrocarbons by anodic differential pulse voltammetry at the glassy carbon electrode in sulfolane and acetonitrile as solvents
 Author: Cozlee, J. F.; Kazi, G. H.; Spurgeon, J. C.
 Location: Dep. Chem., Univ. Pittsburgh, Pittsburgh, Pa.
 Section: CA089001, CA026000, CA080000 Publ. Class: J
 Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 14 Pages: 2170-3
 Identifiers: arom polynuclear hydrocarbon voltammetry, carbon glassy electrode voltammetry

85191640 CA08524181640S
 Analysis of polynuclear aromatic hydrocarbons in automobile exhaust by supercritical fluid chromatography
 Author: Jentoft, R. E.; Gouw, T. H.
 Location: Chevron Res. Co., Richmond, Calif.
 Section: CA089001, CA026000, CA080000 Publ. Class: J
 Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 14 Pages: 2195-9
 Identifiers: exhaust arom polynuclear hydrocarbon detn, chromatog supercrit polynuclear hydrocarbon

85172333 CA08523172333Q
 Gas chromatographic quantitation of polynuclear aromatic hydrocarbons in tobacco smoke
 Author: Severson, R. F.; Snook, M. E.; Arrandale, R. F.; Chortyk, O. T.
 Location: Tob. Lab., ARS, Athens, Ga.
 Section: CA004001, CA011000, CA080000 Publ. Class: J
 Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 13 Pages: 1868-72
 Identifiers: tobacco smoke hydrocarbon gas chromatog

85171202 CA08522171202J
 Detection and determination of polynuclear aromatic hydrocarbons by luminescence spectrometry utilizing the Sipol'ski effect at 77 degrees K. Part III. Luminescence excitation spectra
 Author: Farooq, R.; Kirksight, G. F.
 Location: Dep. Chem., Imp. Coll. Sci. Technol., London, Engl.
 Section: CA080005, CA072200 Publ. Class: J
 Journal: Analyst (London) Coden: ANALAD Publ: 76 Series: 101 Issue: 1204 Pages: 868-73
 Identifiers: polynuclear arom hydrocarbon detection, luminescence excitation polynuclear hydrocarbon, spectrometer luminescence excitation Sipol'ski

85165904 CA08522165904R
 Analysis of polynuclear aromatic hydrocarbons in complex mixtures
 Author: Lee, Milton Lafayette
 Location: Indiana Univ., Bloomington, Indiana
 Section: CA089005, CA026000 Publ. Class: D
 Coden: DABBA Publ: 76 Pages: 281 pp.
 Citation: Dis. Abstr. Int. 8 1976, 36(11), 5548
 Avail: Xerox Univ. Microfilms, Ann Arbor, Mich., Order No. 76-11,369
 Identifiers: arom hydrocarbon detn air, carcinogen detn air

8511937 CA0851711937Y
 A chromatographic analysis for polynuclear aromatic hydrocarbons in small quantities of cigaret smoke condensate
 Author: Severson, R. F.; Snook, M. E.; Chortyk, O. T.; Arrandale, R. F.
 Location: Tob. Lab., ARS, Athens, Ga.
 Section: CA011007, CA080000 Publ. Class: J
 Journal: Beitr. Tabakforsch. Coden: RETAAY Publ: 76 Series: 8 Issue: 5 Pages: 273-22
 Identifiers: tobacco smoke hydrocarbon analysis

8508579 CA08514098579Y
Gas chromatography/mass spectrometry and nuclear magnetic resonance determination of polynuclear aromatic hydrocarbons in airborne particulates

Author: Lee, M. L.; Novotny, M.; Bartle, K. D.
Location: Dep. Chem., Indiana Univ., Bloomington, Indiana
Section: CA089001, CA080000 Publ Class: J
Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 11 Pages: 1866-72

Identifiers: hydrocarbon detn airborne particulate, arom hydrocarbon detn airborne particulate, gas chromatog arom hydrocarbon particulate, mass spectrometry arom hydrocarbon particulate, MS8 arom hydrocarbon particulate

8509039 CA0851309039X
High-pressure liquid chromatography of polynuclear aromatic hydrocarbon constituents of smoke

Author: Heiberger, A. F.; Chortyk, O. T.
Location: Tobacco Lab., ARS, Athens, Ga.
Section: CA011007 Publ Class: J
Journal: Recent Adv. Tob. Sci. Coden: RAISDZ Publ: 75 Series: 1 Issue: New Tech. Smoke Chem. Phys. Pages: 72-96

Identifiers: review chromatog tobacco smoke, arom hydrocarbon tobacco smoke review

8508650 CA0851208650S

Detection and determination of polynuclear aromatic hydrocarbons by luminescence spectrometry utilizing the Spol'skii effect at 77 degree K. Part II. An evaluation of excitation sources, sample cells and detection systems
Author: Causey, B. S.; Kirkbright, G. F.; De Lima, C. G.
Location: Dep. Chem., Imp. Coll. Sci. Technol., London, Engl.
Section: CA080006 Publ Class: J
Journal: Analyst (London) Coden: ANALAO Publ: 78 Series: 101 Issue: 1202 Pages: 367-78

Identifiers: polynuclear hydrocarbon detn luminescence, arom hydrocarbon detn luminescence, luminescence spectrometry hydrocarbon instrumentation, excitation source luminescence hydrocarbon, cryostat cell luminescence hydrocarbon

85087425 CA08510087425R

Selective monitoring of polynuclear aromatic hydrocarbons by high pressure liquid chromatography with a variable wavelength detector

Author: Krstulovic, Ante M.; Rosle, Douglas M.; Brown, Phyllis B.
Location: Dep. Chem., Univ. Rhode Island, Kingston, R. I.
Section: CA089001, CA047000, CA080000 Publ Class: J

Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 9 Pages: 1383-6
Identifiers: chromatog monitoring polynuclear arom hydrocarbon, UV detector chromatog arom hydrocarbon, environment polynuclear arom analysis

85051548 CA08508051548X

Factors affecting the extraction and analysis of polynuclear aromatic hydrocarbons in water
Author: Acheson, M. A.; Harrison, R. M.; Perry, R.; Wellings, R. A.
Location: Imp. Coll., London, Engl.
Section: CA081002 Publ Class: J
Journal: Water Res. Coden: WATRAG Publ: 76 Series: 10 Issue: 3 Pages: 207-12

Identifiers: arom hydrocarbon detn water

85041648 CA08507041648P

Gas chromatographic analysis of polynuclear aromatic hydrocarbons in shellfish on short, wall-coated glass capillary columns

Author: Oruska, Francis J.; Volkoff, Aaron W.; Combs, Michael E.; Larosa, Richard H.; Novotny, Milton L.
Location: Canada Cent. Indust. Waters, Burlington, Ont.
Section: CA004001, CA780000 Publ Class: J
Journal: Anal. Lett. Coden: ANALBP Publ: 76 Series: 9 Issue: 5 Pages: 451-60

Identifiers: arom hydrocarbon gas chromatog, shellfish arom hydrocarbon detn

84189093 CA08426189093F

X-ray excited optical luminescence of polynuclear aromatic hydrocarbons

Author: D'Silva, A. P.; Destreich, G. J.; Fassel, V. A.
Location: Ames Lab., Iowa State Univ., Ames, Iowa
Section: CA080006, CA004000 Publ Class: J
Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 6 Pages: 915-17

Identifiers: x ray excited optical luminescence, luminescence detection polynuclear arom hydrocarbon, perylene luminescence detection, benzopyrene luminescence detection, coronene luminescence detection

- DIALOG File3: CA Search - 1972 thru 1978 (Copr. Am. Chem. Soc.) (Item 16 of 45) User 3631 2apr81 31
- 84184306 CA084261843061
The determination of polynuclear aromatic hydrocarbons by gas-liquid chromatography
Author: Harrison, E. K.; Powell, C. I. G.
Location: Natl. Smokeless Fuels Ltd., Harrow/Middlesex, Engl.
Section: CA088002, CA080000 Publ Class: J
Journal: Ann. Occup. Hyg. Coden: ANHYA3 Publ: 75
Series: 18 Issue: 3 Pages: 199-206
Identifiers: dust polynuclear arom hydrocarbon, benzopyrene dust chromatog, benzopyrene dust chromatog, anthracene dust chromatog, benzo[a]pyrene dust chromatog
- 84189452 CA08424189452U
Polynuclear aromatic hydrocarbons in the environment. I. Determination of polynuclear aromatic hydrocarbons in water by mass fragmentography
Author: Matsushima, Hajime; Nanya, Takahisa
Location: Dep. Chem., Tokyo Metropol. Univ., Tokyo, Japan
Section: CA081002 Publ Class: J
Journal: Bunsenki Kagaku Coden: BNSKAK Publ: 75
Series: 24 Issue: 8 Pages: 808-11 Language: Japan
Identifiers: carcinogen hydrocarbon detn river water, fluoranthene detn river water, pyrene detn river water, benzopyrene detn river water
- 84100819 CA08419100819K
Gas chromatography/mass spectrometry and nuclear magnetic resonance spectrometric studies of carcinogenic polynuclear aromatic hydrocarbons in tobacco and marijuana smoke condensates
Author: Lee, M. L.; Novotny, Milos; Bartle, K. D.
Location: Dep. Chem., Indiana Univ., Bloomington, Indiana
Section: CA004007 Publ Class: J
Journal: Anal. Chem. Coden: ANCHAM Publ: 76 Series: 48 Issue: 2 Pages: 405-16
Identifiers: tobacco smoke arom hydrocarbon, marijuana smoke arom hydrocarbon
- 84085022 CA08412085022R
High-pressure liquid chromatography of polynuclear aromatic hydrocarbons of cigarette smoke condensate
Author: Huebner, A. F.; Snook, M. E.; Chortyk, D. T.
Location: Tob. Lab., ARS, Athens, Ga.
Section: CA004001 Publ Class: J
Journal: Anal. Chem. Acta Coden: ACACAM Publ: 75
Series: 80 Issue: 2 Pages: 303-9
Identifiers: arom hydrocarbon liq chromatog, cigaret smoke condensate liq chromatog
- 84071660 CA08411071660K
Resolution of polynuclear aromatic hydrocarbons by packed column GLC
Author: Grisset, M. H.; Kubota, H.; Guerin, M. R.
Location: Anal. Chem. Div., Oak Ridge Natl. Lab., Oak Ridge, Tenn.
Section: CA011007, CA098000 Publ Class: J
Journal: Anal. Lett. Coden: ANALAP Publ: 75 Series: 8 Issue: 12 Pages: 949-57
Identifiers: benzo[a]anthracene tobacco smoke, phenanthrene detn tobacco smoke, chromatog hydrocarbon tobacco smoke
- 84049362 CA08408049362T
Thin-layer chromatographic determination of polynuclear aromatic hydrocarbons (in waste gases)
Author: Nikolaeva, N. N.; Koroleva, K. I.; Korunov, A. A.
Location: USSR
Section: CA088001, CA080000 Publ Class: J
Journal: Tr. Vses. N.-I. I. Prikl. Tekhnol. In-Ts Elektrogon. Izdell Coden: 04J005 Publ: 75 Issue: 3 Pages: 125-30 Language: Russ
Citation: Ref. Zh., Khim. 1975, Abstr. No. 18P90
Identifiers: hydrocarbon detn waste gas, chromatog hydrocarbon waste gas
- 83188012 CA08322188012W
Supercritical fluid chromatography of polynuclear aromatic hydrocarbon
Author: Fuzita, Kazunori; Shiohara, Ikuro; Nakazawa, Fumito
Location: Hitachi Res. Lab., Hitachi, Ltd., Hitachi, Japan
Section: CA080004, CA081000 Publ Class: J
Journal: Nippon Kagaku Kaishi Coden: NKAK86 Publ: 75 Issue: 8 Pages: 1348-51 Language: Japan
Identifiers: supercrit fluid chromatog arom hydrocarbon, chromatog polynuclear arom hydrocarbon, benzene chromatog, naphthalene chromatog, phenanthrene chromatog, pyrene chromatog, chrysene chromatog, triphenylene chromatog, coal tar supercrit fluid chromatog

83173304 CA08221173304H
Use of the spot-tilt effect for the determination of trace amounts of polynuclear aromatic hydrocarbons
Author: Kiriabright, G. F.; De Lima, C. G.
Location: Chem. Dep., Imp. Coll., London, Engl.
Section: CA004001 Publ. Class: J
Journal: Proc. Soc. Anal. Chem. Coden: PAYCAL Publ: 74
Series: 11 Issue: 3 Pages: 85-80
Identifiers: arom hydrocarbon detn Spot-tilt effect

83084320 CA08207084320H
Determination of the fate of polynuclear aromatic hydrocarbons in natural water systems
Author: McGinnis, Paul B.; Snodgrass, Vernon L.
Location: Dep. Civ. Eng., Univ. Illinois, Urbana, Ill.
Section: CA004007, CA081000 Publ. Class: J
Journal: Res. Rep. - Univ. Ill. Urbana-Champaign, Water Resour. Cent. Coden: IUMRAH Publ: 74 Series: 80, Pages: 84 pp.
Identifiers: arom hydrocarbon decomp natural water, benzanthracene decomp natural water, benzpyrene decomp natural water

82152815 CA08223152815H
Trace polynuclear aromatic hydrocarbon analysis
Author: Naenn, E. D.; Flechtach, N.
Location: Bur. Foods, Food Drug Adm., Washington, D. C.
Section: CA017000, CA004000 Publ. Class: C
Journal: Contrib. Chem. Food Supplies, Invited Sci. Contrib. Pap. Symp. Coden: 28XVAZ Publ: 74 Pages: 209-25
Meeting Date: 73
Publisher: Butterworth Address: London, Engl
Avail: Morton, I.; Rhodes, D. M
Identifiers: review hydrocarbon detn food, carcinogen detn food review

82150310 CA08223150310H
Preliminary results on the use of Tenax for the extraction of pesticides and polynuclear aromatic hydrocarbons from surfaces and drinking waters for analytical purposes
Author: Lenti, V.; Puccelli, G.; Orselli, A.
Location: Inst. Ig. "G. Senerelli", Univ. Rome, Rome, Italy
Section: CA005001 Publ. Class: J
Journal: J. Chromatogr. Coden: JOCRAH Publ: 75
Series: 108 Issue: 1 Pages: 119-24
Identifiers: TenaxGC pesticide hydrocarbon detn

82127657 CA08221127657H

Determination of four- and five-ring condensed hydrocarbon. II. Analysis of polynuclear aromatic compounds 'n n-paraffin feed oil for yeast fermentation
Author: McGinnis, Edgar L.
Location: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CA016001, CA026000, CA023000 Publ. Class: J
Journal: J. Agric. Food Chem. Coden: JAFCAU Publ: 75
Series: 23 Issue: 2 Pages: 228-9
Identifiers: arom analysis paraffin yeast protein

82127656 CA08221127656H
Determination of four- and five-ring condensed hydrocarbons. I. Analysis of polynuclear aromatic hydrocarbons in yeast produced by growth on both n-hydrocarbon and dextrose feeds
Author: McGinnis, Edgar L.; Norris, Matthew S.
Location: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CA016001, CA026000 Publ. Class: J
Journal: J. Agric. Food Chem. Coden: JAFCAU Publ: 75
Series: 23 Issue: 2 Pages: 221-5
Identifiers: arom hydrocarbon anal yeast

82127264 CA08220127264H
Profiles of the polynuclear aromatic fraction from engine oils obtained by capillary-column gas-liquid chromatography and nitrogen-selective detection
Author: Lee, M. L.; Bartle, K. D.; Novotny, M. V.
Location: Dep. Chem., Indiana Univ., Bloomington, Indiana
Section: CA081007, CA080000 Publ. Class: J
Journal: Anal. Chem. Coden: ANCHAM Publ: 78 Series: 47 Issue: 3 Pages: 840-3
Identifiers: lubricating oil identification gas chromatog. polynuclear arom detn lubricant

82089679 CA08214089679S
High resolution GLC (gas-liquid chromatography) profiles of urban air pollutant polynuclear aromatic hydrocarbons
Author: Bartle, K. D.; Lee, M. L.; Novotny, M.
Location: Dep. Chem., Indiana Univ., Bloomington, Indiana
Section: CA080002, CA080000 Publ. Class: J
Journal: Int. J. Environ. Anal. Chem. Coden: IJEAAS Publ: 74 Series: 3 Issue: 4 Pages: 349-56
Identifiers: arom hydrocarbon detn air

81175296 CA08126178296Z
 Determination of polynuclear aromatic hydrocarbons in
 airborne particulate matter
 Author: British Coke Research Assoc.
 Location: Chesterfield/Derbyshire, Eng.
 Section: CA080001, CA080000 Pubi Class: J
 Journal: Coke Res. Rep. Coden: CKRAJ
 Series: 76, Pages: 12 pp.
 Identifiers: benzopyrene
 layer chromatog benzopyrene

81186002 CA0812418000Z
 Methods for fractionation, analytical separation, and
 identification of polynuclear aromatic hydrocarbons in complex
 mixtures
 Author: Novotny, M.; Lee, M. L.; Bartle, K. D.
 Location: Dep. Chem., Indiana Univ., Bloomington, Indiana
 Section: CA080001, CA080000 Pubi Class: J
 Journal: J. Chromatogr. Sci. Coden: JCHSBJ
 Series: 12, Issue: 10, Pages: 808-12
 Identifiers: hydrocarbon detn airborne particulate,
 chromatog hydrocarbon airborne particulate

81104109 CA08117104109Z
 Detection and determination of polynuclear aromatic
 hydrocarbons by luminescence spectrometry utilizing the
 Spontall effect at 77 deg.K
 Author: Mirabright, G. F.; De Lima, C. G.
 Location: Chem. Dep., Imp. Coll., London, Eng.
 Section: CA022002, CA080000 Pubi Class: J
 Journal: Analyst (London) Coden: ANALAO
 Series: 99, Issue: 1179, Pages: 338-54
 Identifiers: Spontall effect detn arom. THF matrix
 Spontall effect

81082240 CA0811608240Z
 Determination of the fate of polynuclear aromatic
 hydrocarbons in natural water systems
 Author: McInnes, Paul R.; Snodgrass, Vernon L.
 Location: Water Resour. Cent., Univ. Illinois, Urbana, Ill.
 Section: CA041001, CA080000 Pubi Class: J
 Journal: U. S. W. T. S., PG Rep. Coden: XPRSCA
 Series: 74, Issue: 22366/60A, Pages: 62 pp.
 Identifiers: Spontall effect detn arom. THF matrix
 Spontall effect

78126473 CA07824126473Z

Analysis of polynuclear aromatic hydrocarbons, some
 heterocyclics, and aliphatics with a single gas chromatograph
 column
 Author: Lane, D. A.; Mos, M. K.; Katz, Morris
 Location: Cent. Res. Environ. Qual., York Univ., Downsview,
 Ont.
 Section: CA080002, CA080000 Pubi Class: J
 Journal: Anal. Chem. Coden: ANCHAM
 Series: 48, Issue: 9, Pages: 1778-8
 Identifiers: hydrocarbon detn chromatog column

78111446 CA07818111446Z
 Selective gas chromatographic detector for polynuclear
 aromatics based on ultraviolet fluorescence
 Author: Robinson, J. M.; Goodbread, Jon P.
 Location: Dep. Chem., Louisiana State Univ., Baton Rouge,
 La.
 Section: CA080004, CA080000 Pubi Class: J
 Journal: Anal. Chem. Coden: ANCHAM
 Series: 56, Issue: 2, Pages: 239-44
 Identifiers: gas chromatog polynuclear arom. UV fluorescence
 detector gas chromatog

78090392 CA07816090392Z
 Rapid methods of analysis for trace quantities of
 polynuclear aromatic hydrocarbons and phenols in automobile
 exhaust, gasoline, and crankcase oil
 Author: Brown, R. A.; Seal, T. D.; King, W. H., Jr.; Dietz,
 W. A.; Kellner, J. R.
 Location: Esso Res. Eng. Co., Linden, N. J.
 Section: CA080002, CA078000, CA080000, CA081000 Pubi
 Class: T
 Journal: U. S. Nat. Tech. Inform. Serv., PG Rep. Coden:
 XPRSCA
 Series: 71, Issue: No. 219025/4, Pages: 86 pp.
 Identifiers: Spontall effect detn arom. THF matrix
 Spontall effect

78184608 CA07824184608Z
 Analysis of polynuclear aromatic hydrocarbons by liquid
 phase chromatography under pressure
 Author: Joubert, Michel; Martin, Georges; Guiochon
 Location: Lab. Chim. Anal. Phys., Ec. Polytech., Paris, Fr.
 Section: CA080004, CA080000 Pubi Class: J
 Journal: Anal. Chem. Coden: ANCHAM
 Series: 50, Issue: 168-78, Language: Fr
 Identifiers: arom polycyclic hydrocarbon chromatog

76120701 CA0761120701H
Determination of photochemical and photobiological properties of polynuclear aromatic compounds and their relation to carcinogenic hazard

Author: Verheest, David
Location: Univ. Cincinnati, Cincinnati, Ohio
Section: CA060013 Publ Class: D
Codon: DARS40 Publ: 72 Pages: 178 pp.
Citation: Diss. Abstr. Int. 8 1973, 33(6), 3689
Avail: Univ. Microfilms, Ann Arbor, Mich., Order No. 73-3816
Identifiers: polynuclear aromatic compounds photochemical properties, carcinogen polynuclear aromatic compounds

76061763 CA07610061763T
Detection of some polynuclear aromatic hydrocarbons and determination of benz(a)pyrene in Tehran atmosphere
Author: Akbari, V.; Aghaie, M.; Dervich, M. R.; Khorgasht, M.
Location: Sci. Fac., Tehran Univ., Tehran, Iran
Section: CA090002, CA038000 Publ Class: J
Journal: Atmos. Environ. Codon: ATENBP
Series: 6 Issue: 12 Pages: 949-52
Identifiers: benzopyrene, dinitro, perylene, dinitro, benzanthracene, dinitro, pyrene, dinitro, phenanthrene, dinitro, coronene, dinitro, air analysis polynuclear aromatic

77117675 CA07718117675E
Determination of the correction factor in the estimation of polynuclear aromatic hydrocarbons separated by thin-layer chromatography
Author: Chatot, G.; Dangy-Caye, R.; Fontanges, R.; Obaton, P.
Location: Div. Microbiol., Cent. Rech. Serv. Sante Armees, Lyons, Fr.
Section: CA090001, CA080000 Publ Class: J
Journal: Chromatographia Codon: CHROM7
Series: 5 Issue: 5 Pages: 480-3 Language: Fr
Identifiers: thin layer chromatography hydrocarbon, aromatic hydrocarbon, dinitro

77108369 CA07718108369V
Determination of polynuclear aromatic hydrocarbons in chemical and petrochemical industry pollution
Author: Stepanova, M. I.; Il'ina, R. I.; Shaposhnikov, Yu. K.
Location: Sci.-Res. Gas Inst., Dzerzhinsk, USSR
Section: CA090002, CA081000, CA080000 Publ Class: J
Journal: Zh. Anal. Khim. Codon: ZAKHIS
Series: 27 Issue: 6 Pages: 1201-4 Language: Russ
Identifiers: aromatic hydrocarbon, dinitro, petrochemical waste,

condensed aromatic chromatography

76089296 CA0761709296W
Separation of polynuclear aromatic hydrocarbons by gas-solid chromatography on graphitized carbon black deposited on Chromosorb W
Author: Frycka, J.
Location: Res. Inst. Coal Tar Chem., Valasske Mezirici, Czech.
Section: CA026000, CA080000 Publ Class: J
Journal: J. Chromatogr. Codon: JOCRAM
Series: 65 Issue: 2 Pages: 432-4
Identifiers: carbon, gas chromatography, aromatic, gas chromatography polycyclic aromatic

76074813 CA07614074813K
Group analysis of industrial mixtures of polynuclear aromatic hydrocarbons by rapid liquid-phase chromatography
Author: Martin, M.; Lohsac, J.; Guichon, G.
Location: Lab. Chim. Anal. Phys., Ec. Polytech., Paris, Fr.
Section: CA081000, CA080000, CA025000 Publ Class: J
Journal: Chromatographia Codon: CHROM7
Series: 5 Issue: 1 Pages: 33-41 Language: Fr
Identifiers: aromatic steam cracking, chromatography, aromatic, analysis aromatic cracking

76067866 CA07612067866Q
Trace fluorometric determination of polynuclear aromatic hydrocarbons in natural water
Author: Keegan, Richard E.
Location: Univ. New Hampshire, Durham, N. H.
Section: CA080000 Publ Class: D
Codon: DARS40 Publ: 71 Pages: 156 pp.
Citation: Diss. Abstr. Int. 8 1971, 32(4), 2048
Avail: Univ. Microfilms, Ann Arbor, Mich., Order No. 71-28,412
Identifiers: fluorometric, aromatic polynuclear aromatic

APPENDIX B
PROJECT QUALITY CONTROL PLAN

PROJECT QUALITY CONTROL PLAN

APPENDIX B

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1.0 INTRODUCTION

This document describes the quality control procedures to be used for the methods development and analysis efforts required in this project. The plan described in the following six sections complies with the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) Quality Assurance (QA) Program and with Environmental Science and Engineering, Inc. (ESE) policy. ESE supports an active, comprehensive QA program within its Technical Operations Division, which is described in the Division's Operations Manual. A major focus of the ESE QA program is the development of a project Quality Assurance/Quality Control (QA/QC) plan describing the specific application of ESE procedures to control and monitor any project.

The specific objectives of this plan are to describe in detail the process for controlling the validity of the data generated for documentation of the analytical methods developed. The Project QA Plan provides a mechanism for documentation of the limits of precision, accuracy, and sensitivity of all analytical systems generating data.

The prospective analytical approach for each analyte will be described in the proposed method reports which will be submitted for approval as required by USATHAMA. The analytical systems controls and data validation procedures described in this Quality Control Plan will be employed to ensure valid, properly formatted data defining the precision, accuracy, and sensitivity of each method. Reports will also be submitted to USATHAMA documenting the methods in both natural and standard media.

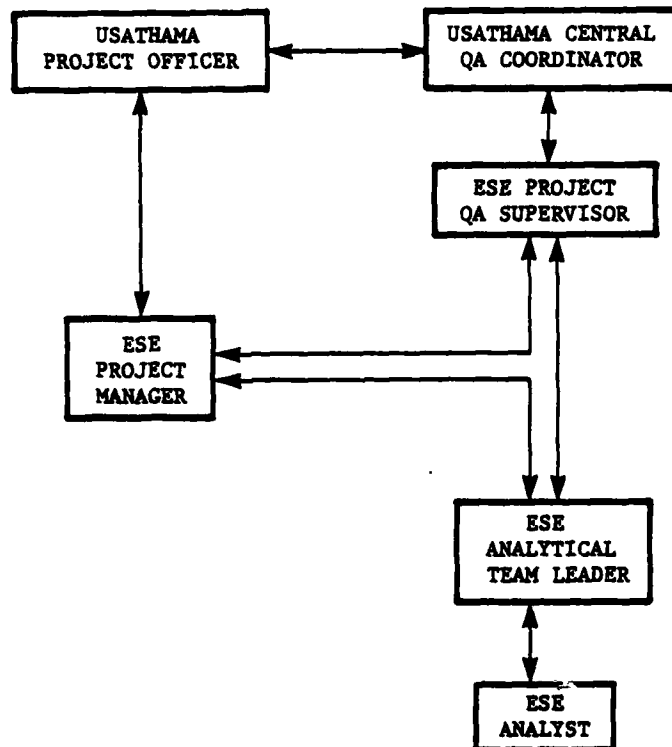
2.0 ORGANIZATION AND RESPONSIBILITIES FOR QUALITY ASSURANCE

The Quality Control Plan functions according to the USATHAMA central-laboratory/field-laboratory concept. ESE acts as the field laboratory which is monitored by the USATHAMA Central Laboratory QA Coordinator. The overall QA/QC organization to provide valid analytical methods to the commander of USATHAMA is shown in Figure 2-1. The function of the plan and QA responsibilities of each of the project participants are outlined in the following subsections.

2.1 OVERALL PLAN FUNCTION

Figure 2-1 depicts the manner in which the ESE Project Quality Assurance/Quality Control (QA/QC) Supervisor monitors the conduct of the project. In this position, the QA/QC Supervisor is not directly subordinate to anyone responsible for analytical methods development; the supervisor reports to the ESE Project Manager and the USATHAMA Central Laboratory QA Coordinator. The specific responsibilities of the QA/QC Supervisor are detailed in Paragraph 2.2.2.

The analyst, under the supervision of the Analytical Team Leader, performs the analyses associated with methods development and submits the results to the Analytical Team Leader for approval. The Analytical Team Leader writes the methods development reports in approved USATHAMA format. The Analytical Team Leader also writes the Proposed Method Development Plan in the USATHAMA format prior to the beginning of work on a method. The Method Development Plan will be submitted to USATHAMA for approval before work begins.



SOURCE: Environmental Science and Engineering, Inc., 1988.

Figure 2 - 1
LABORATORY QA/QC ORGANIZATION
AND FUNCTION

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

The personnel having a direct role in project QA/QC are the ESE Project QA/QC Supervisor, the USATHAMA Central Laboratory QA Coordinator, the ESE Project Manager, the ESE Analytical Team Leader, and the analysts.

2.2 QA/QC RESPONSIBILITIES

2.2.1 USATHAMA CENTRAL LABORATORY QA COORDINATOR

The Central Laboratory of USATHAMA is responsible to the Commander of the Agency for the quality of data collected in support of its programs. The USATHAMA Central Laboratory QA Coordinator, therefore, has the following responsibilities in fulfilling this objective:

1. To provide technical evaluation of field laboratory QA/QC procedures;
2. To provide Standard Analytical Reference Materials (SARMS) with supporting documentation to field laboratories;
3. To provide QA training as required;
4. To provide technical evaluation of field laboratory methods development documentation;
5. To notify the USATHAMA Project Officer, ESE Project Director, ESE Project Manager, and ESE QA/QC Supervisor when a situation exists which precludes statistical control of results; and
6. To provide continuous review of the implementation of the field laboratory QA/QC plan and report the findings to the USATHAMA Project Officer.

2.2.2 ESE PROJECT QA/QC SUPERVISOR

The ESE Project QA/QC Supervisor is responsible to the ESE Project Manager and the USATHAMA Central Laboratory QA Coordinator to monitor the quality of all data reported to USATHAMA in the method report and documentation. The supervisor's specific responsibilities are:

1. To provide an independent overview of the QC practices of the project team from beginning of the project through acceptance of the final report;

2. To ensure that the team completes all QC requirements of the project plan;
3. To approve each method report in the USATHAMA format, and to ensure that the documentation data are correct;
4. To audit data files for correct entry of all data;
5. To assure the availability of SARMS or, where unavailable, to approve the use of interim reference materials;
6. To arrange for and report on purity analyses and stability checks on interim reference materials;
7. To assure the delivery of interim reference materials to the Central QC Laboratory;
8. To establish and maintain liaison between the ESE Project Team and the USATHAMA Central QA Coordinator; and
9. To maintain a vigil of the entire laboratory in order to detect conditions which might jeopardize control of the various analytical systems.

2.4.2 ESE PROJECT MANAGER

The ESE Project Manager is responsible for effective day-to-day management of the total project staff, as well as direct communication and liaison with the USATHAMA Project Officer. The Project Manager's responsibility specific to QA/QC is to approve all QA/QC procedures to be used in the conduct of the project, to provide additional authority when required to support the ESE Project QA/QC Supervisor, and to approve of any revisions to the project QC plan.

The Project Manager is responsible for effective day-to-day coordination of all USATHAMA activity, guidance and technical support in resolution of QC problems, and support of QA/QC preparation of unknown reference samples. This manager also provides additional authority, when needed, to support the QA/QC Supervisor in analytical matters.

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2.2.4 ESE ANALYTICAL LEADER

The ESE Analytical Leader is responsible for provision of accurate laboratory data produced by analysts under his supervision. He is responsible to the ESE Project QA/QC Supervisor to ensure that all quality control procedures are followed and documentation provided. The QA role of the Analytical Leader is, therefore, to assist the QA supervisor in enforcing QA/QC procedures.

2.2.5 ESE ANALYSTS AND SAMPLING PERSONNEL

The following sections describe the QA/QC procedures required to define and document the validity of data forthcoming from the conduct of this project. It is the responsibility of the analysts to perform the required QA/QC procedures and to document all observations in logbooks in permanent ink. It is also the responsibility of the analyst to perform preliminary QC checks to ensure that each batch of data being generated meets all analytical systems criteria. The analyst must also bring any unusual observation or analytical problem to the immediate attention of his/her Analytical Leader or the ESE Project QA/QC Supervisor.

Each analyst is responsible for ensuring that sufficient quantities of reagents of adequate quality are available for the performance of the required analyses.

AMD.1/QC3.1

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3.0 DOCUMENT CONTROL AND REVISIONS

3.1 RATIONALE

The Project Quality Assurance Plan includes a system for documenting and updating all sampling, analytical, and data handling procedures used by ESE for this project. This system uses a standardized indexing format and provides for easy replacement of pages, if techniques and procedures are changed.

3.2 STANDARD OPERATING PROCEDURES

The standardized indexing format includes, at the top of each page, the following information:

1. Section Number,
2. Revision Number, and
3. Date of Revision.

Section numbers within a document are in numerical order. Revision number represents the most current version of a section, with the original version listed as "0". The date represents the date of the latest version. The text of each major section begins on a new page. If revisions to a section involve expansion which adds pages, the additional pages will be numbered 1a, 1b, 1c, etc. For example, if Page 4 were revised and expanded to include an extra paragraph, the overflow would appear on a page designated 4a. The original Page 4 would then be removed from the manual and replaced by revised Page 4 and Page 4a. This system allows expansion within a section without renumbering the entire document.

The Table of Contents follows the same structure as the text and contains a space for "Revision." When a revision to the text is made,

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the Table of Contents page is updated by retyping, or by striking out the old revision number and printing in the current revision number.

Proposed revisions will be reviewed by the Project QA/QC Supervisor, Project Manager, and Project Director and then sent to the USATHAMA Central QA Laboratory Coordinator and USATHAMA Project Officer for final approval.

4.0 ANALYST TRAINING AND CERTIFICATION

4.1 RATIONALE

Accurate and precise analyses can be conducted only by well-trained analysts who correctly operate instruments, thoroughly understand analytical methods, use good analytical technique, and understand and practice necessary QC procedures. While the necessary training may initially be obtained from education, experience, or on-the-job training, it is imperative that the analyst's capabilities be verified prior to conducting analyses and reviewed periodically thereafter.

4.2 STANDARD OPERATING PROCEDURES

The QA/QC Supervisor will provide "test" samples and assist ESE Technical Operations Management when possible in the training of sampling team members and analysts; however, such training is the ultimate responsibility of ESE Technical Operations Management.

Direct responsibility for analytical training rests with the ESE administrative management level of Group Leader.

Each analyst will be reviewed by his/her Group Leader to ensure:

1. Working knowledge of QC policies,
2. Preparation of standards and spikes,
3. Acceptable analysis of reference samples,
4. Acceptable analysis of replicates and spikes,
5. Detection limit and standard calibration requirements, and
6. Knowledge of preventive maintenance techniques.

This evaluation must be done for every analysis the analyst performs on this project.

AMD.1/QC4.2

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A list of qualified personnel for each sampling and analytical task will be provided by the appropriate Group Leaders to the ESE QA/QC Supervisor.

The QA/QC Supervisor will keep a logbook arranged by type of analysis (e.g., Autoanalyzer, atomic absorption, gas chromatography, GC/MS, etc.). Analysts' names will be entered under the qualified headings with the Group Leader's initials and date certified (Figure 4-1). At annual intervals, each Group Leader will review the capabilities of each analyst and recommend whether certification should be continued.

4.3 PERFORMANCE AUDIT

During the conduct of this project, the QA/QC Supervisor will inspect the laboratory occasionally to determine if analyses are being performed only by certified analysts. Data reports require the name of the analyst on the report sheet. Reruns of samples may be required if certified analysts did not perform the analysis.

4.4 ANALYST CERTIFICATION PROCEDURES

The confidence in any analytical method is limited if the analyst has not demonstrated skill in performing the analysis. Analysts will, therefore, not only be trained in QC techniques, but also be required to qualify to run analyses. The qualification test results for certifying analysts must be statistically valid and must include evaluation of precision and accuracy.

Analysts will demonstrate their proficiency in conducting chemical analyses by analyzing spiked standard samples using approved analytical methods. Proficiency will be demonstrated for each analyte to be analyzed by the analyst prior to conducting analyses of natural samples.

For any analytical method, analysts or an analytical team consisting of specific individuals will be considered to be certified to run a particular analysis if they have been involved in developing the precision

AUDITOR

Analyst	Analysis	Certified		Date	Comment
		Yes	No		

SOURCE: Environmental Science and Engineering, Inc., 1988.

**ESE ANALYST CERTIFICATION AUDIT
PAGE**

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

AMD.1/QC4.3

Section Number 4
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and accuracy data needed for method documentation. The precision and accuracy data generated during method documentation must be acceptable to the Analytical Team Leader and the QA/QC Supervisor.

The analytical team responsible for gas chromatographic (GC) and high-performance liquid chromatographic (HPLC) analyses will usually consist of a technician performing the sample work-up and extraction and a chemist performing the subsequent instrumental analysis of the extract. Should this team fail to pass the required QC tests of precision and accuracy for a certain batch of samples, corrective action will require the investigation of the instrumental analysis separate from the extraction. The chemist performing the instrumental analysis is considered certified if he/she meets the instrumental calibration QC criteria. The extraction procedure will then be identified as out of control. The extraction technician can gain recertification by repeating the extraction in question and meeting the QC criteria. If the analyst fails to produce acceptable results, analysis is stopped until the reason for failure is identified and corrected.

5.0 LABORATORY AND METHOD CERTIFICATION

5.1 RATIONALE

Each new method developed requires documentation including precision and accuracy data. A specified detection limit must be achievable for a particular analyte. This detection limit must be statistically meaningful and consistently obtainable. This section outlines the testing procedures which will be used to define the detection limit, precision, and accuracy of each analytical method, according to standardized criteria.

5.2 METHOD CERTIFICATION

The following paragraphs describe the procedures to be used to certify analytical methods. All methods certification and documentation data will be developed initially in standard matrices and subsequently in natural samples.

The standard matrix for documentation of organic analysis will be deionized, organic-free (ASTM Type IV) water containing 100 milligrams per liter (mg/L) each of sulfate and chloride prepared as follows:

1. Add 1.48 grams (g) of dried reagent-grade anhydrous sodium sulfate to a 1-liter (L) volumetric flask and dilute to volume;
2. Add 1.65 g of dried reagent grade sodium chloride to a 1-L volumetric flask and dilute to volume; and
3. Transfer 100 milliliters (ml) of each (1 and 2) to a 1-L flask and dilute to volume.

The resulting solution contains 100 mg/L each of chloride and sulfate ions. This water will be used for blanks or will be spiked with the compound(s) of interest prior to processing through the complete analytical protocol.

The natural water sample used in method documentation can be any uncontaminated natural water sample, preferably a surface water or a composite of surface and ground water, which is available in sufficient quantities to conduct the natural sample testing for all the analytical methods developed under this contract. The same natural water should be used for all analytical methods documentation.

The data for documentation of analyses in soils will be developed using a standard soil matrix. The standard soil will consist of a homogeneous sample of sufficient size to provide a single continuous source for all method documentation. An aliquot of sieved (Paragraph 7.1.3.2) standard soil will be carried through each set of documentation samples to act as a blank. Added concentrations of the subject analyte(s) will be dissolved in a volume of solvent just sufficient to wet the soil. This solution is poured over the subsample of soil and allowed to stand for 1 hour prior to beginning analysis.

The natural soil sample to be used in method documentation can be any uncontaminated soil, preferably a composite of several soil types, which is dried, sieved, and available in sufficient quantities for all the method documentation work to be performed in this project.

Quantitative analytical methods will be developed in this project to quantitate the level of contamination of specific analytes in various environmental matrices. The process of quantitative documentation requires the detection limit of the analytical method to be determined and the precision and accuracy documented to a statistically reliable degree.

Certification of a quantitative method requires analysis of separate batches of standard media (water, soil, etc.) samples spiked at the following concentrations: 0.5X, 1.0X, 2.0X, 5.0X, and 10X, where X is the required/desired detection limit. Each level is spiked and analyzed once on 4 separate days. A blank sample of the standard matrix is also analyzed with each batch.

AMD.1/QC5.3

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A plot of found-versus-target concentration (amount spiked) is generated, and the detection limit is calculated using the methods of Hubaux and Vos. The precision of the method will be the standard error of the best-fit linear regression line of found-versus-target concentration values for the data generated over the 4 days of certification testing. The accuracy of the method will be the slope of the best-fit linear regression line of found-versus-target concentrations. The method will be written in the USATHAMA format and will be submitted to USATHAMA for approval along with the certification data.

6.0 SAMPLE COLLECTION

6.1 RATIONALE

As part of the verification of the developed methods in natural water/soil samples, it may be necessary to collect contaminated or "blank" natural samples for analysis by the methods developed during the project.

6.2 QA PROCEDURES FOR SAMPLING

A field team and leader will be designated by the Project Manager. The Field Team Leader is responsible for proper sampling, labeling of samples, preservation, and shipment of samples to the laboratory in a proper manner.

Water samples will be stored and shipped in amber glass bottles with Teflon®-lined lids. Soil samples will be contained in glass Mason jars. These containers will be prepared by thorough washing with hot detergent and water, triple rinsing with tap water, triple rinsing with deionized water, rinsing with methanol, air drying, and baking at 100°C for several hours.

Sampling locations will be determined in discussions between the Project Manager and the USATHAMA Project Officer.

All collected samples will be stored under refrigeration at 4°C at all times prior to subsampling or analysis.

AMD.1/QC7.1

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7.0 ANALYTICAL SYSTEMS CONTROL

7.1 GENERAL PROCEDURES

7.1.1 NOTEBOOKS AND INSTRUMENT LOGBOOKS

The ultimate repository for information concerning analyses performed in the laboratory is the analyst's personal laboratory notebook and the instrument logbooks.

Each analyst is required to have a personal notebook which is designated by a unique number. Each analyst is responsible for maintaining complete laboratory notes. The ESE QA/QC Supervisor maintains a list of assigned notebook numbers and audits laboratory notebooks without notice. The list of assigned notebooks contains the following information:

1. Notebook number,
2. Assignee,
3. Responsible Group Leader, and
4. Disposition or location and date.

Laboratory notebooks for this project will not be taken from ESE without written permission of the Analytical Team Leader and the ESE Project Manager. After completed notebooks are approved by the analyst, QA/QC Supervisor, and Group Leader, custody is transferred to the Analytical Team Leader. Every entry into the notebook should be dated and signed. Each Group Leader is responsible for ensuring that the notebook entries are counter-signed. Entries in the personal notebook will vary depending on the role of the individual in the laboratory and the type of work being performed. At a minimum, the personal notebook should contain:

1. A reference to or a description of the procedures used for sample work-up or analysis,

2. A summary of the samples extracted or analyzed,
3. Weighings and calculations of standard concentrations, and
4. Information on spiking procedures, and observations and comments on the procedures or samples.

The logbook consists of a bound notebook containing the preventive maintenance schedule.

Each time an instrument is used for sample analysis, the following information is entered:

1. Date of analysis;
2. Project name and number;
3. Number of samples analyzed, and type of sample;
4. Time spent on analysis (start to finish);
5. Preventive maintenance performed, if any;
6. Time spent on preventive maintenance;
7. Instrument calibration performed, if any; and
8. Name of analyst.

Additional notes are made in the instrument logs when required. These notes are particularly important when abnormal instrument or analytical performance is observed. It is the analyst's responsibility to ensure that instrument logs are properly filled out and kept up to date. The QA/QC Supervisor monitors and audits the status of instrument logbooks.

Copies of all pages of instrument logbooks for instruments used in this project will be compiled monthly by the analyst and approved by the QA/QC Supervisor. This instrument documentation record becomes a part of the permanent project QA/QC record.

7.1.2 STANDARDS AND SARMS

All materials used to prepare calibration standards and spiking standards must be Standard Analytical Reference Materials (SARMS)

supplied by USATHAMA. SARMS or interim SARMS are materials that have undergone extensive purity and stability checks. Interim reference materials may be used when analyses must be run before a SARM is available. However, the following precautions must be taken:

1. The interim reference material will be stored at 0°C, and a portion will be retained for comparison with the approved SARM when available;
2. The following data will be recorded as a minimum description of the material:
 - a. Infrared spectrum;
 - b. Melting point, decomposition point, or boiling point;
 - c. NMR spectrum;
 - d. Elemental analysis; and
 - e. GC or LC (by difference) analysis.

In cases where SARMS are difficult to obtain or only small amounts are available, interim SARMS or standards may be used for all calibration and spiking work, as long as the purity and response of such materials can be compared to the purity of the SARM. All reference compounds used in this project will be stored at 0°C and protected from light. The QA/QC Supervisor or Analytical Team Leader will request SARMS as required. The QA/QC Supervisor maintains a record of receipt of SARMS and monitors their use. A record of SARM material usage is maintained which identifies the analyst and date of use.

7.1.3 SAMPLE PREPARATION

The following paragraphs describe the preparation of water, soil, and sediment samples for analysis.

7.1.3.1 Water Samples

Prior to analysis, groundwater samples will be filtered through a 0.45-micron filter (constructed of a material which is suitable for the intended analysis) to remove suspended particulate matter. Surface water samples will not be filtered before analysis. Many organic

compounds adsorb on particulate matter. Therefore, it is desirable to detect contaminants migrating in the suspended fraction of the water column.

7.1.3.2 Soil Samples

Prior to analysis, soil samples will be sieved through a 30-mesh (500-um) brass sieve to remove rocks and debris.

Prior to sieving, soil samples should be spread out on the dull side of aluminum foil to air dry if sufficiently wet.

Soil samples must be properly subsampled before any analysis is performed. All subsampling must be accomplished with the aid of a riffle or by proper quartering techniques according to ASTM Specification D346.

A moisture determination in accordance with ASTM Specification D2216-71, Laboratory Determination of Moisture Content of Soil, will be made on each solid sample so that analytical data can be reported on a moisture-free basis.

7.1.4 STANDARD SAMPLES

Preparation of standard water and soil for methods development and analytical systems control have been described in Paragraph 5.2. Standard water for inorganic analysis consists of deionized water. Standard water for organic analysis consists of deionized organic-free water containing 100 mg/L each of sulfate and chloride. Standard samples for soil and sediment analysis consist of samples of an approved standard soil.

7.1.5 INSTRUMENT CALIBRATION AND OPERATING PROCEDURES

A calibration procedure establishes the relationship between an accurately known calibration standard and the measurement of that

standard by an instrument or analytical procedure. Calibration is not to be confused with standardization. Standards are run each time an instrument or procedure is used, while calibration is performed only at specified intervals.

Operating procedures must be available for all equipment and analytical instrumentation. Such procedures are generally provided by the manufacturer.

Written procedures for the operation and calibration of instrumentation are provided to the analyst in the laboratory to help minimize possible measurement inconsistencies due to differing techniques, conditions, and choice of standards. The procedures include the following information:

1. The specific instrument (or group of instruments) and analysis for which the procedure is applicable;
2. An explanation of theoretical considerations pertinent to the understanding of both the calibration procedure and the analysis;
3. Fundamental calibration specifications;
4. A list of requisite standards and equipment for the procedure;
5. Complete presentation of the procedure in a clear, step-by-step manner; and
6. Specific instructions for obtaining and recording calibration information.

An up-to-date report for each calibration standard used in the calibration system should be provided. If calibration services are performed by a commercial laboratory on a contract basis, copies of reports issued by them should be maintained on file.

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All contractor calibration reports are kept in a suitable file by the QA Supervisor and contain the following information:

1. Report number;
2. Identification or serial number of the calibration standard to which the report pertains;
3. Conditions under which the calibration was performed (temperature, relative humidity, etc.);
4. Accuracy of calibration standards (expressed in percentage or other suitable terms);
5. Deviation or corrections; and
6. Corrections that must be applied if standard conditions of temperature, etc., are not met or differ from those at place of calibration.

Contracts for calibration services should require the contractor to supply records on traceability of their calibration standards.

All equipment to be calibrated should have affixed to it in plain sight a tag bearing the following information:

Description: _____
Ident. No.: _____
Last Calibrated: _____
Calibrated By: _____
Calibration Expires: _____

NOTE: Use of this instrument beyond the calibration expiration date is prohibited.

When the equipment size or its intended use limits the application of labels, an identifying code should be applied.

Equipment past due for calibration should be removed from service either physically or, if this is impractical, impounded by tagging or other means.

7.2 SPECIFIC ANALYTICAL SYSTEMS CONTROL: GC AND HPLC ANALYSES

7.2.1 INSTRUMENT CALIBRATION

Before analyses are performed on the gas chromatograph, the gas flow rates through the column and detector are measured and the carrier gas flow controllers are calibrated. For roto-meter controllers, the position of the floating ball is measured versus the gas flow through the column and a graph of flow rate versus ball position is drawn. Electronic flow controllers are calibrated according to manufacturer's specifications, and the agreement between the set flow rate and the measured flow through the column is checked. A table of measured versus set flow rate is prepared. The flow rate through the column and detectors is measured using a volumetric bubblemeter supplied by the manufacturer. A packed column should be in the instrument, and the oven and the injector should be at ambient temperature. The flow-rate setting is checked at the start of an analytical run and periodically calibrated thereafter, but at least once every 6 months. A record of the flow rate calibration including charts and tables is kept in the instrument logbook.

Temperature calibration of the detector, injector, and oven zones in a GC is accomplished before any analyses are performed. The temperature in these zones is measured by a calibrated thermocouple placed into the appropriate zone. The temperature calibration is recorded in the instrument logbook.

For high pressure liquid chromatographs, the system flow rates are calibrated before any analyses are conducted. The calibration is performed by measuring the liquid flow at the outlet of the detector system using a volumetric measurement such as a Class A volumetric flask. In two pump gradient systems, the flow rates should be checked

with each pump working independently at a 50/50 gradient mixture. A record of this calibration should be kept in the instrument log book.

HPLC columns should be checked for efficiency, before any analysis is conducted, according to manufacturer's specifications or another approved consistent procedure. A record of the column's efficiency and resolution between adjacent peaks should be kept in the instrument notebook.

Calibration is performed when a new instrument arrives in the laboratory and is repeated at periodic intervals thereafter. The instrument sensitivity calibration is performed according to the respective manufacturer's instructions. This sensitivity or performance check is repeated when instrument performance deterioration is suspected. When no manufacturer-supplied sensitivity check procedures are available, the instrument response to a known concentration standard under defined conditions is to be used to check the sensitivity.

The instrument logbooks should contain:

1. Data of last calibration of flow and temperature controllers, and historical curves,
2. Detector calibration calculations and information,
3. A log of the type of analysis run on the instrument including:
 - a. Column conditions and temperature zones for GC and column type, conditions and flow rates for HPLC.
 - b. Sample numbers or other identification of samples analyzed.
 - c. Reference to a notebook page describing the analysis performed.
 - d. Date of analysis.
 - e. Detector used--FID, ECD, UV, fluorescence, etc.
4. Service records.

7.2.2 PEAK MEASUREMENTS

Three primary means of measuring chromatographic peak response may be used: area, peak height, and peak height x width-at-half-height. Peak height is preferred in those cases where very noisy signals make the use of electronic integrators difficult in complex chromatograms. In this project, all these means of peak response measurement may be used. However, once the choice of peak response measurement is made, the same mode of peak response measurement will be used consistently throughout the project for that particular analysis. The choice of method used for a particular analysis is made by the analyst after consultation with his/her supervisor.

Analyte peaks on a chromatogram must be identified. The peaks of interest must be marked to definitively specify which peaks were used in the quantitation and calculations.

The primary means of peak identification for an analyte in the chromatographic methods is that the peak of interest falls within a predetermined retention time window of the retention time of an authentic standard of that particular analyte. Usually a retention window of 5% will be used for peak identification. However, for temperature-programmed or solvent-programmed runs, a retention window of ± 2 standard deviation units around the average value of the standard peak retention time should be used as a confirmatory check. The average peak retention time is determined from analytical standards run that day.

7.2.3 SAMPLE DOCUMENTATION

An analyst's extraction logsheet will be filled out by the analyst performing the sample extraction and will accompany the batch of samples throughout the analysis procedure. (An example of this extraction logsheet is given in Figure 7-1). The data on this sheet will include, at a minimum, the following:

Project Name: _____
Project No.: _____
Sample Type: _____
Analysis: _____
Extraction Procedure: _____

Logbook No.: _____
Plant Code: _____
Analyst: _____

[illegible]

Figure 7-1
ESE EXTRACTION LOGSHEET

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

1. Notebook or literature reference to procedure used for extraction,
2. Type of sample matrix,
3. Date of extraction,
4. Volume or weight of sample taken for analysis,
5. Dry-weight of the sample—if necessary,
6. Final volume of extract,
7. Project number and name,
8. Notes and comments affecting the extraction procedure, and
9. Name or initials of the analyst.

7.2.4 CHROMATOGRAPHIC LOGSHEETS

Two types of chromatographic logsheets are used. One sheet is the standard curve sheet (Figure 7-2) which lists the standards, their concentrations, and the respective peak heights or areas. The second sheet, the chromatographic data sheet (Figure 7-3), lists the samples in order of their injection with the factors needed for calculation of the concentrations.

7.3 ANALYTICAL SYSTEMS QUALITY CONTROL PROCEDURES

The following describes the quality control procedures and requirements for analyses conducted during this project. These quality control requirements are in addition to any specific calibration requirements presented in Subsection 7.2.

An initial instrumental standardization will be performed before any samples are analyzed. Calibration standards will be prepared and analyzed in the concentration series 0 (blank), 0.5X, X, 2X, 5X, and 10X where X is the concentration in the extract or sample being analyzed corresponding to the desired detection limit. For example, if the desired detection limit in the matrix is 1 microgram per liter (ug/L) and a thousandfold concentration is required before introduction into an instrument, X would be 1 ug/L. The data from the initial calibrations during documentation will be averaged and used to calculate: (1) the

QAD194.3/DATA-STCURV4.1

ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.
STANDARD CURVE DATA SHEET FOR GC/NPLC ANALYSES

Project: _____ Date: _____
 Project Number: _____ Analyst: _____
 Plant/ID: _____ Logbook No.: _____ Page: _____
 Compound: _____ Retention Time: _____

Std. No.	Inj. Vol.	Conc. /ul	Am. Inj.	Area Peak or Height	Normalized Response Attn/Factor	Conc. /ul	Am. Inj.	Area Peak or Height	Normalized Response Attn/Factor	Chromatogram Number
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Std. Used	Curve No. 1	Curve No. 2	Curve No. 3
Corr. Coef.			
Intercept			
Slope			

SOURCE: Environmental Science and Engineering, Inc., 1988.

Figure 7 - 2
EEG STANDARD CURVE DATA SHEET FOR GC/NPLC ANALYSES

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.
CHROMATOGRAPHIC LOG SHEET
SAMPLE NO.

Project: _____
 Project No.: _____
 Analyst: _____
 Sample Type: _____
 Log Book No.: _____
 Date: _____
 Pages: _____

Retention Time Window (Z)

[illegible]

SOURCE: Environmental Science and Engineering, Inc., 1988.

Figure 7 - 3
ESE CHROMATOGRAPHIC LOGSHEET

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

average instrumental Hubaux and Vos detection limit, and (2) the average slope of the calibration curve.

The average slope will be used to initiate and set up the control limits. During the development of the method certification data for each analyte, each instrument will be controlled using a standard curve consisting of three standards plus one procedure blank analyzed at the beginning of the analytical batch. A single standard (mid-scale) will be analyzed at the end of the analysis to measure instrument drift. An instrument shall be considered out of control if the slope of the calibration curve decreases by more than 30 percent. The response values for the mid-scale standard analyzed before and after the run must agree within 30 percent. The correlation coefficient for the linear regression of the calibration curve must always exceed 0.995. The value of each of these checks will be recorded in the analyst's notebook.

The analysts are responsible for ensuring that each routine standardization meets the QC criteria. Failure of the analytical system to meet all QC criteria requires immediate corrective action, which may include rerunning samples judged out of control.

All data reported using the calibration curve must be bracketed on the upper and lower end by standards.

At least one procedure blank sample will be included with each batch of samples.

7.4 DEVELOPMENT OF ANALYTICAL METHODS

Development of a method will be initiated by submitting Documentation for Proposed Methods Development to the USATHAMA Chemistry Group for approval prior to development activity (Appendix 4 of USATHAMA QA Program, 1980). Each of the methods proposals will contain the following information, organized in the format outlined below:

DOCUMENTATION FOR PROPOSED METHOD DEVELOPMENT:
(ANALYTE) IN (SOIL/WATER)

1. SUBMITTING ORGANIZATION.
2. JUSTIFICATION OF WORK.
3. OUTLINE OF PROPOSED EFFORT.
4. PROPOSED SAMPLES TO BE USED IN DEVELOPMENT.
5. ESTIMATE OF RESOURCES REQUIRED.
6. PROPOSED SOURCE OF RESOURCES.
7. IMPACT OF REPROGRAMMING USATHAMA FUNDS.
8. ESTIMATED TIME TO COMPLETE WORK AND SUBMIT REPORT.

The USATHAMA Chemistry Group will evaluate the proposed approach for technical soundness and economy of effort. The Chemistry Group will then request that ESE proceed with the method development as proposed or with recommended modifications.

ESE personnel will investigate the proposed procedures to be included in the method. Should any of the proposed procedures approved by the Chemistry Group be found to be inadequate for the method, alternative procedures will be investigated after approval by the Chemistry Group.

7.5 CHARACTERIZATION OF ANALYTICAL METHODS

When the analytical procedures have been finalized by ESE, the procedures will be documented according to the requirements specified in Appendix 2 of the USATHAMA QA Program (August 1980).

ESE will generate precision and accuracy data on the proposed method in standard samples and in natural media.

Using the precision and accuracy data, the detection limit will be calculated as well as the sensitivity at the detection limit for inclusion in the documentation of the method.

Full documentation of the method will be submitted to the USATHAMA Chemistry Group. The Chemistry Group will review the documentation for completeness and comprehension. Based on this review, the Development Laboratory will make any necessary modifications prior to approval of the method by the USATHAMA Chemistry Group.

These method documentation data will include estimates of the standard deviation, percent inaccuracy, and percent imprecision.

1. The standard deviations will be calculated at each target concentration according to:

$$\text{Standard deviation} = S = \left[\frac{\sum X_i^2 - \frac{(\sum X_i)^2}{n}}{n-1} \right]^{1/2}$$

where X_i = the i th found concentration

n = total number of X values

and $\sum$ = summation from $i = 1$ to $i = n$

2. The percent inaccuracy will be calculated at each target concentration according to

$$\text{percent inaccuracy} = \frac{x - TC}{TC} \times 100$$

where x = average found concentration at the particular TC
and TC = target concentration.

3. The percent imprecision will be calculated at each target concentration according to

$$\text{percent imprecision} = \frac{s}{\bar{x}} \times 100$$

where s = standard deviation

and $\bar{x}$ = average found concentration at the particular target concentration.

Upon final approval of the documented method, the Chemistry Group will assign a number to the method.

The format below (USATHAMA QA Program, August 1980) will be followed for submittal of all method documentation data.

TITLE: ANALYTE(S) IN (WATER/SOIL) SAMPLES

1. Application: State analytes that can be analyzed by this method and media in which the analytes are contained (e.g., TNT in soil). The media should be the original matrices to be analyzed (e.g., soil, air, water, biological tissue, etc.) rather than intermediates in the procedure (e.g., bubbler from air sampling, extract from soil, etc.).
 - a. Tested Concentration Range: State concentration range in the original matrix that was tested for this validation (e.g., 1 to 20 ug/L in water, 5 to 100 mg/m³ in air, etc.).
 - b. Sensitivity: Response (peak height, area, etc.) observed for absolute quantity of analyte (state quantity) at the detection limit (e.g., 1,500 area units for 40 picograms).
 - c. Detection Limit: Limit of detection for complete analytical method, determined from precision and accuracy data generated from spiked standard samples (standard water, soil, etc.) and calculated according to Hubaux and Vos, expressed in terms of concentration in original medium (soil, water, etc.).
 - d. Interferences: State any observed interferences or any interferences anticipated based on the method of analysis.
 - e. Analysis Rate: State the estimated number of samples that can be analyzed by this method in an 8-hour day after instrument calibration.

2. Chemistry:

- a. List physical and chemical properties of analyte(s), including Chemical Abstracts Service Registry number.
- b. Describe in detail any chemical reactions involved in the analytical method (such as conversion of organic nitrogen to ammonia followed by conversion to ammonium chloride in hydrochloric acid).

3. Apparatus:

- a. Instrumentation: List makes and models of instruments, as well as specific characteristics (such as detectors).
- b. Parameters: List operating parameters of instruments, as well as chromatography columns.
- c. Hardware/Glassware: List miscellaneous equipment. Include sources for specialty or trademarked items.
- d. Chemicals: List chemicals necessary. State sources of analytical reference materials.

4. Standards:

- a. Calibration standards: Describe, in detail, the step-by-step procedure for preparing instrument calibration standards to include proper storage and shelf life.
- b. Control spikes: Describe, in detail, the step-by-step procedure for preparing spikes of control samples.

5. Procedure: Describe, in detail, the step-by-step procedure for analyzing control and actual samples, as well as instrument calibration procedures. Include instructions for constructing necessary graphs.6. Calculations: Describe, in detail, the manner by which the concentrations in the original matrix are calculated from the responses obtained in the analysis.

7. References: List any references used as a source for the procedures.
8. Data:
 - a. Tabulate precision and accuracy data by indicating found concentrations (uncorrected) for each target concentration by day.
 - b. Tabulate average found value, standard deviation, percent imprecision, and percent inaccuracy for each target value.
 - c. Plot the found concentration versus the target concentration (include linear regression, confidence bounds, and Hubaux and Vos detection limit annotated).
 - d. Plot the standard deviation versus the target concentration.
 - e. Plot the percent inaccuracy versus the target concentration.
 - f. Plot the percent imprecision versus the target concentration.

APPENDIX C
COMPUTER PROGRAM FOR REDUCTION
OF HPLC SCREEN DATA

APPENDIX C
COMPUTER PROGRAM FOR REDUCTION OF HPLC SCREEN DATA

The various subsections of the program are described below:

Steps 10 through 300--Calculates the ug/L of the analytes in the original sample from the raw data and stores the peak heights for later calculation of ratios.

Steps 400 through 430--Lists the analyte names under the file variables H1(I) and H2(I) which are the first and last parts of the compound name, respectively.

Steps 600 through 710--Lists a set of calibration curve data for an analyte on a particular detector.

Steps 992 through 1446--Calculates the calibration data for all of the analytes and stores the resultant slope, intercept, and correlation coefficient for subsequent use under a compound index number, I, and a file number, FI, for each detector:

FI = 1 for the LC-75

FI = 2 for the 254 nm

FI = 3 for fluorescence

The compound index number, I, is equivalent to the X index number used in the calibration curve data. The correlation coefficient is stored under Gu(X). The slope and intercept are stored under KB(X) and KC(X), respectively.

Steps 1495 through 1860--Calculates the detector ratios for each analyte in a single chromatographic run. B1(I), B2(I), and B3(I) are the peak heights of component I on the LC-75, 254 nm, and

fluorescence detectors, respectively. The ratio of the peak height of component I on the LC-75 detector to the peak height on the 254 nm detector is stored as T(I) for responses normalized to the ortho-nitrotoluene response and V(I) for the unnormalized response. H(I) and S(I) are used for the unnormalized 254 nm to fluorescence ratio and LC-75 to fluorescence ratio, respectively.

Steps 1899 through 1932--Subroutine for manual entry of peak heights for the 254 nm detector.

Steps 1969 to 1998--Manual entry of peak heights for fluorescence detector. (Manual entry for LC-75 peak heights uses Steps 130 to 150.)

Step 2082--Defines a special function key for initiation of the ratio program at 992.

Step 2086--Defines a special function key for initiation of the data reduction program at Step 15.

An example of the output of the ratio calculation program beginning at Step 1495 is presented below. Ratios unnormalized and normalized responses are calculated.

Step 2090--Defines a special function key for initiation of the detector ratio program at 1495.

The following is a listing of the input and output of a typical data set calculation for the LC-75 data. In the input section, the program provides a series of prompting statements to set up the data file, and the peak heights are entered for each prompted compound. The output section is titled report and contains the concentration in the sample, the peak height as a check for input errors, and the ratio of the peak height to the peak height of ortho-nitrotoluene.

RUN 1495
WHAT LEVEL
? 19

ENTER OMT RESPONSE AT LC-75
? 519
ENTER OMT RESPONSE AT 254nm
? 568

RESPONSE RATIOS LEVEL 19.
RATIOS WITH OMT

COMPOUND	LC-75/254
MX	7.1311492
PDX	3.1915717
THB	3.9389201
DNB	2.0388309
TETVL	A.
DNP	1.4372199

TNT	2.578323
2,6 DNT	2.068702
2,4 DNT	2.4518561
OMT	1.
NAPHT	1.535228
ACENAPHTHV	1.4738333
FLUORENE	0.1774753
ACENAPTH	0.5486892
PHENANTH	0.401843
ANTHRACENE	0.0078672
FLUORANTH	2.6348618
PYRENE	0.5352884
CHRYSENE	0.4791968
BZ(B)FLUOR	1.12634
BZ(K)FLUOR	1.383748
B(B)PYRENE	1.7397807
BBZ(A)ANT	13.643839
ICD)PVR	1.0171319

RATIOS WITHOUT OMT

COMPOUND	LC-75/254	254/FL	LC-75/FL
MX	4.5834872	A.	0.
PDX	2.9015830	0.	0.
THB	3.592893	0.	0.
DNB	1.0593564	A.	0.
TETVL	0.	0.	0.
DNP	1.118784	0.	0.
TNT	2.3448621	0.	0.
2,6 DNT	1.0865979	0.	0.
2,4 DNT	2.2352941	0.	0.
OMT	0.9119718	0.	0.
NAPHT	1.4888847	26.848989	37.579545
ACENAPHTHV	1.3448945	0.	0.
FLUORENE	7.4576271	1.475	11.
ACENAPTH	0.5883891	0.	0.
PHENANTH	0.3664695	29.216849	10.78679
ANTHRACENE	0.006445	231.86517	1.494382
FLUORANTH	2.4029197	0.5445151	1.3884261
PYRENE	0.4881497	9.1898485	4.4469697
CHRYSENE	0.437814	13.493976	6.7718843
BZ(B)FLUOR	1.0271903	1.0091463	1.0265854
BZ(K)FLUOR	1.2619392	0.4859353	0.6132208
B(B)PYRENE	1.586588	1.2854639	2.007732
BBZ(A)ANT	12.442797	0.5673877	7.0588942
ICD)PVR	0.9275956	1.5947712	1.4793828

EP000 10

IF LC-75 INPUT 1
 IF 254 nm INPUT 2
 IF FLUORESCENCE INPUT 3
 ? 1
 WHAT LEVEL IS THE SAMPLE
 ? 10
 HOW MANY COMPOUNDS
 ? 24
 THE CHROMATOGRAM NUMBER IS? 154
 ENTER 1 FOR NATURAL, 2 FOR STANDARD? 2
 ENTER 1 FOR UNCLEARED, 2 FOR CLEARED? 1
 THE RESPONSE OF DNT IS? 518
 ENTER PEAK HEIGHTS
 MX 7635
 PK 3859
 TH 7723
 DN 6240
 TETRA 0
 DN 2649
 TH 2112
 2.6 DNT 2013
 2.4 DNT 2310
 DNT 518
 NAPHT 3307
 ACENAPHTH 3414
 FLUORENE 440
 ACENAPHTH 1206
 PHENANTH 2449
 ANTHRACENE 133
 FLUORANTH 1446
 PYRENE 1174
 CHRYSENE 562
 BZ(B)FLUOR 680
 BZ(K)FLUOR 872
 B(B)PYRENE 779
 BZ(AM)ANT 5073
 I(CD)PVR 679

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REPORT

CHROMATOGRAM NUMBER 154.
 LEVEL 10.
 PERKIN ELMER LC-75
 STANDARD WATER UNCLEARED

COMPOUND	CONC(ug/L)	RESPONSE	RATIO
MX	20.297539	7635.	14.739202
PK	40.769014	3859.	7.4490069
TH	30.291212	7723.	14.909266
DN	37.413637	6240.	12.046332
TETRA	0.	0.	0.
DN	40.707002	2649.	7.0444015
TH	20.404333	2112.	4.0772201
2.6 DNT	37.413637	2013.	3.8061004
2.4 DNT	31.044370	2310.	4.4749023
DNT	0.	518.	1.
NAPHT	30.000100	3307.	6.3041699
ACENAPHTH	112.4290	3414.	6.5907326
FLUORENE	2.0241490	440.	0.0494200
ACENAPHTH	37.753739	1206.	2.4026235
PHENANTH	24.254340	2449.	6.0969112
ANTHRACENE	0.	133.	0.2567560
FLUORANTH	7.5549302	1446.	3.1776062
PYRENE	32.925103	1174.	2.2664092
CHRYSENE	1.9729443	562.	1.0049421
BZ(B)FLUOR	2.0659135	680.	1.2127413
BZ(K)FLUOR	4.8204703	872.	1.6023977
B(B)PYRENE	1.664967	779.	1.503061
BZ(AM)ANT	30.80055	5073.	11.227020
I(CD)PVR	5.377005	679.	1.2100100

WHAT LEVEL
2 5

ENTER ONT RESPONSE AT LC-75
2 380
ENTER ONT RESPONSE AT 254nm
2 464

RESPONSE RATIOS LEVEL 5.
RATIOS WITH ONT

COMPOUND	LC-75/254
MAX	2.8249453
RDX	2.7262448
TNB	4.477834
DNB	2.3721701
TETRAVL	0.
DNP	2.2671351
TNT	2.7718938
2,6 DNT	2.3902681
2,4 DNT	1.5642592
ONT	1.0021598
NAPHT	2.4980702
ACENAPHTHY	1.6818546
FLUORENE	23.233918
ACENAPTH	0.6680679
PHENANTH	0.4434122
ANTHRACENE	0.
FLUORANTH	2.9672892
PYRENE	0.5880805
CHRYSENE	0.4952439
9Z(8)FLUOR	1.208555
9Z(8)FLUOR	1.5893414
8(8)PYRENE	2.1527002
DBZ(AM)ANT	15.573191
1(CD)PYR	1.1622952

RATIOS WITHOUT ONT

COMPOUND	LC-75/254	254/FL	LC-75/FL
MAX	6.5742483	0.	0.
RDX	3.8516527	0.	0.
TNB	3.6671916	0.	0.
DNB	1.9427253	0.	0.
TETRAVL	0.	0.	0.
DNP	1.8567854	0.	0.
TNT	2.2788053	0.	0.
2,6 DNT	1.9575472	0.	0.
2,4 DNT	1.2810743	0.	0.
ONT	0.8287343	0.	0.
NAPHT	2.8458333	21.818182	44.636264
ACENAPHTHY	1.377381	0.	0.
FLUORENE	19.827778	0.	0.
ACENAPTH	0.5471246	78.25	42.8125
PHENANTH	0.3631393	31.5	11.438889
ANTHRACENE	0.	191.6	0.
FLUORANTH	2.4381875	0.5622733	1.3663843
PYRENE	0.4816176	0.16	3.93
CHRYSENE	0.4055877	16.21875	6.578125
9Z(8)FLUOR	0.9988878	1.8641283	1.8541082
9Z(8)FLUOR	1.3816158	0.4261668	0.5547054
8(8)PYRENE	1.7629938	1.2895442	2.2734584
DBZ(AM)ANT	12.753906	0.6236297	7.953715
1(CD)PYR	0.9518797	1.6181695	1.5326877

LIST

```

10 REM HPLC SCREEN DATA REDUCTION
15 !!!
20 !"IF LC-75 INPUT 1"
40 !"IF 254 nm INPUT 2"
50 !"IF FLUORESCENCE INPUT 3"
60 INPUT FI
65 !"WHAT LEVEL IS THE SAMPLE"
66 INPUT J
70 !"HOW MANY COMPOUNDS"
80 INPUT A
90 !"THE CHROMATOGRAM NUMBER IS";
100 INPUT T
105 !"ENTER 1 FOR NATURAL, 2 FOR STANDARD";
107 INPUT B
108 !"ENTER 1 FOR UNCLEANNED, 2 FOR CLEANED";
109 INPUT D
110 !"THE RESPONSE OF ONT IS";
115 INPUT X
120 !"ENTER PEAK HEIGHTS"
122 IF FI=2 THEN GOTO 1099
123 IF FI=3 THEN GOTO 1969
130 FOR I=1 TO A
140 !S.03H1(I);H2(I);
141 INPUT B1(I)
150 NEXT
160 FOR I=1 TO A
162 C(I)=(B1(I)/X)*KB(I)+KC(I)*0.2
165 IF B1(I)=0 THEN C(I)=0
168 IF C(I)<0 THEN C(I)=0
173 R(I)=B1(I)/X
174 P(I)=B1(I)
175 NEXT
180 !!!
185 !TAB30***REPORT***
187 !!!
190 !TAB23"CHROMATOGRAM NUMBER";T
195 !TAB33"LEVEL";J
200 IF FI=1 THEN !TAB25***PERKIN ELMER LC-75***
210 IF FI=2 THEN !TAB25***ALTEX 254nm U.V.***
220 IF FI=3 THEN !TAB25***PERKIN ELMER FLUORESCENCE***
230 IF B=1 AND D=1 THEN !TAB24"NATURAL WATER UNCLEANNED"
240 IF B=1 AND D=2 THEN !TAB27"NATURAL WATER CLEANED"
250 IF B=2 AND D=1 THEN !TAB24"STANDARD WATER UNCLEANNED"
260 IF B=2 AND D=2 THEN !TAB 27"STANDARD WATER CLEANED"
270 !!
280 !"COMPOUND";TAB15"CONC(us/L)"*TAB35"RESPONSE"*TAB33"RATIO"
283 FOR I=1 TO A
285 !S.03H1(I);H2(I);
286 !TAB5C(I)*TAB25P(I)*TAB44R(I)
287 NEXT
290 !!!!
300 END
350 END
400 FOR I=1 TO 24
410 !S.03H1(I);H2(I)
420 NEXT
430 END
440 !"WHAT FILE"
442 INPUT FI
445 !"ENTER N.V. DETECTION LIMITS"

```

```

446 FOR I=1 TO 24
447 !*5.03H1(I);H2(I);
448 INPUT D(I)
449 NEXT
450 FOR I=1 TO 24
451 IF KB(I)=0 THEN KB(I)=1
452 X(I)=D(I)*5
453 A(I)=(X(I)-KC(I))/KB(I)
454 IF X(I)=0 THEN A(I)=0
455 IF FI=2 THEN S(I)=A(I)*580
456 IF FI=1 THEN S(I)=A(I)*410
457 IF FI=3 THEN S(I)=A(I)
458 IF X(I)=0 THEN A(I)=0
459 NEXT
460 !!!
470 !TAB35***REPORT***
475 !!!
490 !"ANALYTE"TAB20"INTEGRATOR COUNTS"TAB50"NANOGRAMS"
485 FOR I=1 TO 24
490 !*5.03H1(I);H2(I);
500 !TAB13S(I)TAB42X(I)
510 NEXT
520 END
600 !"WHAT FILE"
610 !"INPUT 1 FOR LC-75 "
620 !"INPUT 2 FOR 254nm "
630 !"INPUT 3 FOR FLUORESCENCE"
640 INPUT FI
650 !"HOW MANY CALIBRATION CURVES"
660 INPUT A
670 !"COMPOUND"TAB20"SLOPE"TAB40"INTERCEPT"TAB55"CORR COEFF"
673 A=24
680 FOR I=1 TO A
690 !*5.03H1(I);H2(I);
691 !TAB7KB(I)TAB29KC(I)TAB45Gu(I)
700 NEXT
705 !!!
710 END
992 !!!
995 !"WHAT FILE"
997 INPUT FI
1000 DIM C(50)
1005 DIM R(50)
1006 !"WHAT COMPOUND NUMBER"
1007 INPUT X
1009 !"LIST CONC. RESPONSE"
1010 FOR I=1 TO 50
1015 N=I
1020 INPUT C(I);R(I)
1030 IF C(I) AND R(I) <0 THEN GOTO 1200
1040 NEXT
1200 N=N-1
1210 S=0: T=0: F=0: D=0
1220 FOR I=1 TO N
1230 S=S+C(I)
1260 T=T+R(I)
1290 F=F+C(I)**2
1320 D=D+C(I)*R(I)
1330 NEXT
1340 A=D-(S*T/N)
1350 B=F-(S**2/N)
1360 KA(X)=A/B

```



```

1370 KC(X)=(S-KB(X)*T)/N
1375 Z=A
1380 FOR I=1 TO N
1390 Z=Z+P(I)**2
1400 NEXT
1410 H(X)=KB(X)*SDR(Z/N-(T/N)**2)/SDR(F/N-(S/N)**2)
1420 Gu(X)=1/H(X)
1435 !"SLOPE"TAB20"INTERCEPT"TAB45"CORR COEFF"
1440 !KB(X);TAB20KC(X);TAB45Gu(X)
1445 !!!!
1446 END
1495 !"WHAT LEVEL"
1497 INPUT R
1499 !!!
1500 !"ENTER ONT RESPONSE AT LC-75"
1501 INPUT X
1502 !"ENTER ONT RESPONSE AT 254nm"
1503 INPUT Y
1510 !TAB20"RESPONSE RATIOS"TAB45"LEVEL":R
1520 !TAB25"***RATIOS WITH ONT***"
1525 !!
1530 !"COMPOUND"TAB17"LC-75/254"
1560 FOR I=1 TO 24
1570 IF B2(I)=0 THEN B2(I)=-9
1580 T(I)=(B1(I)/X)/(B2(I)/Y)
1590 V(I)=B1(I)/B2(I)
1720 IF B3(I)=0 THEN B3(I)=-9
1730 H(I)=B2(I)/B3(I)
1770 S(I)=B1(I)/B3(I)
1790 NEXT
1781 FOR I=1 TO 24
1782 IF B2(I)<0 THEN T(I)=0
1783 !*5.03H1(I);H2(I);
1794 !TAB7T(I)
1785 NEXT
1790 !!!
1800 !TAB22"***RATIOS WITHOUT ONT***"
1810 !!
1820 !"COMPOUND"TAB17"LC-75/254"TAB34"254/FL"TAB51"LC-75/FL"
1830 FOR I=1 TO 24
1835 IF B2(I)<0 THEN H(I)=0
1836 IF B2(I)<0 THEN V(I)=0
1837 IF B3(I)<0 THEN H(I)=0
1838 IF B3(I)<0 THEN S(I)=0
1840 !*5.03H1(I);H2(I);
1841 !TAB7V(I)TAB24H(I)TAB41S(I)
1850 NEXT
1860 END
1899 FOR I=1 TO A
1900 !*5.03H1(I);H2(I);
1901 INPUT B2(I)
1902 NEXT
1903 FOR I=1 TO A
1904 C(I)=((B2(I)/X)*KB(I)+KC(I))*0.2
1907 IF B2(I)=0 THEN C(I)=0
1911 IF C(I)<0 THEN C(I)=0
1917 R(I)=B2(I)/X
1920 NEXT
1927 !!!
1928 FOR I=1 TO A
1929 P(I)=B2(I)

```

```
1930 NEXT
1932 GOTO 185
1969 FOR I=1 TO A
1970 !*5.03H1(I);H2(I);
1971 INPUT B3(I)
1972 NEXT
1973 X=1
1974 FOR I=1 TO A
1975 C(I)=((B3(I)/X)*KB(I)+KC(I))*0.2
1980 IF B3(I)=0 THEN C(I)=0
1983 IF C(I)<0 THEN C(I)=0
1992 R(I)=B3(I)
1996 P(I)=B3(I)
1997 NEXT
1998 GOTO 185
2082 GOTO 992
2086 GOTO 15
2090 GOTO 1495
```

APPENDIX D
ANALYTICAL METHODS

APPENDIX D
TABLE OF CONTENTS

PETN, HMX, AND RDX IN WATER SAMPLES

PETN, HMX, AND RDX IN SOIL SAMPLES

HMX IN WATER SAMPLES

HMX IN SOIL SAMPLES

DPA IN SOIL AND SEDIMENT SAMPLES

UDMH IN WATER SAMPLES

ATNBA IN WATER SAMPLES

ATNBA IN SOIL SAMPLES

35DNP IN WATER SAMPLES

35DNP IN SOIL SAMPLES

35DNA IN WATER SAMPLES

35DNA IN SOIL SAMPLES

TDGCL IN WATER SAMPLES

TDGCL IN SOIL SAMPLES

**HPLC SCREEN OF WATER SAMPLES FOR NITROSUBSTITUTED MUNITION
COMPOUNDS AND PAHs**

I.

I.

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PETN, HMX, AND RDX IN WATER SAMPLES

I.

I.

PETN, HMX, AND RDX IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for PETN, HMX, and RDX.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in natural and standard water are listed below:

<u>Analyte</u>	<u>Range (ug/L)</u>
PETN	1.58 to 31.6
HMX	0.43 to 8.5
RDX	1.26 to 25.2

B. SENSITIVITY

The normalized responses (integrator counts) at the natural water detection limits designated in Section 1(C) are listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
PETN	37700	281.1
HMX	121000	143.7
RDX	173000	256.2

The normalized responses (integrator counts) at the standard water detection limits designated in Section 1(C) are listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
PETN	27179	213.1
HMX	96096	110.4
RDX	68495	91.1

C. DETECTION LIMIT

The detection limits in natural water, calculated according to Hubaux and Vos (1970), are listed below:

<u>Analyte</u>	<u>Detection Limit (ug/L)</u>
PETN	4.5
HMX	2.3
RDX	4.1

The detection limits in standard water, calculated according to Hubaux and Vos (1970), are listed below:

<u>Analyte</u>	<u>Detection Limit (ug/L)</u>
PETN	3.4
BMX	1.8
RDX	1.5

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 215 nm and are extractable from water with methylene chloride.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
PETN	Pentaerythrite tetranitrate	78-11-5
	Pentaerythritol tetranitrate	
	2,2-Bis[(nitrooxy)-methyl]-	
	1,3-Propanediol dinitrate (ester)	
	Nitropentaerythritol	
BMX	Pentrit	2691-41-0
	Cyclotetramethylenetetranitramine	
	Octahydro-1,3,5,7-tetrazocine	
	1,3,5,7-Tetranitro-1,3,5,7-	
	tetrazacyclooctane	
RDX	Octogen	121-84-4
	Cyclotrimethylenetrinitramine	
	Hexogen, T-4, Cyclonite, Hexahydro-1,3,4-trinitro-s-triazine	

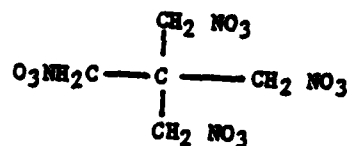
B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte	Formula	Melting Point (°C)	Boiling Point	Density (g/ml)
PETN	$C_5H_8N_4O_{12}$	141	180 at 50 torr	1.77
HMX	$C_4H_8N_8O_8$	276	—	1.77-1.96*
RDX	$C_3H_6N_6O_6$	204.1	—	1.816

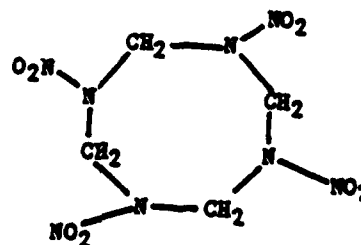
* There are four polymorphic forms of HMX with this range of densities.

Chemical Structures

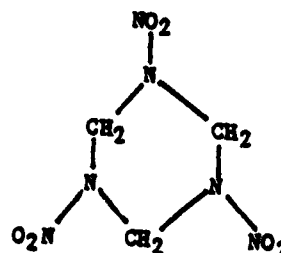
PETN



HMX



RDX



C. CHEMICAL REACTIONS

All of these compounds are highly explosive, and caution should be used in handling. Each compound is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
($\lambda = 215 \text{ nm}$)
2. Column: Zorbax-CN (4.6-mm ID x 25 cm)
Particle size: 7-8 μm
3. Flow Rate/Mobile Phase: 1 ml/min/35% H_2O /65% methanol
4. Temperature: 22°C
5. Injection Volume: 250 μl , fixed loop
6. Retention Times:

<u>Analyte</u>	<u>Retention Time (Minutes)</u>
RDX	7.8
HMX	11.8
PETN	13.9

C. HARDWARE/GLASSWARE

1. 1-liter separatory funnel (Teflon® or glass) (8).
2. 500-ml K-D flask (8).
3. 15-ml K-D receiver (8).
4. 3-ball Snyder column (8).
5. 2-ball micro-Snyder column (8).
6. 10-ml graduated centrifuge tubes (8).
7. Disposable glass pipettes.

D. CHEMICALS

1. Nanograde methylene chloride--J.T. Baker Company.
2. HPLC-grade acetonitrile--J.T. Baker Company.
3. HPLC-grade water--J.T. Baker Company.
4. Anhydrous sodium sulfate--reagent grade.
5. HPLC-grade methanol.

4. STANDARDS

A. CALIBRATION STANDARDS

Separate calibration stock solutions are prepared for each analyte. A composite working calibration standard is prepared from these solutions.

1. The RDX stock calibration standard (6,310 ug/ml) is prepared by weighing 63.1 mg of RDX in a 10-ml volumetric flask, dissolving the RDX in a few ml of acetonitrile, and diluting to the mark with acetonitrile. An intermediate RDX stock calibration standard is prepared by pipetting 1 ml of the RDX stock calibration standard into a 100-ml volumetric flask and diluting to the mark with methanol to give a solution containing 63.1 ug/ml of RDX.
2. The HMX stock calibration standard (5,320 ug/ml) is prepared by weighing 53.2 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile. An intermediate HMX stock calibration standard is prepared by pipetting 1 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 106.4 ug/ml of HMX.
3. The PETN stock calibration standard (3,950 ug/ml) is prepared by weighing 39.5 mg of PETN in a 10-ml volumetric flask, dissolving the PETN in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and

diluting to the mark with acetonitrile. An intermediate PETN stock calibration standard is prepared by pipetting 1 ml of the PETN stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 79.0 ug/ml of PETN.

4. Prepare a series of composite working calibration standards by making dilutions of the intermediate calibration standards with 50% methanol/50% water as follows:

Working Calibration Standard	Intermediate Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
B	RDX	5	50
	HMX	1	
	PETN	5	
C	RDX	5	100
	HMX	1	
	PETN	5	
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration Standard	Concentration (ug/ml)		
	RDX	HMX	PETN
B	6.31	2.13	7.90
C	3.15	1.06	3.95
D	1.26	0.426	1.58
E	0.631	0.213	0.790
F	0.315	0.106	0.395

B. CONTROL SPIKES

1. The working control spike solutions are prepared in the same manner as the working calibration standards using the same letter designations for the different solutions; therefore,

the Working Control Spike Solution B has the same concentration as the Working Calibration Standard B.

2. Pipette 2 ml of the corresponding working control spike solutions into 500 ml of standard or natural water. The solutions used are selected to provide a concentration range of 0.5 to 10 times the desired detection limit.
3. Determine the precision, accuracy, and detection limits for each analyte.

<u>Working Control Spike Used</u>	<u>Analyte Concentration in the Working Control Spike Solution (ug/ml)</u>	<u>Spiked Analyte Concentration in Water (ug/L)</u>
--	--	0.0
B	RDX	25.2
	HMX	8.5
	PETN	31.6
C	RDX	12.6
	HMX	4.26
	PETN	15.8
D	RDX	5.04
	HMX	1.70
	PETN	6.32
E	RDX	2.52
	HMX	0.851
	PETN	3.16
F	RDX	1.26
	HMX	0.426
	PETN	1.58

5. PROCEDURE

A. EXTRACTION

1. Measure 500 ml of the water sample into a 1-L separatory funnel.
2. Check the pH of the sample with pH paper, and adjust the pH to neutral, if necessary.

3. Extract the sample sequentially with three 100-ml portions of methylene chloride. After each portion has been added, shake the funnel vigorously for at least 5 minutes.
4. Let the layers separate for about 2 minutes after each extraction.
5. Draw off the methylene chloride and pass through a glass funnel filled with a small plug of glass wool and about 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
6. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
7. Add a boiling chip (Hengar) to the methylene chloride extract in the flask and attach a 3-ball Snyder column to the apparatus.
8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
9. The balls of the Snyder column should actively chatter when the solvent is evaporating.
10. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of methylene chloride has drained into the receiver, remove the receiver from the K-D flask.
11. Add approximately 2 ml of HPLC methanol to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
12. Repeat Step 11 two times.
13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC acetonitrile. Raise the extract

volume to 1.0 ml in the centrifuge tube with HPLC methanol.
Dilute to 2 ml with HPLC water.

14. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
15. The extract is now ready for chromatography by HPLC.

B. CALIBRATION

1. Inject Working Calibration Standards G, F, E, D, C, and B and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized integrator areas versus nanograms/microliter of each standard to obtain a working curve.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the components of interest.

6. CALCULATIONS

Determine the concentration of RDX according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve (ug/ml),

V_t = Volume of total extract (ml), and

V_s = Volume of initial sample extracted (L).

7. REFERENCES

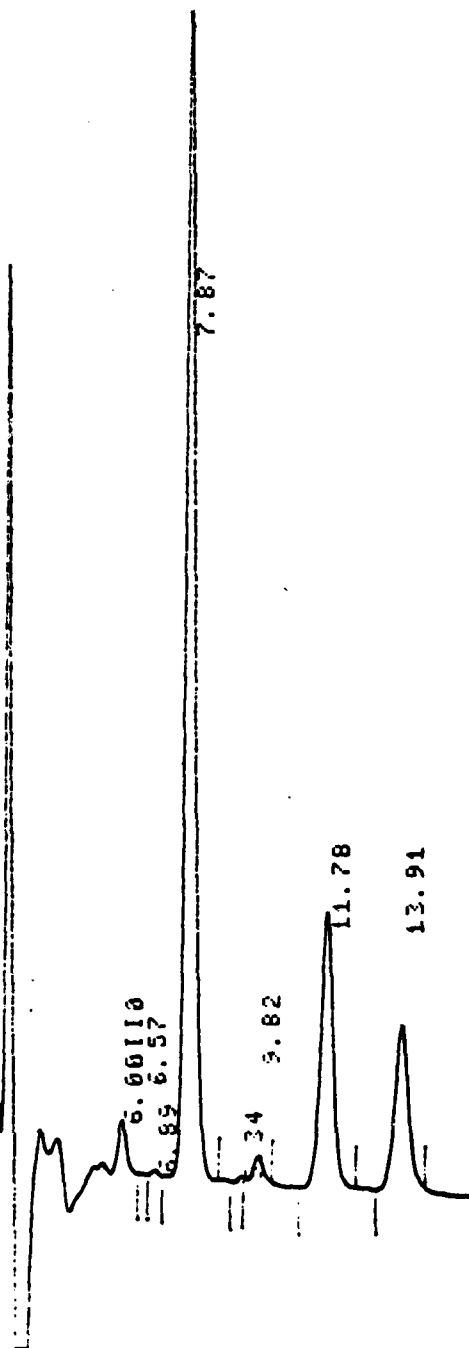
None found.

8. DATA

See attached data sheets.

INJECT TIME 17:22:40

10111.50PM1



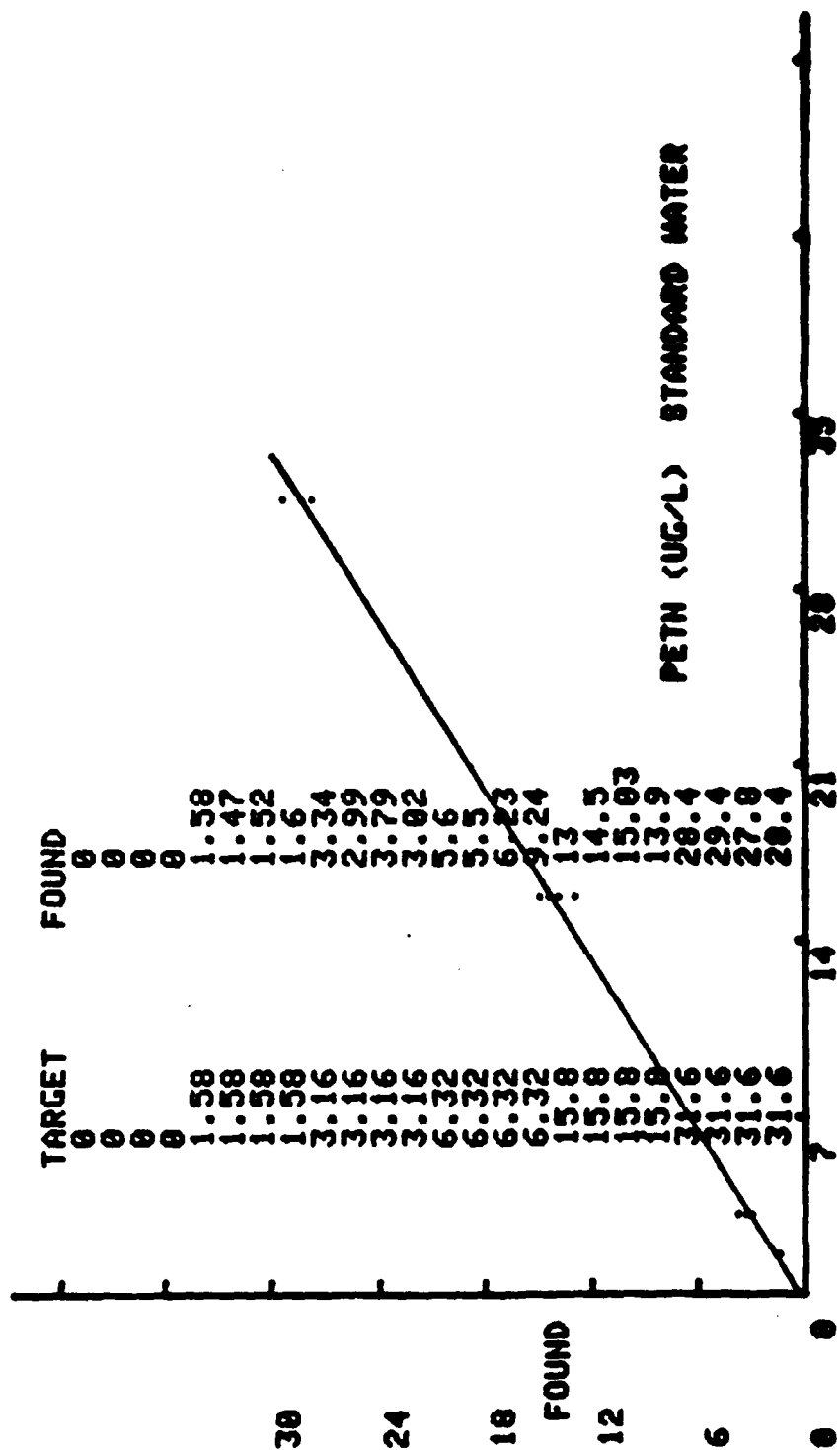
Chromatogram of Standard Water Spiking Experiment

Analyte	Amount Spiked	Retention Time
RDX	12.6 ug/L	7.87 min
HMX	4.3 ug/L	11.78 min
PETN	15.8 ug/L	13.91 min

PETN (UG/L) STANDARD WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.58	1.58	1.47	1.52	1.60
3.16	3.34	2.99	3.79	3.02
6.32	5.60	5.50	6.23	5.24
15.8	13.0	14.5	15.0	13.9
31.6	28.4	29.4	27.8	28.4

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.58	1.54	0.0591	3.83	-2.5734
3.16	3.28	0.372	11.3	3.96
6.32	6.64	1.76	26.5	5.10
15.8	14.1	0.871	6.17	-10.7120
31.6	28.5	0.663	2.33	-5.8101



TARGET

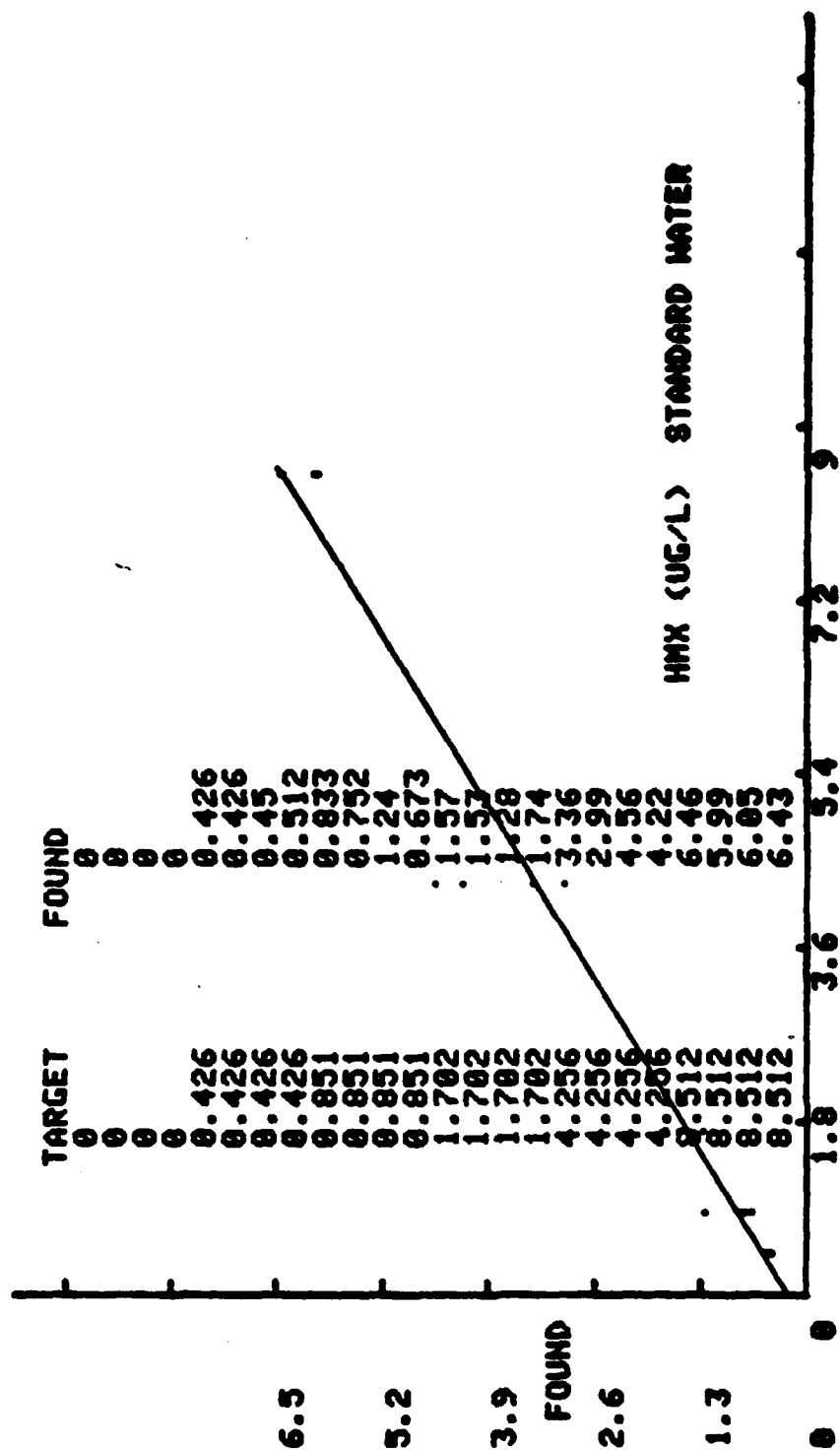
CORR. COEFF. = 0.9963 FOUND = 0.3309 0.09107837 TARGET

DETECTION LIMIT = 3.36307

PNX (UG/L) STANDARD WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.426	0.426	0.426	0.450	0.512
0.851	0.833	0.752	1.24	0.673
1.70	1.57	1.53	1.28	1.74
4.26	3.36	2.99	4.56	4.22
8.51	6.46	5.99	6.05	6.43

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.426	0.453	0.0406	8.95	6.46
0.851	0.874	0.252	28.8	2.76
1.70	1.53	0.190	12.4	-10.1058
4.26	3.78	0.731	19.3	-11.1255
8.51	6.23	0.247	3.96	-26.7796

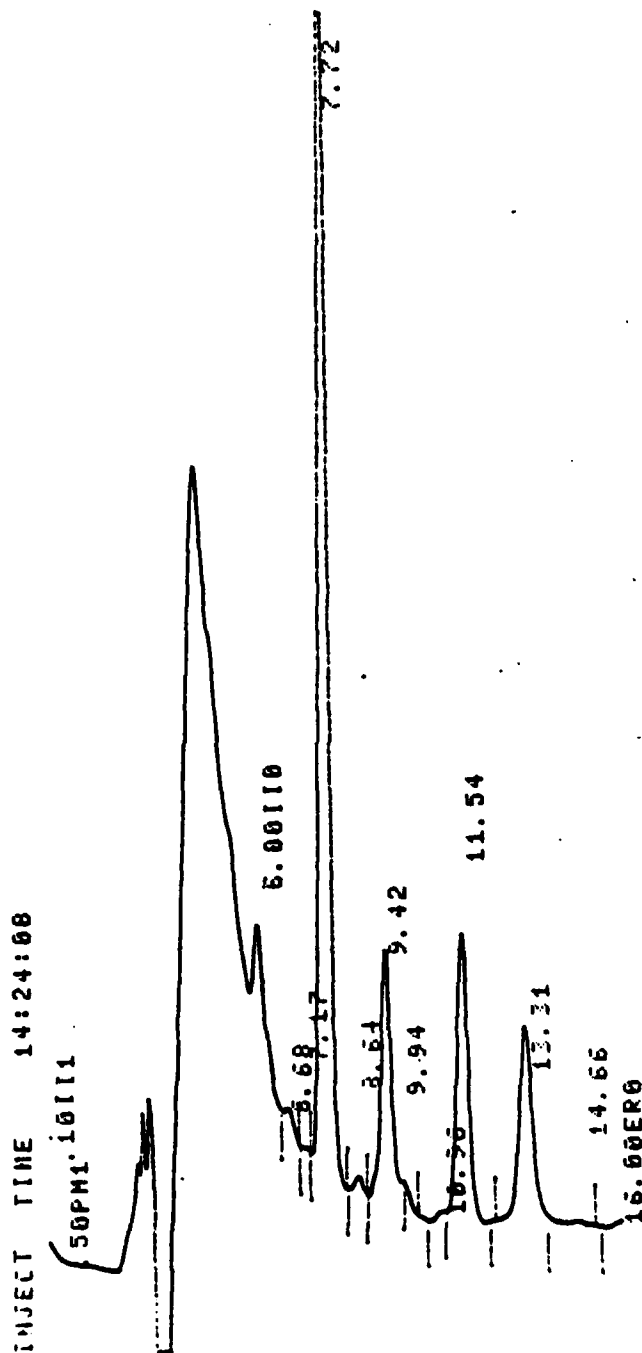


CORR. COEFF. = 0.9050
 DETECTION LIMIT = 1.04791
 TARGET
 FOUND = 0.2205+
 TARGET
 0.73340931

RDX (UG/L) STANDARD WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.351	0.0800	0.370
1.26	1.72	1.48	1.57	2.02
2.52	3.20	2.61	2.62	2.86
5.04	5.66	4.97	5.43	5.08
12.6	12.6	11.7	12.0	11.9
25.2	25.0	24.5	24.0	23.1

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.200	0.188	93.9	0.0000
1.26	1.70	0.237	13.9	34.7
2.52	2.82	0.277	9.81	12.0
5.04	5.28	0.318	6.01	4.86
12.6	12.0	0.393	3.26	-4.4444
25.2	24.1	0.610	3.36	-4.1667



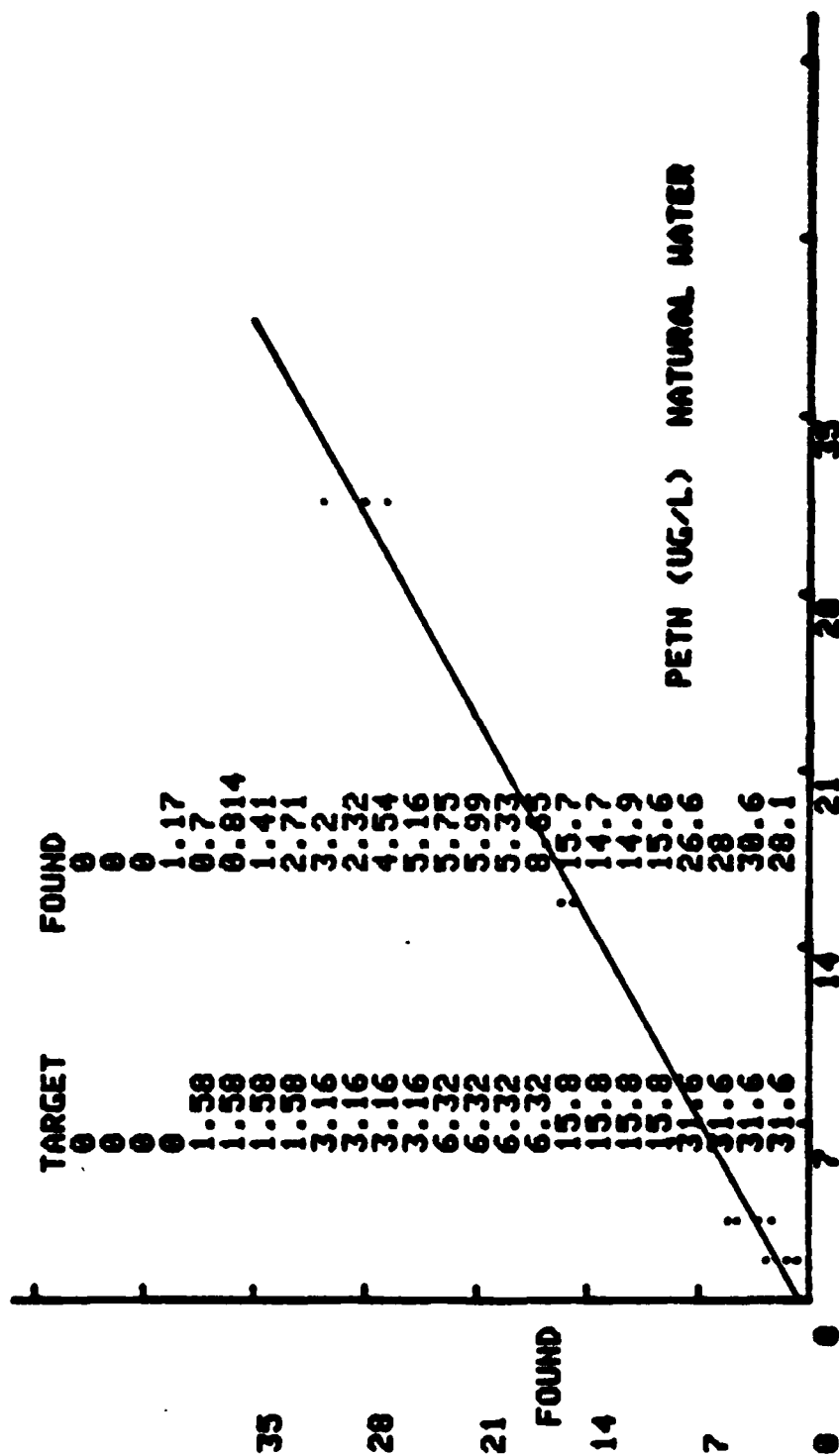
Chromatogram of Natural Water Spiking Experiment

Analyte	Amount Spiked	Retention Time
RDX	12.6 ug/L	7.72 min
HMX	4.3 ug/L	11.54 min
PETN	15.8 ug/L	13.31 min

PETN (UG/L) NATURAL WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	1.17
1.58	0.700	0.814	1.41	2.71
3.16	3.20	2.32	4.54	5.16
6.32	5.75	5.99	5.33	8.65
15.8	15.7	14.7	14.9	15.6
31.6	26.6	28.0	30.6	28.1

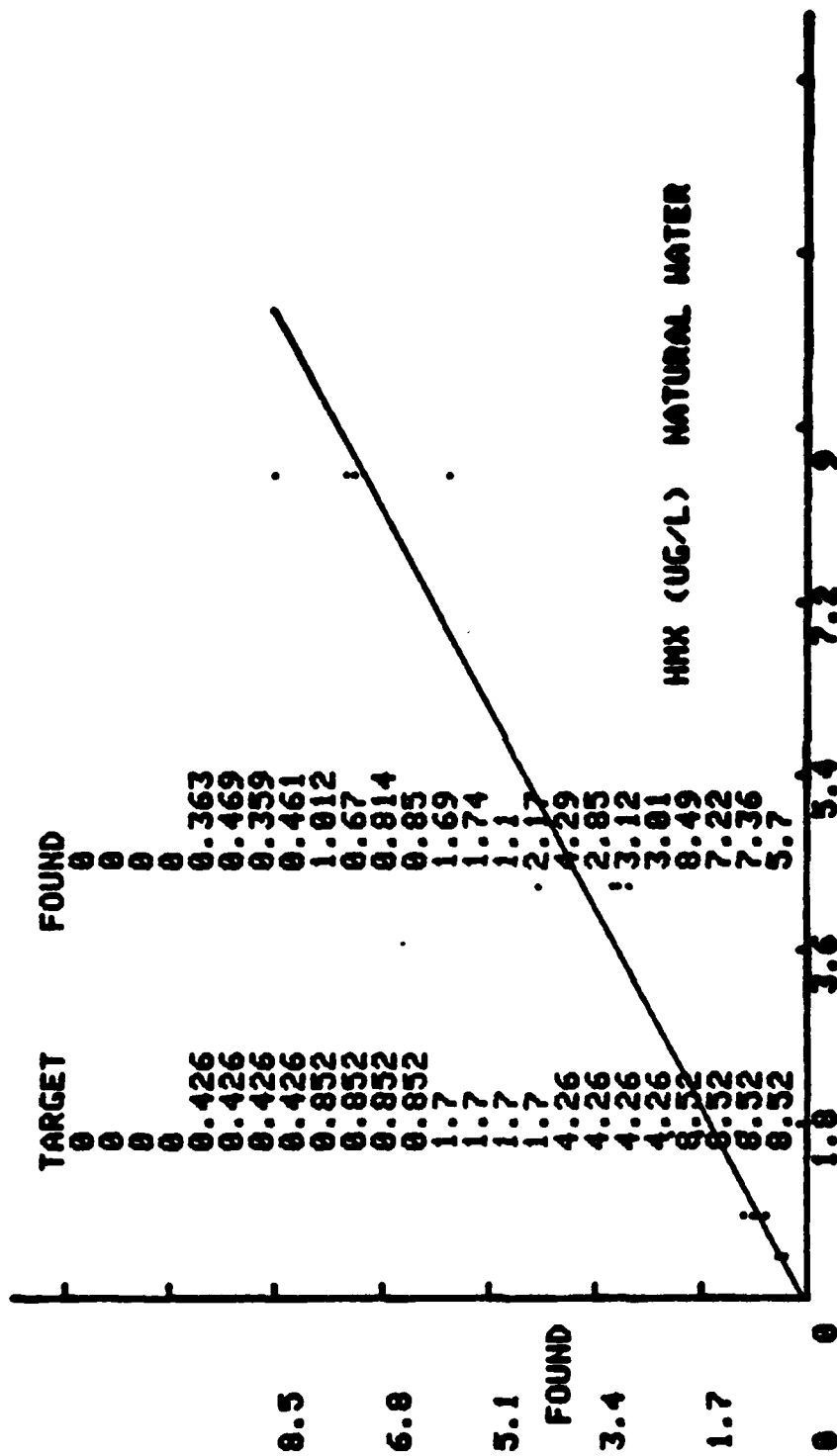
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.292	0.585	200	0.0000
1.58	1.41	0.922	65.4	-10.8544
3.16	3.80	1.28	33.8	20.4
6.32	6.43	1.50	23.4	1.74
15.8	15.2	0.499	3.28	-3.6392
31.6	28.3	1.66	5.87	-10.3639



HNX (UG/L) NATURAL WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.426	0.363	0.469	0.359	0.461
0.852	1.01	0.670	0.814	0.850
1.70	1.69	1.74	1.10	2.17
4.26	4.29	2.85	3.12	3.01
8.52	8.49	7.22	7.36	5.70

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.426	0.413	0.0602	14.6	-3.0517
0.852	0.836	0.140	16.8	-1.8193
1.70	1.67	0.440	26.3	-1.4706
4.26	3.32	0.658	19.8	-22.1244
8.52	7.19	1.15	15.9	-15.5810

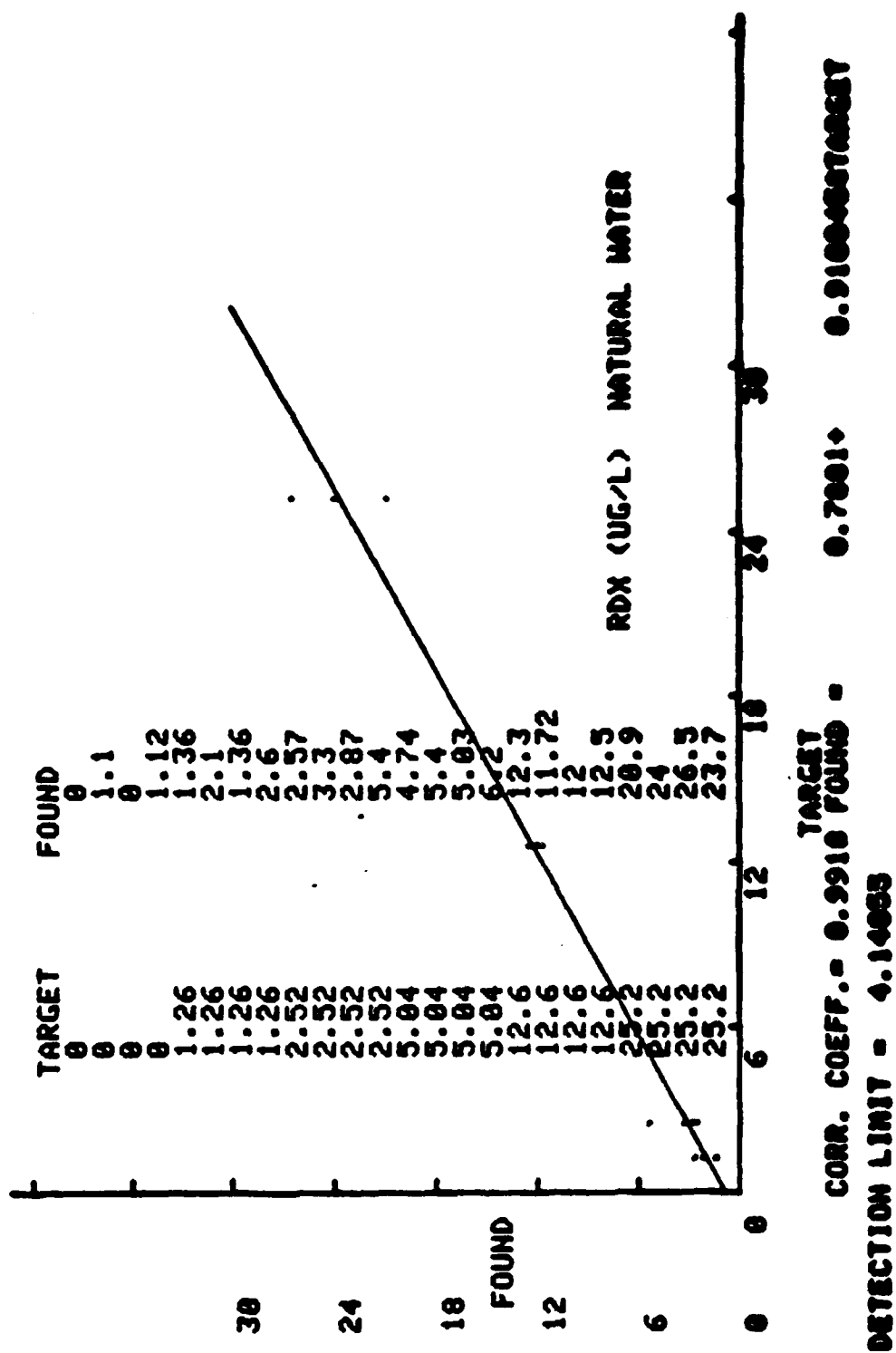


CORR. COEFF. = 0.9706 FOUND - TARGET
 DETECTION LIMIT = 2.20249

POX (UG/L) NATURAL WATER

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	1.10	0.0000	1.12
1.26	1.36	2.10	1.36	2.60
2.52	2.57	3.30	2.87	5.40
5.04	4.74	5.40	5.03	6.20
12.6	12.3	11.7	12.0	12.5
25.2	20.9	24.0	26.5	23.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.555	0.641	115	0.0000
1.26	1.85	0.607	32.7	47.2
2.52	3.53	1.28	36.2	40.3
5.04	5.34	0.632	11.8	6.00
12.6	12.1	0.342	2.62	-3.7302
25.2	23.8	2.29	9.64	-5.6546



1

PETN, HMX, AND RDX IN SOIL SAMPLES

PETN, HMX, AND RDX IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for PETN, HMX, and RDX.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in natural and standard soil are listed below:

<u>Analyte</u>	<u>Range (ug/g)</u>
PETN	0.8 to 16.0
HMX	0.79 to 15.8
RDX	0.96 to 19.2

B. SENSITIVITY

The normalized responses (integrator counts) at the natural soil detection limits designated in Section 1(C) are listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
PETN	53,900	191
HMX	354,000	325
RDX	203,000	224

The normalized responses (integrator counts) at the standard soil detection limits designated in Section 1(C) are listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
PETN	54,600	188
HMX	286,000	261
RDX	128,000	147

C. DETECTION LIMIT

The detection limits in natural soil, calculated according to Hubaux and Vos (1970), are listed below:

<u>Analyte</u>	<u>Detection Limit (ug/g)</u>
PETN	2.3
HMX	4.1
RDX	2.7

The detection limits in standard soil, calculated according to Hubaux and Vos (1970), are listed below:

<u>Analyte</u>	<u>Detection Limit (ug/g)</u>
PETN	2.4
HMX	4.6
RDX	1.9

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 215 nm and are extractable from soil with methylene chloride/acetone. Interferences are minimized by silica-gel cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
PETN	Pentaerythrite tetranitrate	78-11-5
	Pentaerythritol tetranitrate	
	2,2-Bis[(nitrooxy)-methyl]-	
	1,3-Propanediol dinitrate (ester)	

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
	Nitropentaerythritol Pentrit	
HMX	Cyclotetramethylenetetranitramine Octahydro-1,3,5,7-tetrazocine 1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane Octogen	2691-41-0
RDX	Cyclotrimethylenetrinitramine Hexogen, T-4, Cyclonite, Hexahydro-1,3,4-trinitro-s-triazine	121-84-4

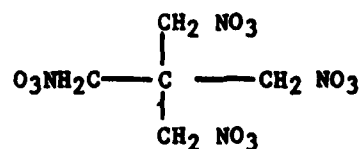
B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point</u>	<u>Density (g/ml)</u>
PETN	$C_5H_8O_{12}N_4$	141	180 at 50 torr	1.77
HMX	$C_4H_8O_8N_8$	276	—	1.77-1.96*
RDX	$C_3H_6O_6N_6$	204.1	—	1.816

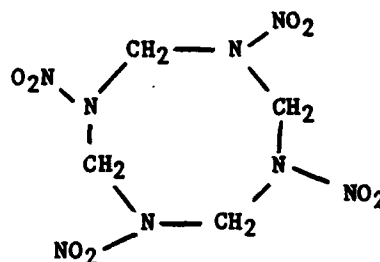
* There are four polymorphic forms of HMX with this range of densities.

Chemical Structures

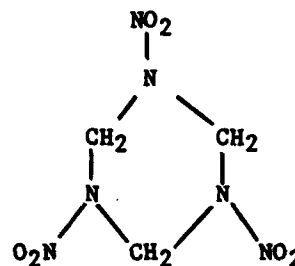
PETN



HMX



RDX



C. CHEMICAL REACTIONS

All of these compounds are highly explosive, and caution should be used in handling. Each compound is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin Elmer LC-75 variable-wavelength detector
($\lambda = 215 \text{ nm}$)
2. Column: Zorbax-CN (4.6-mm ID x 25 cm)
Particle size: 7-8 μm

3. Flow Rate/Mobile Phase: 1 ml/min
35% H₂O/65% methanol

4. Temperature: 22°C

5. Injection Volume: 20 ul, fixed loop

6. Retention Times:

<u>Analyte</u>	<u>Retention Time (Minutes)</u>
RDX	5.4
HMX	6.7
PETN	7.7

C. HARDWARE/GLASSWARE

1. 50-liter centrifuge tubes with Teflon®-lined screw caps (8);
2. 500-ml K-D evaporative flasks (8);
3. 10-ml graduated K-D receivers (8);
4. 3-ball Snyder column (8);
5. 2-ball micro-Snyder column (8);
6. 15-ml graduated centrifuge tubes (8); and
7. 10-ml glass or polyethylene syringes with Luer-lock attachments (10).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Anhydrous sodium sulfate--reagent grade;
5. Nanograde hexane;
6. Nanograde acetone; and
7. Silica-Gel Sep-Paks®--Waters Associates.

4. STANDARDS

A. CALIBRATION STANDARDS

Separate calibration stock solutions are prepared for each analyte. A composite working calibration standard is prepared from these solutions.

1. The RDX stock calibration standard (1.92 mg/ml) is prepared by weighing 47.9 mg of RDX into a 25-ml volumetric flask, dissolving the RDX in a few ml of acetonitrile, and diluting to the mark with acetonitrile.
2. The HMX stock calibration standard (7.91 mg/ml) is prepared by weighing 79.1 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile. An intermediate HMX stock calibration standard is prepared by pipetting 5 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 791 ug/ml of HMX.
3. The PETN stock calibration standard (8.0 mg/ml) is prepared by placing the entire SARM solution (200 mg PETN) in a 25-ml volumetric flask and diluting to the mark with acetonitrile. An intermediate PETN stock calibration standard is prepared by pipetting 5 ml of the PETN stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 800 ug/ml of PETN.
4. Prepare a series of composite working calibration standards by making dilutions of the intermediate calibration standards for PETN and HMX and the stock calibration standard for RDX. Dilute with 50% methanol/50% water as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
B	RDx (stock)	1	25
	HMX	2	
	PETN	2	
C	RDx (stock)	1	50
	HMX	2	
	PETN	2	
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration Standard	Concentration (ug/ml)		
	RDx	HMX	PETN
B	76.8	63.3	64
C	38.4	31.6	32
D	15.3	12.7	12.8
E	7.7	6.3	6.4
F	3.8	3.2	3.2

B. CONTROL SPIKES

1. Prepare Control Spike Solution A for RDx by diluting 5 ml of the calibration standard stock (concentration 1.92 mg/ml) to 50 ml with acetone.
2. Prepare Control Spike Solution B for RDx by diluting 5 ml of the stock control spike solution to 50 ml with acetone.
3. Prepare Control Spike Solution C for HMX and PETN by combining 1 ml each of the calibration standard stock for HMX and the calibration standard stock for PETN in a 25-ml volumetric flask and diluting to volume with acetone.
4. Prepare Control Spike Solution D for HMX and PETN by diluting 5 ml of Control Spike Solution C to 50 ml with acetone.

<u>Control Spike Solution</u>	<u>Concentration (ug/ml)</u>
A (RDX)	192
B (RDX)	19.2
C (HMX, PETN)	316, 320
D (HMX, PETN)	31.6, 32

<u>Control Spike Solution</u>	<u>Standard Diluted</u>	<u>Dilution (ml)</u>	<u>Final Volume (ml)</u>
A (RDX)	Stock Calibration	5	50
B (RDX)	Control Spike A	5	50
C (HMX, PETN)	Stock Calibration	1, 1	25
D (HMX, PETN)	Control Spike C	5	50

5. Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)
6. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon®-lined screw cap.
7. Pipette a known amount of the control spike solutions for RDX, HMX, and PETN onto the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>RDX Control Spike Solution</u>	<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Soil (ug/g)</u>
		<u>RDX</u>
B	1.0	0.96
B	2.0	1.9
B	4.0	3.8
A	1.0	9.6
A	2.0	19

HMX/PETN Control Spike Solution	Spike Volume (ml)	Concentration of Spiked Soil (ug/g)	
		HMX	PETN
D	0.5	0.79	0.80
D	1.0	1.6	1.6
D	2.0	3.2	3.2
C	0.5	7.9	8.0
C	1.0	16	16

8. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

1. Add 35 ml of 20% acetone in methylene chloride to the centrifuge tube.
2. Cap the tube and shake for 3 to 5 minutes.
3. Extract the sample sequentially with three 35-ml portions of the methylene chloride/acetone mixture.
4. Decant off the methylene chloride/acetone mixture each time and pass through a glass funnel filled with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
5. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.

6. Add a boiling chip (Teflon®) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
7. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
8. The balls of the Snyder column should actively chatter when the solvent is evaporating.
9. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of solvent has drained back into the receiver, remove the receiver from the K-D flask.
10. Add approximately 2 ml of nanograde hexane to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
11. Repeat Step 10 twice.
12. Detach the micro-Snyder column from the receiver. With a dispo pipette, transfer the extract into a 10-ml glass syringe fitted with a silica-gel Sep-Pak®. Rinse the receiver three times with 2 ml of 20% methylene chloride in hexane solution, transferring each rinse to the 10-ml syringe. Set aside the receiver for later use.
13. Pass the combined rinses through the silica-gel Sep-Pak® at a rate of approximately 1 to 2 ml/min, discarding the eluate.
14. Quantitatively rinse the K-D receiver from Step 12 three times with a total of 1 to 2 ml of 50% methanol in methylene chloride solution, transferring each rinse to the 10-ml syringe fitted with the silica-gel Sep-Pak®. Add 50% methanol in methylene chloride to the syringe to make a total volume of 10 ml.

15. Elute the silica-gel Sep-Pak®, with the 10-ml total volume of 50% methanol in methylene chloride, into another 10-ml K-D receiver at a rate of 1 to 2 ml/min.
16. Add a Teflon® boiling chip to eluate, attach a 2-ball micro-Snyder column, and concentrate the sample on a water bath heated to 80°C. When the apparent volume of the solution is about 0.5 ml, remove the apparatus from the water bath.
17. Detach the micro-Snyder, and add approximately 2 ml of HPLC methanol to the receiver. Reconcentrate the sample to 0.5 ml.
18. Repeat Step 17 twice.
19. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC methanol. Raise the extract volume to exactly 2.5 ml in the centrifuge tube with HPLC methanol. Dilute to 5 ml with HPLC water.
20. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
21. The extract is now ready for chromatography by HPLC.

C. ANALYSIS

1. Inject 20 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.

- B. Determine the concentration of RDX, HMX, and PETN according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

W_s = Weight of initial sample extracted (g).

7. REFERENCES

None found.

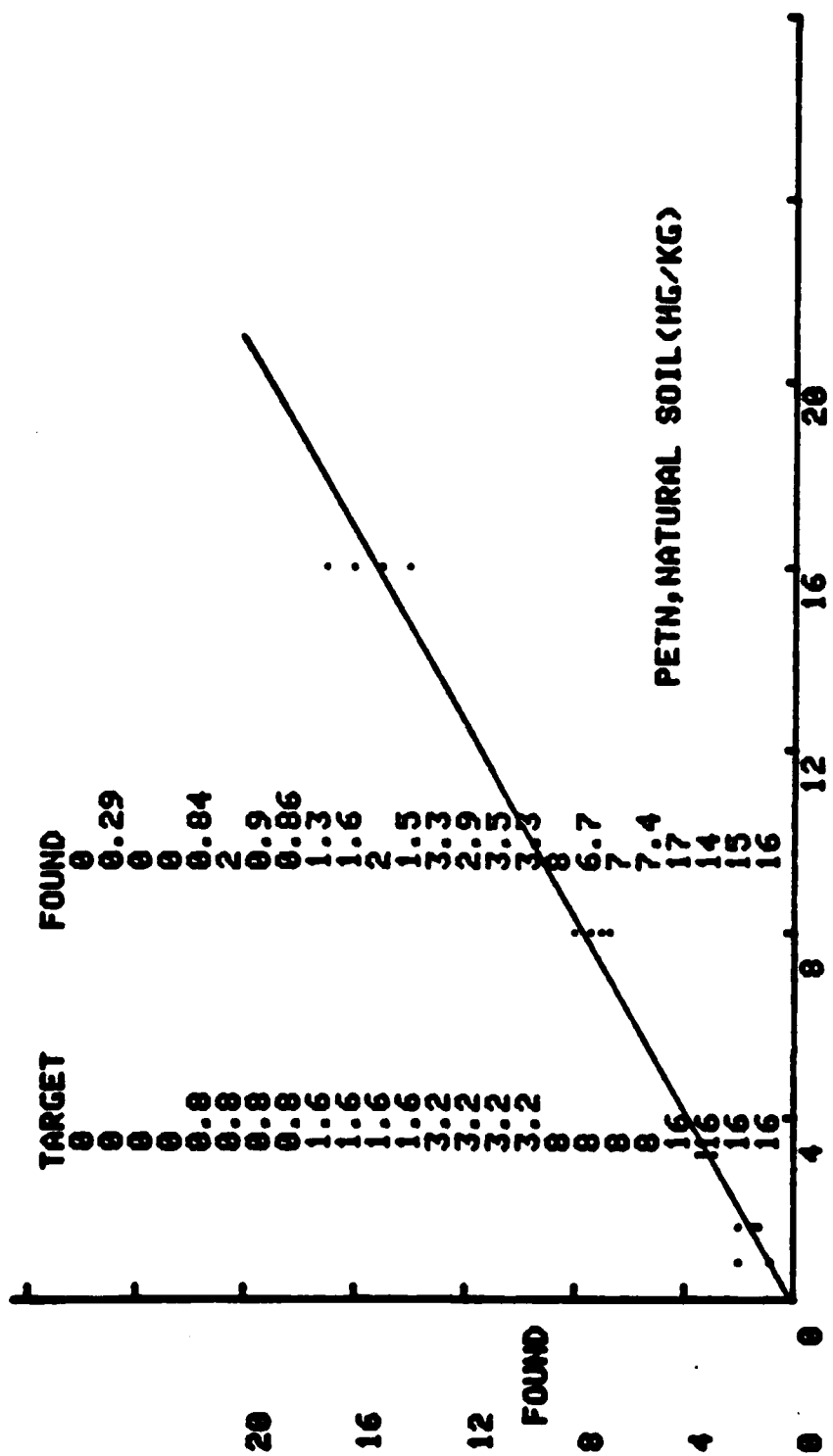
8. DATA

See attached data sheets.

PETN, NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.290	0.0000	0.0000
0.800	0.840	2.00	0.900	0.860
1.60	1.30	1.60	2.00	1.50
3.20	3.30	2.90	3.50	3.30
8.00	8.00	6.70	7.00	7.40
16.0	17.0	14.0	15.0	16.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0725	0.145	200	0.0000
0.800	1.15	0.567	49.3	43.7
1.60	1.60	0.294	18.4	0.0000
3.20	3.25	0.252	7.74	1.56
8.00	7.27	0.562	7.73	-9.0625
16.0	15.5	1.29	8.33	-3.1250

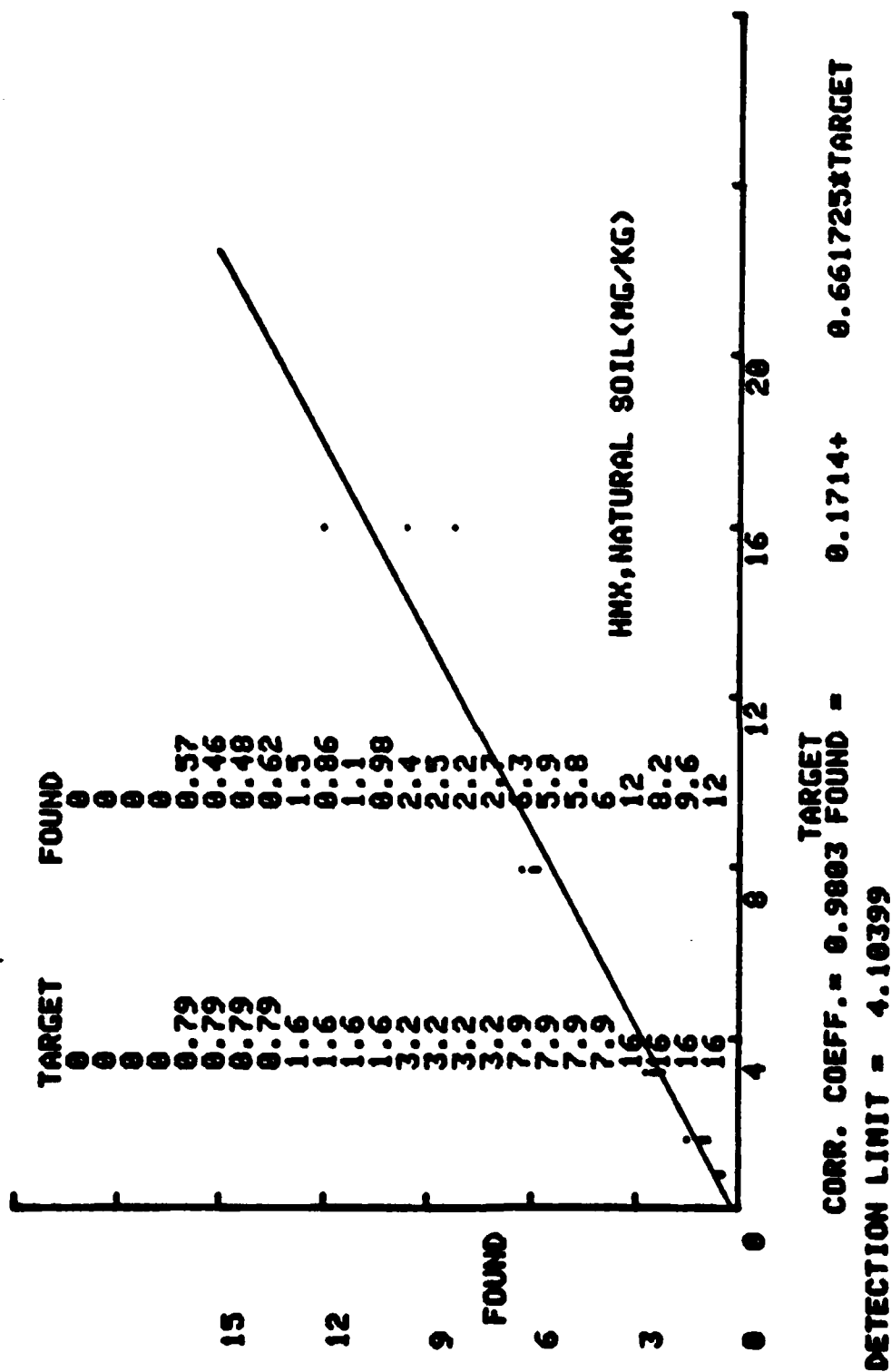


CORR. COEFF. = 0.9936
 DETECTION LIMIT = 2.31215
 TARGET
 FOUND = 0.1285+ 0.948522xTARGET

HMX, NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.790	0.570	0.460	0.480	0.620
1.60	1.50	0.860	1.10	0.980
3.20	2.40	2.50	2.20	2.70
7.90	6.30	5.90	5.80	6.00
16.0	12.0	8.20	9.60	12.0

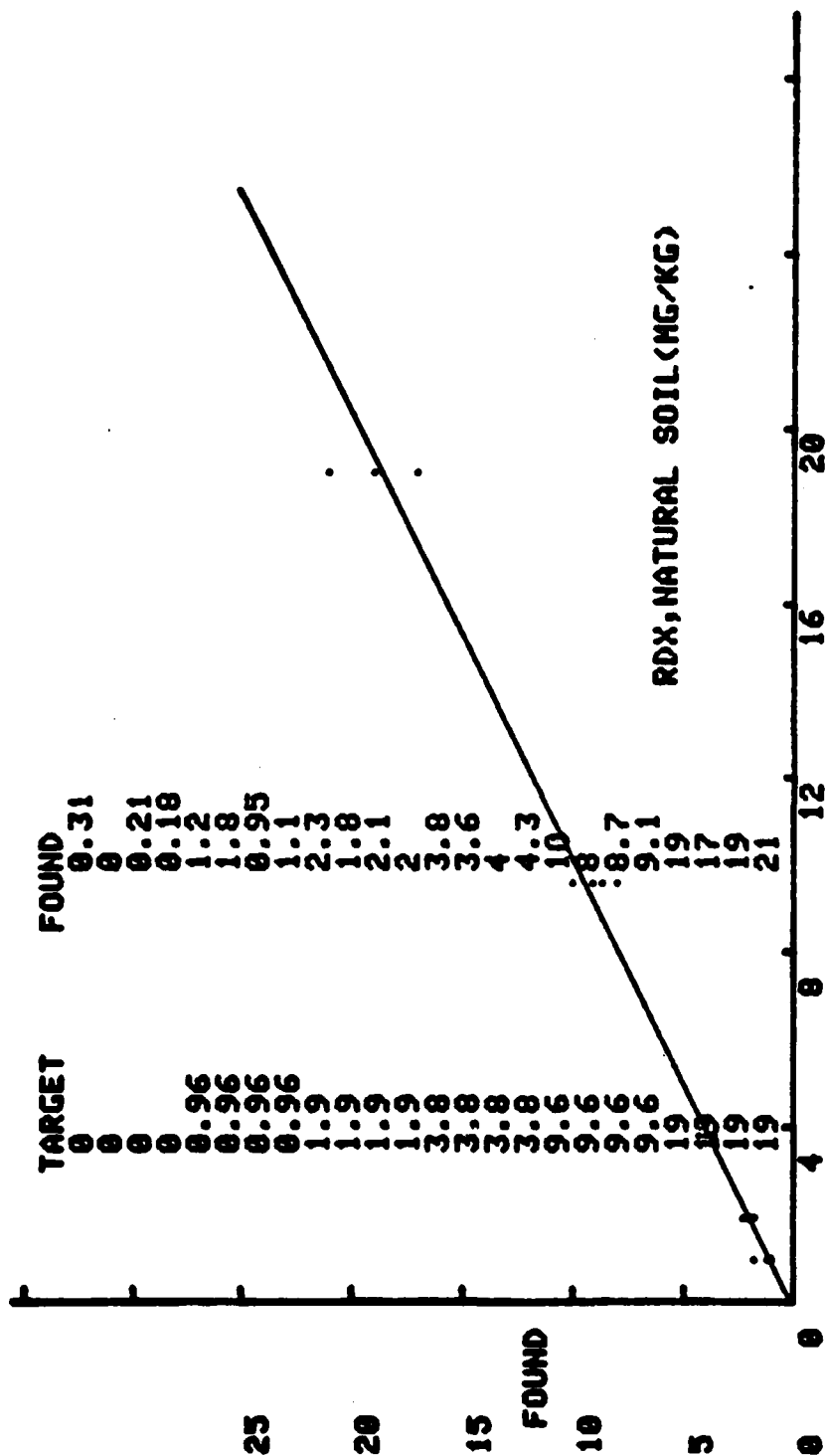
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.790	0.532	0.0754	14.2	-32.5949
1.60	1.11	0.278	25.0	-30.6250
3.20	2.45	0.208	8.50	-23.4375
7.90	6.00	0.216	3.60	-24.0506
16.0	10.4	1.88	18.0	-34.6875



RDX, NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.310	0.0000	0.210	0.180
0.960	1.20	1.80	0.950	1.10
1.90	2.30	1.80	2.10	2.00
3.80	3.80	3.60	4.00	4.30
9.60	10.00	8.00	8.70	9.10
19.0	19.0	17.0	19.0	21.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.175	0.129	73.8	0.0000
0.960	1.26	0.373	29.5	31.5
1.90	2.05	0.208	10.2	7.89
3.80	3.92	0.299	7.61	3.29
9.60	8.95	0.835	9.33	-6.7708
19.0	19.0	1.63	8.59	-0.0000

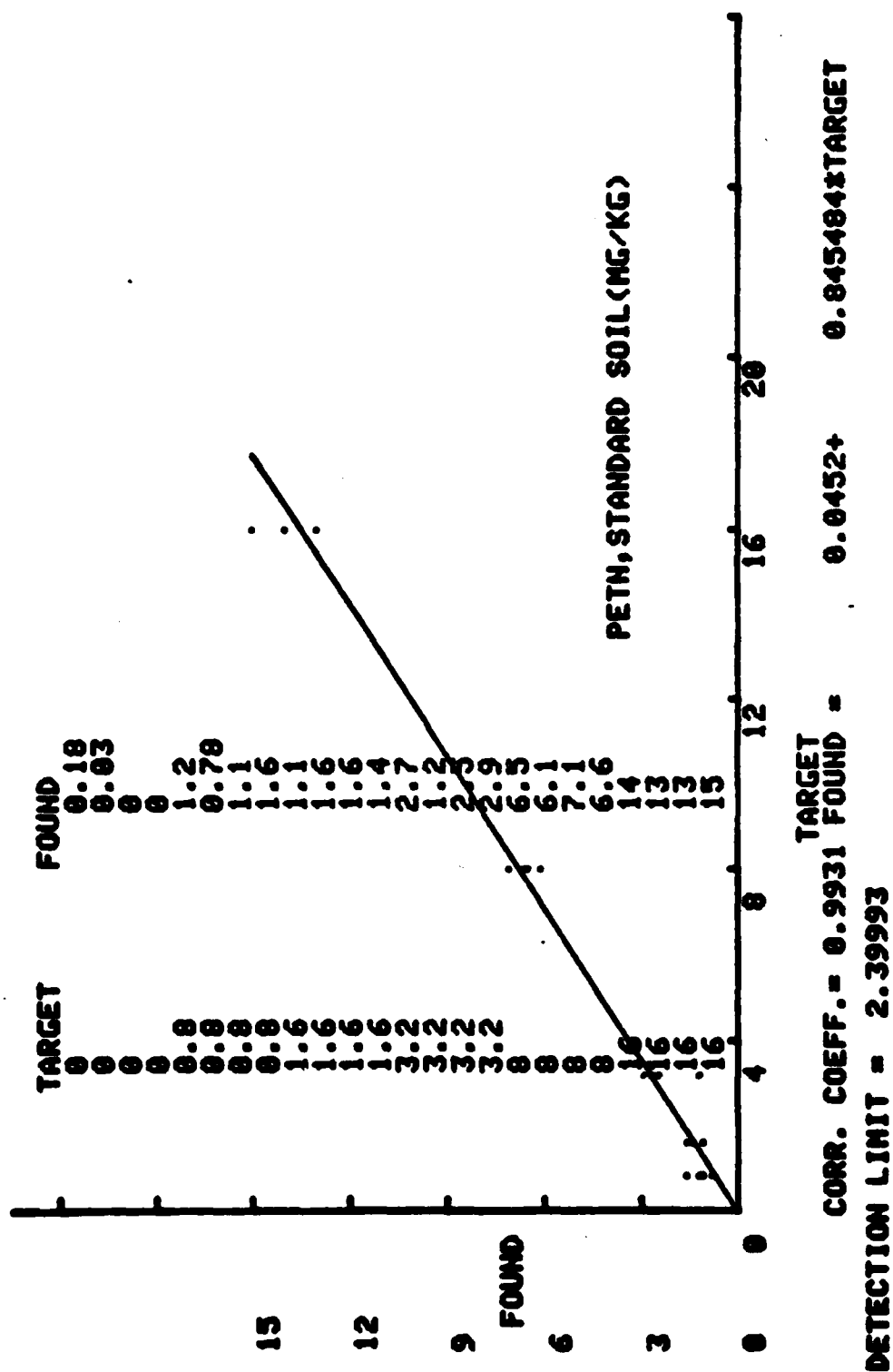


CORR. COEFF. = 0.9938
 DETECTION LIMIT = 2.71923
 TARGET FOUND = 0.1456+
 0.978137*TARGET

PETN, STANDARD SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.180	0.0300	0.0000	0.0000
0.800	1.20	0.780	1.10	1.60
1.60	1.10	1.60	1.60	1.40
3.20	2.70	1.20	2.50	2.90
8.00	6.50	6.10	7.10	6.60
16.0	14.0	13.0	13.0	15.0

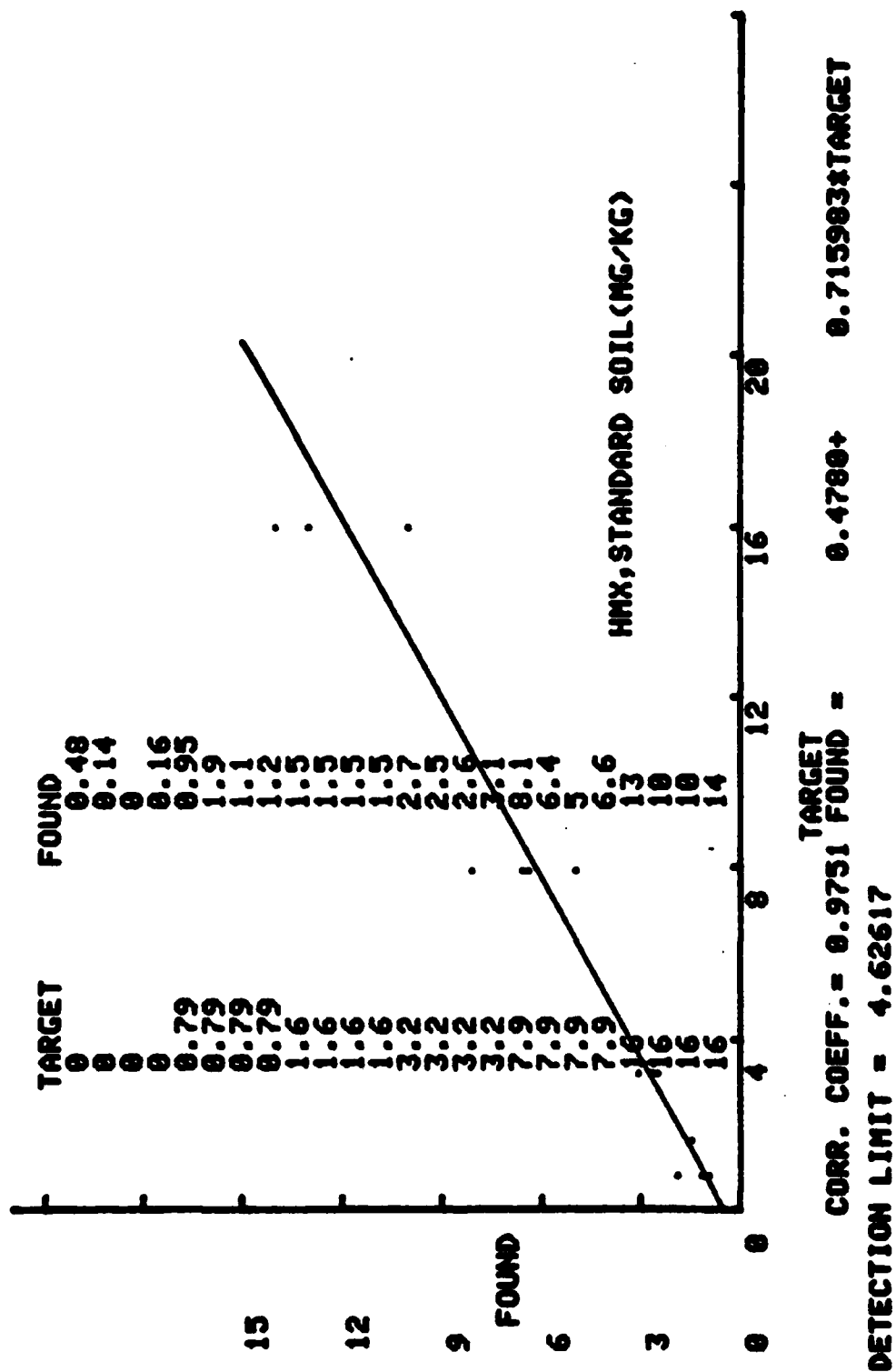
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0525	0.0862	164	0.0000
0.800	1.17	0.338	28.9	46.2
1.60	1.42	0.236	16.6	-10.9375
3.20	2.32	0.768	33.0	-27.3438
8.00	6.57	0.411	6.26	-17.8125
16.0	13.8	0.957	6.96	-14.0625



HMx, STANDARD SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.480	0.140	0.0000	0.160
0.790	0.950	1.90	1.10	1.20
1.60	1.50	1.50	1.50	1.50
3.20	2.70	2.50	2.60	3.10
7.90	8.10	6.40	5.00	6.60
16.0	13.0	10.00	10.00	14.0

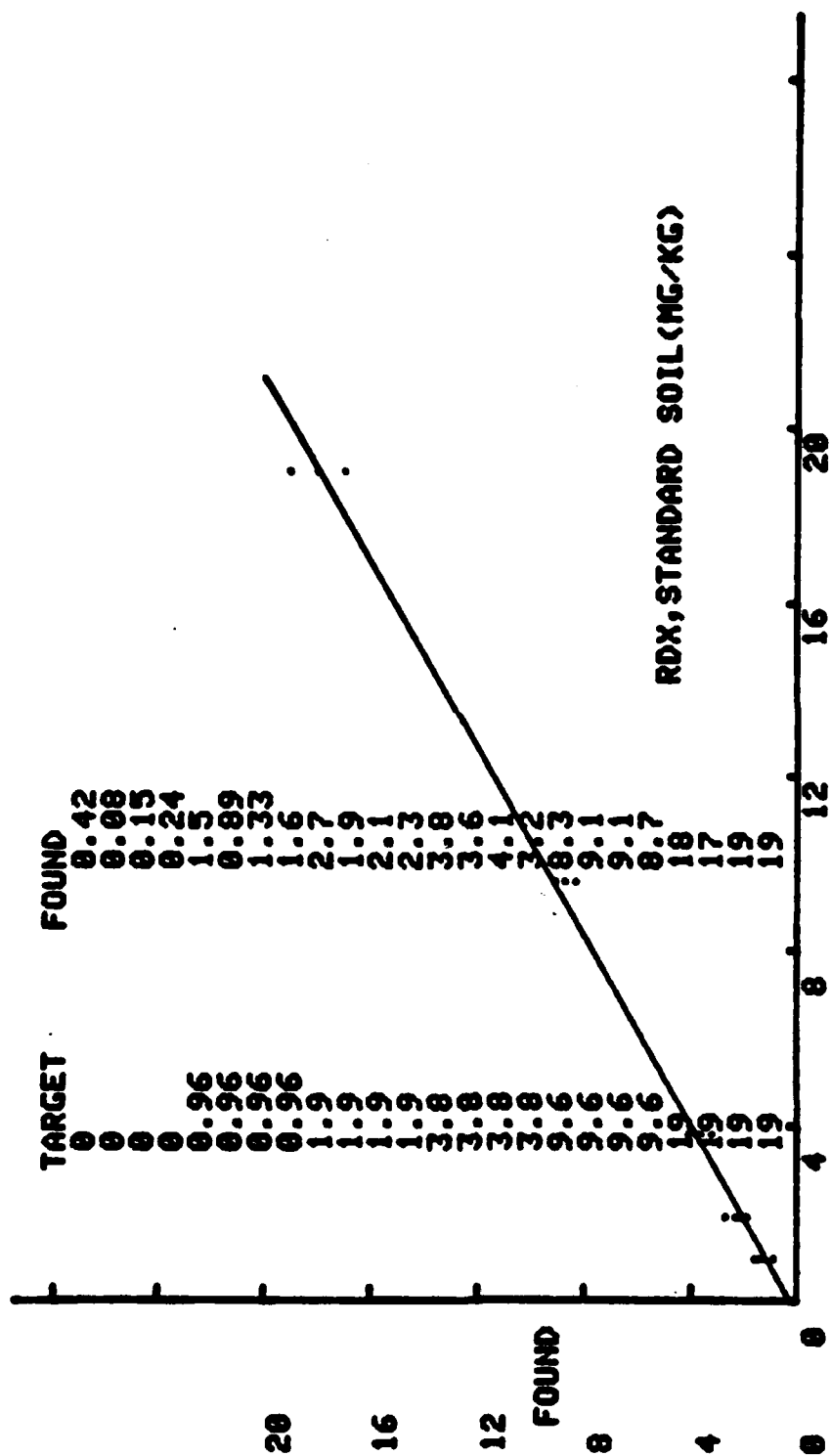
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.195	0.203	104	0.0000
0.790	1.29	0.421	32.7	63.0
1.60	1.50	0.0000	0.0000	-6.2500
3.20	2.72	0.263	9.65	-14.8438
7.90	6.52	1.27	19.4	-17.4051
16.0	11.8	2.06	17.5	-26.5625



RDX, STANDARD SOIL

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.420	0.0800	0.150	0.240
0.960	1.50	0.890	1.33	1.60
1.90	2.70	1.90	2.10	2.30
3.80	3.80	3.60	4.10	3.20
9.60	8.30	9.10	9.10	8.70
19.0	18.0	17.0	19.0	19.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.222	0.147	66.1	0.0000
0.960	1.33	0.314	23.6	38.5
1.90	2.25	0.342	15.2	18.4
3.80	3.67	0.377	10.3	-3.2895
9.60	8.80	0.383	4.35	-8.3333
19.0	18.3	0.957	5.25	-3.9474



CORR. COEFF. = 0.9970
 DETECTION LIMIT = 1.89484
 TARGET FOUND = 0.2568+
 TARGET = 0.935527

HMX IN WATER SAMPLES

HMX IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative determination of HMX in environmental water samples.

A. TESTED CONCENTRATION RANGE

The tested concentration range in standard and natural water is 0.63 to 12.7 ug/L.

B. SENSITIVITY

The normalized response (integrator counts corrected for attenuation) at the natural water detection limit is 55,000 area counts corresponding to a quantity of 61.3 ng, and 165,000 area counts for 185 ng at the standard water detection limit.

C. DETECTION LIMIT

The detection limits in standard and natural water samples calculated by the Hubaux and Vos procedure are 3.0 ug/L and 0.98 ug/L, respectively.

D. INTERFERENCES

No interferences were encountered in samples of natural water. However, this method may be subject to interferences from neutral, methylene chloride-extractable species which absorb light at 230 nm.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze six extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

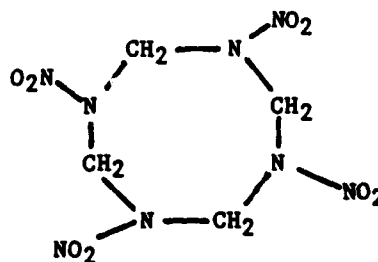
<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
HMX	Cyclotetramethylenetetranitramine Octahydro-1,3,5,7-tetrazocine 1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane Octogen	2691-41-0

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point</u>	<u>Density (g/ml)</u>
HMX	$C_4H_8O_8N_8$	276	—	1.77-1.96*

* There are four polymorphic forms of HMX with this range of densities.

Chemical Structure



C. CHEMICAL REACTIONS

HMX is highly explosive, and caution should be used in handling.
HMX is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 230 nm)
2. Column: Ultrasphere-CN (4.6-mm ID x 25 cm)
Particle size: 5 μ m
3. Flow Rate/Mobile Phase: 1 ml/min
35% water/65% methanol
4. Temperature: 22°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 6.9 minutes

C. HARDWARE/GLASSWARE

1. 1-liter separatory funnels (Teflon® or glass) (8).
2. 500-ml K-D flasks (8).
3. 15-ml K-D receivers (8).
4. 3-ball Snyder columns (8).
5. 2-ball micro-Snyder columns (8).
6. 10-ml graduated centrifuge tubes (8).
7. Disposable glass pipette.

D. CHEMICALS

1. Nanograde methylene chloride--J.T. Baker Company.
2. HPLC-grade acetonitrile--J.T. Baker Company.
3. HPLC-grade water--J.T. Baker Company.
4. Anhydrous sodium sulfate--reagent grade.
5. HPLC-grade methanol.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The HMX stock calibration standard (7.9 mg/ml) is prepared by weighing 79.1 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few milliliters of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile.
2. An intermediate stock calibration standard is prepared by pipetting 1 ml of the HMX stock calibration standard into a 25-ml volumetric flask and diluting to the mark with methanol to give a solution containing 316.4 ug/ml of HMX.
3. Prepare the working calibration standards by making dilutions of the intermediate stock calibration standard and Working Calibration Standard A with 50% methanol/50% water as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Intermediate Stock	1	100
B	Intermediate Stock	0.5	100
C	Working Standard A	5	25
D	Working Standard A	5	50
E	Working Standard A	5	100

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	3.16
B	1.58
C	0.63
D	0.32
E	0.16

B. CONTROL SPIKES

1. Prepare the stock control spiking solution (316.4 ug/ml) by diluting 1 ml of the stock calibration standard

(concentration: 7.9 mg/ml) to volume with acetonitrile in a 25-ml volumetric flask.

2. Prepare the working control spike solutions as follows:

<u>Working Control Spike Solution</u>	<u>Solution Used</u>	<u>Volume (ml)</u>	<u>Final Volume (ml)</u>
A	Stock control spike standard	1	50
B	Working Control Spike Standard A	5	50

3. Pipet a known amount of the working control spike solutions into standard water. The quantity spiked should be selected to provide a concentration of 0.5 to 10 times the detection limit.
4. Determine the accuracy and detection limit for the analyte in standard water by pipetting the working control spike solutions into 500 ml of standard water and analyzing according to the procedure outlined in Section 5.

<u>Working Control Spike Solution</u>	<u>Volume Spiked (ml)</u>	<u>Concentration (ug/L)</u>
--	--	0
B	0.5	0.63
B	1.0	1.27
B	2.0	2.53
A	0.5	6.33
A	1.0	12.7

5. PROCEDURE

A. EXTRACTION

1. Measure 500 ml of the water sample into a 1-L separatory funnel.
2. Check the pH of the sample with pH paper, and adjust the pH to neutral, if necessary.

3. Extract the sample sequentially with three 100-ml portions of methylene chloride. After each portion has been added, shake the funnel vigorously for at least 5 minutes.
4. Let the layers separate for about 2 minutes after each extraction.
5. Draw off the methylene chloride and pass through a glass funnel containing a small plug of glass wool and about 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
6. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
7. Add a boiling chip (Hengar) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
9. The balls of the Snyder column should chatter actively when the solvent is evaporating.
10. When the apparent volume of the solution remaining in the receiver is approximately 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of methylene chloride has drained into the receiver, remove the receiver from the K-D flask.
11. Add approximately 2 ml of HPLC-grade methanol to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
12. Repeat Step 11 two times.
13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade acetonitrile. Raise the extract volume to 1.0 ml in the centrifuge tube with HPLC-grade methanol. Dilute to 2 ml with HPLC-grade water.

14. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
15. The extract is now ready for HPLC analysis.

B. CALIBRATION

1. Inject Working Calibration Standards E, D, C, B, and A and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard C at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized integrator areas versus nanograms injected of each standard to obtain a working curve.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the HMX peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of HMX according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

V_s = Volume of initial sample extracted (L).

7. REFERENCES

None found.

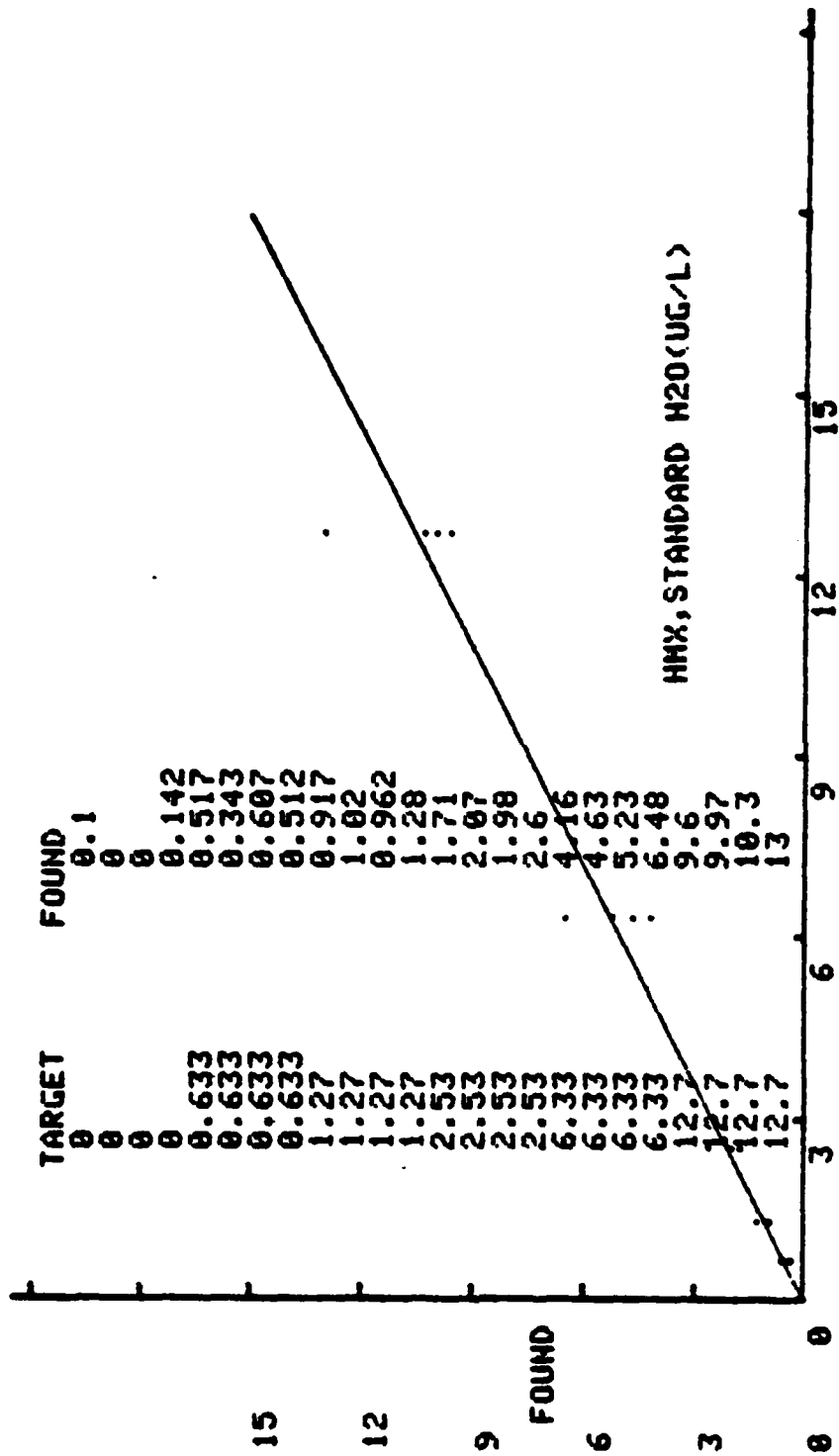
8. DATA

See attached data sheets.

HNX, STANDARD H2O (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.1000	0.0000	0.0000	0.142
0.633	0.517	0.343	0.607	0.512
1.27	0.917	1.02	0.962	1.28
2.53	1.71	2.07	1.98	2.60
6.33	4.16	4.63	5.23	6.48
12.7	9.60	9.97	10.3	13.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0005	0.0719	119	0.0000
0.633	0.495	0.110	22.3	-21.8404
1.27	1.04	0.162	15.5	-17.7362
2.53	2.09	0.373	17.8	-17.3913
6.33	5.12	1.00	19.6	-19.0363
12.7	10.7	1.55	14.4	-15.6102

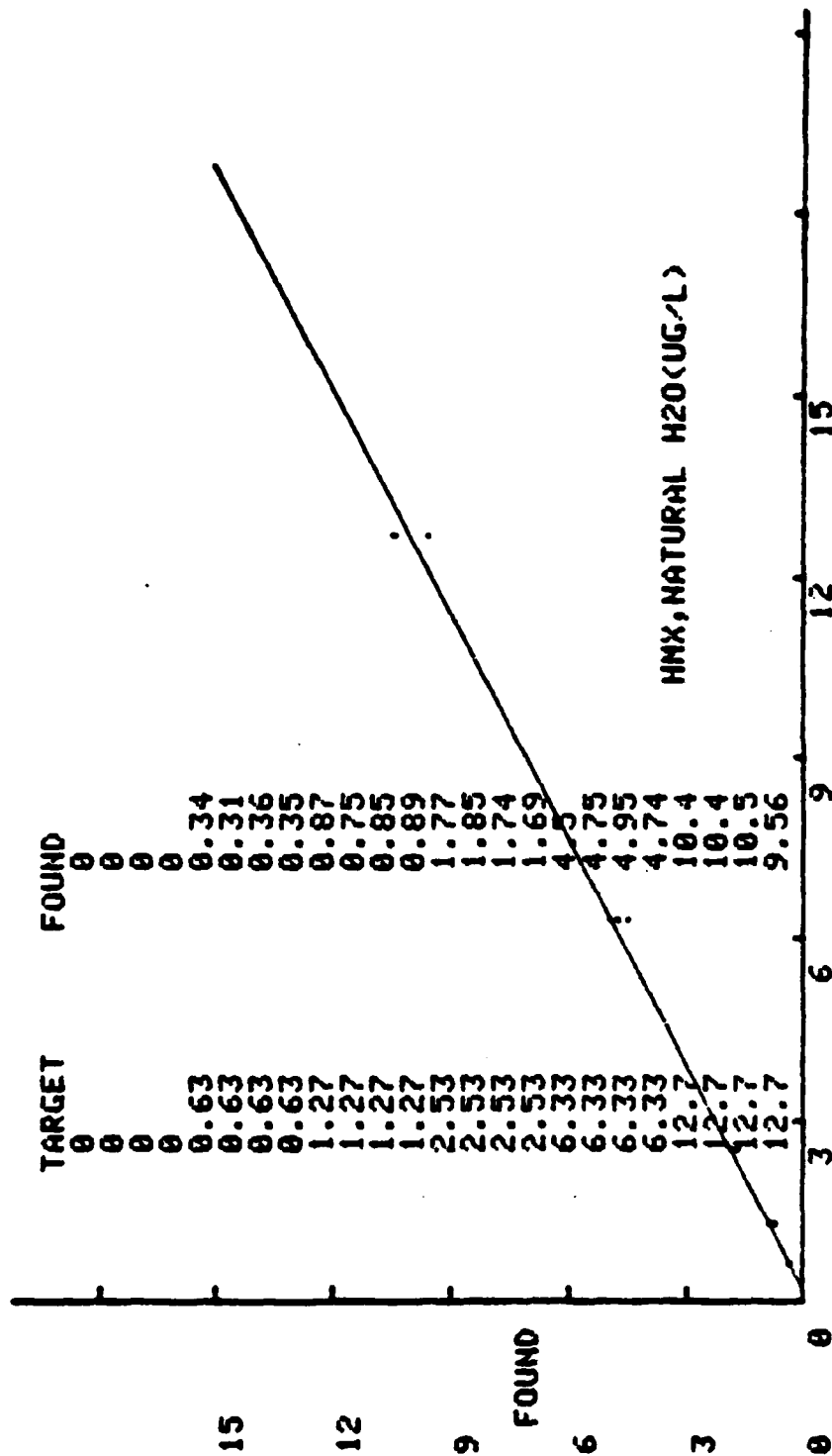


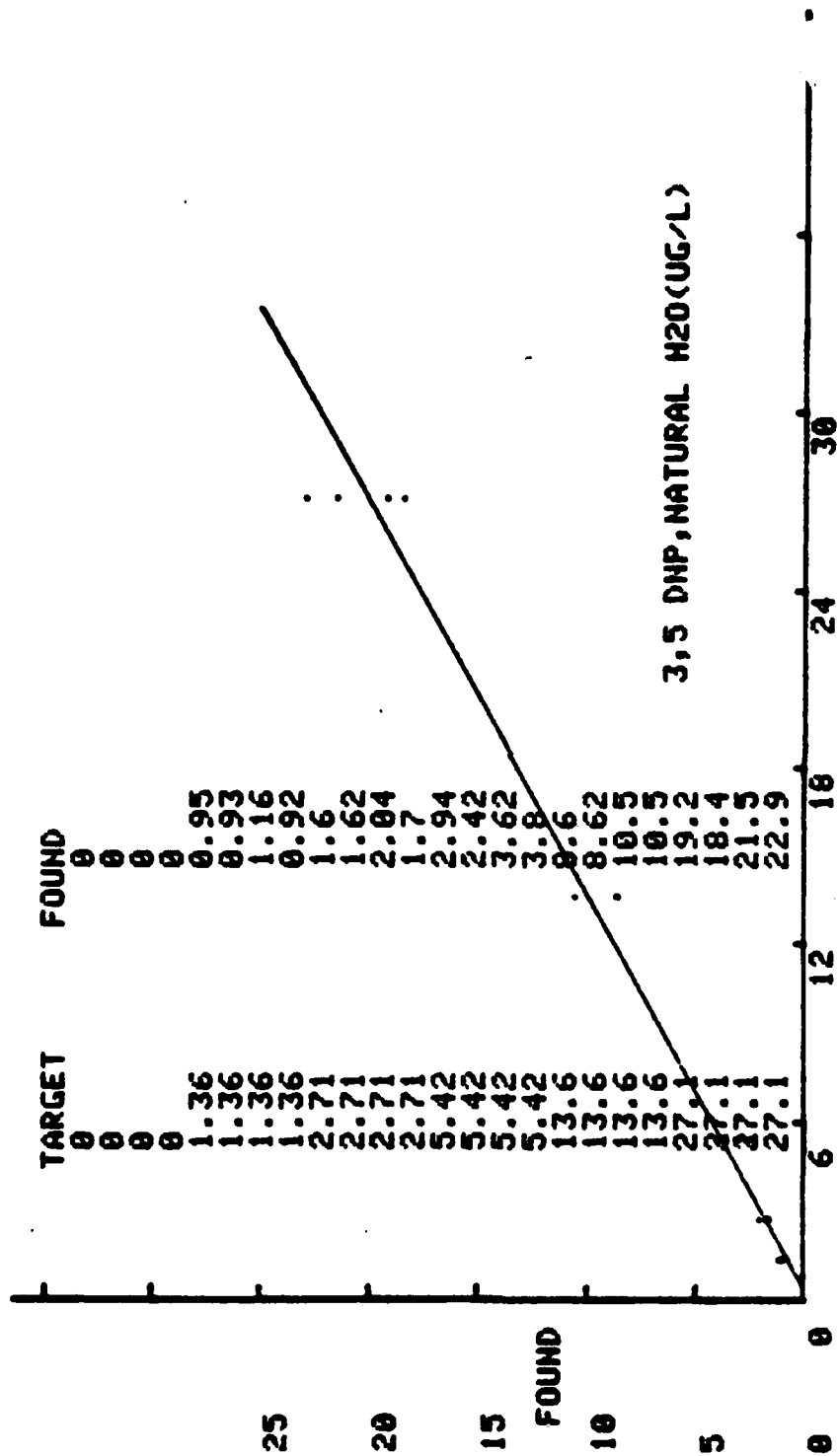
CORR. COEFF. = 0.9840
 DETECTION LIMIT = 2.92953
 TARGET
 FOUND = -0.0281 + 0.839673 * TARGET

HMX, NATURAL H₂O (UG/L)

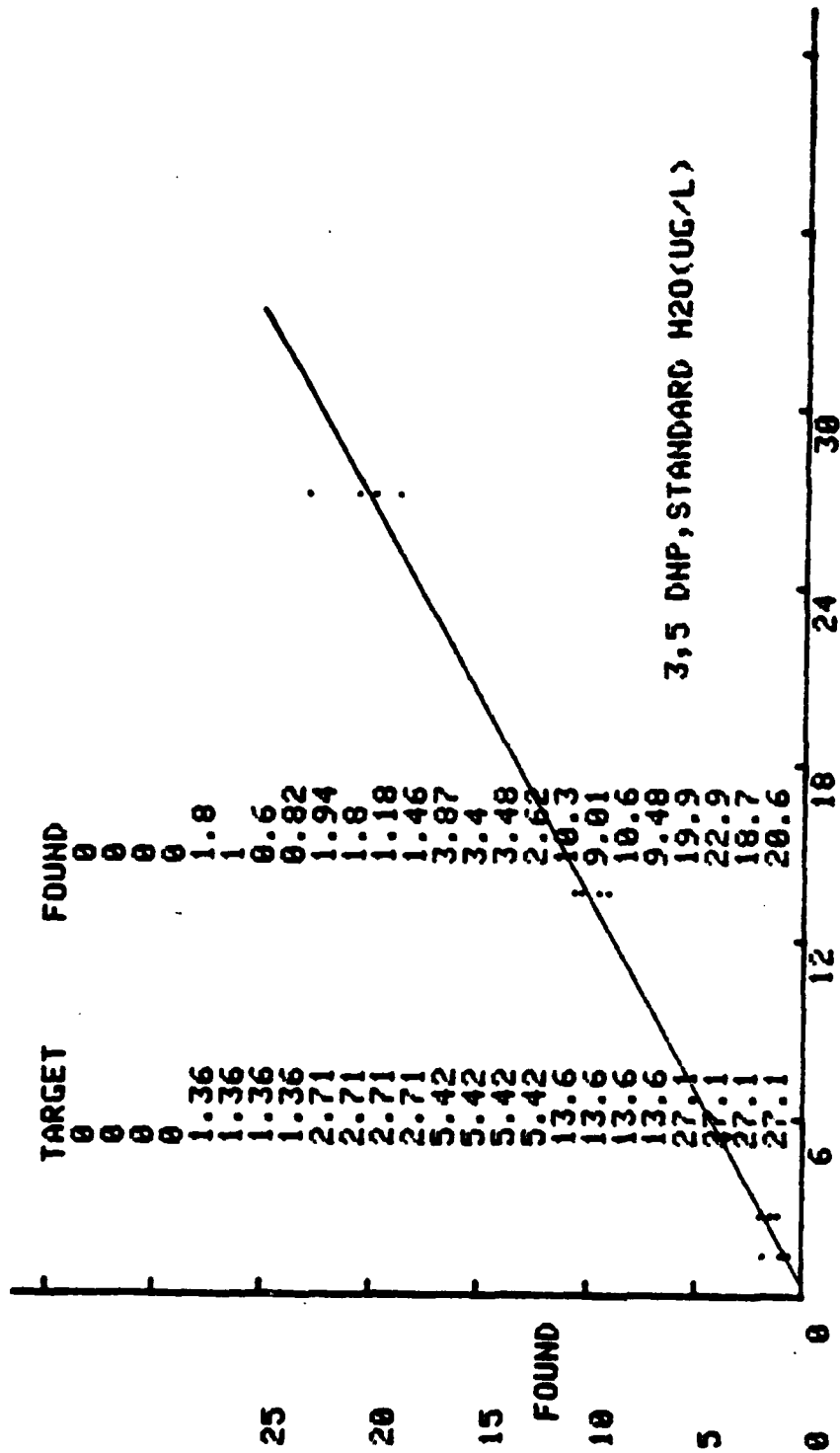
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.630	0.340	0.310	0.360	0.350
1.27	0.870	0.750	0.850	0.890
2.53	1.77	1.85	1.74	1.69
6.33	4.50	4.75	4.95	4.74
12.7	10.4	10.4	10.5	9.56

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.630	0.340	0.0216	6.35	-46.0318
1.27	0.840	0.0622	7.40	-33.8583
2.53	1.76	0.0670	3.80	-30.3360
6.33	4.73	0.184	3.89	-25.1975
12.7	10.2	0.439	4.30	-19.5669

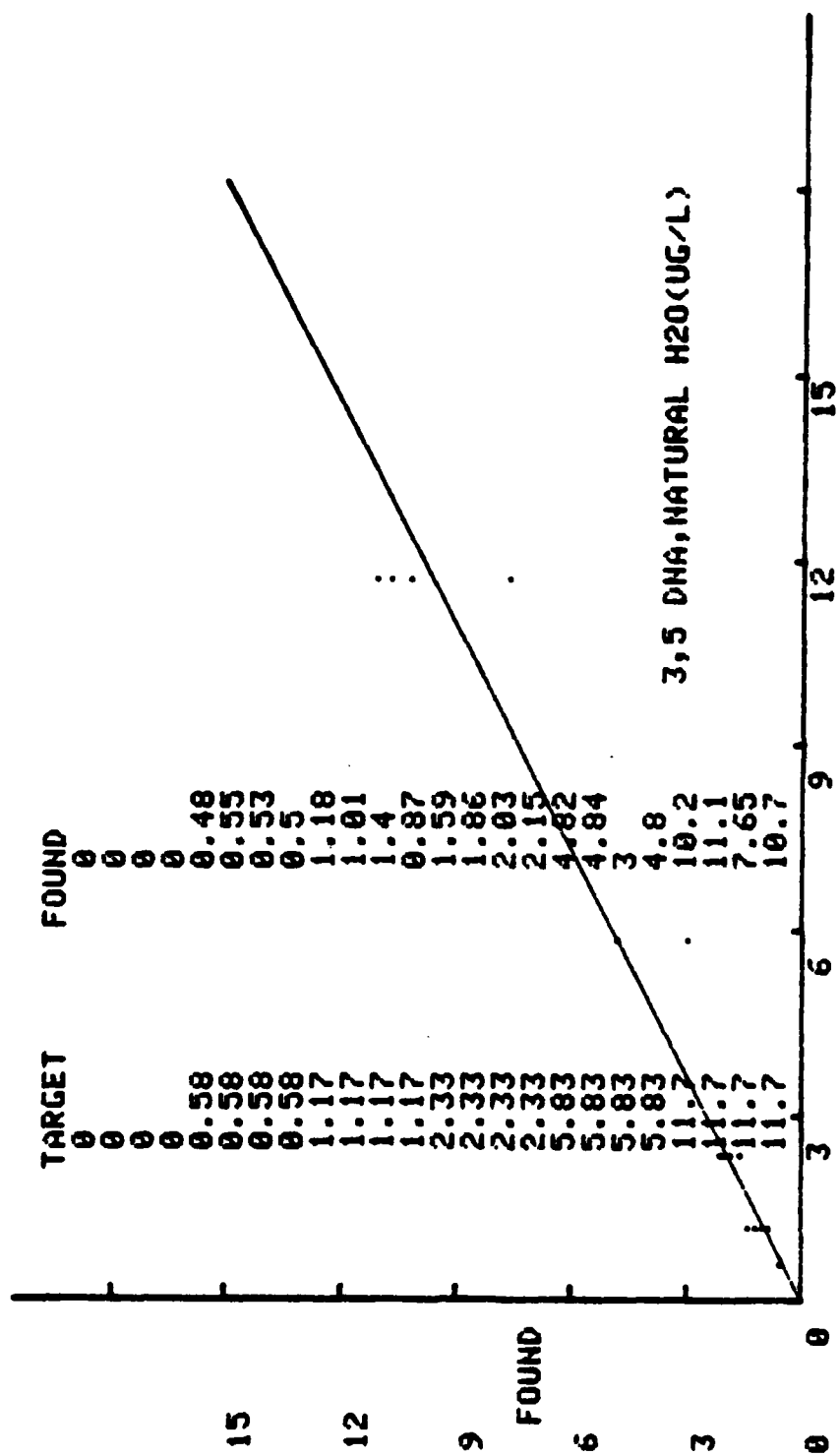




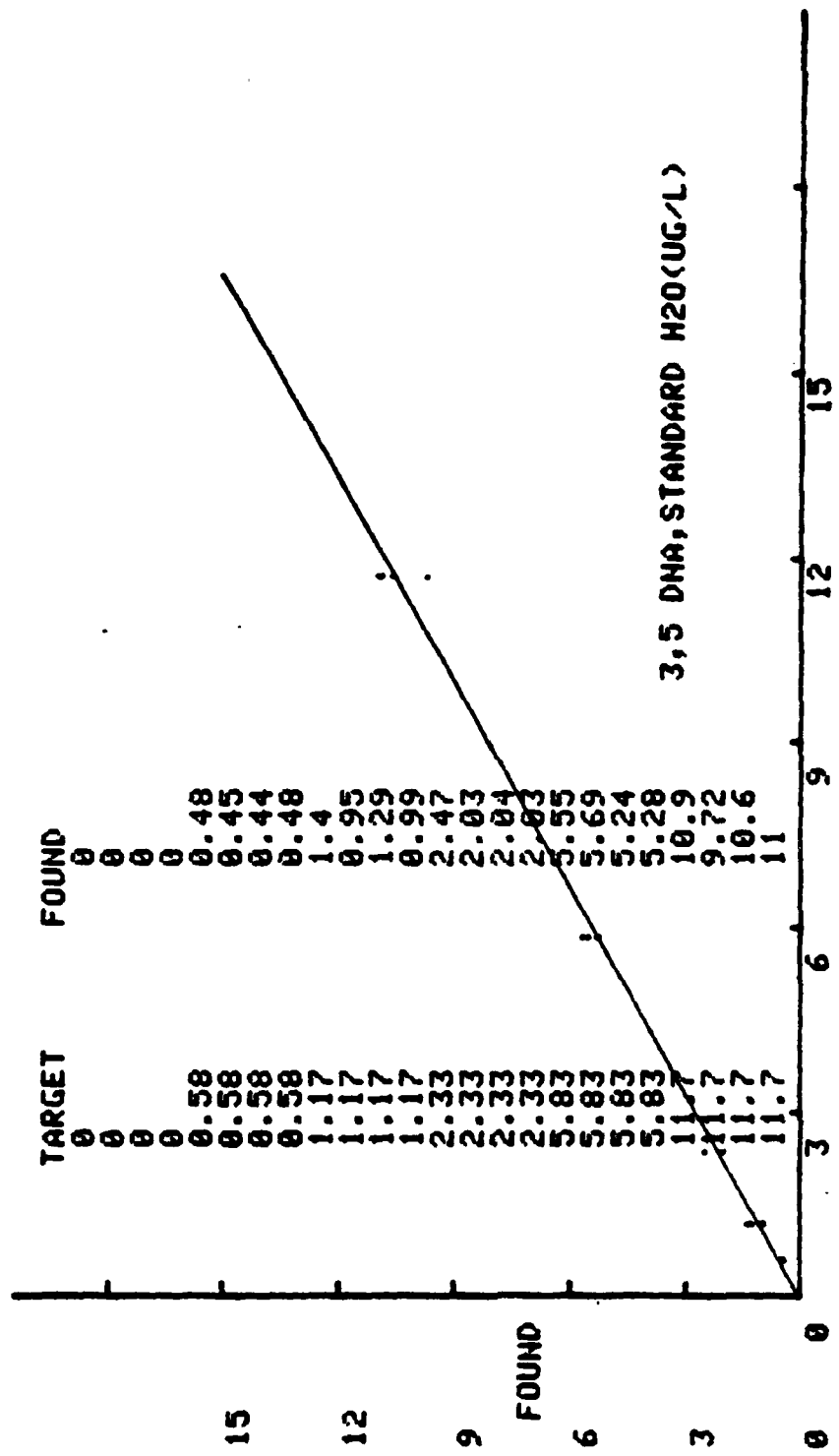
CORR. COEFF. = 0.9916
 DETECTION LIMIT = 4.49756
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 FOUND = -0.3444+
 TARGET
 0.758044xTARGET



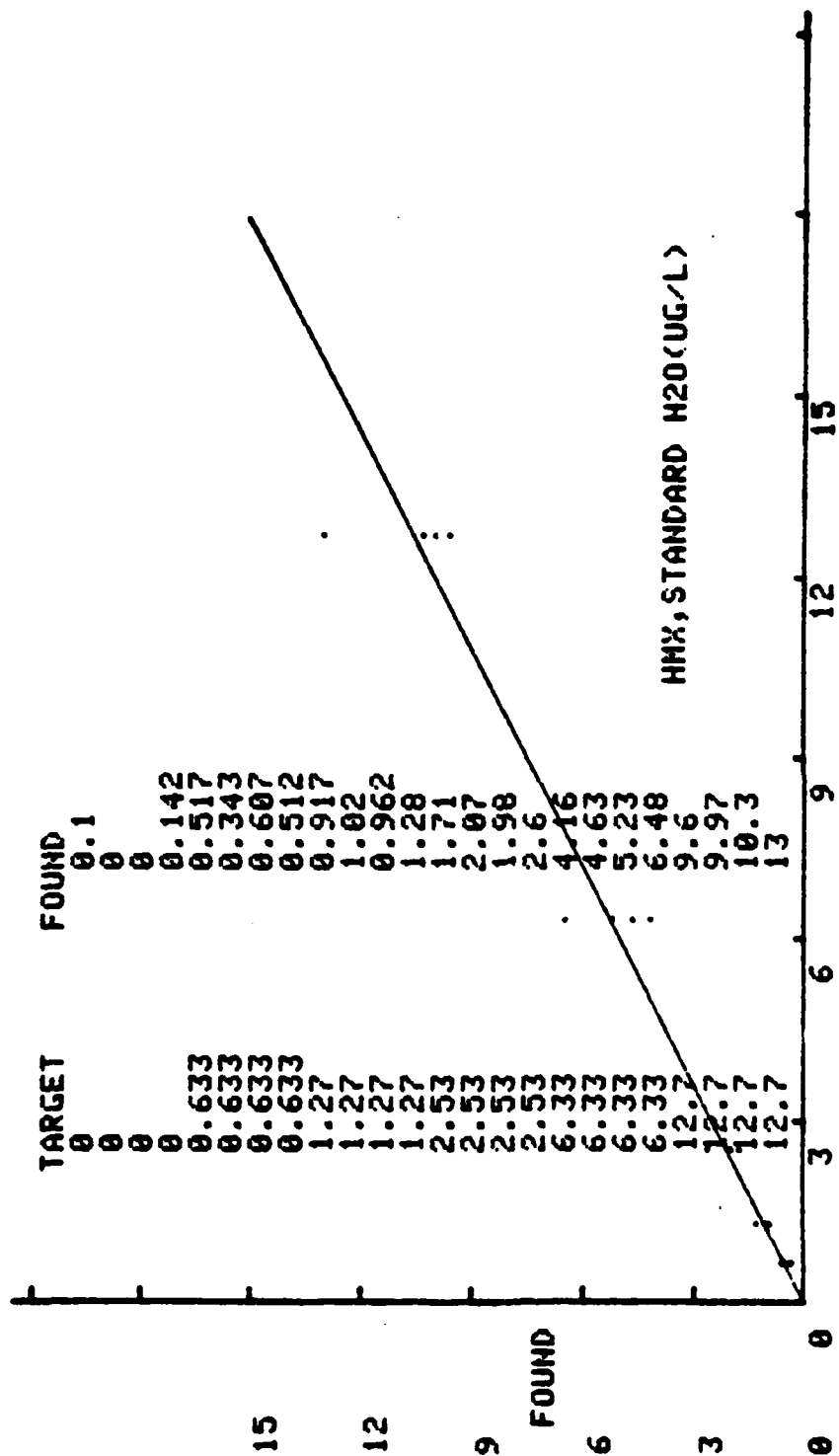
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 DETECTION LIMIT = 3.79772
 TARGET
 FOUND = -0.3102+ 0.761630 TARGET



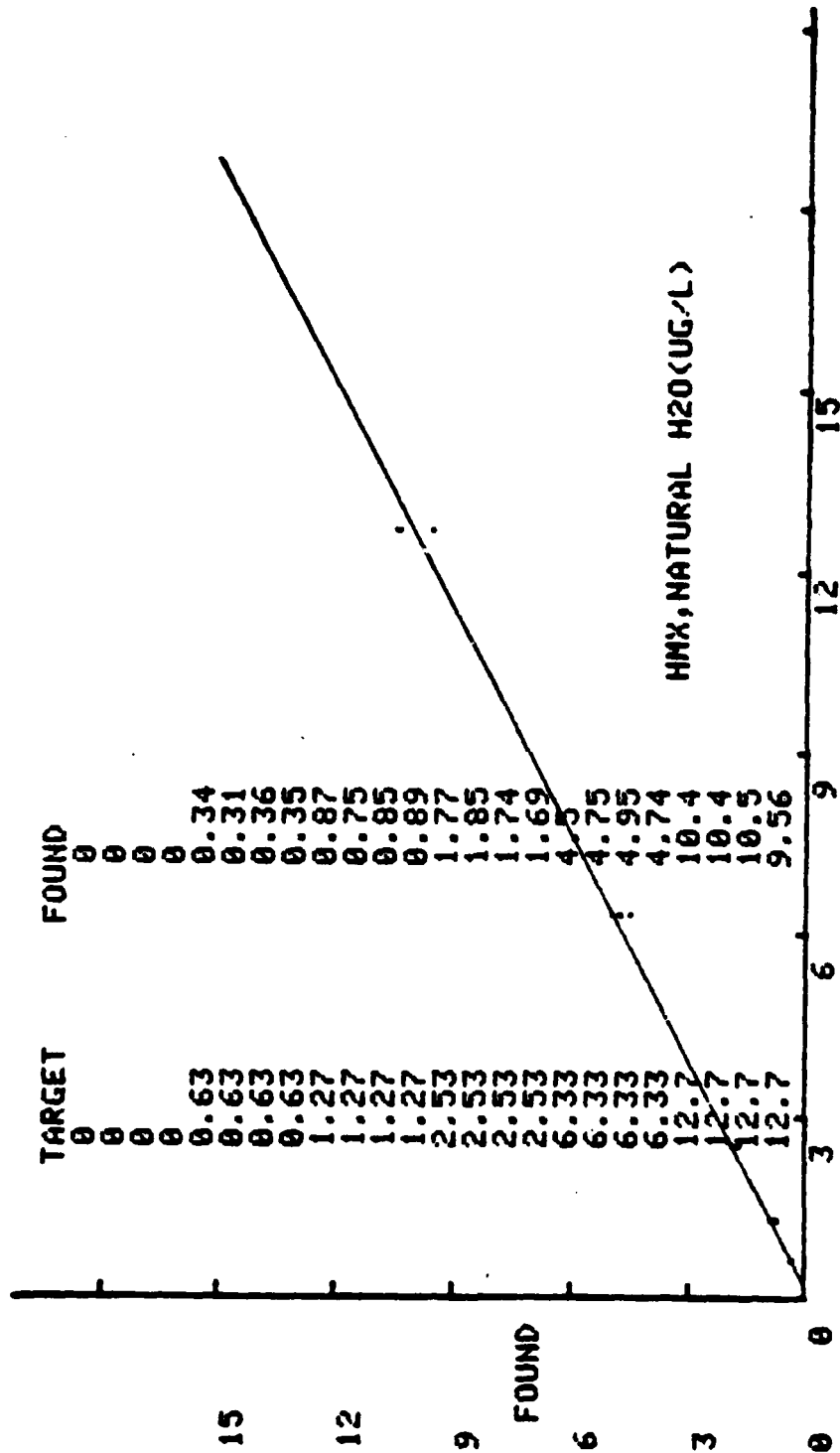
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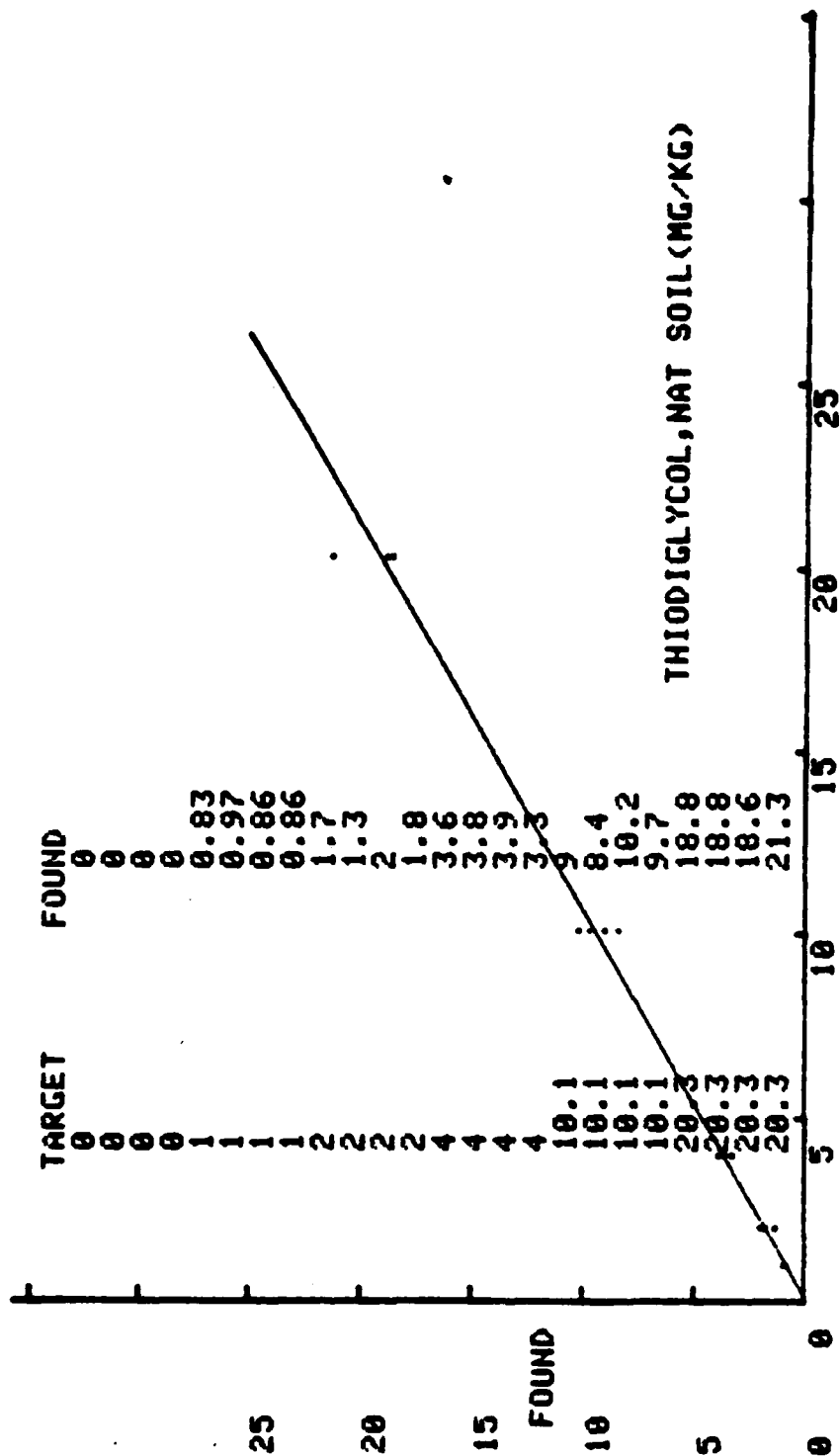
CORR. COEFF. = 0.9976
 DETECTION LIMIT = 1.03759
 TARGET FOUND = 0.0334
 TARGET



CORR. COEFF. = 0.9840
 DETECTION LIMIT = 2.92953
 TARGET
 FOUND = -0.0281 + 0.839673 * TARGET



CORR. COEFF. = 0.9982
 DETECTION LIMIT = 0.9687
 TARGET
 FOUND = -0.1807+ 0.808901 TARGET



CORR. COEFF. = 0.9966
 DETECTION LIMIT = 2.15007
 TARGET FOUND = -0.1365+
 0.953861 * TARGET

[illegible]

HMX IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for HMX.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.79 to 15.8 ug/g.

B. SENSITIVITY

The normalized response (integrator count) at the natural soil detection limit, designated in Section 1(C), is listed below:

<u>Integrator Counts</u>	<u>Nanograms</u>
339,000	389

The normalized response (integrator count) at the standard soil detection limit, designated in Section 1(C), is listed below:

<u>Integrator Counts</u>	<u>Nanograms</u>
327,000	375

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 4.9 ug/g.

The detection limit in standard soil, calculated according to Hubaux and Vos (1970), is 4.7 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb UV light at 230 nm and are extractable from soil with methylene chloride/acetone. Interferences are minimized by silica-gel cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

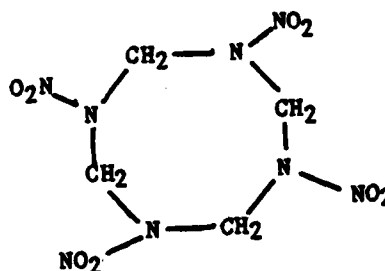
<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
HMX	Cyclotetramethylenetetranitramine	2691-41-0
	Octahydro-1,3,5,7-tetrazocine	
	1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane	
	Octogen	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml)</u>
HMX	$C_4H_8O_8N_8$	276	—	1.77-1.96*

* There are four polymorphic forms of HMX with this range of densities.

Chemical Structure



C. CHEMICAL REACTION

HMX is highly explosive, and caution should be used in its handling. HMX is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 230 nm)
2. Column: Zorbax-CN (4.6-mm ID x 25 cm)
Particle size: 7-8 μ m
3. Flow Rate/Mobile Phase: 1 ml/min
35% H₂O/65% methanol
4. Temperature: 22°C
5. Injection Volume: 20- μ l, fixed loop
6. Retention Time: 6.7 minutes

C. **HARDWARE/GLASSWARE**

1. 50-ml centrifuge tubes with Teflon[®]-lined screw caps (8);
2. 500-ml K-D evaporative flasks (8);
3. 10-ml graduated K-D receivers (8);
4. 3-ball Snyder columns (8);
5. 2-ball micro-Snyder columns (8);
6. 15-ml graduated centrifuge tubes (8); and
7. 10-ml glass or polyethylene syringes with Luer-lock attachments (10).

D. **CHEMICALS**

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Anhydrous sodium sulfate--reagent grade;
5. Nanograde hexane;
6. Nanograde acetone; and
7. Silica-Gel Sep-Paks[®]--Waters Associates.

4. **STANDARDS**

A. **CALIBRATION STANDARDS**

1. The HMX stock calibration standard (7.91 mg/ml) is prepared by weighing 79.1 mg of HMX into a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile.
2. An intermediate HMX stock calibration standard is prepared by pipetting 5 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 791 ug/ml of HMX.
3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standards for HMX. Dilute with 50% methanol/50% water as follows:

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<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
B	HMX (intermediate)	2	25
C	HMX (intermediate)	2	50
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
B	63.3
C	31.6
D	12.7
E	6.3
F	3.2

B. CONTROL SPIKES

1. Prepare Control Spike Solution A for HMX by pipetting 1 ml of the calibration standard stock for HMX into a 25-ml volumetric flask and diluting to volume with acetone.
2. Prepare Control Spike Solution B for HMX by diluting 5 ml of Control Spike Solution A to 50 ml with acetone.

<u>Control Spike Solution</u>	<u>Concentration (ug/ml)</u>
A (HMX)	316
B (HMX)	31.6

3. Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)

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4. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon®-lined screw cap.
5. Pipette a known amount of the control spike solution for HMX onto the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>HMX Control Spike Solution</u>	<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Soil (ug/g)</u>
B	0.5	0.79
B	1.0	1.6
B	2.0	3.2
A	0.5	7.9
A	1.0	16

6. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

1. Add 35 ml of 20% acetone in methylene chloride to the centrifuge tube.
2. Cap the tube and shake for 3 to 5 minutes.
3. Extract the sample sequentially with three 35-ml portions of the methylene chloride/acetone mixture.

4. Decant off the methylene chloride/acetone mixture each time, and pass through a glass funnel filled with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
5. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
6. Add a boiling chip (Teflon®) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
7. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
8. The balls of the Snyder column should actively chatter when the solvent is evaporating.
9. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After approximately 1 ml of solvent has drained back into the receiver, remove the receiver from the K-D flask.
10. Add approximately 2 ml of nanograde hexane to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
11. Repeat Step 10 twice.
12. Detach the micro-Snyder column from the receiver. With a dispo pipette, transfer the extract into a 10-ml glass syringe fitted with a silica-gel Sep-Pak®. Rinse the receiver three times with 2 ml of 20% methylene chloride in hexane solution, transferring each rinse to the 10-ml syringe. Set aside the receiver for later use.

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13. Pass the combined rinses through the silica-gel Sep-Pak® at a rate of approximately 1 to 2 ml/min, discarding the eluate.
14. Quantitatively rinse the K-D receiver from Step 12 three times with a total of 1 to 2 ml of 50% methanol in methylene chloride solution, transferring each rinse to the 10-ml syringe fitted with the silica-gel Sep-Pak®. Add 50% methanol in methylene chloride to the syringe to make a total volume of 10 ml.
15. Elute the silica-gel Sep-Pak®, with the 10-ml total volume of 50% methanol in methylene chloride, into another 10-ml K-D receiver at a rate of 1 to 2 ml/min.
16. Add a Teflon® boiling chip to eluate, attach a 2-ball micro-Snyder column, and concentrate the sample in a water bath heated to 80°C. When the apparent volume of the solution is about 0.5 ml, remove the apparatus from the water bath.
17. Detach the micro-Snyder, and add approximately 2 ml of HPLC methanol to the receiver. Reconcentrate the sample to 0.5 ml.
18. Repeat Step 17 twice.
19. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC methanol. Raise the extract volume to exactly 2.5 ml in the centrifuge tube with HPLC methanol. Dilute to 5 ml with HPLC water.
20. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
21. The extract is now ready for chromatography by HPLC.

C. ANALYSIS

1. Inject 20 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard-versus-peak area counts.
- B. Determine the concentration of HMX according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

W_s = Weight of initial sample extracted (g).

7. REFERENCES

None found.

8. DATA

See attached data sheets.

HMX, STANDARD SOIL (UG/G)

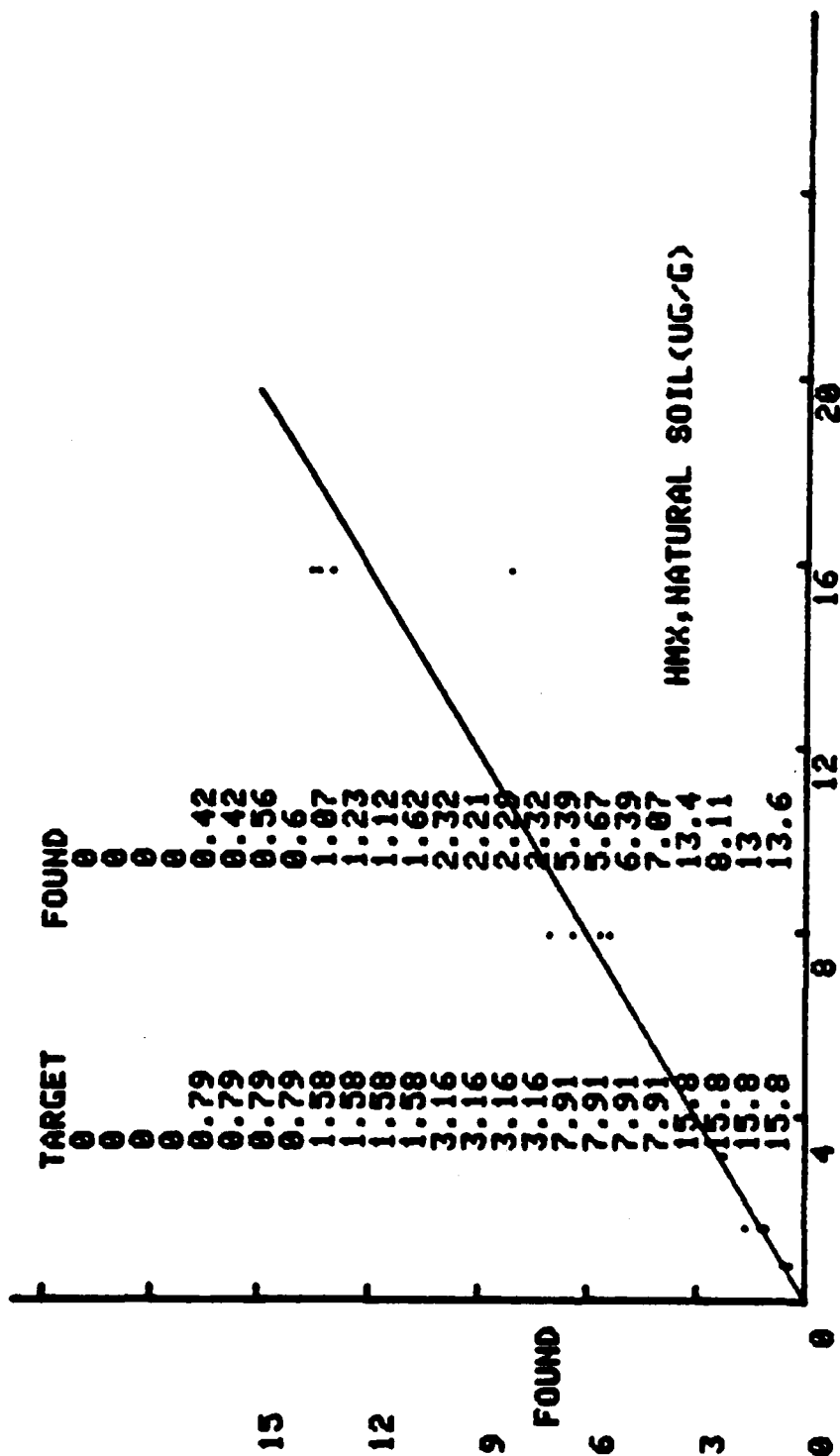
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0530	0.0000	0.0000	0.0000
0.790	0.620	0.660	0.680	0.850
1.58	1.37	1.41	1.64	1.03
3.16	3.30	3.06	3.22	2.32
7.91	6.16	6.03	4.90	6.91
15.8	13.1	13.5	8.32	10.9

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0132	0.0265	200	0.0000
0.790	0.702	0.101	14.4	-11.0760
1.58	1.36	0.252	18.5	-13.7658
3.16	2.97	0.448	15.1	-5.8544
7.91	6.00	0.830	13.8	-24.1467
15.8	11.5	2.39	20.9	-27.4209

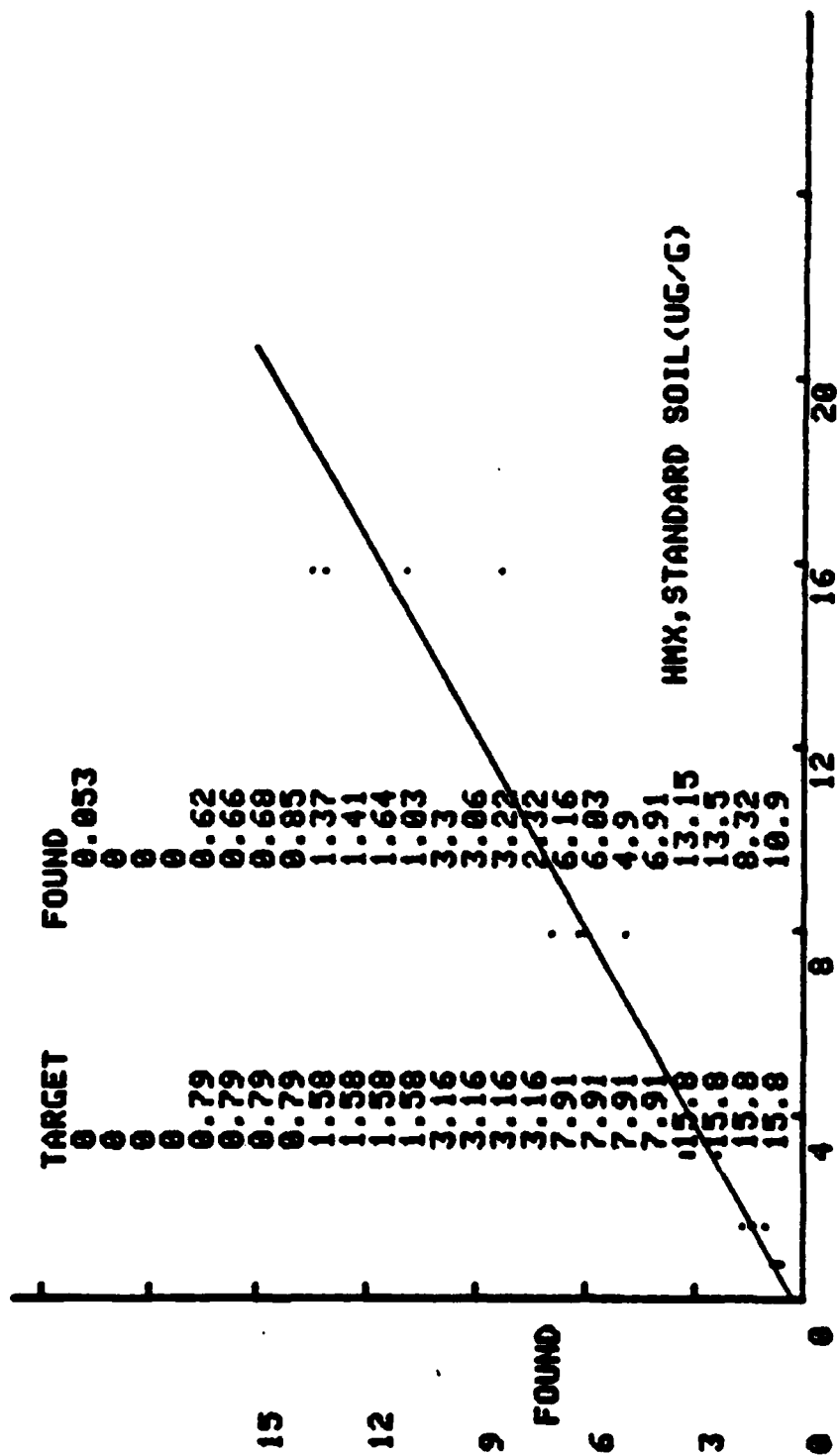
HMX, NATURAL SOIL (UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.790	0.420	0.420	0.560	0.600
1.58	1.07	1.23	1.12	1.62
3.16	2.32	2.21	2.29	2.32
7.91	5.39	5.67	6.39	7.07
15.8	13.4	8.11	13.0	13.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.790	0.500	0.0938	18.8	-36.7089
1.58	1.26	0.249	19.8	-20.2532
3.16	2.28	0.0520	2.27	-27.6899
7.91	6.13	0.755	12.3	-22.5032
15.8	12.0	2.62	21.8	-23.8766



CORR. COEFF. = 0.9745
 DETECTION LIMIT = 4.63496
 TARGET
 FOUND = -0.0296+ 0.765401 * TARGET



CORR. COEFF. = 0.9728 TARGET FOUND = 0.2613+ 0.716582*TARGET
DETECTION LIMIT = 4.78998

DPA IN SOIL AND SEDIMENT SAMPLES

DPA IN SOIL AND SEDIMENT SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of DPA in environmental soil and sediment samples.

This method was developed and tested in two soil matrices: a red clay obtained from the Alabama Army Ammunition Plant and a topsoil from North Central Florida. These two soils are subsequently referred to as the standard soil and the natural soil, respectively.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges are:

<u>Matrix</u>	<u>Tested Concentration Range (ug/g)</u>
Standard Soil	1.3 to 26
Natural Soil	1.5 to 30

B. SENSITIVITY

The normalized response (integrator counts x attenuation at the documented detection limits designated in Part C below are:

<u>Matrix</u>	<u>Integrator Counts</u>	<u>Quantity (picograms)</u>
Standard Soil	1,221	1,500
Natural Soil	1,627	1,600

C. DETECTION LIMITS

The detection limits, calculated according to Hubaux and Vos (1970), are:

7/22/82

<u>Matrix</u>	<u>Concentration (ug/g)</u>
Standard Soil	1.5
Natural Soil	1.6

D. INTERFERENCES

This method contains no cleanup step and thus may be subject to interferences from nitrogen- or phosphorus-containing compounds which co-elute with the chromatographic peak for DPA. The detection method used for this work is, however, very selective for nitrogen (and phosphorus), with a selectivity ratio of 1,000:1 for nitrogen versus carbon. This method has been tested on a variety of soil matrices with no apparent interferences.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 10 extractions in an 8-hour day.

2. CHEMISTRY**A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER**

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
DPA	--	122-39-4

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTES

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml at 20°C)</u>
DPA	C ₁₂ H ₁₁ N	52-54	302	1.16

C. CHEMICAL REACTIONS

None found.

3. APPARATUS

A. INSTRUMENTATION

Perkin Elmer Sigma 2 GC interfaced to a Spectra Physics 4100 Computing Integrator.

B. GC PARAMETERS

1. Detector: Nitrogen-phosphorus specific; bead current setting--6.8 to 7.0.

2. Column: 1/4-inch x 2-mm ID x 10-ft glass
Packing: 1.5% OV-17 on 80/100 mesh supelcoport

3. Gas Flow

Carrier: Nitrogen (50 ml/minute)
Detector: Hydrogen (1-2 ml/minute)
Air (100 ml/minute)

4. Temperature

Injector: 250°C
Detector: 300°C
Oven Temperature: Isothermal at 160°C

5. Injection Volume: 5 ul

6. Retention Time: 4.0 minutes

C. HARDWARE/GLASSWARE

1. 50-ml, Teflon®-lined capped centrifuge tube.
2. Glass disposable pipettes.
3. Centrifuge, capable of handling 50-ml centrifuge tubes.
4. Class A volumetric flasks: 50 ml, 10 ml.
5. Metal spatula.

D. CHEMICALS AND REAGENTS

1. Toluene, nanograde, distilled in glass.
2. Methanol, nanograde, distilled in glass.
3. 0.01N sodium hydroxide solution.
4. Analytical standards (SARMs or equivalent).

4. STANDARDS

A. CALIBRATION STANDARDS

1. Prepare a calibration standard stock (11.7 mg/ml) by weighing 117 mg of the DPA SARM in a single 10-ml volumetric flask.

Dissolve in a few ml of toluene and dilute to volume with toluene. Wrap the flask in foil and store in a freezer. No evidence of degradation was observed over a 6-month period.

2. Prepare the dilute stock calibration standard by pipetting 1 ml of the calibration standard stock and diluting to volume with toluene in a 100-ml volumetric flask. Label this solution and store in an amber, septum-sealed vial at 4°C.
3. Prepare the working stock calibration standards by making dilutions with toluene of dilute stock calibration standard as follows.

<u>Working Stock Calibration Standard</u>	<u>Dilute Stock Calibration Standard</u>	<u>Final Volume (ml)</u>	<u>Concentra- tion (ng/g)</u>
A	100 ul	50	234
B	200 ul	50	468
C	400 ul	50	937
D	1.0 ml	50	2,340
E	2.0 ml	50	4,680
F	4.0 ml	50	9,360
G	10.0 ml	50	23,400

Store standards in amber septum-sealed vials at 4°C until ready to use.

B. CONTROL SPIKES

1. Prepare the stock control spike solution (2.57 mg/ml) by weighing approximately 25.7 mg of the DPA SARM into a single 10-ml volumetric flask.

Dissolve in a few ml of methanol and dilute to volume with methanol.

2. Dilute the stock control spike solution by pipetting 1 ml into a 10-ml volumetric flask and diluting to volume with methanol to obtain the working stock control spike solution.
3. Pipet a known amount of the working control spike into a standard soil sample. The quantity spiked should be selected to approximately double the expected concentration or to provide a concentration of 0.5 to 10 times the detection limit. The precision, accuracy, and detection limits were determined by spiking 10-g samples of soil at the following levels:

<u>Volume Spiked (ul)</u>	<u>Concentration (ug/g)</u>
0	0
50	1.28
100	2.57
200	5.14
500	12.8
1,000	25.7

4. After spiking the soil, enough toluene is added to just wet the sample. The mixture is allowed to air-dry for 1 hour before analysis.
5. Perform the procedures in Section 5, starting with Step B2.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let all soil samples air-dry on the dull side of aluminum foil until they can be sieved through a 30-mesh sieve.
2. Sediment samples are extracted wet.
3. Determine the dry weight of the sediment or soil by weighing 5 g of sample into a tared beaker. Dry the soil at 105°C until constant weight. Record the dry weight. Calculate the percent moisture.

B. EXTRACTION

1. Weigh 10 g of soil or 15 g of wet sediment (to the nearest 0.1 g) into a 50-ml centrifuge tube.
2. Add 20 ml of 0.01N sodium hydroxide, 10 ml of toluene, and 5 ml of methanol to the tube and cap. Shake the tube vigorously for 3 minutes.
3. Place the tube in the centrifuge, and centrifuge at medium speed ("5") until the solids separate from the solvent (10 minutes).
4. Withdraw the toluene/methanol layer (top layer) using a glass pipet and transfer to a 50-ml volumetric flask.
5. Repeat the extraction with 10 ml of toluene and 5 ml of methanol two more times.
6. Combine the toluene extracts in the 50-ml volumetric flask.
7. Dilute the extract with toluene to the 50-ml mark.

8. Shake the flask and transfer approximately 1 ml to a septum-sealed vial. Cap securely with septum and seal. Protect from light by wrapping the sample vial with foil.

C. CALIBRATION

1. Inject the working standards singly at the beginning of the analytical run and one standard at the conclusion of the analytical run.
2. Plot the normalized peak height versus micrograms injected of each standard to obtain a working curve.

D. ANALYSIS

Inject 5 ul of the toluene extract onto the GC column.

6. CALCULATIONS

- A. Determine the concentration of the analyte according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{(V_i)(W)}$$

where A = Amount of analyte found (ug), determined from the standard curve,

V_t = Volume of total extract (ml),

V_i = Volume of extract injected (ml), and

W = Sample weight (g).

- B. Correct for percent moisture in the original soil/sediment sample.

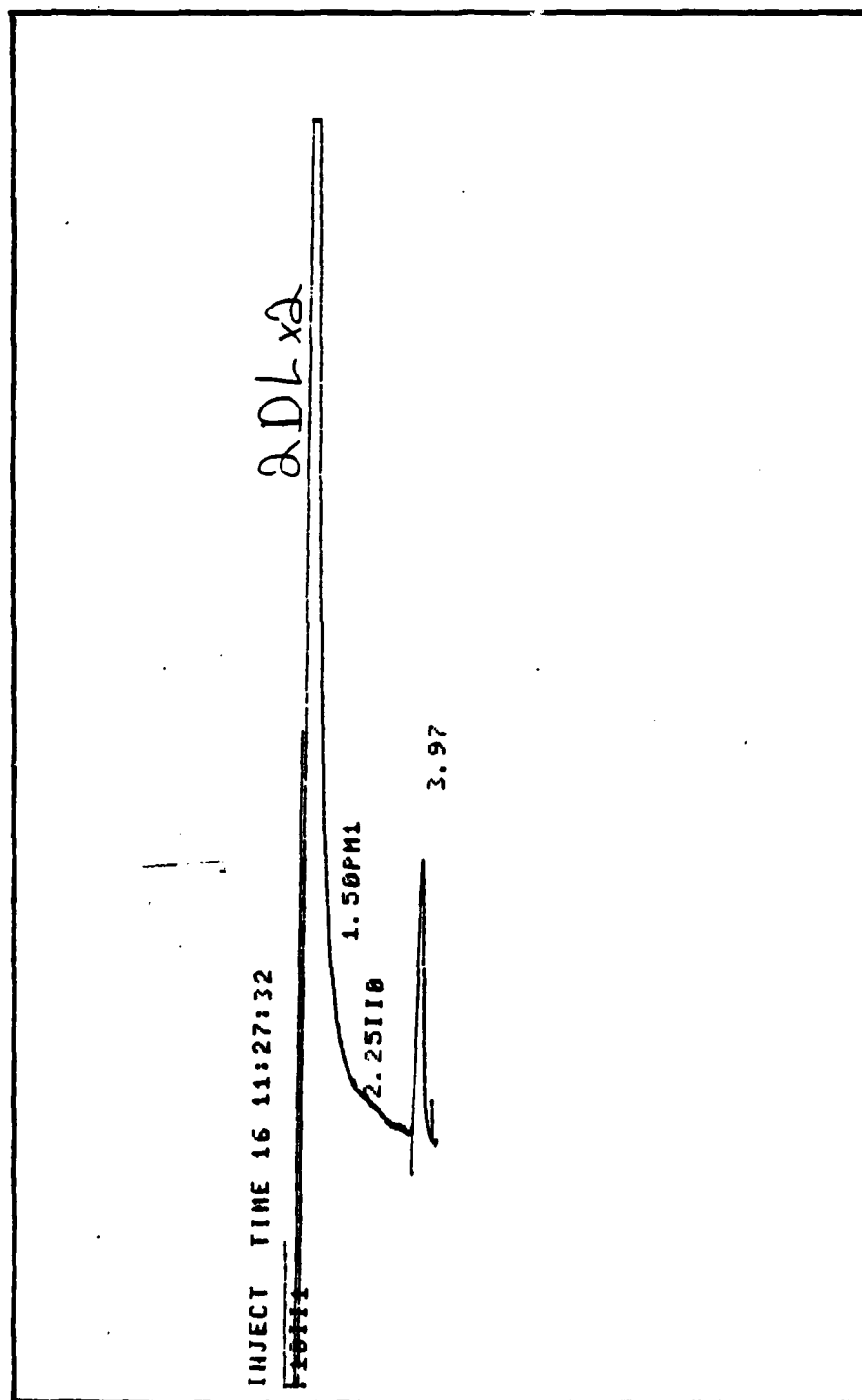
7. REFERENCES

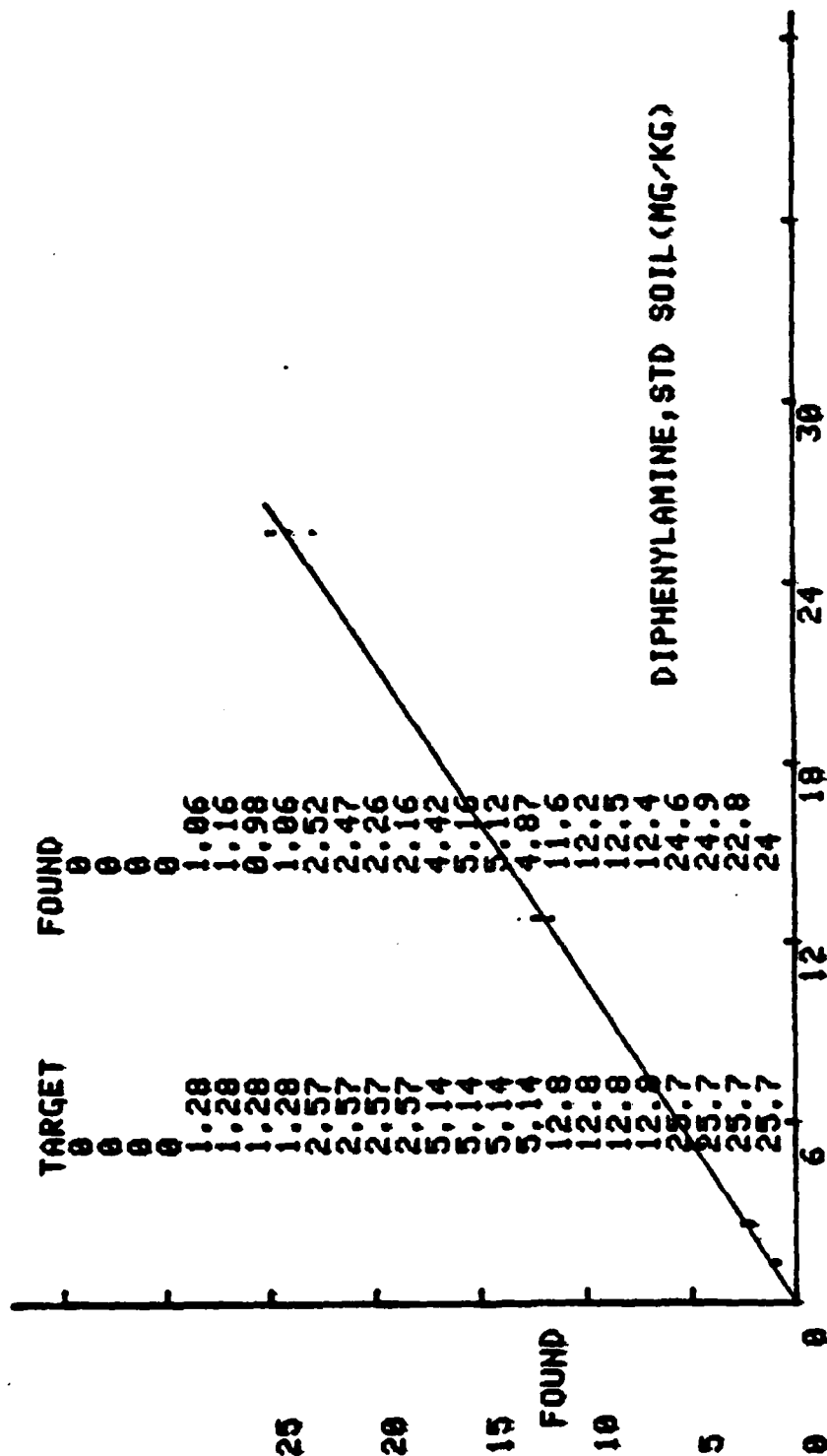
None found.

8. DATA

See attached data sheets.

Figure 1
Chromatogram of Diphenylamine
(2340 pg injected).



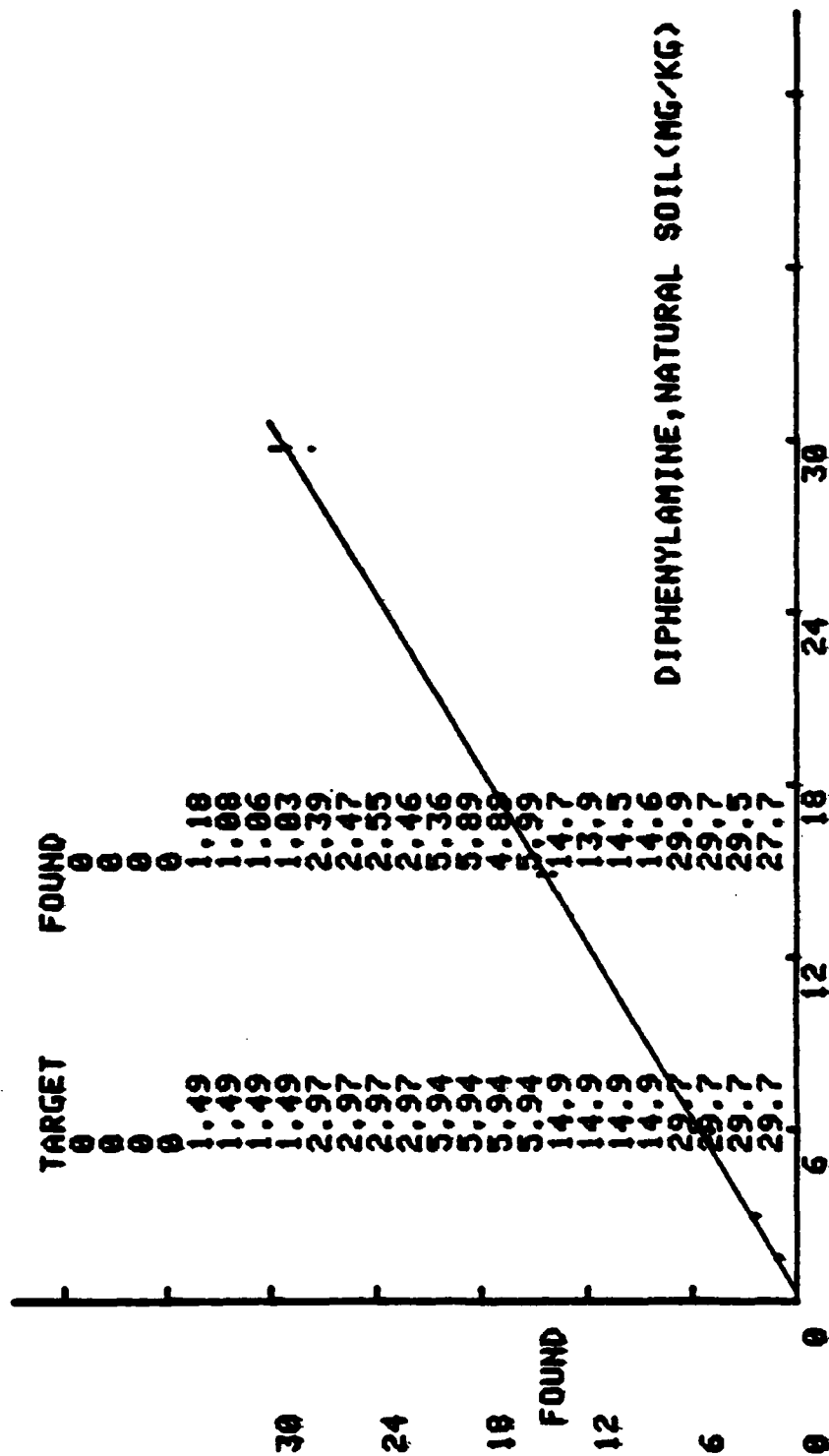


CORR. COEFF. = 0.9989
 DETECTION LIMIT = 1.52955
 TARGET
 FOUND = -0.0185+ 0.940639 * TARGET

DIPHENYLAMINE, STD SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.22	1.06	1.16	0.980	1.06
2.57	2.52	2.47	2.26	2.16
5.14	4.42	5.16	5.12	4.87
12.2	11.6	12.2	12.5	12.4
25.7	24.6	24.9	22.8	24.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.22	1.06	0.0737	6.92	-16.7969
2.57	2.35	0.171	7.26	-8.4630
5.14	4.89	0.340	6.95	-4.8152
12.2	12.2	0.403	3.31	-4.6828
25.7	24.1	0.929	3.86	-6.3230



CORR. COEFF. = 0.9991
 DETECTION LIMIT = 1.64848
 TARGET
 FOUND = -0.3036+
 0.991534*TARGET

DIPHENYLAMINE, NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.49	1.18	1.08	1.06	1.03
2.97	2.39	2.47	2.55	2.46
5.94	5.36	5.89	4.89	5.99
14.9	14.7	13.9	14.5	14.6
29.7	29.9	29.7	29.5	27.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.49	1.05	0.0650	5.98	-27.0134
2.97	2.47	0.0655	2.66	-16.9192
5.94	5.53	0.510	9.21	-6.8603
14.9	14.4	0.359	2.49	-3.1879
29.7	29.2	1.01	3.47	-1.6835

UDMH IN WATER SAMPLES

UDMH IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for UDMH.

A. TESTED CONCENTRATION RANGE

The tested concentration range for standard and natural water is 5 to 107 ug/L.

B. SENSITIVITY

The normalized responses (peak heights) at the detection limits designated in Section C (below) are 325 mm for 1.36 ng in natural water and 423 mm for 2.01 ng in standard water.

C. DETECTION LIMITS

The detection limits, calculated according to Hubaux and Vos (1970), are 11 ug/L for natural water and 16 ug/L for standard water.

D. INTERFERENCES

This method may be subject to interferences from compounds which can be readily oxidized under an electrochemical potential of +0.9 volt. Phenolic compounds are included in this class; however, chromatographic conditions were selected to minimize interferences from the commonly found priority pollutant phenols.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 samples in an 8-hour day.

2. CHEMISTRY

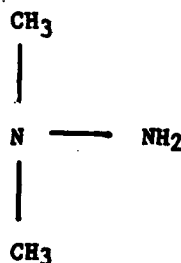
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
UDMH	asym-dimethylhydrazine	
	1,1-dimethylhydrazine	57-14-7
	unsym-dimethylhydrazine	
	N,N-dimethylhydrazine	
	Dimazine	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Molecular Formula</u>	<u>Melting Point</u>	<u>Boiling Point</u>	<u>Density (g/ml)</u>
UDMH	C ₂ H ₈ N ₂	-58°C	63.9°C	0.791

Chemical Structure



C. CHEMICAL REACTIONS

UDMH is a powerful reducing agent used as the base in rocket fuel formulations. UDMH is highly corrosive and irritating to skin, eyes, and mucous membranes. UDMH is readily oxidized in alkaline solution by a number of oxidants (HgO, halogens, and halates) to produce tetrazine. Tetrazines are inherently unstable and split out N₂ under thermal or protolytic conditions. In acidic solutions, UDMH reacts to form diazenium salts, (CH₃)₂N⁺ = NH X⁻, which react as dienophiles with

conjugated dienes in the Diels-Alder reaction. Hydrazines reduce many commonly found metal ions to lower valence states or to the metals themselves. Over 23 metal ions have been shown to react with hydrazines.

The kinetics of the decomposition of UDMH in aqueous solutions were briefly examined, and it has been found that the half-life for the disappearance of UDMH is less than 1 day. For this reason, standards for the UDMH analysis must be prepared fresh daily, immediately before the analysis is begun. Samples should be analyzed as soon as possible after collection. Chromatograms which illustrate the decomposition of UDMH in water are presented in Figure 1.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Bioanalytical Systems Model EC-2A Electrochemical Detector interfaced to a linear strip-chart recorder.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Bioanalytical Systems, Inc. Model EC-2A Electrochemical Detector with a glassy carbon electrode
Potential = +0.9 volt
2. Column: Zorbax C-8 (4.6-mm ID x 25 cm)
Particle size = 5-6 μ m
3. Flow Rate/Mobile Phase: 1 ml/min/50% acetonitrile/50% 0.09 M PO_4^{2-} buffered to pH = 7
4. Temperature: 22°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 7.1 minutes

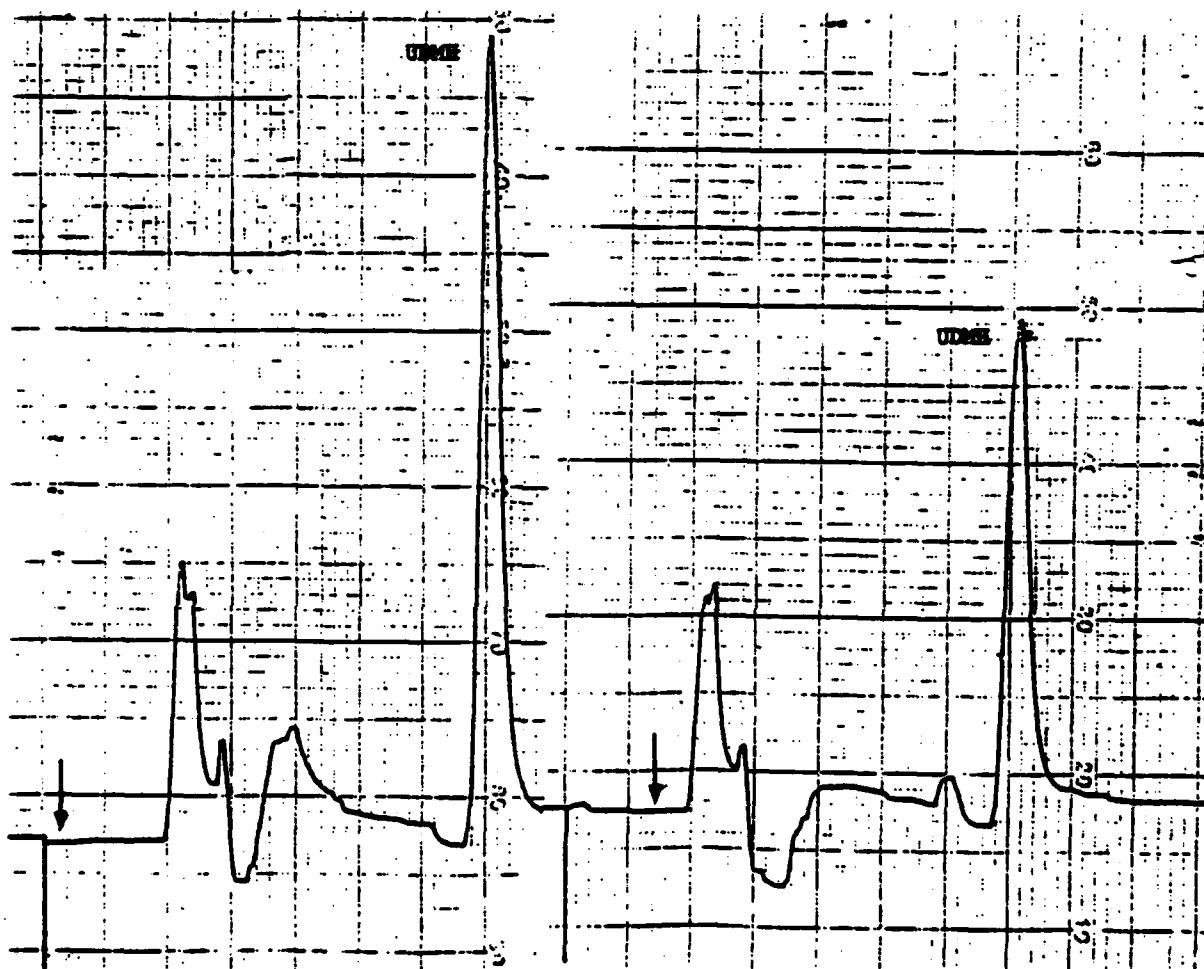


Figure 1. Chromatograms of UDME at 50 ug/L in Natural Surface Water
Time = 0 hours on left and 24 hours on right (pH = 6.0)

C. **HARDWARE/GLASSWARE**

1. 25-ml volumetric flask (1)
2. 50-ml volumetric flask (10)

D. **CHEMICALS**

1. HPLC-grade acetonitrile, J.T. Baker Company.
2. HPLC-grade water, J.T. Baker Company.
3. Potassium dihydrogenphosphate, J.T. Baker Company.
4. Potassium monohydrogenphosphate, J.T. Baker Company.

4. **STANDARDS**

A. **CALIBRATION STANDARDS**

1. Prepare a stock calibration standard (6.72 mg/ml) by weighing 168 mg of the UDMH SARM in a 25-ml volumetric flask.
2. Dissolve the UDMH in a few ml of HPLC-grade acetonitrile, and dilute to volume with acetonitrile. Wrap the flask in foil, and store at 4°C.
3. Prepare Intermediate Stock Calibration Standard A by pipetting 1 ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with acetonitrile. Transfer the solution to an amber, septum-sealed vial, and store at 4°C.
4. Prepare Intermediate Stock Calibration Standard B by pipetting 1 ml of Intermediate Stock Calibration Standard A into a 50-ml volumetric flask and diluting to volume with acetonitrile. Transfer this solution to an amber, septum-sealed vial, and store at 4°C.
5. Prepare a series of working calibration standards by making dilutions with 50% acetonitrile/50% 0.09 M phosphate-buffered water (pH = 7) as follows:

Working Calibration Standard	Concentration (ng/g)	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
C	54	Intermediate Stock B	1	50
D	27	Intermediate Stock B	0.5	50
E	10.8	Working Standard C	10	50
F	5.4	Working Standard D	10	50
G	2.7	Working Standard D	5	50

The standards must be prepared fresh daily.

B. CONTROL SPIKES

1. Pipet a known amount of Intermediate Stock Calibration Standard B into 25 ml of standard or natural water samples, and analyze by the procedure outlined in Section 5 below. The quantity spiked should be selected to provide a concentration of 0.5 to 10 times the detection limit.
2. Determine the precision, accuracy, and detection limit for the analyte.

Concentration (ng/g)	Volume (ml) of Standard B Spiked into 25 ml
--	0.0
5.36	0.050
10.7	0.100
21.4	0.200
53.6	0.500
107.2	1.00

5. PROCEDURE

A. SAMPLE PREPARATION

1. Pipet 25 ml of the water sample into a 50-ml volumetric flask.
2. Add approximately 15 ml of HPLC-grade acetonitrile to the volumetric flask, shake until thoroughly mixed, and wait until all the gas bubbles formed on mixing disappear (approximately 5 minutes).

3. Dilute to the 50-ml mark with HPLC-grade acetonitrile.
4. The sample is now ready for analysis by HPLC.

B. CALIBRATION

1. Inject 250 ul of each of the working calibration standards and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard E at the end of the analytical run to verify constancy of the instrument response.
2. Plot the normalized peak height (mm) versus the concentration for each standard to obtain a working calibration curve.

C. ANALYSIS

1. Inject 250 ul of the sample onto the LC column.
2. Perform analysis of the sample according to the conditions given in Section 3B.
3. Measure the peak height (mm) for the UDMH peak.

6. CALCULATIONS

Calculate the concentration of UDMH according to the following equation:

$$\text{Concentration ug/L} = 2A/1000$$

where: A is the concentration (ug/ml) obtained from the calibration curve.

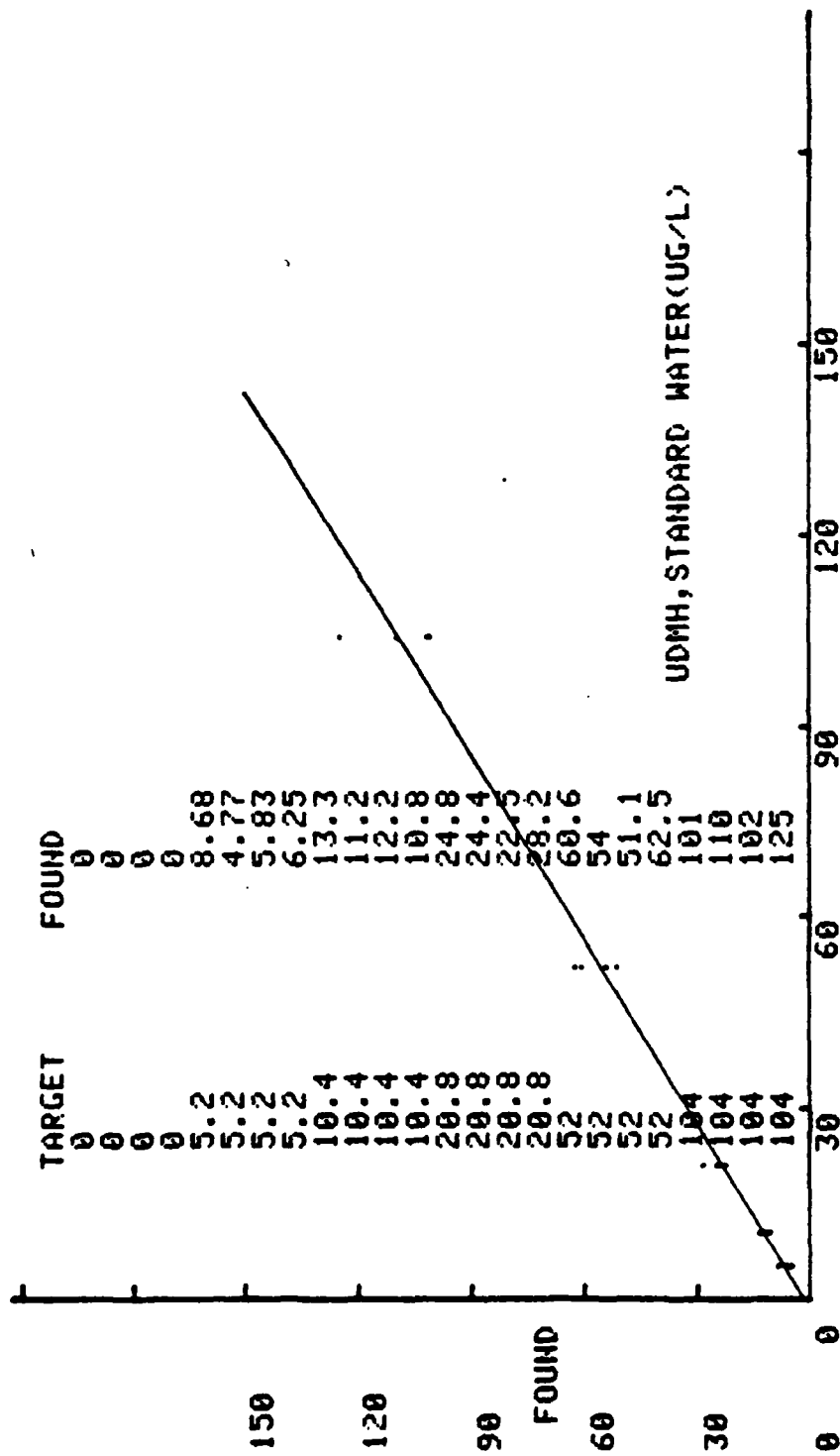
The injection volume is 250 ul, and the factor of 2 is due to the dilution of the sample with acetonitrile. This dilution is necessary to match sample and mobile phase compositions to minimize baseline disturbances arising from the sample injection.

7. REFERENCES

None found.

8. DATA

See attached data sheets. The target values reported are the averages of the values obtained over the 4 successive days of spiking experiments. Since UDMH decomposes rapidly, the standards were prepared fresh each day, and exact duplication of the target values was not practically feasible. The actual target values were closely spaced about the averages with a relative standard deviation of 2 percent.

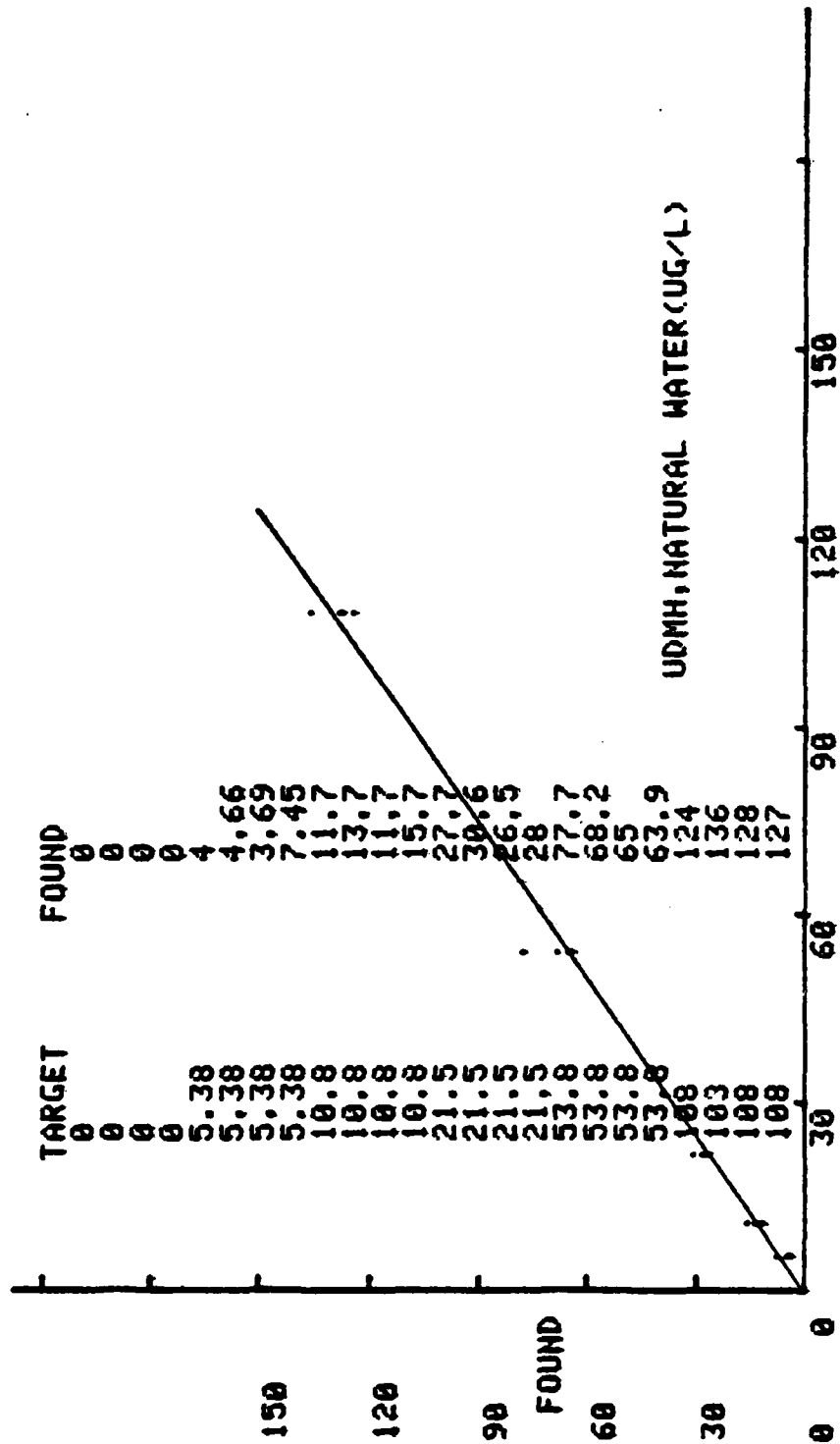


CORR. COEFF. = 0.9927
 DETECTION LIMIT = 16.10962
 TARGET FOUND = 1.3358+ 1.048689*TARGET

UDMH, STANDARD WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
5.20	8.68	4.77	5.63	6.25
10.4	13.3	11.2	12.2	10.8
20.8	24.8	24.4	22.5	28.2
52.0	60.6	54.0	51.1	62.5
104	101	110	102	125

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
5.20	6.38	1.65	25.9	22.7
10.4	11.9	1.12	9.41	14.2
20.8	25.0	2.37	9.50	20.1
52.0	57.0	5.39	9.44	9.71
104	110	11.1	10.1	5.29



CORR. COEFF. = 0.9956
 DETECTION LIMIT = 10.98899
 TARGET = 0.6207+
 1.203508 * TARGET

UDMH, NATURAL WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
5.38	4.00	4.66	3.69	7.45
10.8	11.7	13.7	11.7	15.7
21.5	27.7	30.6	26.5	28.0
53.8	77.7	68.2	65.0	63.9
108	124	136	128	127

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
5.38	4.95	1.72	34.6	-7.9926
10.8	13.2	1.91	14.5	22.2
21.5	28.2	1.73	6.12	31.2
53.8	68.7	6.27	9.13	27.7
108	129	5.12	3.98	19.2

ATNBA IN WATER SAMPLES

ATNBA IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for ATNBA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water is listed below in ug/L.

<u>Analyte</u>	<u>Range (ug/L)</u>
ATNBA	0.59 to 11.8

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1C is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
ATNBA	107,111	248

The normalized response (integrator counts) at the standard water detection limit designated in Section 1C is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
ATNBA	160,343	371

C. DETECTION LIMIT

The detection limit in natural water, calculated according to Hubaux and Vos (1970), is listed below:

<u>Analyte</u>	<u>Detection Limit (ug/L)</u>
ATNBA	2.2

AMD.4/TNBAH20.2
7/22/82

The detection limit in standard water, calculated according to Hubaux and Vos (1970), is listed below:

<u>Analyte</u>	<u>Detection Limit (ug/L)</u>
ATNBA	3.3

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 254 nanometers and are extractable from water at pH = 5 with methylene chloride/acetone. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 8 extractions in an 8-hour day.

2. CHEMISTRY

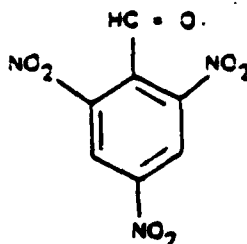
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
ATNBA	2,4,6-trinitrobenzene carbonal	606-34-8
	2,4,6-trinitrobenzene carbox-aldehyde	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml)</u>
ATNBA	C ₇ H ₃ O ₇ N ₃	119	---	---

Chemical Structure



C. CHEMICAL REACTIONS

ATNBA is explosive and rapidly decomposes in light; it must be protected by use of amber glass or aluminum foil. It decomposes rapidly to 135TNB in basic solution via a red-colored intermediate. It is slowly decomposed to 135TNB by dissolved oxygen, water, or upon heating with water or alcohol.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 UV detector (λ = 254 nm).
2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)
Particle size: 5 μ m

3. Flow Rate/Mobile Phase: 1 ml/min
45% H₂O/55% methanol

4. Temperature: 25°C
5. Injection Volume: 250 ul, fixed loop
6. Retention Times:

<u>Analyte</u>	<u>Retention Time (minutes)</u>
ATNBA	5.8
135TNB (decomposition product)	6.4

C. HARDWARE/GLASSWARE

- 1-L Teflon® separatory funnels with screw caps (8);
- 250-ml flat-bottomed boiling flasks (8);
- Glass filter funnels (8);
- Rotary evaporation with controlled-temperature water baths (4);
- Cold trap and vacuum pump;
- 15-ml graduated centrifuge tubes (8); and
- 5-ml glass syringes with Luer-lock attachments (8).

D. CHEMICALS

- Nanograde methylene chloride;
- HPLC-grade methyl alcohol--J.T. Baker Company;
- HPLC-grade water--J.T. Baker Company;
- Reagent-grade anhydrous sodium sulfate;
- Nanograde pentane;
- HPLC-grade acetonitrile--J.T. Baker Company;
- Florisil® Sep-Paks®--Waters Associates;
- 85% phosphoric acid--ACS;
- Colorphast® indicator sticks (MCB Manufacturing Chemists, Inc.); and
- Pasteur pipettes.

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4. STANDARDS

A. CALIBRATION STANDARDS

1. The ATNBA stock calibration standard (1.06 mg/ml) is prepared by weighing 10.6 mg of ATNBA into a 10-ml volumetric flask, dissolving the ATNBA in a few ml of acetonitrile, and diluting to the mark. The stock solution should be stored under nitrogen, in an amber vial in a freezer. It should be replaced on a monthly basis or sooner if degradation is observed (bright pink color or appearance of 135TNB on chromatogram).
2. An intermediate calibration standard is prepared by diluting 1 ml of the stock solution to 100 ml with acetonitrile that has been vacuum degassed. This standard should be stored under nitrogen, in an amber vial, in the freezer. It should be replaced on a weekly basis, or sooner if degradation is observed.
3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standard. The standards should be made fresh just prior to analysis. Dilute with a 50% acetonitrile/50% water solution (that has been vacuum degassed and kept under nitrogen) as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Intermediate stock	5.0	10.0
B	Intermediate stock	2.0	10.0
C	Intermediate stock	1.0	10.0
D	Intermediate stock	0.50	10.0
E	Intermediate stock	0.20	10.0
F	Intermediate stock	0.10	10.0

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	5.30
B	2.12
C	1.06
D	0.530
E	0.212
F	0.106

B. CONTROL SPIKES

1. Use the intermediate calibration standard as the control spiking solution.
2. Measure out 900 ml of water into a 1-L separatory funnel.
3. Pipet a known amount of the control spike solution into the sample and mix thoroughly. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Water (ug/L)</u>
0.05	0.59
0.1	1.18
0.2	2.36
0.5	5.89
1.0	11.78

4. Adjust the pH of the sample to 5 with 85% H_3PO_4 .
5. Extract the samples according to the procedure presented in Section 5B.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Measure 900 ml of the sample into a 1-L Teflon® separatory funnel.
2. Adjust the sample pH to 3 with 85% H₃PO₄ by dropwise addition.

B. EXTRACTION

1. Add 60 ml of methylene chloride to the separatory funnel.
2. Extract the sample by shaking the funnel for 3 minutes and then allowing 10 minutes phase separation time. Emulsions formed should be separated by centrifuging or sonication.
3. Drain the methylene chloride through a glass funnel filled with a small plug of glass wool and approximately 20 g of anhydrous sodium sulfate into a 250-ml boiling flask.
4. Repeat Steps 1 through 3 twice more and then combine extracts. The extracts should be protected from light by covering the boiling flask with aluminum foil.
5. After the third extract has been transferred to the flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
6. Concentrate the methylene chloride extract by rotary evaporation on a 35°C water bath.
7. When the apparent volume of the solution remaining in the flask is about 1 ml, remove the apparatus from the rotovap. Using a Pasteur pipette, transfer the extract to a 15-ml graduated centrifuge tube. Rinse the flask with an additional 1 ml of methylene chloride. Transfer the rinse to the centrifuge tube and raise the volume to 2.5 ml.
8. Raise to a final volume of 5 ml by adding nanograde pentane. Mix.
9. Equip a 5-ml syringe with a Florisil® Sep-Pak®. Pour the extract into the syringe, and pass through the Sep-Pak®, discarding the eluent.

10. Measure 5 ml of a 20% acetonitrile/80% methylene chloride solution in the centrifuge tube, and pour into the syringe. Elute the Sep-Pak® into the original boiling flask.
11. Evaporate the extract on the rotovap to an approximate volume of 1 ml. Add 10 ml of acetonitrile, and reduce the volume to 1 ml. Add a further 10 ml of acetonitrile, and reduce the extract to a final volume of 0.5 ml.
12. Transfer the 0.5 ml of extract to a 15-ml graduated centrifuge tube using a Pasteur pipette. Rinse the flask with an additional 0.5 ml of acetonitrile (which has been vacuum degassed and stored under nitrogen) and transfer to the centrifuge tube. Raise to a final volume of 2.0 ml with HPLC-grade water (vacuum degassed and stored under nitrogen).
13. Transfer to a 3-ml amber, septum-sealed vial for storage at 4°C.
14. The extract is now ready for chromatography by HPLC. The extract must be analyzed within 24 hours of time of extraction.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3B.
3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of ATNBA according to the following formula:

AMD.4/TNBAH20.9
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$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

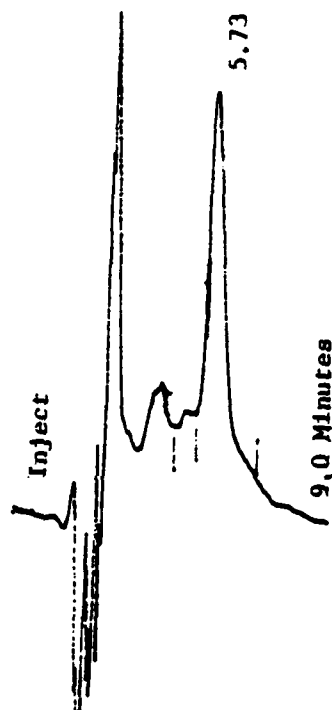
V_s = Volume of initial sample extracted (L).

7. REFERENCES

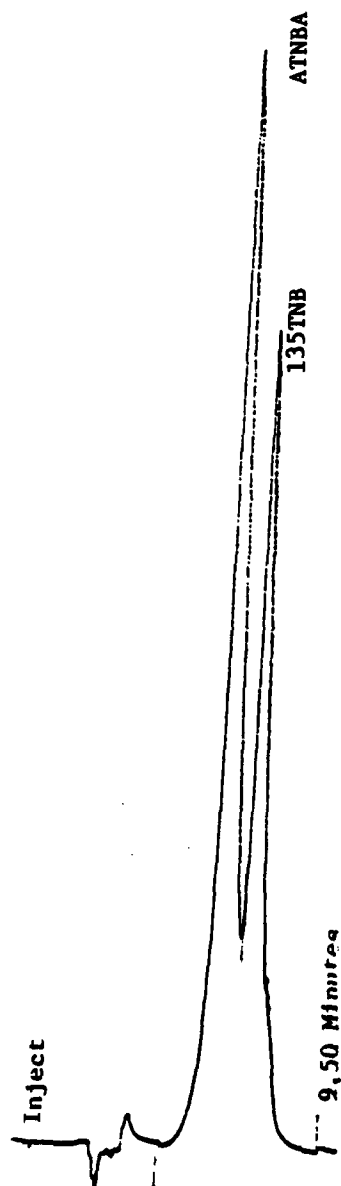
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8. DATA

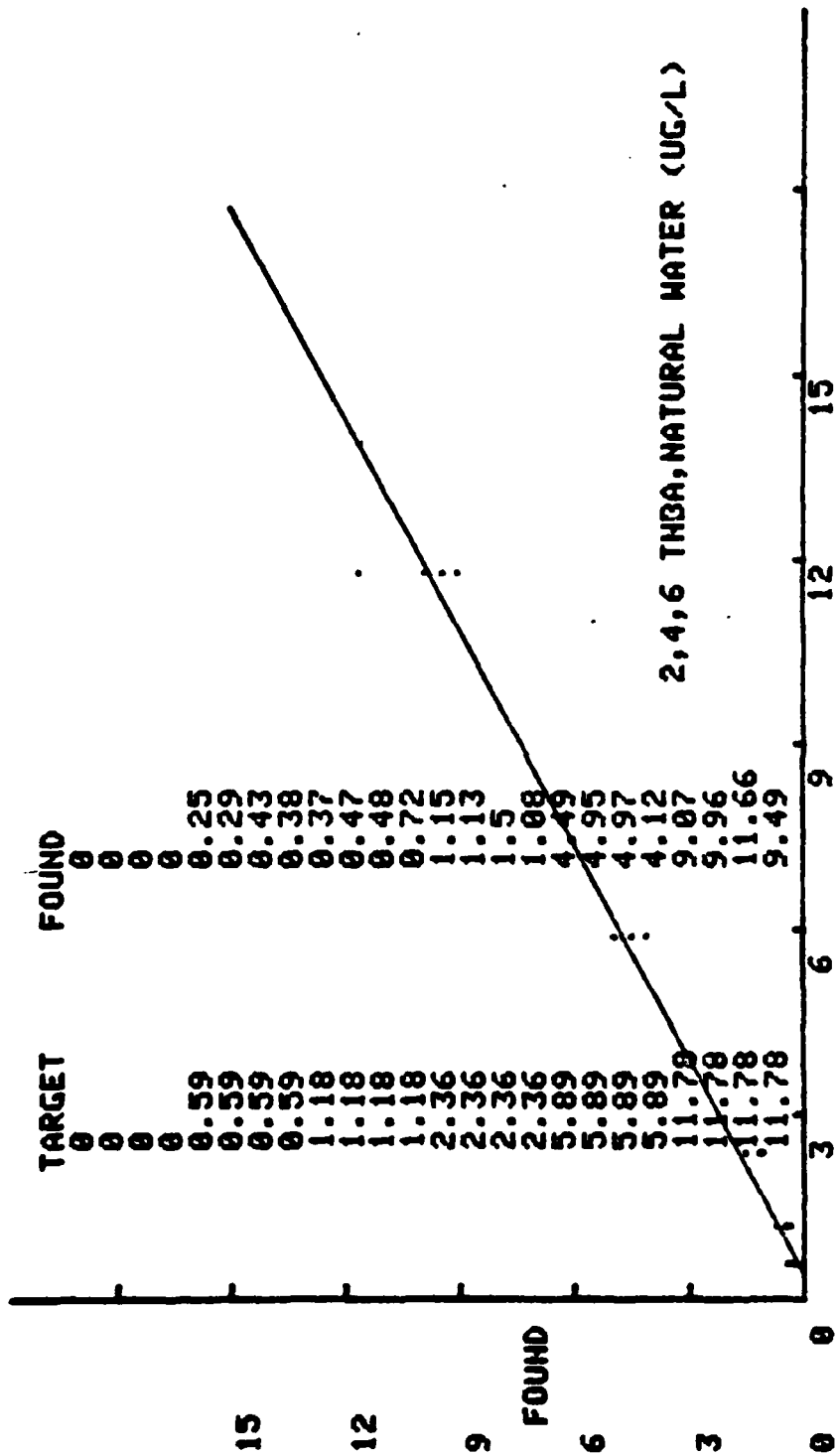
See attached data sheets.



Chromatogram of a Natural Water Extract Spiked at the
2.36-ug/L Level with ATNBA



Chromatogram of ATNBA and 135TNB (Decomposition Product)
Under the Conditions Given in Section 3B

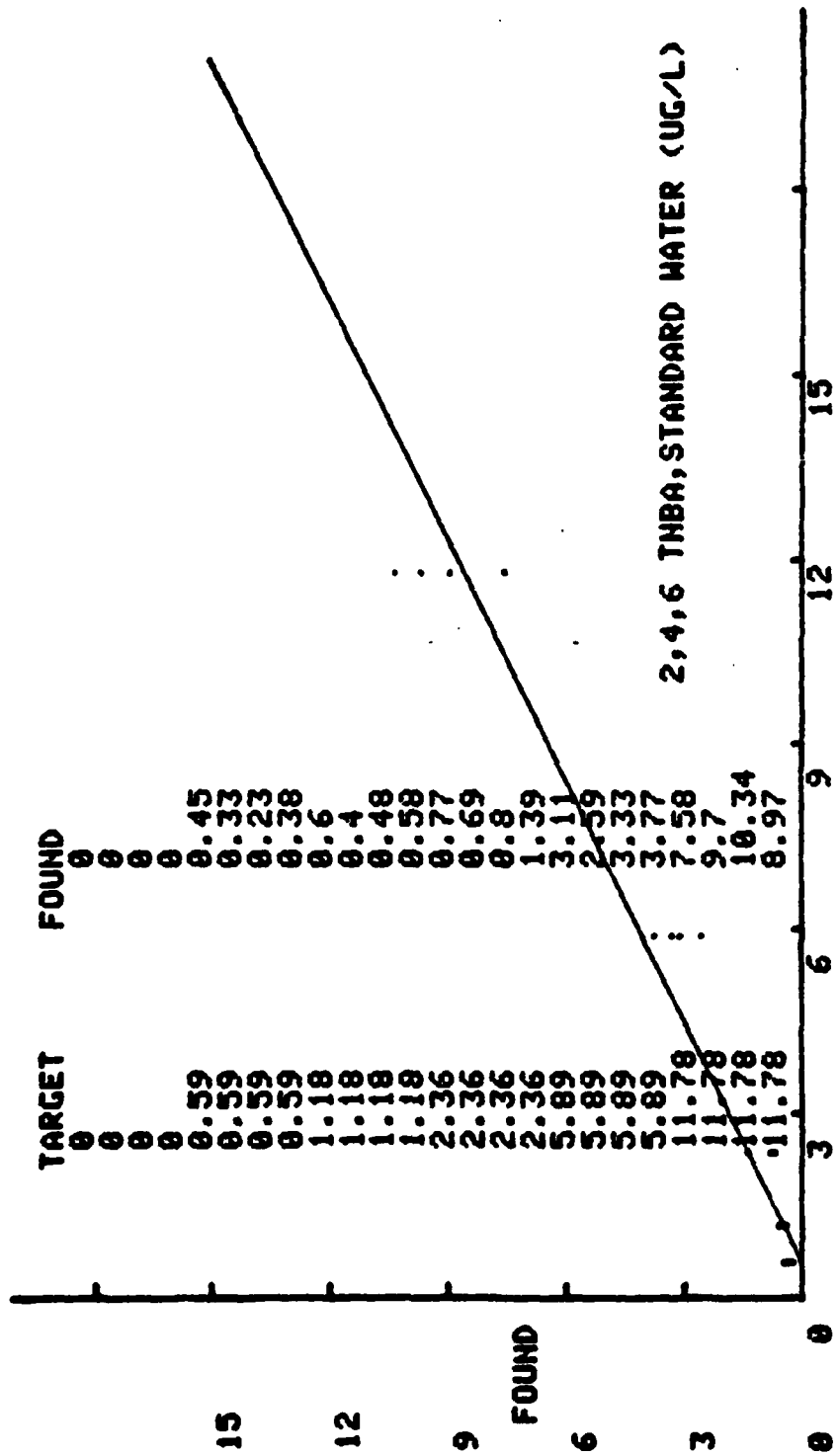


CORR. COEFF. = 0.9898
 DETECTION LIMIT = 2.16146
 TARGET FOUND = -0.3746+
 TARGET = 0.870997*TARGET

2,4,6 TNBA, NATURAL WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.590	0.250	0.290	0.430	0.380
1.16	0.370	0.470	0.480	0.720
2.36	1.15	1.13	1.50	1.08
5.89	4.49	4.95	4.97	4.12
11.8	9.07	9.96	11.7	9.49

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.590	0.337	0.0822	24.4	-42.7966
1.16	0.510	0.149	29.1	-56.7797
2.36	1.21	0.192	15.8	-48.5170
5.89	4.63	0.407	8.79	-21.3497
11.8	10.0	1.14	11.3	-14.7284



CORR. COEFF. = 0.9767 TARGET FOUND = -0.4459+ 0.770549xTARGET

DETECTION LIMIT = 3.29857

2,4,6 TNBA, STANDARD WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.590	0.450	0.330	0.230	0.380
1.18	0.600	0.400	0.480	0.580
2.36	0.770	0.690	0.800	1.39
5.89	3.11	2.59	3.33	3.77
11.8	7.58	9.70	10.3	8.97

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.590	0.347	0.0925	26.6	-41.1017
1.18	0.515	0.0929	18.0	-56.3559
2.36	0.912	0.322	35.3	-61.3348
5.89	3.20	0.491	15.3	-45.6706
11.8	9.15	1.19	13.0	-22.3472

ATNBA IN SOIL SAMPLES

ATNBA IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for ATNBA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is listed below in ug/g:

<u>Analyte</u>	<u>Range (ug/g)</u>
ATNBA	0.51 to 10.2

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section 1C is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
ATNBA	750,970	1,780

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1C is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
ATNBA	429,880	1,010

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is listed below:

<u>Analyte</u>	<u>Detection Limit (ug/g)</u>
ATNBA	3.5

The detection limit in standard soil, calculated according to Hubaux and Vos (1970), is listed below:

<u>Analyte</u>	<u>Detection Limit (ug/g)</u>
ATNBA	2.0

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 254 nm and are extractable from soil with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 8 extractions in an 8-hour day.

2. CHEMISTRY

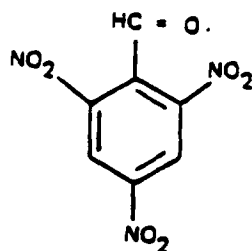
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
ATNBA	2,4,6-trinitrobenzene carbonal 2,4,6-trinitrobenzene carbox- aldehyde	606-34-8

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml)</u>
ATNBA	C ₇ H ₃ O ₇ N ₃	119	--	--

Chemical Structure



C. CHEMICAL REACTIONS

ATNBA is explosive and rapidly decomposed by light, and its solutions must be protected by use of amber glass or aluminum foil wrapping. It decomposes rapidly to 135TNB in basic solution via a red-colored intermediate. ATNBA is slowly decomposed to 135TNB by dissolved oxygen, water, or upon heating with water or alcohol.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 UV detector ($\lambda = 254 \text{ nm}$)
2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)
Particle size: 5 μm

3. Flow Rate/Mobile Phase: 1 ml/min
45% H₂O/55% methanol

4. Temperature: 25°C
5. Injection Volume: 250 ul, fixed loop
6. Retention Times:

<u>Analyte</u>	<u>Retention Time (minutes)</u>
ATNBA	5.8
135TNB (decomposition product)	6.4

C. HARDWARE/GLASSWARE

1. 50-ml centrifuge tubes with Teflon®-lined screw caps (8);
2. 250-ml boiling flasks (8);
3. Glass filter funnels (8);
4. Rotary evaporator (8), with controlled water bath, manifold, cold trap, and vacuum pump;
5. 15-ml graduated centrifuge tubes (8);
6. 5-ml glass syringes with Luer-lock attachments (8); and
7. Centrifuge (capacity for 50-ml tubes).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Reagent-grade anhydrous sodium sulfate;
5. Nanograde pentane;
6. HPLC-grade acetonitrile--J.T. Baker Company; and
7. Florisil® Sep-Paks®--Waters Associates.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The ATNBA stock calibration standard (1.02 mg/ml) is prepared by weighing 10.2 mg of ATNBA into a 10-ml

volumetric flask, dissolving the ATNBA in a few ml of acetonitrile, and diluting to the mark with acetonitrile. The stock solution should be stored under nitrogen, in an amber vial, in a freezer. The stock solution should be replaced on a monthly basis or sooner if degradation is observed (bright pink color or appearance of 135TNB on chromatogram).

2. An intermediate calibration standard is prepared by diluting 2 ml of the stock solution to 10 ml with acetonitrile. This standard should be stored under nitrogen, in a septum-sealed, amber vial in the freezer. The standard should be replaced on a weekly basis or sooner if degradation is observed.
3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standard. The working calibration standards should be made fresh just prior to analysis. Dilute with a 50% methanol/50% water solution which has been vacuum degassed and stored under nitrogen. Prepare the dilutions as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Intermediate Calibration Standard	1	10
B	Intermediate Calibration Standard	0.5	10
C	Intermediate Calibration Standard	0.1	5
D	Intermediate Calibration Standard	0.1	10
E	Intermediate Calibration Standard	0.05	10
F	Working Calibration Standard C	1	10

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	20.4
B	10.2
C	4.08
D	2.04
E	1.02
F	0.408

B. CONTROL SPIKES

1. Use the intermediate calibration standard as the control spiking solution.
2. Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)
3. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon®-lined screw cap.
4. Pipette a known amount of the control spike solutions into 3 ml of methylene chloride and quantitatively transfer to the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Soil (ug/g)</u>
0.05	0.51
0.1	1.02
0.2	2.04
0.5	5.1
1.0	10.2

5. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.
6. Extract the sample according to the procedure presented in Section 5B.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

1. Add 30 ml of methylene chloride to the centrifuge tube.
2. Cap the tube, shake for 3 minutes, and centrifuge for 5 minutes at 2,000 rpm.
3. Decant off the methylene chloride and pass through a glass funnel filled with a small plug of glass wool and approximately 20 g of anhydrous sodium sulfate into a 250-ml boiling flask. In event of a wet sediment, the methylene chloride layer should be withdrawn by use of a Pasteur pipet and the aqueous layer retained.
4. Steps 1 through 3 should be repeated twice more and the methylene chloride extracts combined. The extracts in the boiling flask should be protected from light by covering with aluminum foil.
5. After the third extract has been transferred to the boiling flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
6. Concentrate the methylene chloride extract by rotary evaporation on a 35°C water bath.
7. When the apparent volume of the solution remaining in the flask is about 1 ml, remove the apparatus from the rotovap. Transfer the extract to a 15-ml graduated centrifuge tube using a Pasteur pipet. Rinse the boiling flask with an additional 1 ml of methylene chloride, and transfer the rinses to the centrifuge tube. Raise the volume of the extract to 2.5 ml with methylene chloride.

8. Raise to a final volume of 5 ml by adding nanograde pentane. Mix.
9. Equip a 5-ml glass syringe with a Florisil® Sep-Pak®. Pour the extract into the syringe and pass through the Sep-Pak®, discarding the eluent.
10. Put 5 ml of a 20% acetonitrile/80% methylene chloride solution in the centrifuge tube and pour into the syringe. Elute the Sep-Pak® into the original boiling flask.
11. Evaporate the extract on a rotovap to an approximate volume of 1 ml. Add 10 ml of acetonitrile, and reduce the volume to 1 ml. Repeat with a further addition of 10 ml of acetonitrile and a final volume of 1 ml.
12. Transfer the 1-ml extract to a 15-ml graduated centrifuge tube using a Pasteur pipet. Rinse the flask with an additional 2 ml of acetonitrile (which has been vacuum degassed and stored under nitrogen) and transfer to the centrifuge tube. Raise to a volume of 4 ml with acetonitrile and to a final volume of 10 ml with HPLC-grade water (vacuum degassed and stored under nitrogen).
13. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C. Fill the vial to minimize headspace.
14. The extract is now ready for chromatography by HPLC. The extract should be analyzed within 24 hours and preferably immediately.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3B.
3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of ATNBA according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

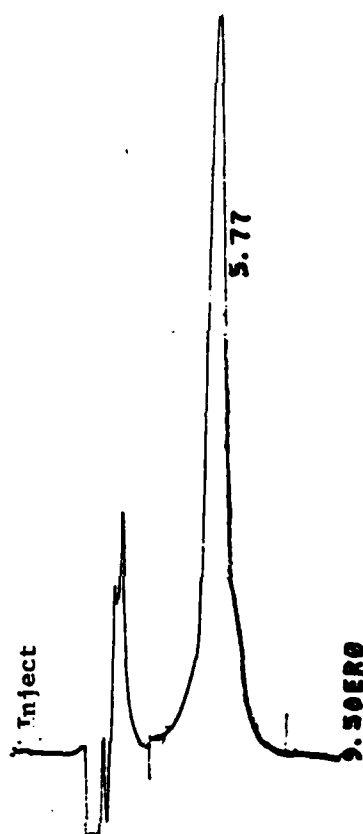
W_s = Weight of initial sample extracted (g).

7. REFERENCES

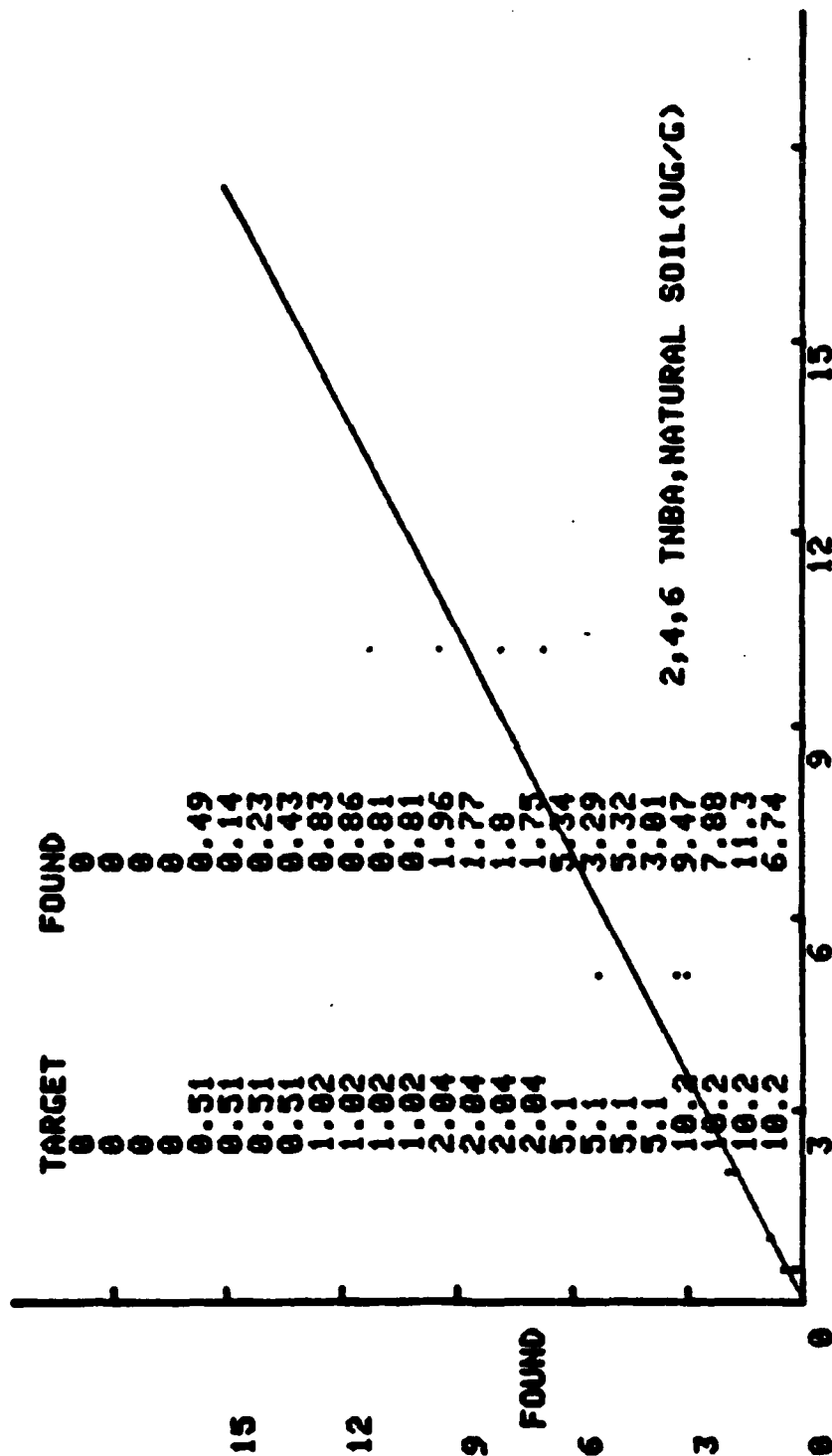
None found.

8. DATA

See attached data sheets.



Chromatogram of an Extract of Natural Soil Spiked with 2.04 ug/g of ATNBA

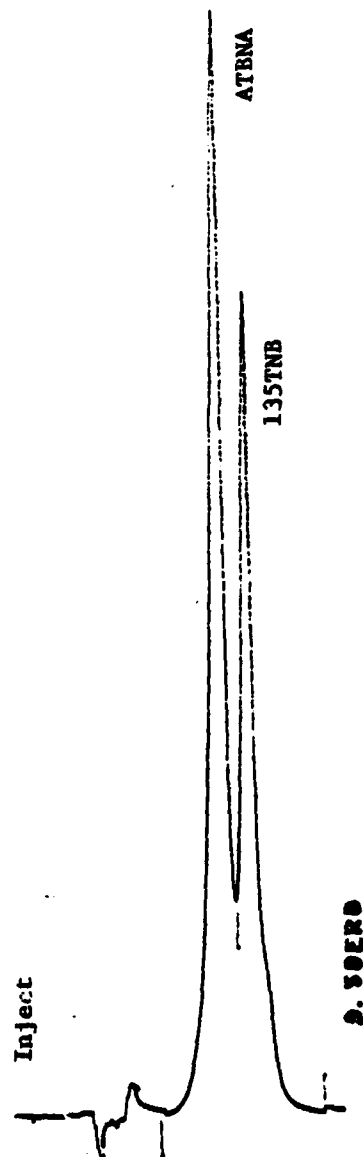


CORR. COEFF. = 0.9632
 DETECTION LIMIT = 3.51893
 TARGET FOUND = -0.0527+ 0.867697 * TARGET

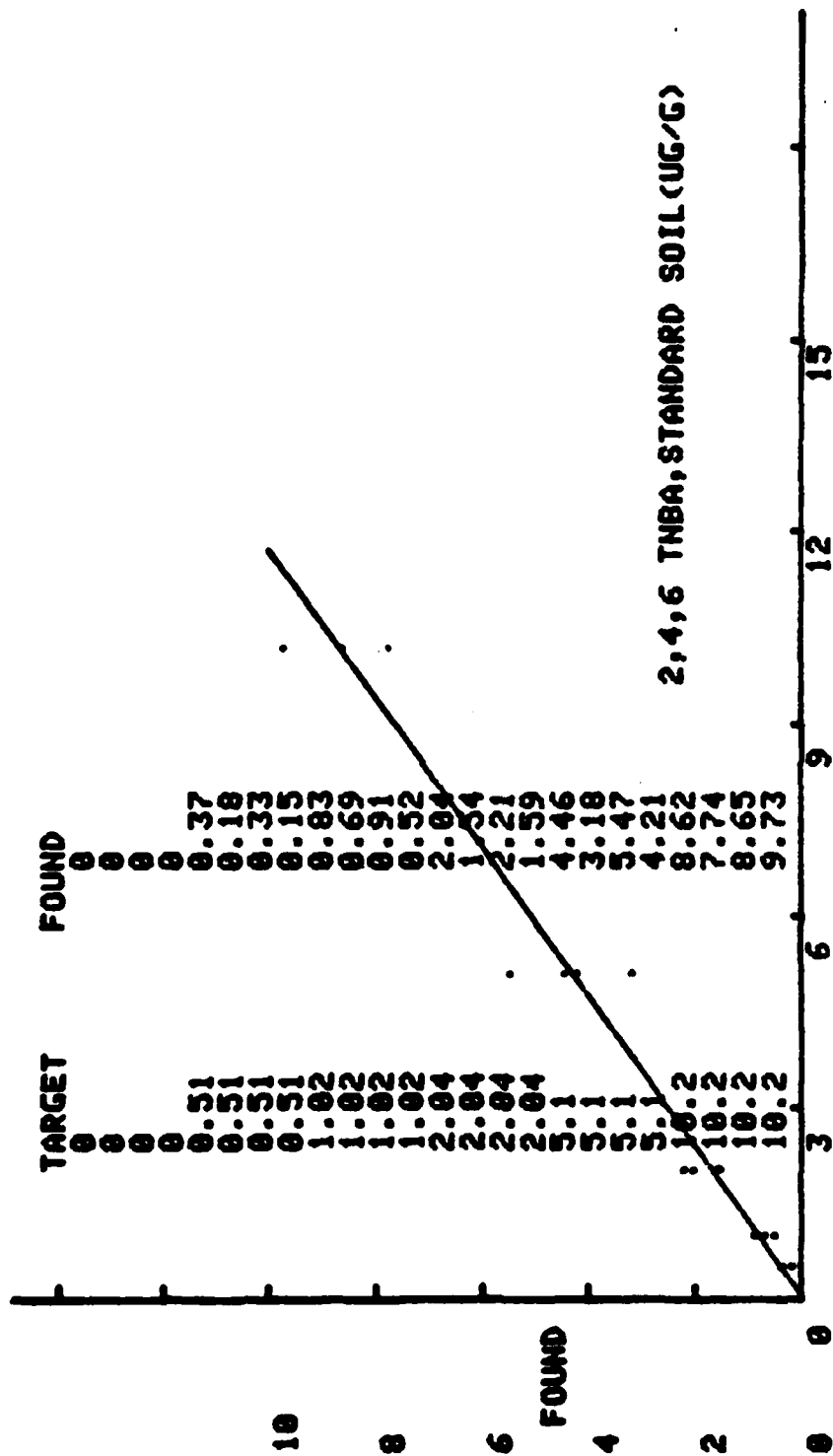
2,4,6 TBA, NATURAL SOIL (UG/G)

TARGET CONCENTRATION	DAY			
	1	2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.510	0.490	0.140	0.230	0.450
1.00	0.830	0.860	0.810	0.810
2.04	1.96	1.77	1.80	1.75
5.10	5.34	3.29	5.32	3.01
10.2	9.47	7.88	11.3	6.74

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.510	0.322	0.165	51.1	-36.7647
1.00	0.827	0.0236	2.86	-18.8726
2.04	1.82	0.0956	5.25	-10.7843
5.10	4.24	1.26	29.6	-16.8627
10.2	8.85	1.98	22.4	-13.2598



Chromatogram of ATNBA and 135TNB (Decomposition Product)
under the Conditions Given in Section 3B



CORR. COEFF. = 0.9885
 DETECTION LIMIT = 1.99144
 TARGET FOUND = -0.0602+ 0.859353*TARGET

3.4.6 TARA STANDARD SOIL (UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.310	0.370	0.180	0.330	0.150
1.02	0.830	0.690	0.910	0.520
2.04	2.04	1.54	2.21	1.59
5.10	4.46	3.18	5.47	4.21
10.2	8.62	7.74	8.65	9.73

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.310	0.257	0.109	42.2	-49.5058
1.02	0.737	0.171	23.2	-27.6961
2.04	1.84	0.331	18.0	-9.5588
5.10	4.33	0.940	21.7	-15.0980
10.2	8.68	0.815	9.38	-14.8529

35DNP IN WATER SAMPLES

35DNP IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNP.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.61 to 12.2 ug/L.

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNP	69,000	510

The normalized response (integrator counts) at the standard water detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNP	58,000	431

C. DETECTION LIMIT

The detection limit in natural water calculated according to Hubaux and Vos (1970), is 4.5 ug/L. The detection limit in standard water is 3.8 ug/L.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 340 nm and are extractable from water with methylene chloride. Interferences are minimized by a base-neutral pre-extraction of the sample and a Florisil® Sep-Pak® cleanup.

AMD.3/DINH20.2
7/22/82

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

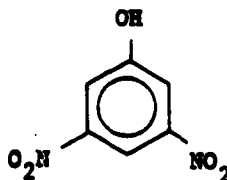
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
35DNP	None	586-11-8

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Acid Dissociation Constant</u>
35DNP	$C_6H_3O_5N_2$	126	$pk_a = 6.7$

Chemical Structure



C. CHEMICAL REACTIONS

35DNP undergoes normal phenolic reactions such as acid dissociation. No reactions which adversely affected the stability of the compound were observed.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual pump liquid chromatograph equipped with an Altex Model 153 UV detector and 340-nm filter interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 fixed-wavelength detector equipped with a 340-nm filter. ($\lambda = 340 \text{ nm}$)
2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)
Particle size: 5 μm
3. Flow Rate/Mobile Phase: 1 ml/min
30% water/70% methanol/
0.03 M H_3PO_4
4. Temperature: 25°C
5. Injection Volume: 250 μl , fixed loop
6. Retention Time: 6.0 minutes

C. HARDWARE/GLASSWARE

1. 1-L Teflon® separatory funnels with screw caps (6);
2. Short-stemmed glass funnels (6);
3. 500-ml K-D evaporative flasks, acid washed (6);
4. 3-ball Snyder columns (6);
5. 2-ball micro-Snyder columns (6);
6. 10-ml graduated centrifuge tubes (6);
7. 5-ml glass syringes with Luer-lock tips (6); and
8. 1-liter graduated cylinders (6).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Acid-washed, anhydrous sodium sulfate (reagent grade)--
prepared as follows: in a 500-ml, round-bottom flask,
slurry 100 g of anhydrous sodium sulfate with 200 ml of
diethyl ether containing 0.1 ml of concentrated sulfuric
acid. Attach the flask to a rotary evaporator and remove
the ether by vacuum evaporation. Store the treated sodium
sulfate at 130°C.

5. Acid-treated glass wool--Supelco 2-0383;
6. 6N NaOH--weigh out 240 g of reagent-grade NaOH pellets and dissolve in 1 L of organic-free water;
7. 85% phosphoric acid, reagent grade;
8. 6N HCl--dilute concentrated HCl 1:1 with water;
9. Florisil® Sep-Paks®--Waters Associates;
10. Sodium chloride, reagent grade;
11. Teflon® boiling chips; and
12. ColorpHast® pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The stock calibration standard (1.22 mg/ml) is prepared by weighing 12.2 mg of 35DNP into a 10-ml volumetric flask, dissolving in a few ml of methanol, and diluting with methanol to the mark.
2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with the HPLC mobile phase (30% water/70% methanol/0.03 M H_3PO_4) as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Stock Calibration Standard	1	50
B	Standard A	5	10
C	Standard A	1	10
D	Standard A	2	25
E	Standard A	1	25
F	Standard A	1	50

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	24.4
B	12.2
C	2.44
D	1.95
E	0.976
F	0.488

B. CONTROL SPIKES

1. Use Working Calibration Standard A as the control spike solution.
2. Measure 900 ml of water into a 1-L Teflon® separatory funnel.
3. Pipet a known amount of the control spike solution into the 900-ml sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Water (ug/L)</u>
0.05	1.36
0.10	2.71
0.20	5.42
0.50	13.6
1.00	27.1

4. Shake the sample to assure a homogeneous mixture before extraction.

5. PROCEDURE

A. EXTRACTION

1. Measure 900 ml of sample to be analyzed and pour into a 1-L separatory funnel.
2. Adjust the sample pH to 12 with 6N NaOH (approximately 3 ml). Add 100 g of NaCl to the sample. Shake the sample to dissolve the salt.

3. Add 100 ml of methylene chloride and shake for 1 minute. Allow the layers to separate at least 10 minutes. Centrifugation or placing the separatory funnel in an ultrasonic bath aids in breaking any emulsions.
4. Discard the methylene chloride extract.
5. Add 3 ml of 85% H_3PO_4 to the sample. Mix well and then adjust the pH to 3 by adding 6N NaOH (approximately 3.5 ml).
6. Extract the sample sequentially with three 100-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times.
7. Pass the extracts through a glass funnel containing approximately 20 g of acid-washed sodium sulfate and an acid-washed glass wool plug. Collect the extracts in an acid-washed K-D apparatus with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls in the Snyder column should chatter actively at the proper evaporation rate.
9. Let the apparent volume of extract in the receiver decrease to approximately 2 ml, then remove the receiver from the bath and let cool. Detach the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
10. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.

11. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
12. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Repeat the concentration by adding 10 ml of methanol and reconcentrating to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and reducing the volume to less than 1.0 ml.
13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver with 0.5 ml of HPLC-grade water. Add two drops of 85% phosphoric acid and raise the extract volume to exactly 2.0 ml in the centrifuge tube with HPLC-grade water.
14. Transfer to a 2-ml septum-sealed vial for storage at 4°C. The extract is now ready for HPLC analysis.

B. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the 35DNP peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNP according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the
sample extract by comparison with the appropriate
standard curve,

V_t = Volume of total extract (ml), and

V_s = Volume of initial sample extracted (L).

7. REFERENCES

None found.

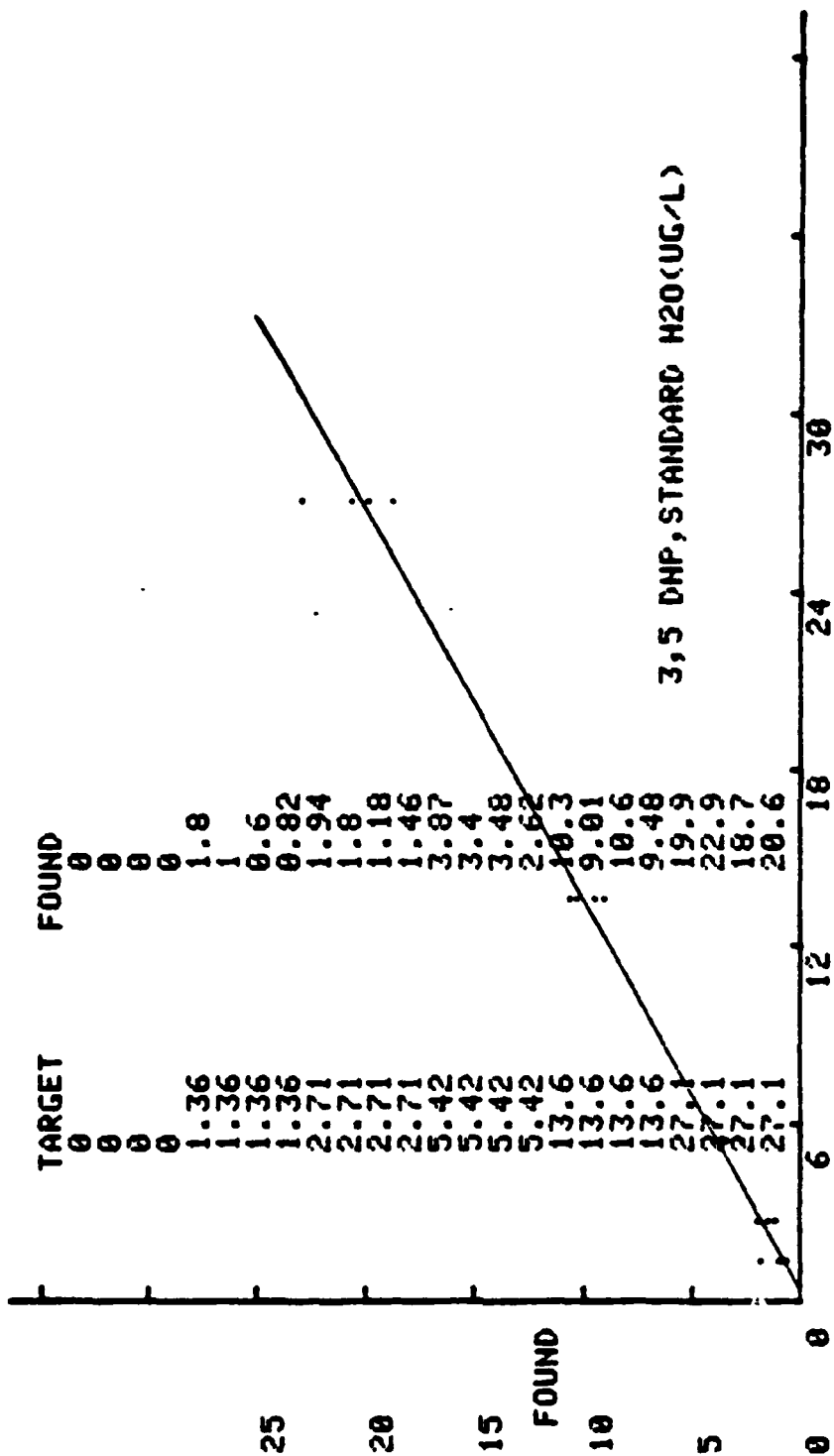
8. DATA

See attached data sheets.

3.5 DEF. STANDARD H2O (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.36	1.86	1.000	0.600	0.820
2.71	1.94	1.80	1.18	1.46
5.42	3.87	3.40	3.48	2.62
13.6	10.3	9.01	10.6	9.46
27.1	19.9	22.9	18.7	20.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.36	1.05	0.523	49.6	-22.4265
2.71	1.59	0.342	21.5	-41.1439
5.42	3.34	0.524	15.7	-38.3363
13.6	9.35	0.732	7.43	-27.5919
27.1	20.5	1.77	6.61	-24.2620

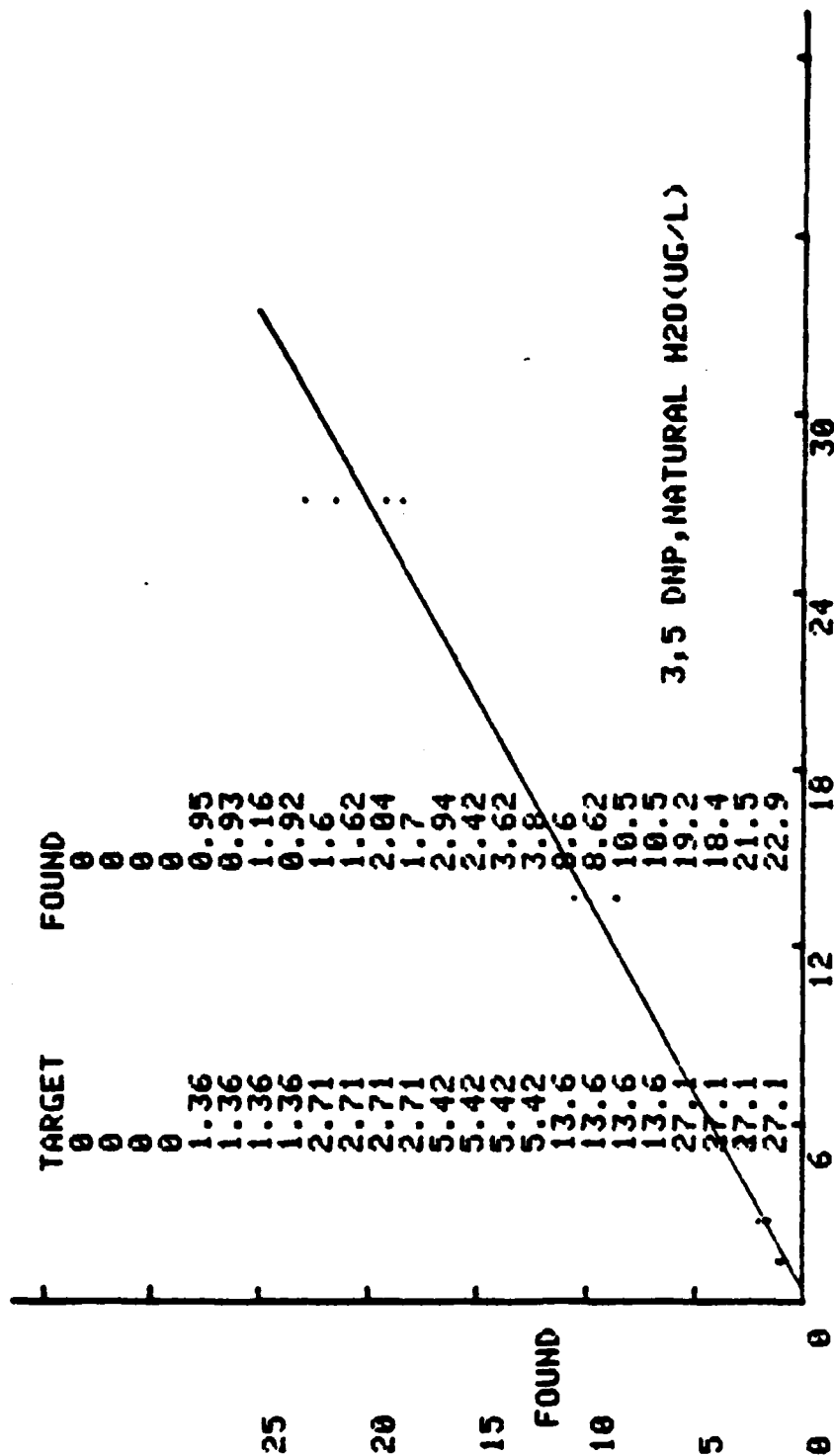


CORR. COEFF. = 0.9940
 DETECTION LIMIT = 3.79772
 TARGET
 FOUND = -0.3102+ 0.761630+TARGET

3,5 DNP, NATURAL H2O (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.36	0.950	0.930	1.16	0.920
2.71	1.60	1.62	2.04	1.70
5.42	2.94	2.42	3.62	3.80
13.6	8.60	8.62	10.5	10.5
27.1	19.2	18.4	21.5	22.9

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.36	0.990	0.114	11.5	-27.2059
2.71	1.74	0.205	11.8	-35.7934
5.42	3.19	0.636	19.9	-41.0517
13.6	9.55	1.09	11.4	-29.7426
27.1	20.5	2.07	10.1	-24.3543



CORR. COEFF. = 0.9916
 DETECTION LIMIT = 4.49756
 TARGET
 FOUND = -0.3444+
 TARGET = 0.758044x

35DNP IN SOIL SAMPLES

35DNP IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNP.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.61 to 12.2 ug/g.

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNP	125,596	850

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNP	140,160	950

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 1.7 ug/g. The detection limit in standard soil is 1.9 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 340 nm and are extractable from water with methylene chloride. Interferences are minimized by a water extraction of the sample and a Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

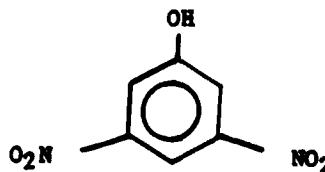
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
35DNP	None	586-11-8

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Acid Dissociation Constant</u>
35DNP	$C_6H_3O_5N_2$	126	$pK_a = 6.7$

Chemical Structure



C. CHEMICAL REACTIONS

35DNP undergoes normal phenolic reactions such as acid dissociation. No reactions which adversely affected the stability of the compound were observed.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector and 340-nm filter interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 fixed-wavelength detector equipped with a 340-nm filter ($\lambda = 340$ nm)
2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)
Particle size: 5 μ m
3. Flow Rate/Mobile Phase: 1 ml/min
30% water/70% methanol
0.03 M H_3PO_4
4. Temperature: 25°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 6.0 minutes

C. HARDWARE/GLASSWARE

1. 250-ml separatory funnels with stoppers, acid washed (6);
2. Short-stemmed glass funnels (6);
3. 500-ml K-D evaporative flasks, acid washed (6);
4. 3-ball Snyder columns (6);
5. 2-ball micro-Snyder columns (6);
6. 10-ml graduated centrifuge tubes (6);
7. 5-ml glass syringes with Luer-lock tips (6);
8. 1-L graduated cylinders (6); and
9. 50-ml centrifuge tubes with Teflon[®]-lined screw caps (6).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Acid-washed, anhydrous sodium sulfate (reagent grade)--
prepared as follows: in a 500-ml, round-bottom flask,
slurry 100 g of anhydrous sodium sulfate with 200 ml of
diethyl ether containing 0.1 ml of concentrated sulfuric
acid. Attach the flask to a rotary evaporator and remove
the ether by vacuum evaporation. Store the treated sodium
sulfate at 130°C.

5. Acid-treated glass wool--Supelco 2-0383;
6. 85% phosphoric acid, reagent grade;
7. 6N HCl-- dilute concentrated HCl 1:1 with water for acid washing glassware;
8. Florisil® Sep-Paks®--Waters Associates;
9. Sodium chloride, reagent grade;
10. Teflon® boiling chips; and
11. ColorpHast® pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The stock calibration standard (1.22 mg/ml) is prepared by weighing 12.2 mg of 35DNP into a 10-ml volumetric flask, dissolving in a few ml of methanol, and diluting with methanol to the mark.
2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with the HPLC mobile phase (30% water/70% methanol/0.03 M H_3PO_4) as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Stock Calibration Standard	5	25
B	Standard A	1	10
C	Standard A	0.5	10
D	Standard B	2	10
E	Standard B	1	10
F	Standard B	0.5	10
G	Standard C	0.5	10

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	244
B	24.4
C	12.2
D	4.88
E	2.44
F	1.22
G	0.61

B. CONTROL SPIKES

1. Use Working Calibration Standard A as the control spike solution.
2. Weigh out 20.0 g of soil into a 50-ml centrifuge tube.
3. Pipet a known amount of the control spike solution (dissolved in a sufficient volume of HPLC-grade water to just wet the soil) into the centrifuge tube. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Volume of Control Spike Solution (ml)</u>	<u>Concentration of Spiked Soil (ug/L)</u>
0.05	0.61
0.10	1.22
0.20	2.44
0.50	6.1
1.00	12.2

4. Shake the sample to mix and let set 1 hour to air dry.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let soil sample air dry on dull side of aluminum foil until it can be sieved through a 30-mesh sieve.

2. Sample the sieved soil by quartering and place 20.0 g into a 50-ml centrifuge tube.

B. EXTRACTION

1. Add 35 ml of HPLC-grade water to the centrifuge tube and mix thoroughly by shaking for 3 minutes. Centrifuge the sample at approximately 3,000 rpm for 15 minutes, and decant the water layer into a 250-ml separatory funnel.
2. Repeat Step 1 twice more and combine the extracts.
3. Adjust the pH of the water extracts to 3 with 85% phosphoric acid.
4. Extract the water extracts sequentially with three 80-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times.
5. Pass the methylene chloride extracts through a glass funnel containing approximately 20 g of acid-washed sodium sulfate and an acid-washed glass wool plug. Collect the extracts in an acid-washed K-D apparatus with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
6. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls in the Snyder column should chatter actively at the proper evaporation rate.
7. Let the apparent volume of extract in the receiver decrease to approximately 2 ml, then remove the receiver from the bath and let cool. Detach the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
8. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional

1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.

9. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
10. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Repeat the concentration by adding 10 ml of methanol and reconcentrating to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and reducing the volume to less than 2.0 ml.
11. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver with 2-ml of HPLC-grade water. Add two drops of 85% phosphoric acid and raise the extract volume to exactly 10.0 ml in the centrifuge tube with HPLC-grade water.
12. Transfer a portion of the sample to a 2-ml septum-sealed vial for storage at 4°C. The extract is now ready for HPLC analysis.

B. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the 35DNP peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNP according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{(W_s)}$$

where: A = Concentration (ug/ml) of analyte found in the
sample extract by comparison with the appropriate
standard curve,

V_t = Volume of total extract (ml), and

W_s = Weight of initial sample extracted (g).

7. REFERENCES

None found.

8. DATA

See attached data sheets.

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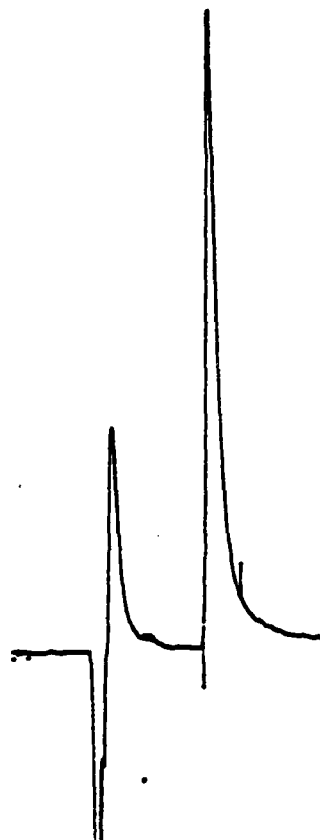
1.

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Chromatogram of an Extract of a Natural Soil Sample
Spiked with 35DNP at 1.2 ug/g

3,5 DINITROPHENOL, STD. SOIL (UG/G)

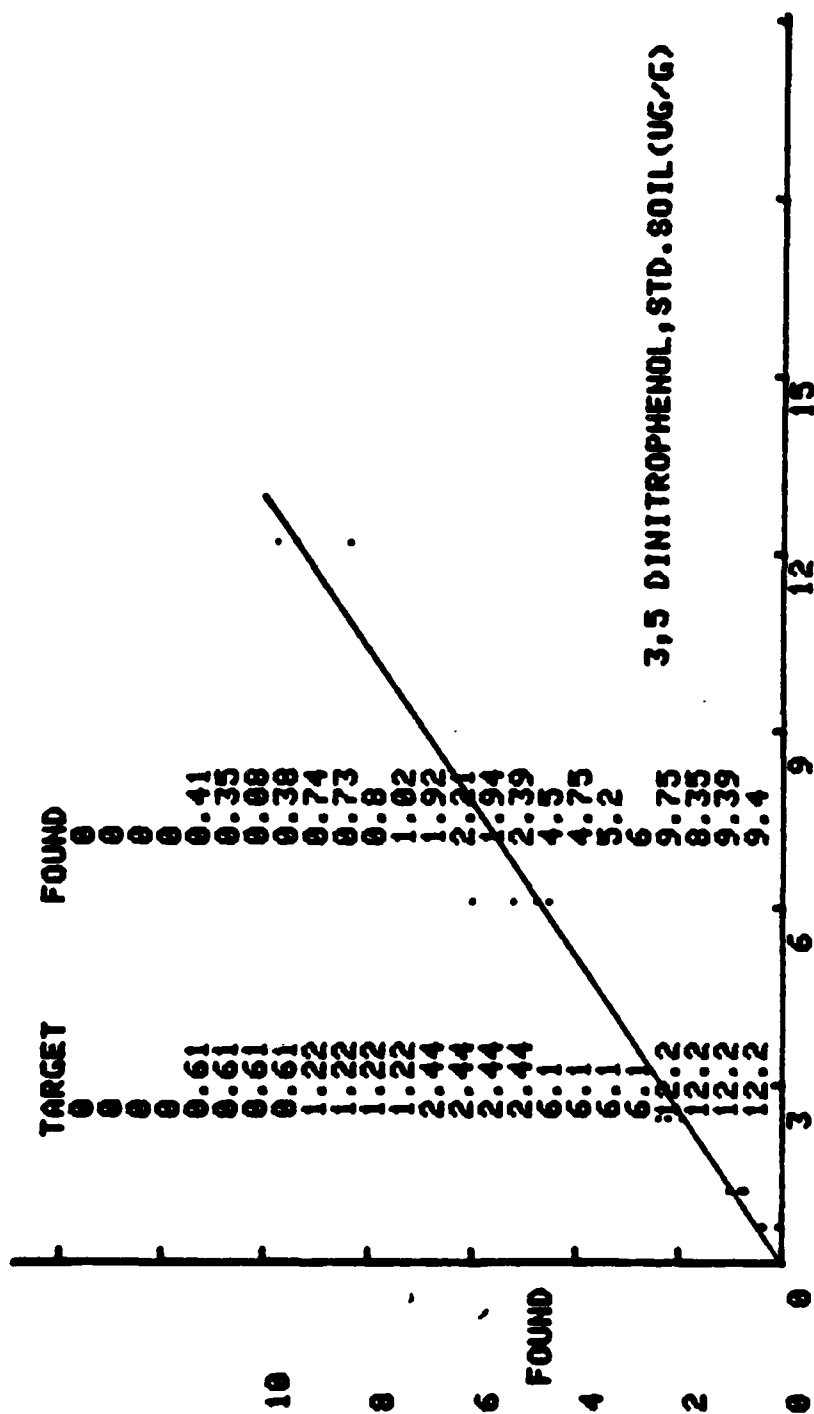
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.610	0.410	0.350	0.0800	0.380
1.22	0.740	0.730	0.800	1.02
2.44	1.92	2.21	1.94	2.35
6.10	4.50	4.75	5.20	6.00
12.2	9.75	8.35	9.39	9.40

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.610	0.305	0.152	49.8	-50.0000
1.22	0.822	0.135	16.4	-32.5820
2.44	2.11	0.226	10.7	-13.3197
6.10	5.11	0.659	12.9	-16.1685
12.2	9.22	0.605	6.56	-24.4057

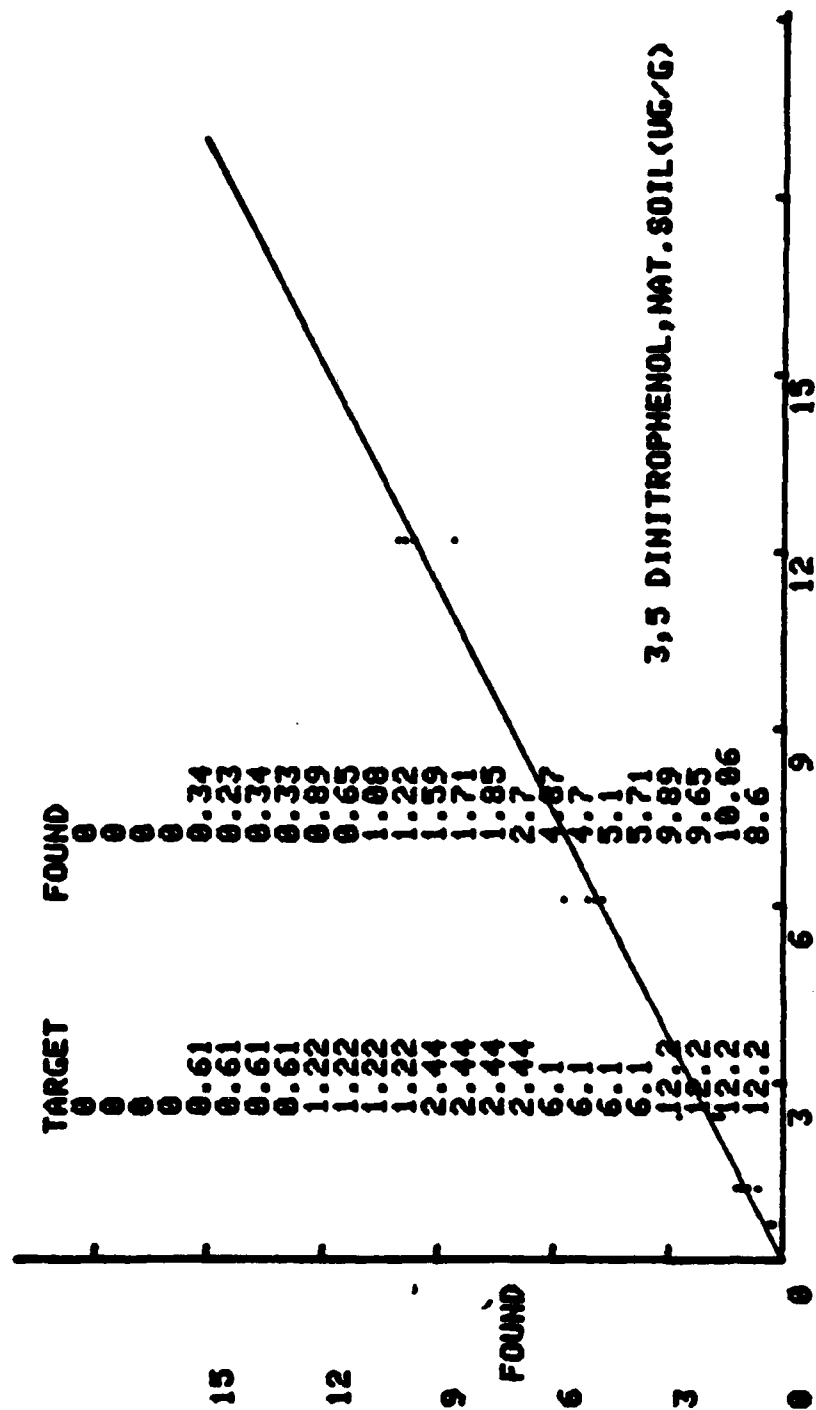
3,5 DINITROPHENOL, NA1. SOIL (UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.610	0.340	0.230	0.340	0.330
1.22	0.890	0.650	1.08	1.22
2.44	1.59	1.71	1.85	2.70
6.10	4.87	4.70	5.10	5.71
12.2	9.89	9.65	10.1	8.60

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.610	0.310	0.0535	17.3	-49.1803
1.22	0.960	0.247	25.7	-21.3115
2.44	1.96	0.503	25.6	-19.5697
6.10	5.09	0.442	8.67	-16.4754
12.2	9.55	0.655	6.86	-21.7213



TARGET
 CORR. COEFF. = 0.9926 FOUND = 0.0321+ 0.770261 * TARGET
 DETECTION LIMIT = 1.89494



TARGET

CORR. COEFF. = 0.9941 FOUND = -0.0062+ 0.793745 TARGET

DETECTION LIMIT = 1.69672

35DNA IN WATER SAMPLES

35DNA IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for 35DNA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water is 0.58 to 11.7 ug/L.

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNA	89,900	342

The normalized response (integrator counts) at the standard water detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNA	31,300	120

C. DETECTION LIMIT

The detection limit in natural water calculated according to Hubaux and Vos (1970), is 3.0 ug/L. The detection limit in standard water is 1.1 ug/L.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile basic and neutral organic compounds which absorb light at 395 nm and are extractable from water with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

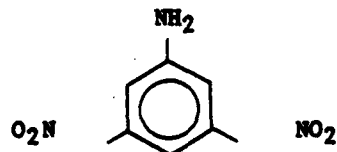
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
35DNA	3,5-dinitroaniline, 1-amino-3,5-dinitrobenzene	618-87-1

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Acid Dissociation Constant</u>
35DNA	$C_6H_5O_4N_3$	160-162	$pK_a = 0.23$

Chemical Structure



C. CHEMICAL REACTIONS

35DNA reacts as a weak base.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 395 nm)
2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm
Particle size: 5 μ m)
3. Flow Rate/Mobile Phase: 1 ml/min
30% water/70% methanol
4. Temperature: 25°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 5.6 minutes

C. HARDWARE/GLASSWARE

1. 1-L glass separatory funnels with stoppers (8);
2. Short-stemmed glass funnels (8);
3. 500-ml K-D evaporative flasks (8);
4. 25-ml graduated K-D receivers (8);
5. 3-ball Snyder columns (8);
6. 2-ball micro-Snyder columns (8);
7. 10-ml graduated centrifuge tubes (8);
8. 5-ml glass syringes with Luer-lock attachments(8);
9. 1-L graduated cylinders (8); and
10. 100-ml graduated cylinder (1).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Anhydrous sodium sulfate, reagent grade;
5. Glass wool;
6. Teflon® boiling chips;
7. Florisil® Sep-Paks®--Waters Associates;

8. 6N sodium hydroxide--weigh out 240 g of reagent-grade NaOH pellets and dissolve in 1 L of HPLC-grade water; and
9. ColorpHast® pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The 35DNA stock calibration standard (1.05 mg/ml) is prepared by weighing 10.5 mg of 35DNA into a 10-ml volumetric flask, dissolving the 35DNA in a few ml of methanol, and diluting with methanol to the mark.
2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with 50% methanol/50% water as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Stock Calibration Standard	1	100
B	Standard A	5	10
C	Standard A	1	10
D	Standard A	2	25
E	Standard A	1	25
F	Standard A	1	50

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	10.5
B	5.25
C	1.05
D	0.84
E	0.42
F	0.21

B. CONTROL SPIKES

1. Use Working Calibration Standard A as the control spike solution.
2. Measure 900 ml of water into a 1-L separatory funnel.
3. Pipet a known amount of the control spike solution into the 900-ml sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Spike Volume (ml)</u>	<u>Concentration of Spiked Water (ug/L)</u>
0.05	0.58
0.10	1.17
0.20	2.33
0.50	5.83
1.00	11.67

4. Shake the sample to assure a homogeneous mixture before extraction.

5. PROCEDURE

A. EXTRACTION

1. Measure 900 ml of sample to be analyzed and pour into a 1-L separatory funnel.
2. Adjust the sample pH to 12 with 6N NaOH (approximately 3 ml).
3. Extract the sample sequentially with three 100-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times. Centrifuge or sonicate to separate any resulting emulsions.
4. Pass the extracts through a glass funnel containing approximately 20 g of sodium sulfate and a glass wool plug. Collect the extracts in a K-D apparatus equipped with a 10-ml receiver. Rinse the sodium sulfate with approximately

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20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.

5. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls should chatter actively at the proper evaporation rate.
6. Let the apparent volume of extract decrease to approximately 2 ml, then remove from the bath, and let cool. Remove the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
7. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.
8. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
9. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver, and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Add an additional 10 ml of methanol and reconcentrate to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and concentrating to a volume of approximately 0.5 ml.
10. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver quantitatively with 0.5 ml of HPLC-grade water. Raise the extract volume to exactly 2.0 ml in the centrifuge tube with HPLC-grade water.
11. Transfer to a 2-ml septum-sealed vial for storage at 4°C.
12. The extract is now ready for HPLC analysis.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the 35DNA peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNA according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

V_s = Volume of initial sample extracted (L).

7. REFERENCES

None found.

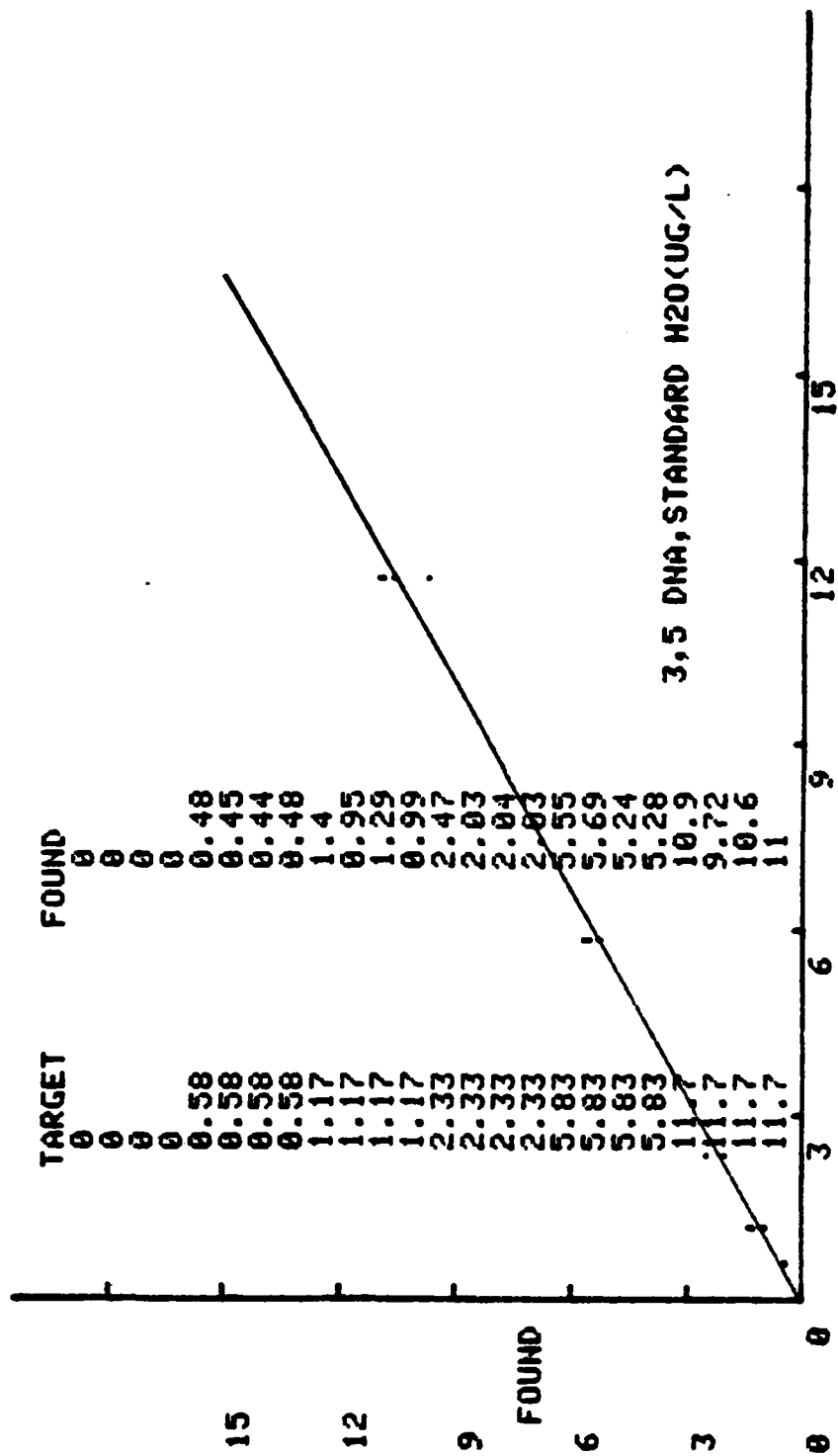
8. DATA

See attached data sheets.

3.5 DIA. STANDARD H₂O (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.560	0.480	0.450	0.440	0.480
1.17	1.40	0.950	1.25	0.990
2.33	2.47	2.03	2.04	2.03
5.66	5.55	5.69	5.24	5.28
11.7	10.9	9.72	10.6	11.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.560	0.462	0.0206	4.46	-20.2586
1.17	1.16	0.222	19.2	-1.0684
2.33	2.14	0.218	10.2	-8.0472
5.66	5.44	0.216	3.97	-6.6895
11.7	10.6	0.582	5.51	-9.7863

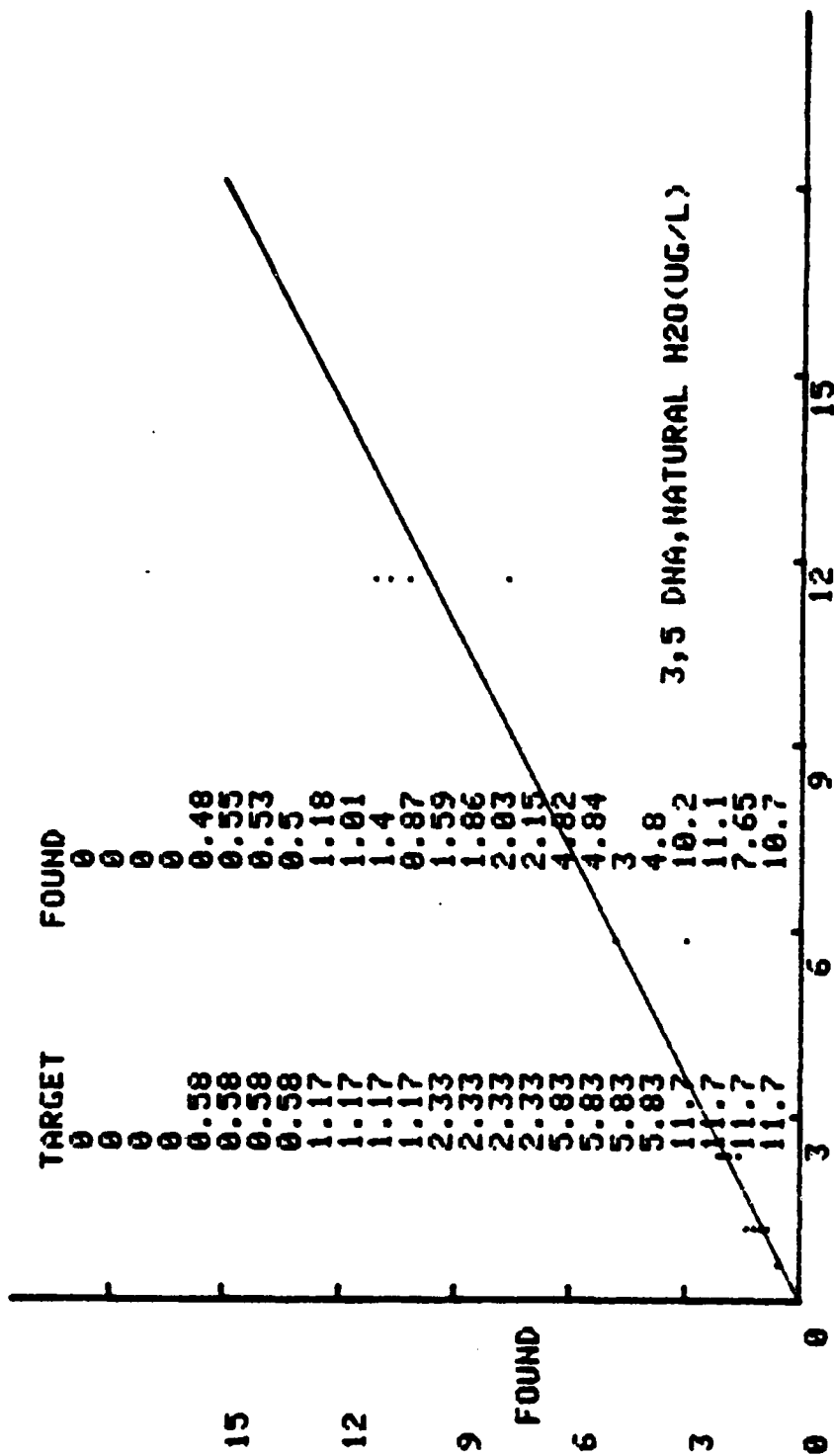


CORR. COEFF. = 0.9976
 DETECTION LIMIT = 1.03759
 TARGET FOUND = 0.0334
 TARGET = 0.904994

3.5 DNA, NATURAL H2O (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.500	0.480	0.550	0.530	0.500
1.17	1.18	1.01	1.40	0.870
2.33	1.59	1.86	2.03	2.15
5.83	4.82	4.84	3.00	4.80
11.7	10.2	11.1	7.65	10.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.500	0.515	0.0311	6.04	-11.2069
1.17	1.11	0.228	20.5	-4.7009
2.33	1.91	0.243	12.7	-18.1331
5.83	4.36	0.910	20.9	-25.1267
11.7	9.91	1.55	15.7	-15.2778



COR. COEFF. = 0.9804
 DETECTION LIMIT = 2.99386

TARGET FOUND = -0.0259+ 0.831576 * TARGET

35DNA IN SOIL SAMPLES

35DNA IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.525 to 10.5 ug/g.

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNA	167,078	550

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1(C) is listed below:

<u>Analyte</u>	<u>Integrator Counts</u>	<u>Nanograms</u>
35DNA	79,742	263

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 1.1 ug/g. The detection limit in standard soil is 0.53 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 395 nm and are extractable from water with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

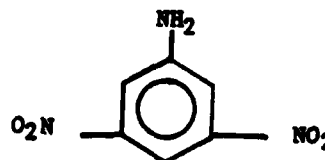
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
35DNA	3,5-dinitroaniline, 1-amino-3,5-dinitrobenzene 3,5-dinitrobenzenamine	618-87-1

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Acid Dissociation Constant</u>
35DNA	$C_6H_5O_4N_3$	160-162	$pK_a = 0.23$

Chemical Structure



C. CHEMICAL REACTIONS

35DNA reacts as a weak base.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 395 nm)
2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm)
Particle size: 5 μ m
3. Flow Rate/Mobile Phase: 1 ml/min
30% water/70% methanol
4. Temperature: 25°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 5.6 minutes

C. HARDWARE/GLASSWARE

1. 250-ml glass separatory funnels with stoppers (6);
2. Short-stemmed glass funnels (6);
3. 500-ml K-D evaporative flasks (6);
4. 10-ml graduated K-D receivers (6);
5. 3-ball Snyder columns (6);
6. 2-ball micro-Snyder columns (6);
7. 10-ml graduated centrifuge tubes (6);
8. 5-ml glass syringes with Luer-lock attachments (6);
9. 50-ml centrifuge tubes with Teflon[®]-lined screw caps; and
10. 100-ml graduated cylinder (1).

D. CHEMICALS

1. Nanograde methylene chloride;
2. HPLC-grade methyl alcohol--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. Anhydrous sodium sulfate, reagent grade;
5. Glass wool;
6. Teflon[®] boiling chips;
7. Florisil[®] Sep-Paks[®]--Waters Associates;

4. STANDARDS

A. CALIBRATION STANDARDS

1. The 35DNA stock calibration standard (1.05 mg/ml) is prepared by weighing 10.5 mg of 35DNA into a 10-ml volumetric flask, dissolving the 35DNA in a few ml of methanol, and diluting with methanol to the mark.
2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with 50% methanol/50% water as follows:

<u>Working Calibration Standard</u>	<u>Standard Diluted</u>	<u>Volume of Standard Used (ml)</u>	<u>Final Volume (ml)</u>
A	Stock Calibration Standard	5	25
B	Standard A	1	10
C	Standard A	0.5	10
D	Standard A	0.2	10
E	Standard A	0.1	10
F	Standard A	0.05	10
G	Standard D	1	10

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>
A	210
B	21.0
C	10.5
D	4.2
E	2.1
F	1.05
G	0.42

B. CONTROL SPIKES

1. Use Working Calibration Standard A as the control spike solution.
2. Weigh out 20.0 g of soil into a 50-ml centrifuge tube.
3. Pipet a known amount of the control spike solution (dissolved in a sufficient volume of HPLC-grade water to just wet the soil) into the centrifuge tube. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

<u>Volume of Control Spike Solution (ml)</u>	<u>Concentration of Spiked Water (ug/L)</u>
0.05	0.525
0.10	1.05
0.20	2.10
0.50	5.25
1.00	10.5

4. Shake the sample to mix and let air dry for 1 hour.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let soil sample air dry on dull side of aluminum foil until it can be sieved through a 30-mesh sieve.
2. Sample the soil by quartering and place 20.0 g into a 50-ml centrifuge tube.

B. EXTRACTION

1. Add 35 ml of HPLC-grade water to the centrifuge tube and shake for 3 minutes. Centrifuge the sample at approximately 3,000 rpm for 15 minutes and decant the water layer into a 250-ml separatory funnel.
2. Repeat Step 1 twice more and combine the extracts.
3. Extract the water extracts sequentially with three 80-ml portions of methylene chloride using 2-minute shake times

and 10-minute separation times. Centrifuge to separate any resulting emulsions.

4. Pass the methylene chloride extracts through a glass funnel containing approximately 20 g of sodium sulfate and a glass wool plug. Collect the extracts in a K-D apparatus equipped with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
5. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls should chatter actively at the proper evaporation rate.
6. Let the apparent volume of extract decrease to approximately 2 ml, then remove from the bath, and let cool. Remove the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
7. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.
8. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
9. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver, and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Add an additional 10 ml of methanol and reconcentrate to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and concentrating to a volume of approximately 2.0 ml.
10. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing

the receiver quantitatively with 2.0 ml of HPLC-grade water. Raise the extract volume to exactly 10.0 ml in the centrifuge tube with HPLC-grade water.

11. Transfer to a 2-ml septum-sealed vial for storage at 4°C.
12. The extract is now ready for HPLC analysis.

B. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample using the conditions given in Section 3(B).
3. Measure the response of the 35DNA peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNA according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample extract by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and

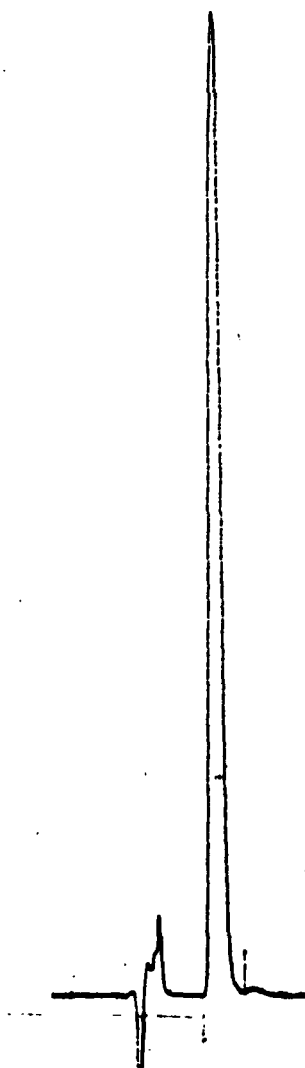
W_s = Weight of initial sample extracted (g).

7. REFERENCES

None found.

8. DATA

See attached data sheets.



Chromatogram of the Extract of a Natural Soil Sample
Spiked with ³⁵DNA at 2.1 ug/g

3,5 DINITROANILINE, STD. SOIL (UG/G)

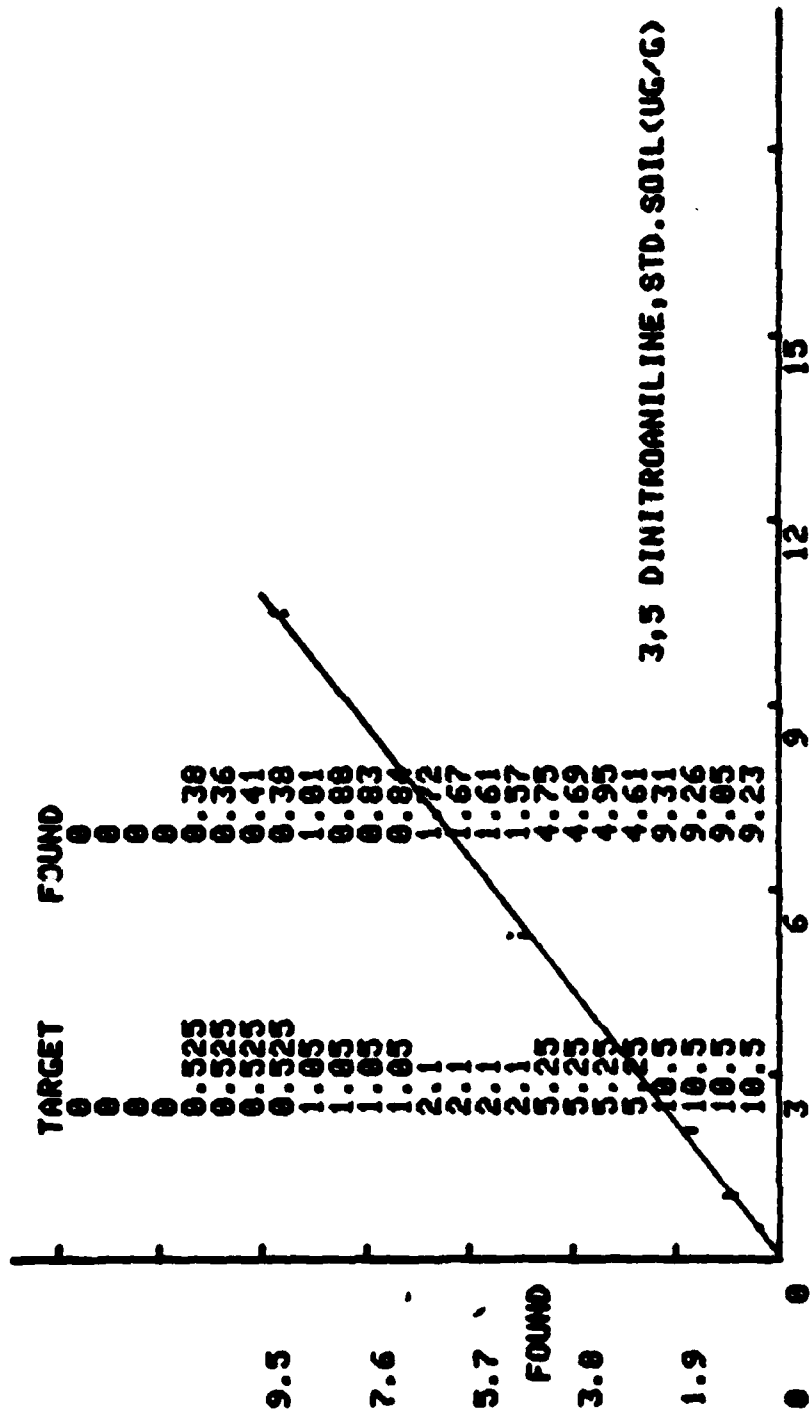
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.525	0.380	0.360	0.410	0.380
1.05	1.01	0.880	0.830	0.840
2.10	1.72	1.67	1.61	1.57
5.25	4.75	4.69	4.95	4.61
10.5	9.31	9.26	9.05	9.23

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.525	0.382	0.0206	5.39	-27.1429
1.05	0.890	0.0829	9.31	-15.2381
2.10	1.64	0.0660	4.02	-21.7857
5.25	4.75	0.145	3.06	-9.5238
10.5	9.21	0.113	1.23	-12.2619

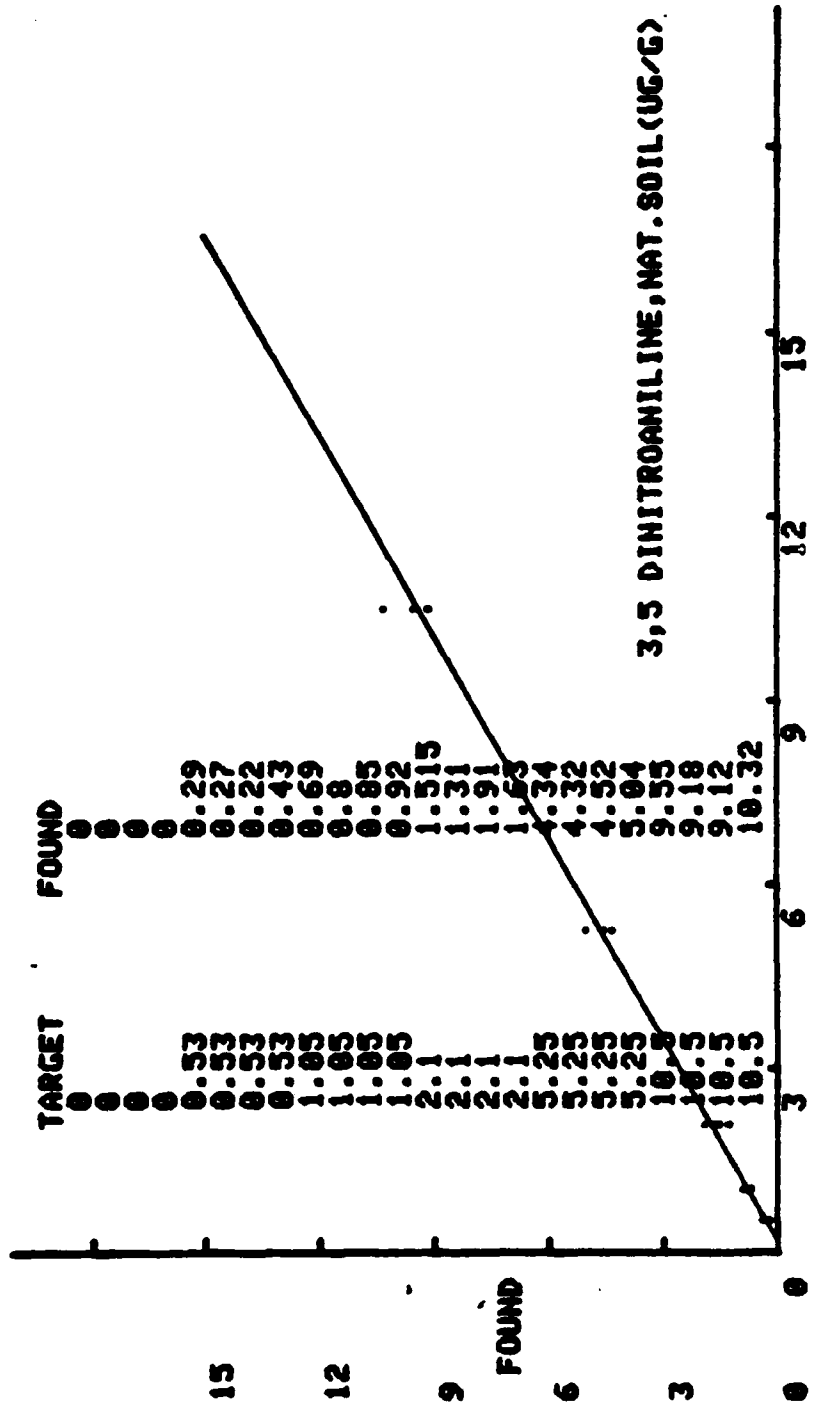
3,5 DINITROANILINE,NAT.SOIL(UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.530	0.290	0.270	0.220	0.430
1.05	0.690	0.800	0.850	0.920
2.10	1.51	1.31	1.91	1.63
5.25	4.34	4.32	4.52	5.04
10.5	9.55	9.18	9.12	10.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.530	0.302	0.0900	29.7	-42.9245
1.05	0.815	0.0968	11.9	-22.3810
2.10	1.59	0.250	15.7	-24.2262
5.25	4.55	0.336	7.37	-13.2381
10.5	9.54	0.552	5.79	-9.1191



CORR. COEFF. = 0.9993 FOUND - TARGET
 DETECTION LIMIT = 0.525 -0.0596+ 0.087256xTARGET



CORR. COEFF. = 0.9967
 DETECTION LIMIT = 1.00175
 TARGET FOUND = -0.1707+ 0.917605 TARGET

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TDGCL IN WATER SAMPLES

TDGCL IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for TDGCL.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural water is 40 to 800 ug/L.

B. SENSITIVITY

The normalized response (peak height in mm times attenuation) at the detection limit designated in Section 1(C) is 4,736 mm for 2,975 ng of TDGCL.

C. DETECTION LIMIT

The detection limit in natural water, calculated according to Hubaux and Vos (1970), is 119 ug/L.

D. INTERFERENCES

This method may be subject to interferences from highly water-soluble compounds which absorb light at 215 nm. Because of the polarity of glycols, it is not possible to concentrate them quantitatively by solvent extraction; therefore, their determination must be carried out in the aqueous phase. TDGCL is concentrated by boiling off the water taking advantage of its high boiling point (165°C). A cleanup of the sample is achieved by column chromatography on Amberlite® XAD-7 resin which removes some of the UV-absorbing interferences occurring in natural surface waters. These interferences have not been observed in ground waters examined and seem to be generated by hydrolysis of high molecular weight substances (possibly humic and fulvic acids) occurring in surface waters.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 6 extracts in an 8-hour day. One analyst can perform 6 extractions and boildowns in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
TDGCL	2,2'-Thiodiethanol Bis (b-hydroxyethyl) Sulfide	111-48-8

B. PHYSICAL AND CHEMICAL PROPERTIES

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml)</u>
TDGCL	C ₄ H ₁₀ O ₂ S	-10	165	1.1819

C. CHEMICAL REACTIONS

None found.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 gradient liquid chromatograph (HPLC) equipped with a Perkin-Elmer LC-75 variable-wavelength, UV-visible detector and interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 215 nm)
2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm)
Particle Size: 5 μ m

3. Flow Rate and Mobile Phase: 1 ml/min of phosphate buffer solution, pH = 3.0
4. Temperature: 22°C
5. Injection Volume: 250 ul, fixed loop
6. Retention Time: 11.8 minutes

C. **HARDWARE/GLASSWARE**

1. 1,000-ml separatory funnel (Teflon® or glass) (6);
2. 600-ml beaker (6);
3. 10-cm diameter watch glass (6);
4. 50-ml beaker (6); and
5. 10-ml graduated centrifuge tubes (6).

D. **CHEMICALS AND REAGENTS**

1. Nanograde methylene chloride--J.T. Baker Company;
2. HPLC-grade acetonitrile--J.T. Baker Company;
3. HPLC-grade water--J.T. Baker Company;
4. 6N sodium hydroxide;
5. 6N sulfuric acid; and
6. Phosphate buffer--5 g of $\text{NH}_4\text{H}_2\text{PO}_4$ and 1 ml of 85% H_3PO_4 in HPLC-grade water (pH = 3.0).

4. **STANDARDS**

A. **CALIBRATION STANDARDS**

1. Prepare a stock calibration standard (10 mg/ml) by weighing out 100 mg of TDGCL into a single 10-ml volumetric flask and diluting to volume with HPLC-grade water. Wrap the flask in foil and store at 4°C.
2. Prepare a dilute stock calibration standard by pipetting 1 ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.

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3. Prepare the working calibration standards by making dilutions of the dilute stock calibration standard using the appropriate volumetric flasks and HPLC-grade water as follows:

<u>Working Calibration Standard</u>	<u>Concentration (ug/ml)</u>	<u>Volume of Dilute Stock Taken (ml)</u>	<u>Final Volume (ml)</u>
B	4.0	1	50
C	8.0	2	50
D	16	4	50
E	40	10	50
F	80	20	50

B. CONTROL SPIKES

1. Prepare the stock control spike standard by weighing 100 mg of TDGCL, transferring to a 10-ml volumetric flask, and diluting to volume with HPLC-grade water.
2. Prepare the working control spike standard by pipetting 2 ml of the stock control spike standard into a 100-ml volumetric flask and diluting to volume with HPLC-grade water.
3. Pipet known amounts of the working control spike standard into standard water. The quantity spiked should be selected to provide a concentration range of 0.5 to 10 times the detection limit.
4. Determine the accuracy and detection limit by pipetting the following amounts of the working control spike standard into 500 ml of standard water and analyzing according to the procedure outlined in Section 5:

<u>Volume of Working Control Spike Standard Spiked (ml)</u>	<u>Concentration of TDGCL (ug/L)</u>
0	0
0.100	40
0.200	80
0.400	160
1.00	400
2.00	800

5. PROCEDURE

A. BOILDOWN AND COLUMN CLEANUP

1. It is important that the following procedures be performed in one 8-hour day.
2. Measure 500 ml of the water sample into a 1-L beaker.
3. Add a boiling chip (Teflon®) and concentrate the sample by boiling the water on a hot plate to a volume of 50 ml. The boildown time should be as rapid as possible and should not exceed 2.5 hours.
4. Adjust the pH of the sample to 3 and transfer the sample to a column (20 cm x 1 cm) packed with Amberlite® XAD-7 resin. The resin is prepared by shaking 50 g of resin with 100 ml of methanol for 15 minutes on a wrist-action shaker. The methanol is decanted, and the operation is repeated sequentially with three 100-ml portions of methanol followed by four 100-ml portions of HPLC-grade water. The column is slurry packed in water. The column flow rate is controlled at a rate of 1 ml/min, and the eluate is collected in a 250-ml beaker. The column is rinsed with 50 ml of HPLC-grade water into the same beaker to give a total volume of approximately 100 ml.
5. The volume of the solution is further reduced by boiling to less than 25 ml, and then quantitatively transferred with rinsing into a 50-ml beaker.
6. The volume of the solution is then reduced to less than 5 ml by boiling on a hot plate.
7. Transfer the solution into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade water. Dilute to the 5 ml mark with HPLC-grade water.
8. Filter the sample through a 0.45-um filter and transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
9. The solution is now ready for chromatography by HPLC.

B. CALIBRATION

1. Inject the Working Calibration Standards B, C, D, E, and F and a blank singly at the beginning of the analytical run. Inject the Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized peak heights versus nanograms injected of each standard to obtain a working curve.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the component of interest.

6. CALCULATIONS

Determine the concentration of TDGCL according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where: A = Nanograms of TDGCL found in the sample by comparison with the appropriate standard curve,

V_t = Final volume of solution (ml),

V_s = Volume of initial sample extracted (ml), and

V_i = Volume injected (ml).

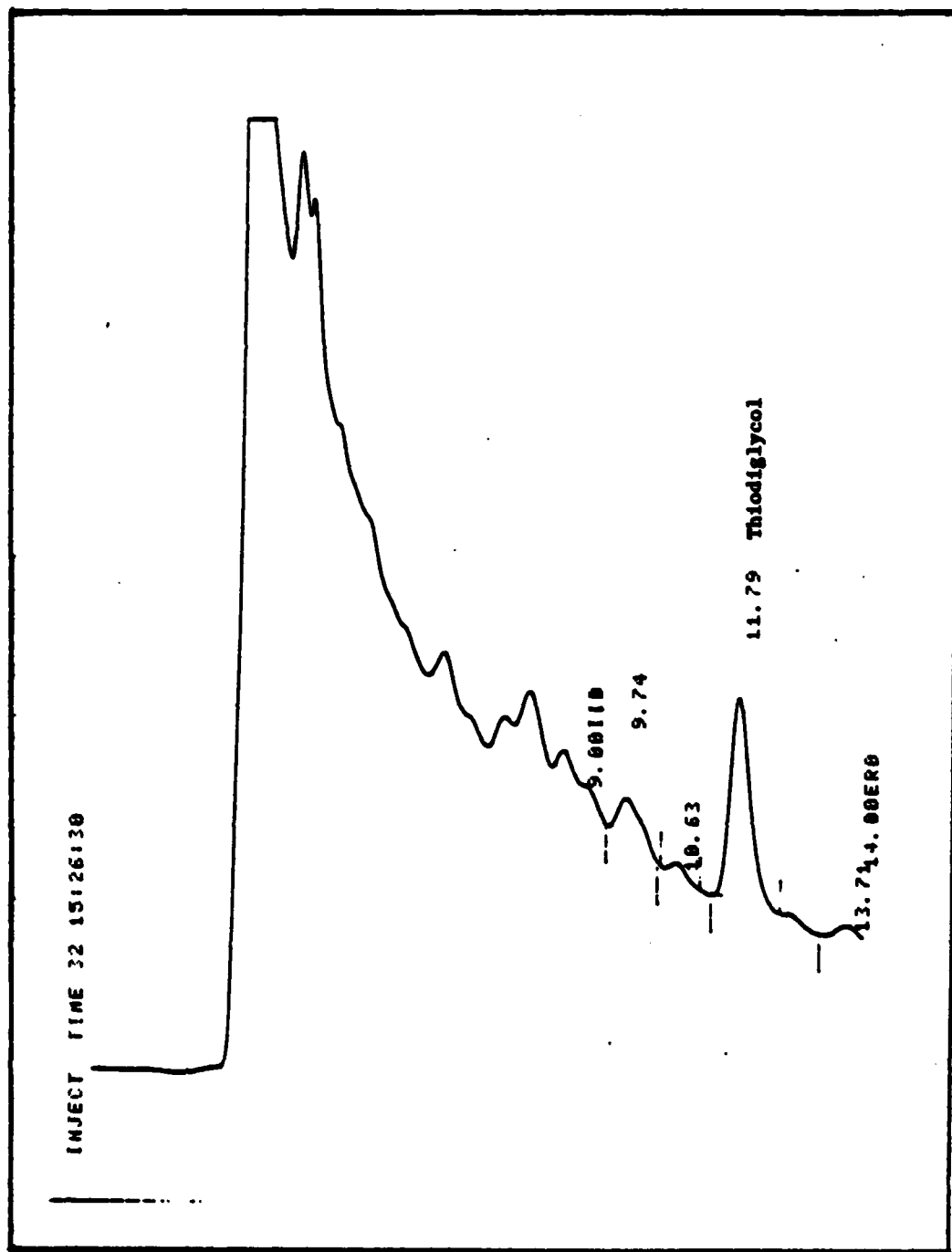
7. REFERENCES

None found.

8. DATA

See attached data sheets.

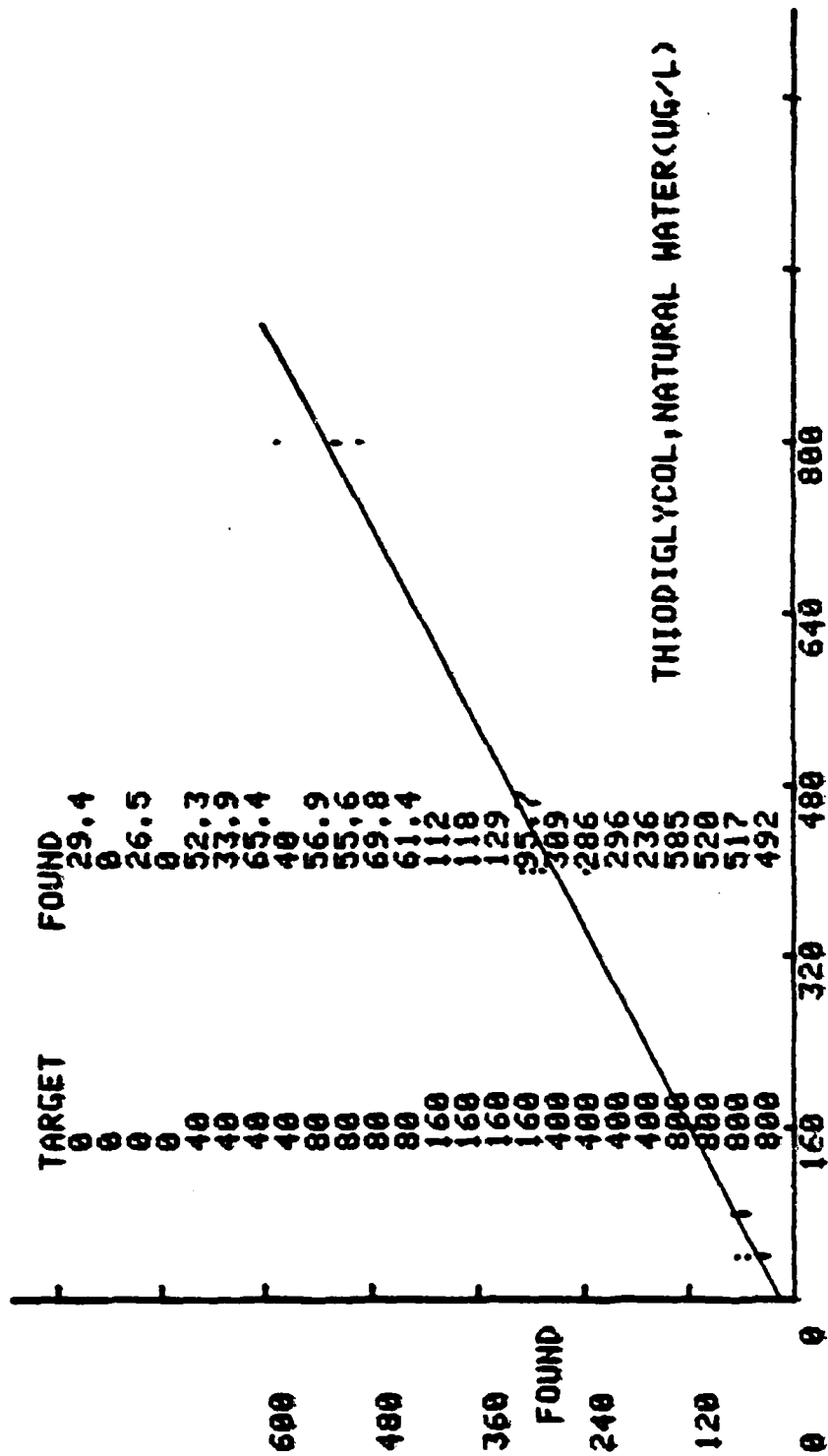
Figure 1
Chromatogram from the analysis of 500 ml of a surface water sample
spiked at 400 ppb.



THIODIGLYCOL, NATURAL WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	29.4	0.0000	26.5	0.0000
40.0	52.3	33.9	65.4	40.0
80.0	56.9	55.6	69.8	61.4
160	112	118	129	95.7
400	309	286	296	236
800	585	520	517	492

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	14.0	16.2	116	0.0000
40.0	47.9	14.0	29.1	19.7
80.0	60.9	6.42	10.5	-23.8436
160	114	13.9	12.2	-28.9531
400	282	31.9	11.3	-29.5625
800	529	39.7	7.51	-33.9375



CORR. COEFF. = 0.9933 FOUND = TARGET
 DETECTION LIMIT = 118.77774
 15.3111+ 0.645175*TARGET

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1.

TDGCL IN SOIL SAMPLES

TDGCL IN NATURAL SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil and sediment samples for TDGCL.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural soil is 1 to 20 ug/g.

B. SENSITIVITY

The normalized response (peak height in mm x attenuation) at the natural soil detection limit is 1,056 mm for 1.02 ug of TDGCL.

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 2.2 ug/g.

D. INTERFERENCES

This method may be subject to interferences from highly water-soluble compounds which absorb light at 215 nm. Because of the polarity of the glycols, it is not possible to extract them from soil quantitatively by organic solvent extraction; therefore, their determination is performed by extraction of the soil with water. TDGCL is concentrated by evaporation of the water by boiling, taking advantage of its high boiling point of 165°C. A cleanup of the sample is achieved by column chromatography of the acidified aqueous extract on Amberlite® XAD-7 resin. Partial sample cleanup is also achieved by using an acidic aqueous extraction; only highly water-soluble substances will be extracted. After column chromatography, the water is neutralized and boildown proceeds.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform 8 extractions and boildowns in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

<u>Analyte</u>	<u>Alternate Nomenclature</u>	<u>CAS Registry Number</u>
TDGCL	2,2'-Thiodiethanol Bis (B-hydroxyethyl) Sulfide	111-48-8

B. PHYSICAL AND CHEMICAL PROPERTIES

<u>Analyte</u>	<u>Formula</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>	<u>Density (g/ml)</u>
TDGCL	C ₄ H ₁₀ O ₂ S	-10	165	1.1819

C. CHEMICAL REACTIONS

None.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 gradient liquid chromatograph (HPLC) equipped with a Perkin-Elmer LC-75 variable-wavelength UV-visible detector and interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector
(λ = 215 nm)

2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm)
Particle Size: 5 μ m
3. Flow Rate and Mobile Phase: 1 ml/min phosphate buffer solution
4. Temperature: 22°C
5. Injection Volume: 250 μ l, fixed loop
6. Retention Time: 12.5 minutes

C. HARDWARE/GLASSWARE

1. 250-ml Erlenmeyer flasks with screw caps (8);
2. 600-ml beakers (8);
3. 10-cm-diameter watch glasses (8);
4. 50-ml beakers (8);
5. 10-ml graduated centrifuge tubes (8);
6. Burrell Model 75 wrist-action shaker (1); and
7. Glass chromatography columns (20 cm x 1-cm ID) (8).

D. CHEMICALS AND REAGENTS

1. HPLC-grade water--J.T. Baker Company;
2. 6N sodium hydroxide;
3. 6N sulfuric acid; and
4. Phosphate buffer--5.75 g of $\text{NH}_4\text{H}_2\text{PO}_4$ and 1 ml of 85% H_3PO_4 in 1 L of HPLC-grade water (pH = 3.0).

4. STANDARDS

A. CALIBRATION STANDARDS

1. Prepare a stock calibration standard (10.13 mg/ml) by weighing out 101.3 mg of TDGCL into a single 10-ml volumetric flask and diluting to volume with HPLC-grade water. Wrap the flask in foil and store at 4°C.
2. Prepare a dilute stock calibration standard by pipetting 1 ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.

7/22/82

3. Prepare the working calibration standards by making dilutions of the dilute stock calibration standard using the appropriate volumetric flasks and HPLC-grade water as follows:

<u>Working Calibration Standard</u>	<u>Volume of Dilute Stock Used (ml)</u>	<u>Final Volume (ml)</u>	<u>Concentration (ug/ml)</u>
B	0.5	50	2.03
C	1	50	4.05
D	2	50	8.10
E	5	50	20.3
F	10	50	40.5

B. CONTROL SPIKES

1. Prepare the stock control spike standard (10.13 mg/ml) by weighing 101.3 mg of TDGCL, transferring to a 10-ml volumetric flask, and diluting to volume with HPLC-grade water.
2. Prepare the working control spike standard by pipetting 1 ml of the stock control spike standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.
3. Pipet known amounts of the working control spike standard into standard soil. The quantity spiked should be selected to provide a concentration range of 0.5 to 10 times the detection limit.
4. Determine the accuracy and detection limit by pipetting the following amounts of the working control spike standard into 10 g of soil and analyzing according to the procedure outlined in Section 5:

<u>Volume of Working Control Spike Standard Spiked (ml)</u>	<u>Concentration of TDGCL (ug/g)</u>
0	0
0.05	1.0
0.1	2.0
0.2	4.0
0.5	10
1.0	20

5. PROCEDURE

A. SAMPLE PREPARATION

1. The soil sample should be air dried on the dull side of aluminum foil and then sieved through a 30-mesh sieve.

B. EXTRACTION

1. It is important that the following procedures be performed in one 8-hour day.
2. Measure 10 g of sieved, dried soil or wet sediment into a tared 250-ml centrifuge tube with screw cap.
3. Add 50 ml of HPLC-grade water and adjust the pH to 3 using 6N H₂SO₄.
4. Cap the tube, shake vigorously by hand for 5 minutes, and centrifuge at 2,250 rpm for 15 minutes.
5. The supernatant liquor is decanted and collected in a 250-ml beaker.
6. Step 5 is repeated twice. After collecting all supernatant water, the pH is adjusted to 3 using 6N H₂SO₄.
7. Quantitatively transfer contents of the 250-ml beaker to a glass column (20 cm x 1-cm ID) packed with Amberlite® XAD-7 resin. (The resin is previously prepared by shaking 50 g of the resin with 100 ml of methanol for 15 minutes on a wrist-action shaker. The methanol is decanted, and the operation is repeated sequentially with three 100-ml portions of methanol followed by four 100-ml portions of

HPLC-grade water. The column is slurry-packed in water. The resin must be cleaned after each sample and may be reused a total of three times.) Pass the sample through the column at maximum flow (approximately 4 ml/min), and collect in a 300-ml beaker. The column is rinsed and eluted to dryness with 50 ml of HPLC-grade water into the same beaker to give a total volume of approximately 250 ml.

8. Add a Teflon® boiling chip, reduce the volume of the water to less than 25 ml by boiling on a hot plate, and quantitatively transfer the solution into a 50-ml beaker.
9. Reduce the volume of the solution to less than 5 ml by boiling on a hot plate.
10. Transfer the solution into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade water, and dilute to the 5-ml mark with the same water.
11. Filter the sample through a 0.45-um filter, and transfer to a 5-ml, amber, septum-sealed vial for storage at 4°C.
12. The solution is now ready for HPLC analysis.

C. CALIBRATION

1. Inject Working Calibration Standards B, C, D, E, and F and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized integrator response versus ng/ul of each standard to obtain a working curve.

D. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the TDGCL peak.

6. CALCULATIONS

Determine the concentration of TDGCL according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where: A = Weight of TDGCL found in the sample by comparison with the appropriate standard curve (ng),

V_t = Final volume of solution (ml),

V_s = Weight of initial sample extracted (g), and

V_i = Volume injected (ul).

7. REFERENCES

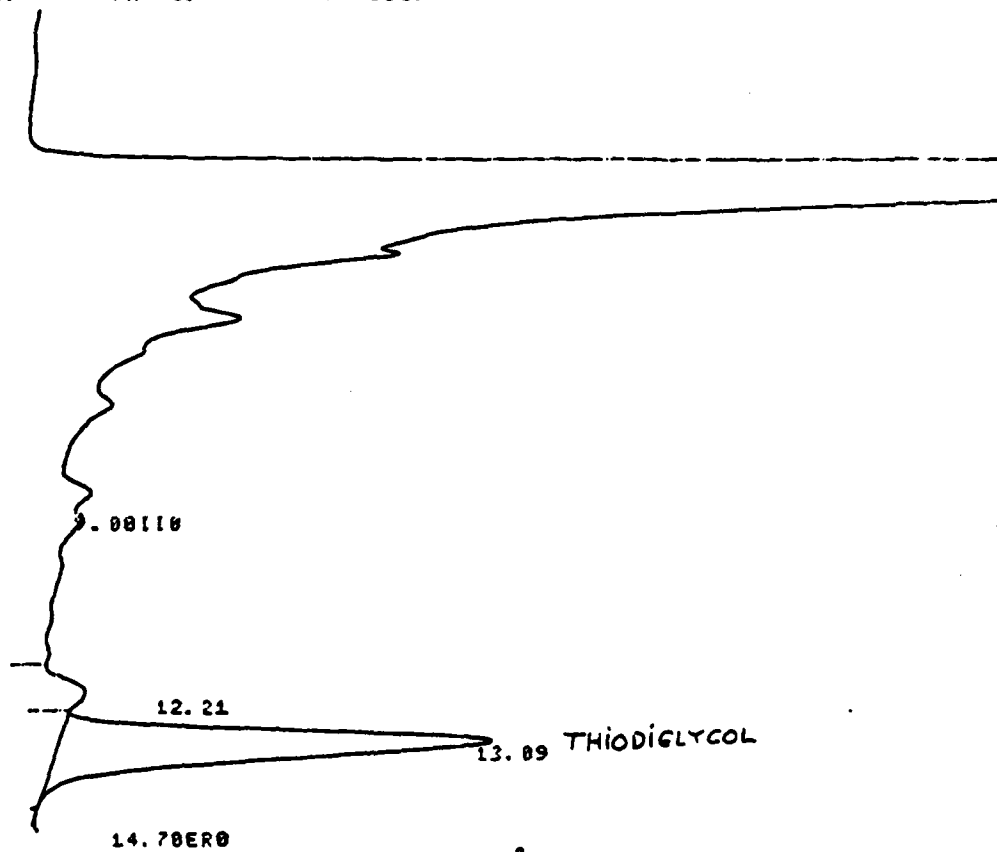
None found.

8. DATA

See attached data sheets.

Chromatogram of Natural Soil Extract
Spiked with Thiodiglycol

INJECT TIME 11 11:31:37
AT= 64. PN= 6. PT= 306.

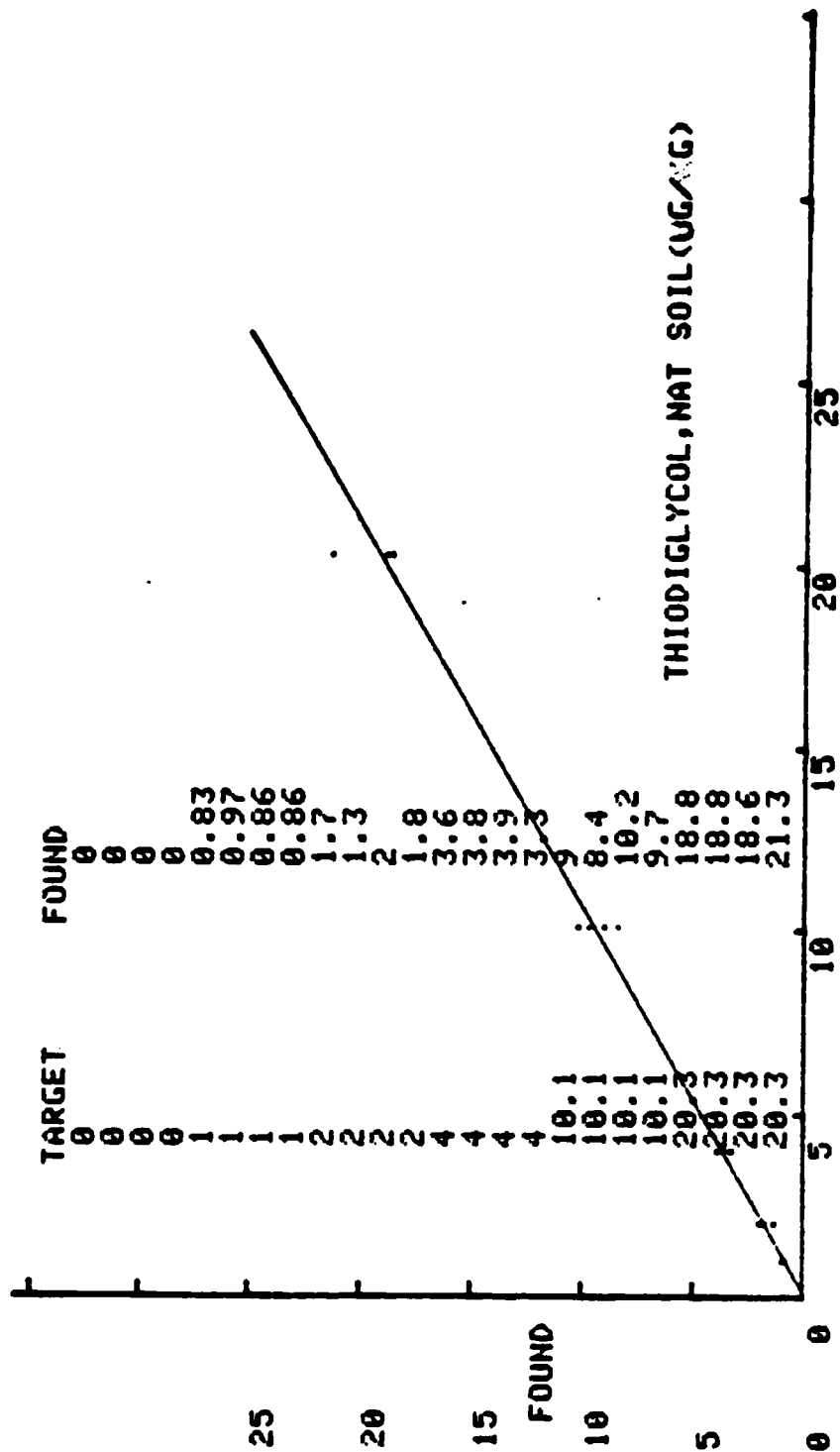


5X 5PME Thiodiglycol

THIODIGLYCOL-NAT SOIL (UG/ G)

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.000	0.830	0.970	0.860	0.860
2.00	1.70	1.30	2.00	1.80
4.00	3.60	3.80	3.90	3.30
10.1	9.00	8.40	10.2	9.70
20.3	18.8	18.8	18.6	21.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.00	0.880	0.0616	7.01	-12.0000
2.00	1.70	0.294	17.3	-15.0000
4.00	3.65	0.265	7.25	-8.7500
10.1	9.32	0.789	8.46	-7.6733
20.3	19.4	1.29	6.64	-4.5567



CORR. COEFF. = 0.9966
 DETECTION LIMIT = 2.15007
 TARGET
 FOUND = -0.1365+
 0.955861 * TARGET

Index

1.

1. **Introduction**

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HPLC SCREEN OF WATER SAMPLES FOR
NITROSUBSTITUTED MUNITION COMPOUNDS AND PAHs

HPLC SCREEN OF WATER SAMPLES
FOR NITROSUBSTITUTED MUNITION COMPOUNDS AND PAHs

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for nitrosubstituted munition organics and PAHs by HPLC. Detector ratios are employed to provide data for the qualitative identification of the analytes. Quantitative documentation was performed on the analytes listed in Table 1 using the detectors and wavelengths indicated. Documentation was performed for samples with and without silica-gel chromatography cleanup.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water for each analyte is listed in Table 2.

B. SENSITIVITY

The normalized responses (integrator peak height counts) at the standard water detection limits (without silica-gel cleanup) designated in Section 1(c) are listed in Table 3.

The normalized responses (integrator peak height counts) at the natural water detection limits (without silica-gel cleanup) designated in Section 1(c) are listed in Table 4.

C. DETECTION LIMIT

The detection limits (DL) in standard and natural water, calculated according to Hubaux and Vos (1970), are listed in Table 5.

The detection limits for standard water samples cleaned by silica-gel chromatography are listed to indicate the quality

Table 1. Analytes and Quantitative Detection Methods

Analyte	Quantitative Detection Method
HMX	UV (230 nm)
RDX	UV (230 nm)
135TNB	UV (230 nm)
13DNB	UV (254 nm)
35DNP	UV (254 nm)
246TNT	UV (254 nm)
26DNT	UV (230 nm)
24DNT	UV (254 nm)
Naphthalene	UV (280 nm)
Acenaphthylene	UV (280 nm)
Acenaphthene	UV (280 nm)
Phenanthrene	UV (280 nm)
Anthracene	UV (254 nm)
Fluoranthene	UV (280 nm)
Pyrene	UV (280 nm)
Chrysene	UV (280 nm)
Benzo(b)fluoranthene	Fluorescence (λ ex 290 nm, λ em > 350 nm)
Benzo(k)fluoranthene	Fluorescence (λ ex 290 nm, λ em > 350 nm)
Benzo(a)pyrene	UV (254 nm)
Indeno(1,2,3-cd)pyrene	UV (280 nm)

Source: ESE, 1982.

Table 2. Tested Concentration Ranges in Natural and Standard Water

Analyte	Concentration Range (ug/L)
HMX	2.0 to 40
RDX	2.0 to 48
135TNB	1.9 to 38
13DNB	2.2 to 44
35DNP	3.6 to 73
246TNT	2.0 to 40
26DNT	2.3 to 46
24DNT	2.0 to 40
Naphthalene	4.1 to 82
Acenaphthylene	6.6 to 131
Acenaphthene	1.5 to 48
Phenanthrene	1.5 to 30
Anthracene	1.2 to 24
Fluoranthene	0.4 to 8.0
Pyrene	1.9 to 38
Chrysene	0.10 to 2.0
Benzo(b)fluoranthene	0.10 to 2.1
Benzo(k)fluoranthene	0.21 to 4.1
Benzo(a)pyrene	0.22 to 4.5
Indeno(1,2,3-cd)pyrene	0.28 to 5.8

Source: ESE, 1982.

Table 3. Sensitivity at Standard Water Detection Limits
Without Cleanup

Analyte	Integrator Peak Height Counts	Nanograms
HMX	2,354	74
RDX	466	30
135TNB	985	30
13DNB	507	31
35DNP	605	56
246TNT	226	29
26DNT	392	46
24DNT	299	29
Naphthalene	1,406	137
Acenaphthylene	514	109
Acenaphthene	272	49
Phenanthrene	593	26
Anthracene	1,786	11
Fluoranthene	185	4.3
Pyrene	241	38
Chrysene	78	2.0
Benzo(b)fluoranthene	633	8.3
Benzo(k)fluoranthene	520	5.8
Benzo(a)pyrene	323	4.2
Indeno(1,2,3-cd)pyrene	164	9.1

Source: ESE, 1982.

Table 4. Sensitivity at Natural Water Detection Limits Without Cleanup

Analyte	Integrator Peak Height Counts	Nanograms
HMX	1,550	48
RDX	464	30
135TNB	357	32
13DNB	871	53
35DNP	1,869	173
246TNT	265	34
26DNT	307	36
24DNT	419	40
Naphthalene	1,047	103
Acenaphthylene	661	140
Acenaphthene	279	51
Phenanthrene	526	24
Anthracene	3,959	23
Fluoranthene	305	8
Pyrene	218	33
Chrysene	60.2	1.6
Benzo(b)fluoranthene	200	2.8
Benzo(k)fluoranthene	619	7.0
Benzo(a)pyrene	722	9.8
Indeno(1,2,3-cd)pyrene	222	12

Source: ESE, 1982.

Table 5. Detection Limits in Standard and Natural Water*

Analyte	Standard Water (ug/L)	Standard Water (After Silica-Gel Cleanup) (ug/L)	Natural Water (ug/L)
HMX	15	11	10
RDX	6	8	6
135TNB	6	6	6
13DNB	6	8	11
35DNP	11	28	34
246TNT	6	11	7
26DNT	9	12	7
24DNT	6	7	7
Naphthalene	27	33	21
Acenaphthylene	22	35	28
Acenaphthene	10	12	10
Phenanthrene	5	5	5
Anthracene	2	5	5
Fluoranthene	0.9	1	2
Pyrene	8	6	7
Chrysene	0.4	0.5	0.3
Benzo(b)fluoranthene	2	0.3	0.6
Benzo(k)fluoranthene	1	0.8	1
Benzo(a)pyrene	0.8	0.9	2
Indeno(1,2,3-cd)pyrene	2	2	2

* Calculated according to Hubaux and Vos, 1970.

Source: ESE, 1982.

of the cleanup technique for each analyte. The detection limits listed for standard water without silica-gel cleanup are the actual detectability criteria to be used for the analytes. The silica-gel cleaned extracts are analyzed only when a suspected target analyte is detected in the noncleaned extracts and when large unresolved background interferences prevent adequate quantitation.

D. INTERFERENCES

This method may be subject to interferences from organic compounds which are extractable from acidic water with methylene chloride. The qualitative analysis via detector ratios should be sufficient to prevent misidentification of unknown peaks. The silica-gel cleanup step provides a mechanism for eliminating possible interferences from highly polar compounds such as fatty acids and carboxylic acids which may be present in the extract. An unidentified fluorescence peak occurred at 25.7 minutes during the standard water documentation. This peak did not appear in either the standard or natural water documentation and did not display a measurable absorbance at 254 or 230 nm. Therefore, the compound should not interfere in the method.

Several compounds were not fully documented because of co-elution with another analyte under the screen conditions, chemical instability, or experimental interferences. However, the retention times and detector ratios for most of these compounds were determined and are listed in Table 6.

E. ANALYSIS RATE

After instrument calibration, which requires approximately 6 hours, one analyst can analyze four extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

Table 6. Retention Times and Detector Ratios for Compounds which were not Adequately Resolved and/or Documented

Analyte	Problem	Retention Time (minutes)	Detector Ratio (Wavelength/Wavelength) (nm)
Tetryl	Co-elutes with nitrobenzene under certain column conditions	(24.4)* 29.5	3.14 (230/280)
NB	Co-elutes with tetryl under certain column conditions	24.8	0.701 (230/280)
ATNBA	Unstable	14.3	2.8 (230/280)
12DNT	Internal standard not documented	52.5	0.8518 (230/280)

* The peak for tetryl undergoes significant shifts in retention time depending on the condition of the ODS column used in the system. Under the preliminary column conditions for documentation, tetryl interfered with nitrobenzene (RT = 24.4 min). After a new ODS column was installed, the RT for tetryl had shifted (RT = 29.5 min).

Source: ESE, 1982.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBERS

The alternate nomenclature and CAS registry numbers for the analytes of interest are listed in Table 7.

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTES

The physical and chemical properties for the analytes of interest are listed in Table 8.

C. CHEMICAL REACTIONS

Caution should be used in handling all of these compounds, especially the standard materials. All of the nitrosubstituted compounds are either explosives or breakdown products of explosives. The PAHs are known carcinogens, teratogens, and mutagens.

3. APPARATUS

A. INSTRUMENTATION

The HPLC instrumentation (see Figure 1) is a gradient elution system with two columns connected in series with a variable-wavelength, UV-visible absorbance detector; a 254-nm absorbance detector; and a fluorescence detector.

An Altex Model 322 dual-pump liquid chromatograph was used as the gradient pumping system. After mixing, the elution solvent was passed through a guard column (5 cm by 4.6 mm) packed with silica gel (Fisher, 60 to 200 mesh) to presaturate the mobile phase with silica and therefore extend column life. The mobile phase was then passed through a 0.25- μ m filter to remove entrained particulates.

An Altex Model 500 autosampler was used as the injection system, and a guard column (5 cm by 4.6 mm) packed with Pelliguard LC-CN pellicular packing (40 μ m) was present in the system. Both the

Table 7. Alternate Nomenclature and CAS Registry Numbers

Analyte	Alternate Nomenclature	CAS Registry Number
HMX	Cyclotetramethylenetetranitramine Octahydro-1,3,5,7-tetrazocine 1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane Octogen	2691-41-0
RDX	Cyclotrimethylenetrinitramine Hexogen, T-4, Cyclonite, Hexahydro-1,3,4-trinitro-s-triazine	121-84-4
135TNB	sym-Trinitrobenzene benzite	25377-32-6
13DNB	m-Dinitrobenzene	99-65-01
35DNP		586-11-8
246TNT	sym-Trinitrotoluene 1-methyl-2,4,6-trinitrobenzene trotyl; Tolit, Trilit	118-96-7
26DNT		606-20-2
24DNT		121-14-2
Naphthalene		91-20-3
Acenaphthylene		208-96-8
Acenaphthene	Naphthyleneethylene	82-32-9
Phenanthrene		85-01-8
Anthracene	p-Naphthalene	120-12-7
Fluoranthene	1,2-Benzacenaphthene Idryl	86-73-7
Pyrene	Benzo(d,e,f)phenanthrene	129-00-0
Chrysene	1,2-Benzophenanthrene Benzo(a)phenanthrene	218-01-9

Table 7. Alternate Nomenclature and CAS Registry Numbers
(Continued, Page 2 of 2)

Analyte	Alternate Nomenclature	CAS Registry Number
Benzo(b)fluoranthene	3,4-Benzofluoranthene Benz(e)acephenanthrylene	205-99-2
Benzo(k)fluoranthene	11,12-Benzofluoranthene	207-08-9
Benzo(a)pyrene	3,4-Benzopyrene	50-32-8
Dibenzo(a,h)anthracene	1,2:5,6-Dibenzanthracene	53-70-3
Indeno(1,2,3-cd)pyrene	Indeno(1,2,3)pyrene 2,3-o-Phenylenepyrene	193-39-5

Source: ESE, 1982.

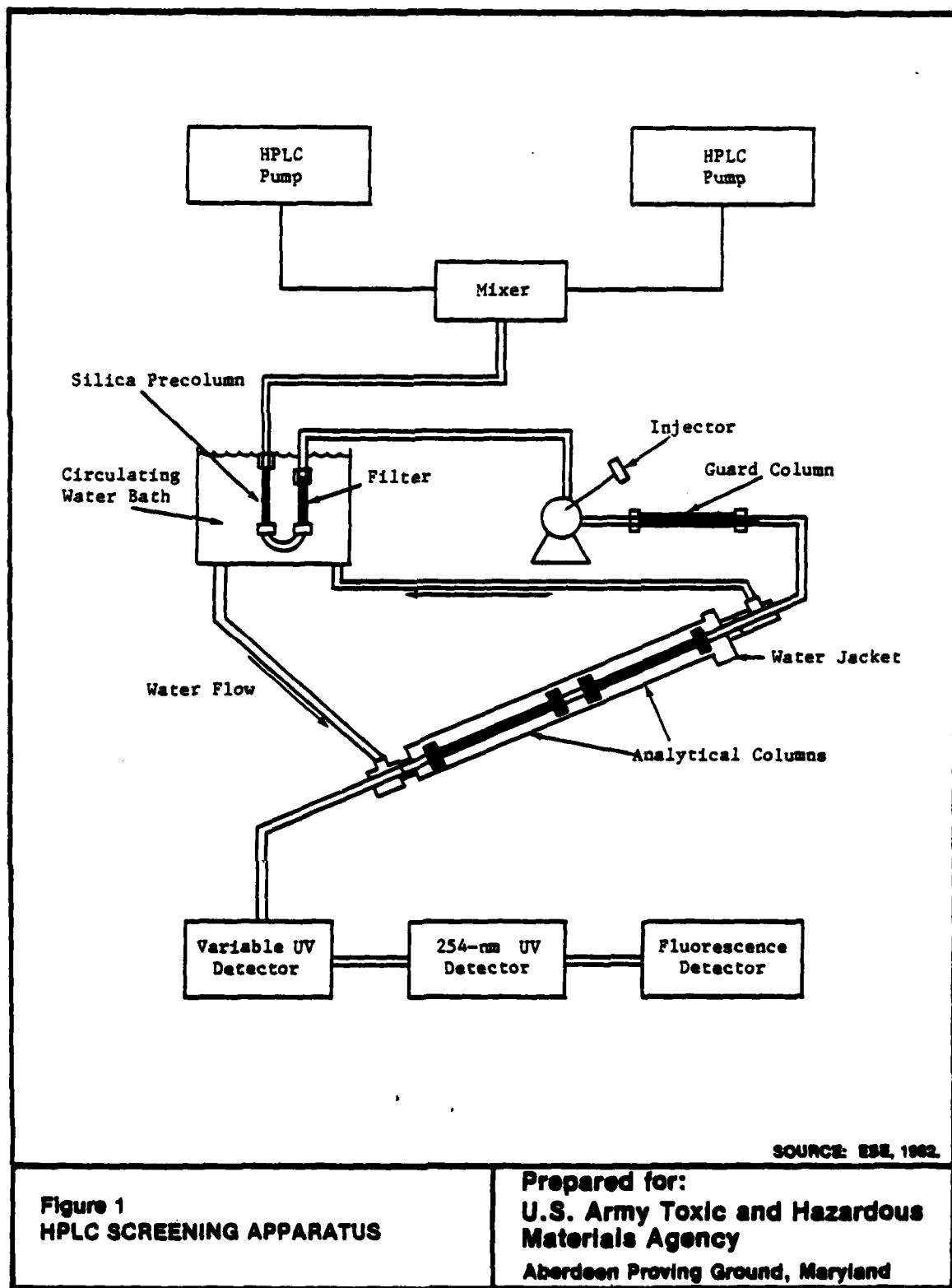
Table 8. Physical and Chemical Properties

Analyte	Formula	Melting Point (°C)	Boiling Point (°C)	Density (g/ml @ 20°C)
HMX	C ₄ H ₈ N ₈ O ₈	276	—	1.77-1.96*
RDX	C ₃ H ₆ N ₆ O ₆	204.1	—	1.816
135TNB	C ₆ H ₃ N ₂ O ₄	122	†	1.69
13DNB	C ₆ H ₄ N ₂ O ₄	90	302	1.57
35DNP	C ₆ H ₄ N ₂ O ₂	126	—	1.702
246TNT	C ₇ H ₅ N ₃ O ₆	80	280†	1.65
26DNT	C ₇ H ₅ N ₂ O ₄	66	—	1.28
24DNT	C ₇ H ₅ N ₂ O ₄	71	300†	1.442
Naphthalene	C ₁₀ H ₈	80.22	210.8 @ 720 torr	1.145
Acenaphthylene	C ₁₂ H ₈	92	265-275†	0.8988
Acenaphthene	C ₁₂ H ₁₀	96	278	1.225
Phenanthrene	C ₁₄ H ₁₀	101	340	1.182
Anthracene	C ₁₄ H ₁₀	216.2	345	1.25
Fluoranthene	C ₁₆ H ₁₀	111	375	1.252
Pyrene	C ₁₆ H ₁₀	149	404	1.271
Chrysene	C ₁₈ H ₁₀	254	448	1.274
Benzo(b)fluoranthene	C ₂₀ H ₁₂	167	—	—
Benzo(k)fluoranthene	C ₂₀ H ₁₂	217	—	—
Benzo(a)pyrene	C ₂₀ H ₁₂	179	312 @ 10 torr	—
Dibenzo(a,h)anthracene	C ₂₂ H ₁₄	269	—	—
Indeno(1,2,3-cd)pyrene	C ₂₂ H ₁₂	162.5	—	—

* There are four polymorphic structures of HMX with this range of densities.

† Decomposes.

Source: ESK, 1982.



injector and the guard column are maintained at room temperature. The silica precolumn and filter are maintained at the same temperature (52°C) as the analytical columns, which consist of an Ultrasphere CN 5- μ m column (25 cm by 4.6-mm ID) connected in series with an Ultrasphere ODS 5- μ m column (25 cm by 4.6-mm ID). The column temperature is maintained at 52°C by use of a circulating water bath (Fisher Scientific Model 80) and a column water jacket from Altech Associates. Any of the commonly available column thermostats capable of handling two columns may be substituted. The Ultrasphere CN column is the first column in the series after the injector followed by the Ultrasphere ODS column.

The column effluent is passed through three detectors in series, each connected by means of low-dead-volume unions and a minimum length of 0.010-inch stainless-steel tubing. The order of the detectors is as follows:

1. Perkin-Elmer LC-75 variable-wavelength spectrometer with autocontrol.
2. Altex Model 153 fixed-wavelength detector set at 254 nm.
3. Perkin-Elmer Fluorescence Spectrometer Model 650-S.

Each detector is connected to a Spectra Physics Model 4100 integrator. An Altex Model 420 microprocessor is used to control the pumps and signal the variable-wavelength detector. The microprocessor initially signals the autosampler to load the sample loop. The autosampler then flushes the sample loop for 60 seconds, injects the sample onto the analytical column, and signals the three integrators to start. After 10 minutes, the microprocessor sends a second flag to the autosampler to reset it prior to the next injection. At 60 minutes, the microprocessor signals the Perkin-Elmer LC-75 with autocontrol to switch detection wavelength from 230 to 280 nm. This wavelength

is reset to 230 nm at 135 minutes by another signal from the microprocessor, and at 137 minutes, a second signal to the LC-75 resets the detector for the next injection.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detectors:

- a. Perkin-Elmer LC-75 variable-wavelength spectrometer with autocontrol set at 230 nm for first 60 minutes of chromatogram then switched to 280 nm.
- b. Altex Model 153 fixed-wavelength detector (254 nm).
- c. Perkin-Elmer 650-S fluorescence spectrometer.
Excitation wavelength is 290 nm with a spectral band pass of 10 nm. Emission is monitored at wavelengths longer than 350 nm by use of a cutoff filter and by setting the emission monochromator in the zero-order mode.

2. Columns

- a. Guard column (5.0 cm by 4.6-mm) packed with Pelliguard LC-CN 40-um pellicular packing.
- b. Ultrasphere CN, 5-um column (25 cm by 4.6-mm ID).
- c. Ultrasphere ODS, 5-um column (25 cm by 4.6-mm ID).

3. Flow Rate: 1.0 ml/min.

4. Mobile Phase: Elution gradient (see Figure 2):

- a. 30% methanol (CH_3OH) in phosphate-buffered water (pH=3) for 50 minutes.
- b. Increase percentage of CH_3OH to 65% over 30 minutes.
- c. At 80 minutes, increase the percentage of CH_3OH to 80% over 20 minutes.
- d. At 100 minutes, increase the percentage of CH_3OH to 90% over 10 minutes, and hold for 10 minutes.
- e. Decrease the percentage of CH_3OH to 30% over 5 minutes, and hold for 15 minutes for reequilibration before next injection.

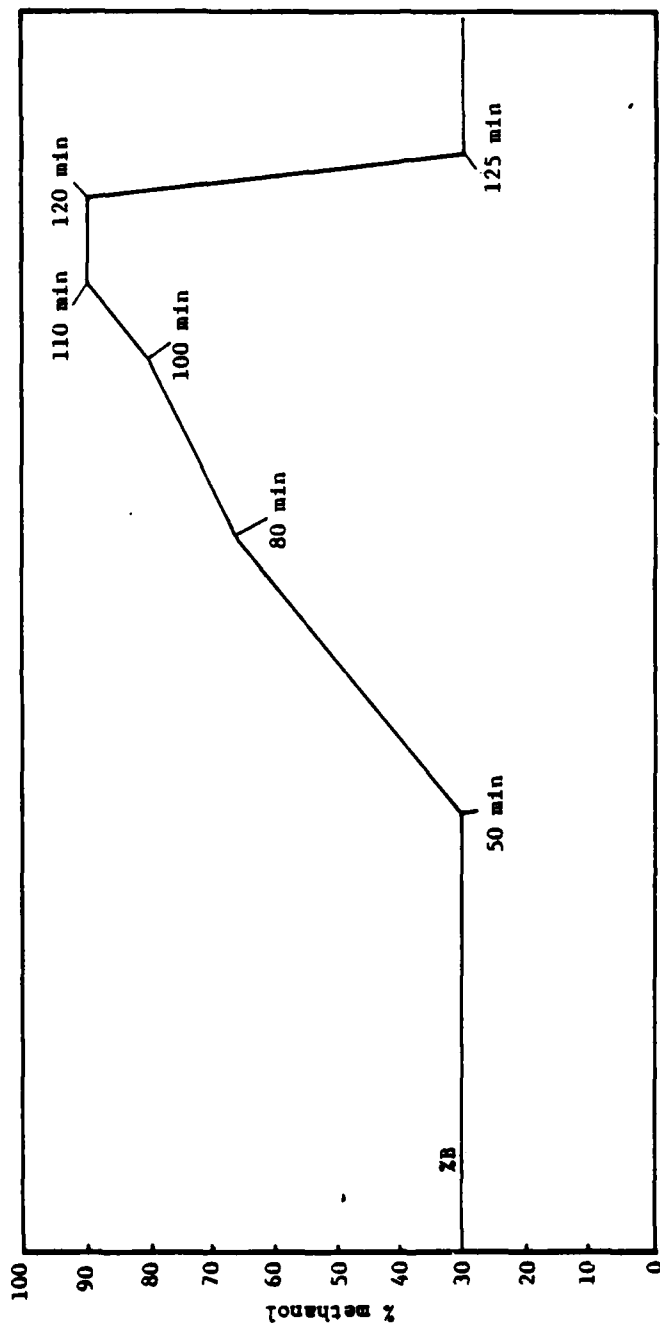


Figure 2
ELUTION PROGRAM FOR HPLC SCREEN

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

5. Temperature: 52°C.
6. Injection Volume: 50 ul, fixed loop.
7. Retention Times (see Table 9).

C. HARDWARE/GLASSWARE

1. 2-liter separatory funnel (Teflon® or glass) (8).
2. 500-ml K-D flask (8).
3. 20-ml K-D receiver (8).
4. Sep-Pak® silica-gel disposable cartridge (8) from Waters Associates.
5. 3-ball Snyder column (8).
6. 2-ball micro-Snyder column (8).
7. 10-ml graduated centrifuge tubes (8).
8. 10-ml syringe with Luer-lock fittings (1).
9. 4-cm glass funnels (8).
10. Disposable glass pipettes.

D. CHEMICALS

1. Nanograde methylene chloride--J.T. Baker Company.
2. Reagent-grade sodium chloride--J.T. Baker Company.
3. HPLC-grade methyl alcohol--J.T. Baker Company.
4. HPLC-grade water--J.T. Baker Company.
5. Anhydrous sodium sulfate--J.T. Baker Company.
6. 85-percent H₃PO₄, reagent-grade--J.T. Baker Company.
7. Teflon® boiling chips.
8. Colorphast® pH indicator sticks--MCB Manufacturing Chemists, Inc.
9. 6N NaOH--240 g of reagent-grade NaOH pellets dissolved in 1 L of organic-free water.
10. 6N HCl--dilute-concentrated, reagent-grade HCl in 1 L with organic-free water.
11. Sulfuric acid, reagent grade--J.T. Baker Company.
12. Sodium thiosulfate, reagent grade--J.T. Baker Company.

Table 9. HPLC Instrumental Parameters: Retention Times

Analyte	Retention Time (Minutes)
HMX	9.2
RDX	13.0
135TNB	16.8
13DNB	20.8
35DNP	26.9
246TNT	29.3
26DNT	35.0
24DNT	36.1
Naphthalene	84.5
Acenaphthylene	89.1
Acenaphthene	95.7
Phenanthrene	97.5
Anthracene	98.8
Fluoranthene	102.2
Pyrene	103.4
Chrysene	108.2
Benzo(b)fluoranthene	112.9
Benzo(k)fluoranthene	113.3
Benzo(a)pyrene	114.0
Dibenzo(a,h)anthracene	116.8
Indeno(1,2,3-cd)pyrene	117.9

Source: ESE, 1982.

4. STANDARDS

A. CALIBRATION STANDARDS

1. Separate stock calibration standards are prepared for each analyte by weighing the indicated amounts of each compound into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile (see Table 10).
2. Intermediate stock calibration standards are prepared for some of the analytes by pipetting the indicated volumes of the stock calibration standards into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile.

<u>Analyte</u>	<u>Volume of Stock Calibration Standard Used (ml)</u>	<u>Final Volume (ml)</u>	<u>Concentration of Intermediate Stock Calibration Standard (ug/ml)</u>
Phenanthrene	2	10	380
Chrysene	1	10	100
Benzo(b)fluoranthene	0.5	10	52
Benzo(k)fluoranthene	1	10	103
Benzo(a)pyrene	1	10	112
Dibenzo(a,h)anthracene	1	10	94
Indeno(1,2,3-cd)pyrene	1	100	36

3. The most concentrated (10x level) composite working calibration standard, F, is prepared from the separate stock and intermediate stock calibration standards. This high-level standard is diluted to yield all of the lower concentration standards. The high-level standard is prepared by pipetting the indicated amounts of the individual stock calibration standards or intermediate stock calibration standards into a single 50-ml volumetric flask. Fifteen milliliters of HPLC-grade acetonitrile are then added to make the final solution approximately 30% with

Table 10. Preparation of Calibration Standards

Analyte	Amount (mg)	Final Volume (ml)	Stock Calibra- tion Standard Concentration (ug/ml)
HMX	10	10	1,000
RDX	10	10	1,000
135TNB	9.5	10	950
13DNB	11	10	1,100
35DNP	18.15	10	1,815
246TNT	10	10	1,000
26DNT	11.4	10	1,140
24DNT	10	10	1,000
Naphthalene	20.61	10	2,061
Acenaphthylene	32.8	10	3,280
Acenaphthene	12.0	10	1,200
Phenanthrene	19.0	10	1,900
Anthracene	30.3	100	303
Fluoranthene	10	10	1,000
Pyrene	9.6	10	960
Chrysene	10	10	1,000
Benzo(b)fluoranthene	10.4	10	1,040
Benzo(k)fluoranthene	10.3	10	1,030
Benzo(a)pyrene	11.2	10	1,120
Dibenzo(a,h)anthracene	9.4	10	940
Indeno(1,2,3-cd)pyrene	36	10	3,600

Source: ESE, 1982.

respect to acetonitrile. The solution is then diluted to the mark using phosphate-buffered, HPLC-grade water (pH = 3) (see Table 11).

4. The lower-level working calibration standards are prepared by pipetting the indicated volumes of the F standard into volumetric flasks and diluting to the mark with 30% acetonitrile in HPLC-grade water buffered with phosphate (pH = 3).

<u>Standard</u>	<u>Volume of F Standard</u>	<u>Final Volume</u>	<u>Nominal Level</u>
A	0	10	Blank
B	0.5	10	0.5x
C	1.0	10	1x
D	2.0	10	2x
E	5.0	10	5x

B. CONTROL SPIKES

1. Intermediate stock spiking solutions are prepared for several of the heavier PAH compounds by pipetting the indicated volumes of the stock calibration standards into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile.

<u>Analyte</u>	<u>Volume of Stock Calibration Standard Used (ml)</u>	<u>Final Volume (ml)</u>	<u>Concentration of Intermediate Stock (ug/ml)</u>
Phenanthrene	2	10	380
Chrysene	1	10	100
Benzo(b)fluoranthene	0.5	10	52
Benzo(k)fluoranthene	1.0	10	103
Benzo(a)pyrene	1.0	10	112
Indeno(1,2,3-cd)pyrene	1.0	100	36

Table 11. Preparation of the Most Concentrated Composite Working Calibration Standard, F

Analyte	Volume of Standard Diluted (ml)	Standard Diluted	Concentration of F Standard (ug/ml)
HMX	0.5	Stock	10
RDX	0.5	Stock	10
135TNB	0.5	Stock	9.5
13DNB	0.5	Stock	11
35DNP	0.5	Stock	18.2
246TNT	0.5	Stock	10
26DNT	0.5	Stock	11.4
24DNT	0.5	Stock	10
Naphthalene	0.5	Stock	20.6
Acenaphthylene	0.5	Stock	32.8
Acenaphthene	0.5	Stock	12
Phenanthrene	1.0	Intermediate Stock	7.6
Anthracene	1.0	Stock	6.1
Fluoranthene	0.1	Stock	2.0
Pyrene	0.5	Stock	9.6
Chrysene	0.25	Intermediate Stock	0.5
Benzo(b)fluoranthene	0.5	Intermediate Stock	0.52
Benzo(k)fluoranthene	0.5	Intermediate Stock	1.03
Benzo(a)pyrene	0.5	Intermediate Stock	1.12
Dibenzo(a,h)anthracene	2.0	Intermediate Stock	3.76
Indeno(1,2,3-cd)pyrene	2.0	Intermediate Stock	1.44

Source: ESE, 1982.

2. The working control spike solutions are prepared by making a composite spike solution for the nitrosubstituted compounds and a separate solution for the PAHs. The indicated volumes (Table 12) of the stock calibration standards or intermediate stock spiking solutions are pipetted into a 25-ml volumetric flask and diluted to volume with HPLC-grade acetonitrile to make the working control spike solutions.
3. The following amounts of both the nitrocomposite and PAH composite working control spike solutions are pipetted into 1 L of standard or natural water. The concentrations spiked are shown in Section 8.

<u>Nominal Level</u>	<u>Volume Spiked (ml)</u>
Blank	0
0.5x	0.1
1x	0.2
2x	0.4
5x	1.0
10x	2.0

4. The precision, accuracy, and detection limits are determined for each analyte.

5. PROCEDURE

A. SAMPLING

1. Samples must be collected in amber-glass containers with Teflon®-lined caps. The bottle must be prerinsed with the sample before collection.
2. Samples must be extracted within 7 days of collection.

Table 12. Preparation of Working Control Spike Solutions

Analyte	Volume of Standard Diluted (ml)	Standard Diluted	Final Volume (ml)	Concentration (ug/ml)
<u>Nitrosubstituted Compounds--Working Control Spike Solution</u>				
HMX	0.5	Stock	25	20
RDX	0.5	Stock	25	20
135TNB	0.5	Stock	25	19
13DNB	0.5	Stock	25	22
35DNP	0.5	Stock	25	36.3
246TNT	0.5	Stock	25	20
26DNT	0.5	Stock	25	22.8
24DNT	0.5	Stock	25	20
<u>PAH Compounds--Working Control Spike Solution</u>				
Naphthalene	0.5	Stock	25	41.2
Acenaphthylene	0.5	Stock	25	65.6
Acenaphthene	0.5	Stock	25	24
Phenanthrene	1	Intermediate	25	15.2
		Stock		
Anthracene	1	Stock	25	12.1
Fluoranthene	0.1	Stock	25	4.0
Pyrene	0.5	Stock	25	19.2
Chrysene	0.25	Intermediate	25	1.0
		Stock		
Benzo(b)fluoranthene	0.5	Intermediate	25	1.04
		Stock		
Benzo(k)fluoranthene	0.5	Intermediate	25	2.06
		Stock		
Benzo(a)pyrene	0.5	Intermediate	25	2.24
		Stock		
Dibenzo(a,h)anthracene	2	Intermediate	25	7.52
		Stock		
Indeno(1,2,3-cd)pyrene	2	Intermediate	25	2.88
		Stock		

Source: ESE, 1982.

B. EXTRACTION

1. Allow samples to warm to room temperature. Mark the water meniscus on the side of the sample container for later determination of the exact sample volume. The sample volume should not be less than 1 L. Do not filter the water.
2. Pour the entire sample into a 2-L glass or Teflon® separatory funnel with Teflon® stopcocks.
3. Check the pH with wide-range pH paper and adjust the pH to less than 3 with 6N HCl.
4. Add 100 g of reagent-grade NaCl, and shake to dissolve the salt.
5. Add 100 ml of methylene chloride to the sample bottle, shake for 30 seconds to rinse the container walls, and transfer the solvent into the separatory funnel.
6. Extract the sample by vigorously shaking the separatory funnel for at least 2 minutes with periodic venting to release any vapor pressure.
7. Allow the organic layer to separate from the water layer for a minimum of 10 minutes.
8. If the emulsion interface between the layers is greater than one-third the volume of the organic layer, centrifugation or placing the separatory funnel in an ultrasonic bath must be employed to break the emulsion. Addition of small amounts of methanol can also aid in dispersing emulsions.
9. Draw off the methylene chloride and pass through a glass funnel fitted with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 25-ml K-D receiver, calibrated at the 25-ml mark.
10. Repeat Steps 5 through 9 two more times.
11. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride. This solvent rinse is added to the K-D apparatus.

12. Add a Teflon[®] boiling chip to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus. Prewet the Snyder column by adding approximately 1 ml of methylene chloride to the top.
13. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
14. The balls of the Snyder column should actively chatter, but the chambers should not flood.
15. When the apparent volume of the liquid in the receiver is less than approximately 15 ml, remove the K-D apparatus from the water bath and allow it to drain at least 10 minutes while cooling.
16. Remove the Snyder column and rinse the K-D flask and its lower joint into the receiver with 1 to 2 ml of methylene chloride.
17. Raise the volume of the methylene chloride in the receiver to the calibrated 20-ml mark.
18. Stopper the receiver with an ungreased ground-glass stopper, and mix the extract by inversion of the receiver.
19. Remove the ground-glass stopper, and pipette 10 ml of the methylene chloride extract into an amber or foil-wrapped 20-ml glass vial. Cap the vial with a Teflon[®]-lined cap, and store at 4°C for later use if a silica-gel cleanup is necessary.
20. Rinse the ground-glass stopper with 1 to 2 ml of methylene chloride into the extract remaining in the receiver.
21. Place a micro-Snyder column on the receiver, and prewet the column with 1 ml of methylene chloride. Add a fresh Teflon[®] boiling chip.
22. Concentrate the extract by gently heating the receiver in an 80°C water bath.

23. When the apparent volume of the liquid is less than approximately 1 ml, remove the receiver from the water bath.
24. Remove the micro-Snyder column and rinse its lower joint into the receiver with 2 ml of HPLC-grade acetonitrile.
25. Reattach the 2-ball micro-Snyder column, and reconcentrate to 0.5 ml.
26. Remove the receiver from the water bath and again add 2 ml of HPLC-grade acetonitrile, rinsing the joint column with the solvent.
27. Again, reconcentrate the sample to 0.5 ml.
28. Repeat Steps 26 and 27.
29. After this third exchange to acetonitrile, the extract is quantitatively transferred to a 10-ml graduated centrifuge tube.
30. The extract volume is then reduced by controlled evaporation under a gentle stream of dry nitrogen to a volume of approximately 0.2 to 0.3 ml.
31. The sides of the graduated centrifuge tube are then rinsed with 0.2 ml of HPLC-grade acetonitrile to yield an approximately 0.6-ml extract volume.
32. Dilute the extract to the calibrated 2-ml mark with phosphate-buffered, HPLC-grade water (pH = 3). This procedure yields a final extract volume of 2 ml in a solution that contains approximately 30% CH₃CN.
33. Transfer this extract to a 1-ml septum-sealed vial for HPLC analysis.
34. The extract is now ready for HPLC analysis.

C. SAMPLE CLEANUP

If it becomes apparent during the HPLC analysis that the sample cleanup is necessary for proper qualitative identification of the sample components, the following procedure is used to help remove possible interferents. The cleanup procedure is

necessary when components are found with the proper retention times but do not have the correct detector ratios, or when large broad-band interferents are noted in the chromatogram. The sample cleanup steps are as follows:

1. The 10-ml extract portion saved in Step 19 of the extraction procedure is transferred to a 10-ml glass syringe with Leur-lock tip.
2. The extract is then passed through a silica-gel Sep-Pak[®] attached to the syringe at a rate of approximately 5 ml/min, and the eluate is collected in a 20-ml K-D receiver.
3. The storage vial is rinsed with 5 ml of 50% CH₃OH in methylene chloride; this extract is passed through the silica-gel Sep-Pak[®] and the eluate collected.
4. The eluates from Steps 2 and 3 may be combined if only general cleanup is desired. The eluates are analyzed separately if sample fractionation is also required.
5. Follow Steps 21 through 34 of the extraction procedure to prepare the final extract for HPLC analysis.

D. CALIBRATION

1. A minimum of three instrument calibration standards and a blank will be run at the beginning of the analytical run. One of these standards will be duplicated at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized integrator areas versus micrograms/milliliter of each standard to obtain a working curve.

E. ANALYSIS

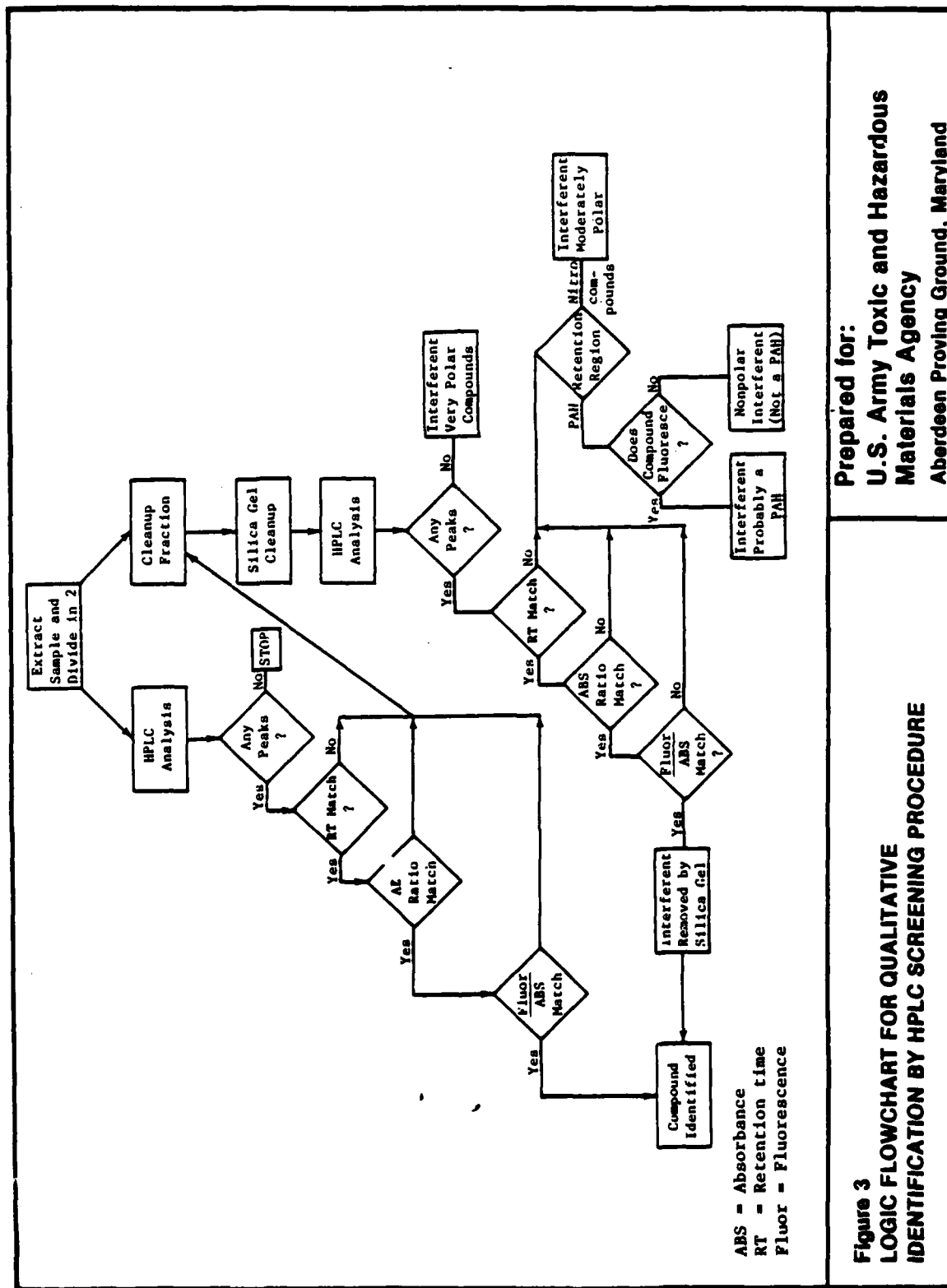
1. Inject 50 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the components of interest on each of the three detectors. Peak heights

rather than areas are used because they are less subject to interferences.

4. Quantitation of the components of interest is carried out according to Section 6 on the particular detector specified for each analyte in Section 1.
5. Qualitative identification of the component of interest is outlined in Figure 3. Detector ratios are calculated for unknown sample components and compared to the ratios obtained during the calibration run. The ratios obtained during documentation should serve as guidelines for the magnitude and variance of the expected ratios. The absolute value of the ratios may vary somewhat on different instrumentation, especially on ratios involving fluorescence because only a relative intensity on a particular instrument is measured. The absorbance ratios should be more consistent among different instruments because the absorbance is a property measured in a more rigorously defined manner than fluorescent intensity.

If the retention time and the ratios match those for one of the standards, the presence of that particular analyte in the sample is confirmed. If peaks are found for which the retention times match but the ratios do not match any of the standards, sample cleanup is conducted. The cleaned fraction is then analyzed by HPLC, and the same criteria are applied for compound verification.

A detector ratio is considered positive if it falls within the 95-percent confidence interval for the ratio obtained during documentation for that particular analyte. The mean detector ratios, 95-percent confidence interval, and lowest level (ppb) at which these ratios could be accurately measured due to instrumental sensitivity limitations are presented in Tables 13 and 14.



Prepared for:
 U.S. Army Toxic and Hazardous
 Materials Agency
 Aberdeen Proving Ground, Maryland

Figure 3
 LOGIC FLOWCHART FOR QUALITATIVE
 IDENTIFICATION BY HPLC SCREENING PROCEDURE

Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silica-Gel Cleanup

Compound	Detector Wavelength (nm)	Mean Absorbance Ratio* $\pm$ 95% CI†	Lowest Concentration for Ratio (ug/L)	Target IL (ug/L)	Documented DI** (ug/L)	Retention Time (minutes)
BMX	230/254	6.6 $\pm$ 0.80	4.0	4.0	15	9.2
RDX	230/254	3.0 $\pm$ 0.25	2.0	4.0	6	13.0
ATBA	230/254	2.8	ND††	ND	ND	14.3
13TNB	230/254	3.6 $\pm$ 0.23	1.9	3.8	6	16.8
13TNB	230/254	1.5 $\pm$ 0.25	2.2	4.4	6	20.8
NB	230/254	0.70	ND	ND	ND	24.8
35DNP	230/254	1.7 $\pm$ 0.58	3.6	7.3	11	26.9
Tetryl	230/254	3.14	ND	ND	ND	29.5
246DNT	230/254	2.3 $\pm$ 0.43	2.0	4.0	6	29.3
26DNT	230/254	1.5 $\pm$ 0.95	2.3	4.6	9	35.0
24DNT	230/254	1.2 $\pm$ 0.31	2.0	4.0	6	36.1
12DNT	230/254	0.852	ND	ND	ND	52.5
Naphthalene	280/254	1.6 $\pm$ 0.8	4.1	8.2	27	84.5
	254/F1***	21 $\pm$ 12	41	8.2	—	—
	280/F1	36 $\pm$ 5.0	41	8.2	—	—
Acenaphthylene	280/254	1.3 $\pm$ 0.28	6.6	13	22	89.1
Acenaphthene	280/254	0.53 $\pm$ 0.13	1.5	4.8	10	95.7
Phenanthrene	280/254	0.35 $\pm$ 0.13	1.5	3.0	5	97.5
	254/F1	29 $\pm$ 5.9	6.1	3.0	—	—
	280/F1	11 $\pm$ 1.6	6.1	3.0	—	—

Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silica-Gel Cleanup
(Continued, Page 2 of 3)

Compound	Detector Wavelength (nm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented IL** (ug/L)	Retention Time (minutes)
Anthracene	280/254	0.0082 ± 0.014	12	2.4	2	98.8
	254/F1	220 ± 56	12	2.4	—	—
	280/F1	1.5 ± 0.31	12	2.4	—	—
Fluoranthene	280/254	2.2 ± 0.64	0.4	0.8	0.9	102.2
	254/F1	0.55 ± 0.16	0.4	0.8	—	—
	280/F1	1.2 ± 0.39	0.4	0.8	—	—
Pyrene	280/254	0.46 ± 0.16	1.9	3.8	8	103.4
	254/F1	7.9 ± 1.5	1.9	3.8	—	—
	280/F1	3.8 ± 1.0	19	3.8	—	—
Chrysene	280/254	0.43 ± 0.15	0.40	0.20	0.4	108.2
	254/F1	15 ± 1.3	1.0	0.20	—	—
	280/F1	6.6 ± 2.5	1.0	0.20	—	—
Benzo(b)fluoranthene	254/280	0.99 ± 0.42	0.21	0.21	2	112.9
	254/F1	0.97 ± 0.27	0.21	0.21	—	—
	280/F1	0.99 ± 0.56	0.21	0.21	—	—
Benzo(k)fluoranthene	280/254	1.3 ± 0.47	0.21	0.41	1	113.3
	254/F1	0.39 ± 0.16	0.21	0.41	—	—
	280/F1	0.50 ± 0.23	0.21	0.41	—	—
Benzo(a)pyrene	280/254	1.6 ± 0.70	0.45	0.45	0.8	114.0
	254/F1	1.3 ± 0.58	0.45	0.45	—	—
	280/F1	2.0 ± 0.83	0.45	0.45	—	—

Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silica-Gel Cleanup
(Continued, Page 3 of 3)

Compound	Detector Wavelength (nm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented DL** (ug/L)	Retention Time (minutes)
Dibenzo(a,h)anthracene	280/254	12 ± 6.4	0.75	1.5	ND	116.8
	254/FI	0.62 ± 0.39	0.75	1.5	—	—
	280/FI	6.9 ± 2.2	0.75	1.5	—	—
Indeno(1,2,3-cd)pyrene	280/254	0.89 ± 0.27	0.58	0.58	2	117.9
	254/FI	1.6 ± 0.34	0.58	0.58	—	—
	280/FI	1.4 ± 0.32	0.58	0.58	—	—

* Determined from peak height measurements.

† CI = Confidence interval (n = 20).

** DL = Detection limit calculated according to Hbbaux and Vos, 1970.

†† Not determined due to co-elution, interference, or instability problems.

*** Fluorescence measured at wavelengths greater than 350 nm.

Source: ESE, 1982.

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Cleanup

Compound	Detector Wavelength (nm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target IL (ug/L)	Documented IL** (ug/L)	Retention Time (minutes)
BAX	230/254	6.6 ± 2.2	4.0	4.0	11	9.2
BDX	230/254	3.0 ± 0.63	2.0	4.0	8	13.0
137NB	230/254	3.6 ± 0.79	1.9	3.8	6	16.8
13NB	230/254	1.9 ± 0.29	2.2	4.4	8	20.8
35NP	230/254	1.6 ± 0.68	3.6	7.3	8	26.9
246NT	230/254	2.3 ± 0.76	2.0	4.0	11	29.3
26NT	230/254	1.7 ± 0.89	2.3	4.6	12	35.0
24NT	230/254	1.1 ± 0.55	2.0	4.0	7	36.1
Naphthalene	280/254	1.6 ± 0.62	4.1	8.2	33	84.5
	254/F1††	22 ± 16	41	8.2	—	—
	280/F1	37 ± 6.0	41	8.2	—	—
Acenaphthylene	280/254	1.4 ± 0.71	6.6	13	35	89.1
Acenaphthene	280/254	0.52 ± 0.47	1.5	4.8	12	95.7
Phenanthrene	280/254	0.35 ± 0.05	1.5	3.0	5	97.5
	254/F1	31 ± 12	6.1	3.0	—	—
	280/F1	10 ± 4.7	6.1	3.0	—	—
Anthracene	280/254	0.0084 ± 0.011	12	2.4	5	98.8
	254/F1	230 ± 72	12	2.4	—	—
	280/F1	1.9 ± 2.5	12	2.4	—	—

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Cleanup (Page 2 of 3)

Compound	Detector Wavelength (nm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target IL (ug/L)	Documented IL** (ug/L)	Retention Time (minutes)
Fluoranthene	280/254	2.3 ± 0.56	0.4	0.8	1	102.2
	254/FI	0.53 ± 0.13	0.4	0.8	—	—
	280/FI	1.2 ± 0.30	0.4	0.8	—	—
Pyrene	280/254	0.48 ± 0.17	1.9	3.8	6	103.4
	254/FI	7.8 ± 1.4	7.7	3.8	—	—
	280/FI	3.7 ± 0.97	7.7	3.8	—	—
Chrysene	280/254	0.40 ± 0.046	0.40	0.20	0.5	108.2
	254/FI	16.2 ± 5.9	1.0	0.20	—	—
	280/FI	6.0 ± 5.0	1.0	0.20	—	—
Benzo(b)fluoranthene	280/254	1.0 ± 0.46	0.21	0.21	0.3	112.9
	254/FI	0.94 ± 0.30	0.21	0.21	—	—
	280/FI	0.96 ± 0.38	0.21	0.21	—	—
Benzo(k)fluoranthene	280/254	1.4 ± 0.71	0.41	0.41	0.8	113.3
	254/FI	0.39 ± 0.26	0.21	0.41	—	—
	280/FI	0.53 ± 0.26	0.41	0.41	—	—
Benzo(a)pyrene	280/254	1.8 ± 0.52	0.45	0.45	0.9	114.0
	254/FI	1.3 ± 0.51	0.45	0.45	—	—
	280/FI	2.0 ± 1.2	0.45	0.45	—	—

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Cleanup (Page 3 of 3)

Compound	Detector Wavelength (nm)	Absorbance Ratio* + 95% C.I.†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented DL** (ug/L)	Retention Time (minutes)
Dibenzo(a,h) anthracene	280/254	13 + 3.7	1.5	1.5	ND***	116.8
	254/FI	0.61 + 0.28	1.5	1.5	—	—
	280/FI	7.7 + 2.9	1.5	1.5	—	—
Indeno(1,2,3-cd)pyrene	280/254	0.88 + 0.38	0.58	0.58	2	117.9
	254/FI	1.6 + 0.77	0.58	0.58	—	—
	280/FI	1.4 + 1.0	0.58	0.58	—	—

* Determined from peak heights measurements.

† CI = Confidence interval (n = 20).

** DL = Detection limit calculated according to Hubaux and Vos, 1970.

†† Fluorescence measured at wavelengths greater than 350 nm.

*** ND = Not determined due to interference problem.

Source: ESE, 1982.

6. CALCULATIONS

Determine the concentration of each analyte according to the following formula:

$$\text{Concentration (ug/g)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve (ug/ml),

V_t = Volume of total extract (ml), and

V_s = Volume of initial sample extracted (L).

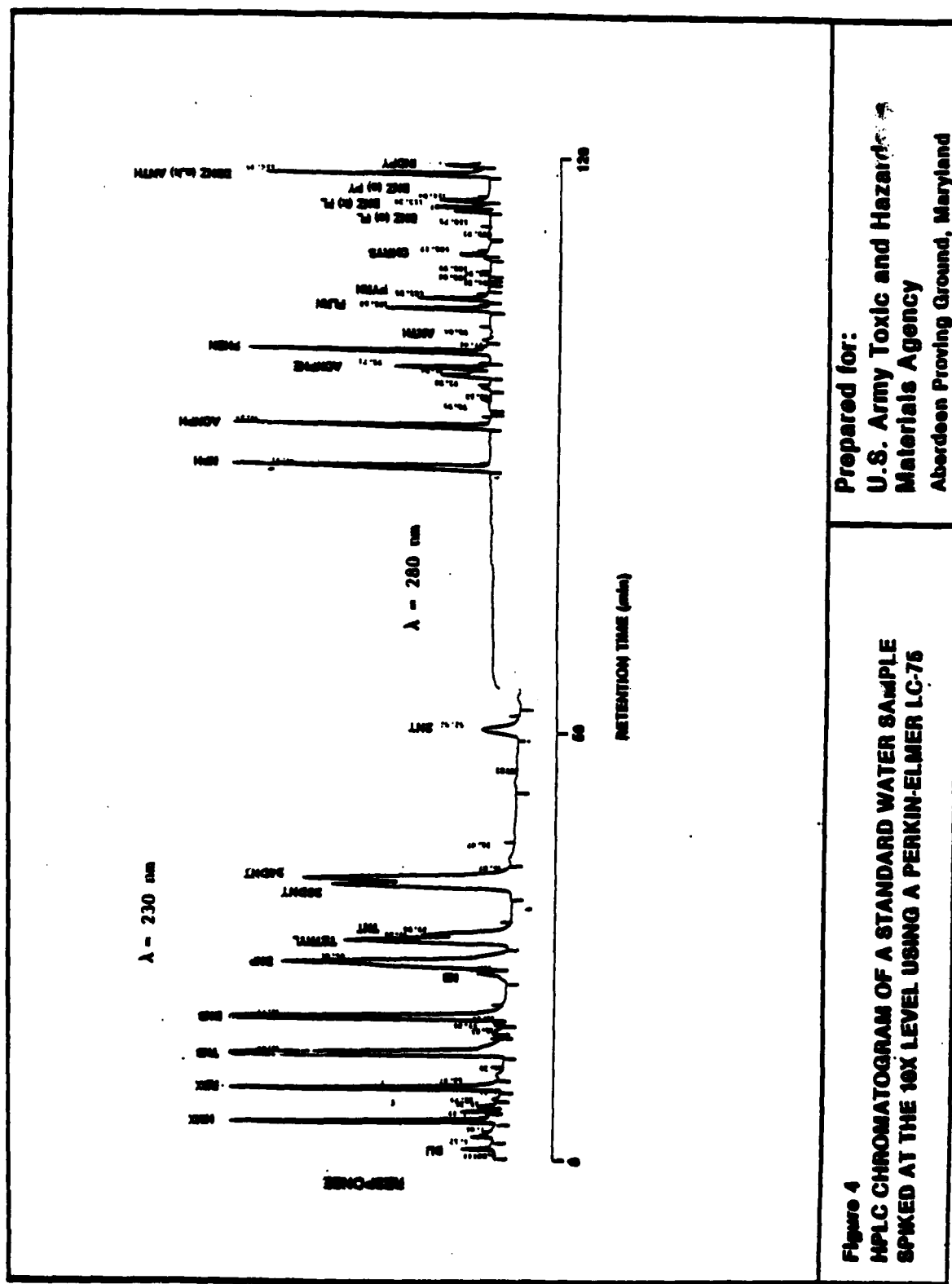
The concentration is corrected for recovery by dividing by the slope of the regression line for observed value versus target value for spiked samples.

7. REFERENCES

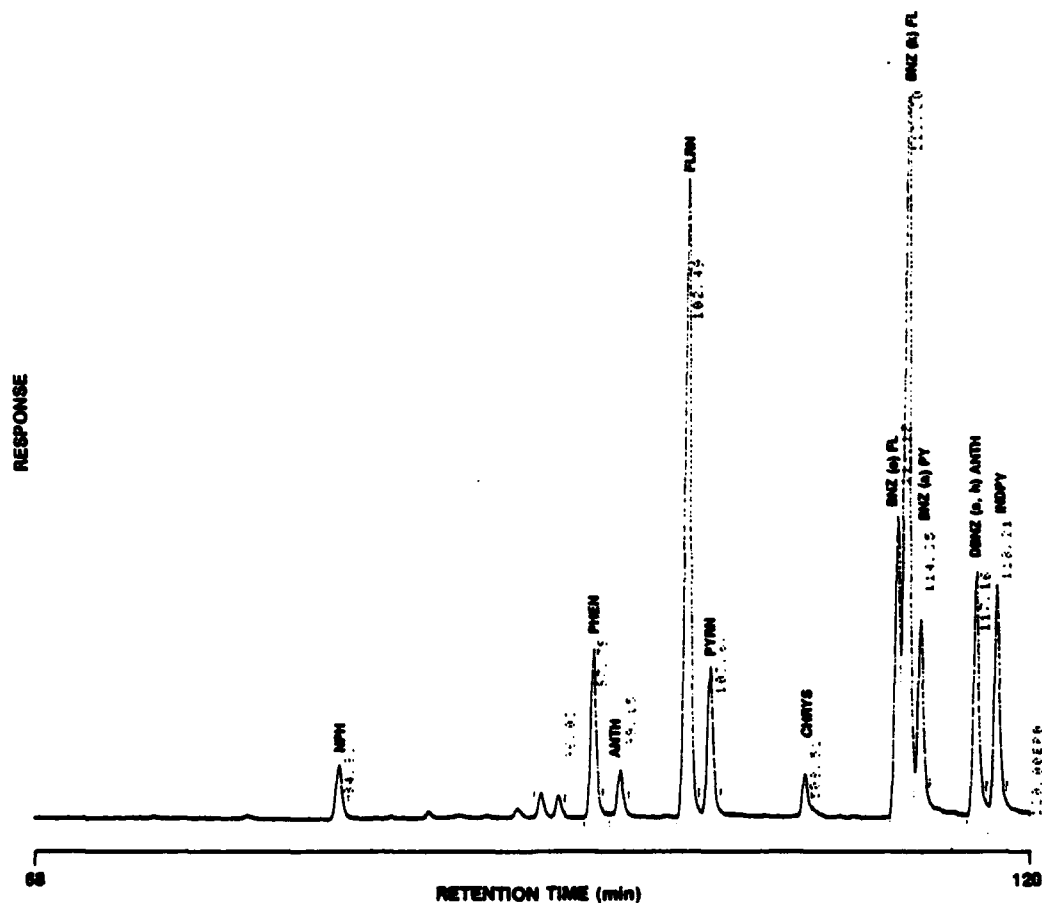
Hubaux, A. and Vos, G. 1970. Anal. Chem. 42, 849-885.

8. DATA

See Figures 4 through 6 and attached data sheets.







Excitation λ = 290 nm
 Emission λ = Filter (>350 nm)

Figure 6
HPLC CHROMATOGRAM OF A STANDARD
WATER SAMPLE SPIKED AT THE 10-DL LEVEL
USING A FLUORESCENCE DETECTOR

Prepared for:
U.S. Army Toxic and Hazardous
Materials Agency
Aberdeen Proving Ground, Maryland

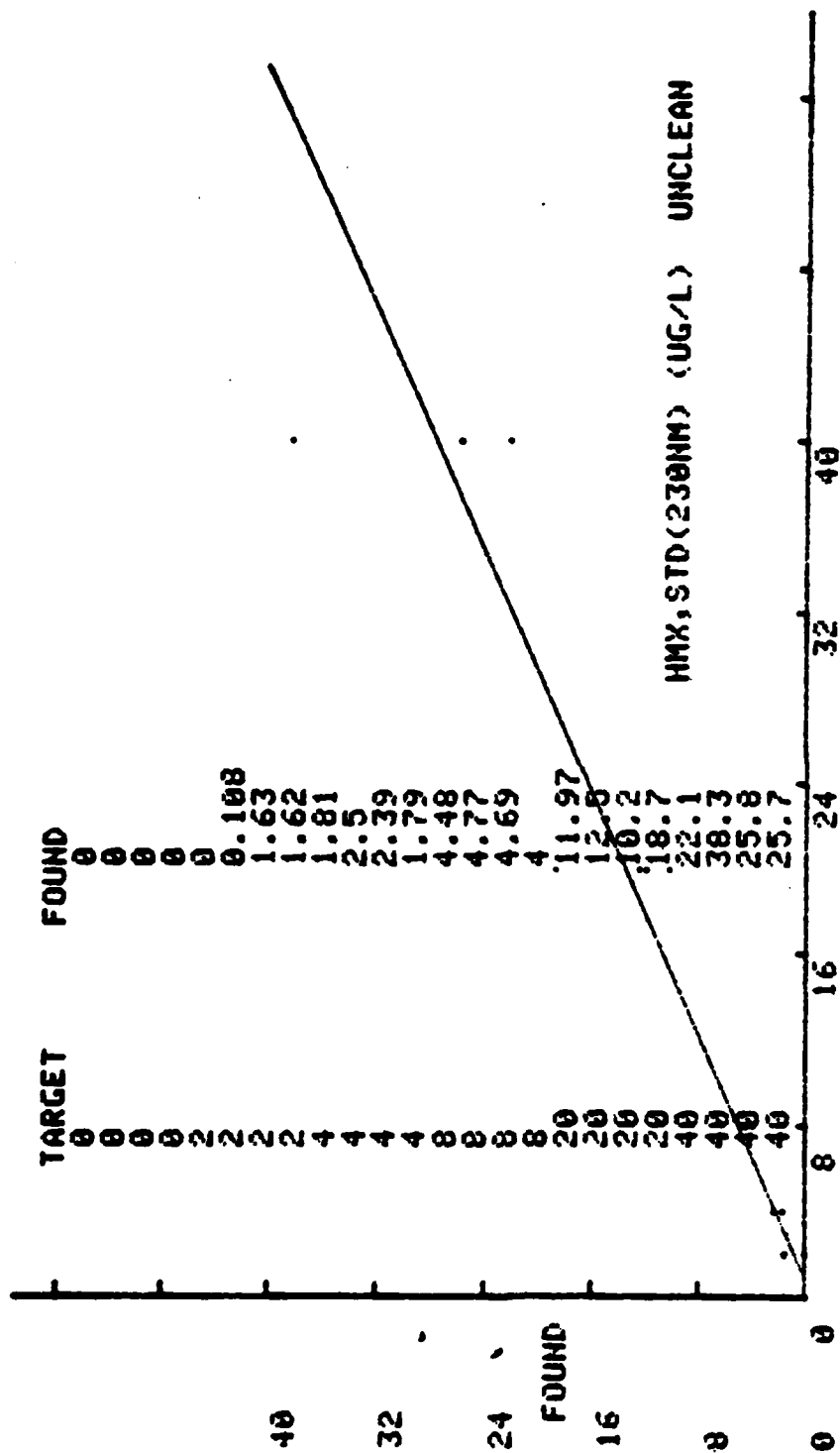
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I.

HMx,STD(230NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.0000	0.108	1.63	1.62
4.00	1.81	2.50	2.39	1.79
8.00	4.48	4.77	4.69	4.00
20.0	12.0	12.5	10.2	18.7
40.0	22.1	38.3	25.8	25.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.839	0.908	108	-58.0
4.00	2.12	0.375	17.7	-46.9
8.00	4.48	0.346	7.71	-43.9
20.0	13.3	3.70	27.8	-33.3
40.0	28.0	7.10	25.4	-30.1



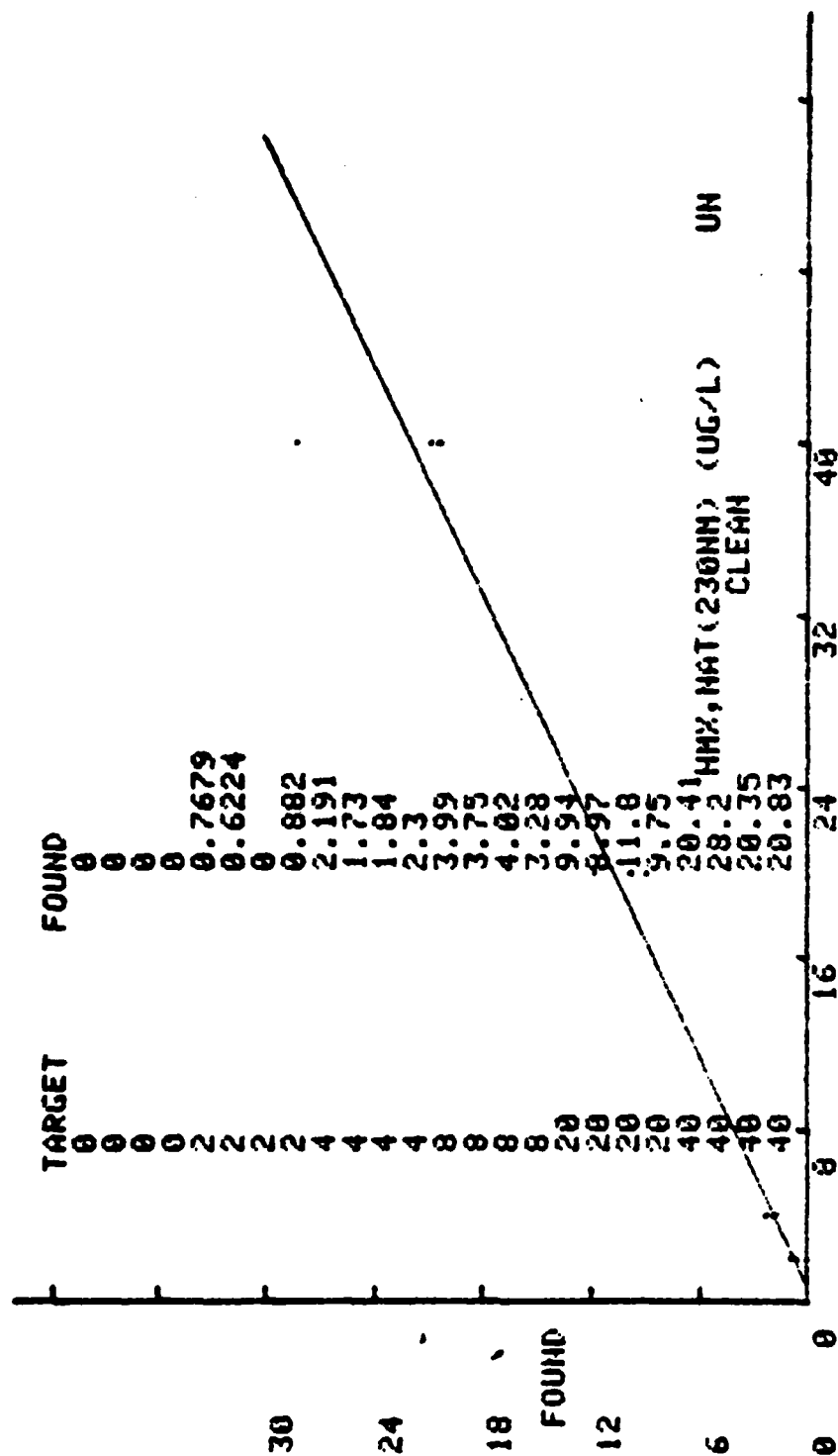
CORR. COEFF. = 0.9603
 DETECTION LIMIT = 14.79678
 TARGET
 -0.6169+ 0.708996+TARGET

HMX,NAT(230NM) (UG/L)

UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.768	0.622	0.0000	0.882
4.00	2.19	1.73	1.84	2.30
8.00	3.99	3.75	4.02	3.28
20.0	9.94	8.97	11.8	9.75
40.0	20.4	28.2	20.3	20.8

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.568	0.393	69.2	-71.6
4.00	2.02	0.273	13.6	-49.6
8.00	3.76	0.342	9.10	-53.0
20.0	10.1	1.20	11.9	-49.4
40.0	22.4	3.84	17.1	-43.0

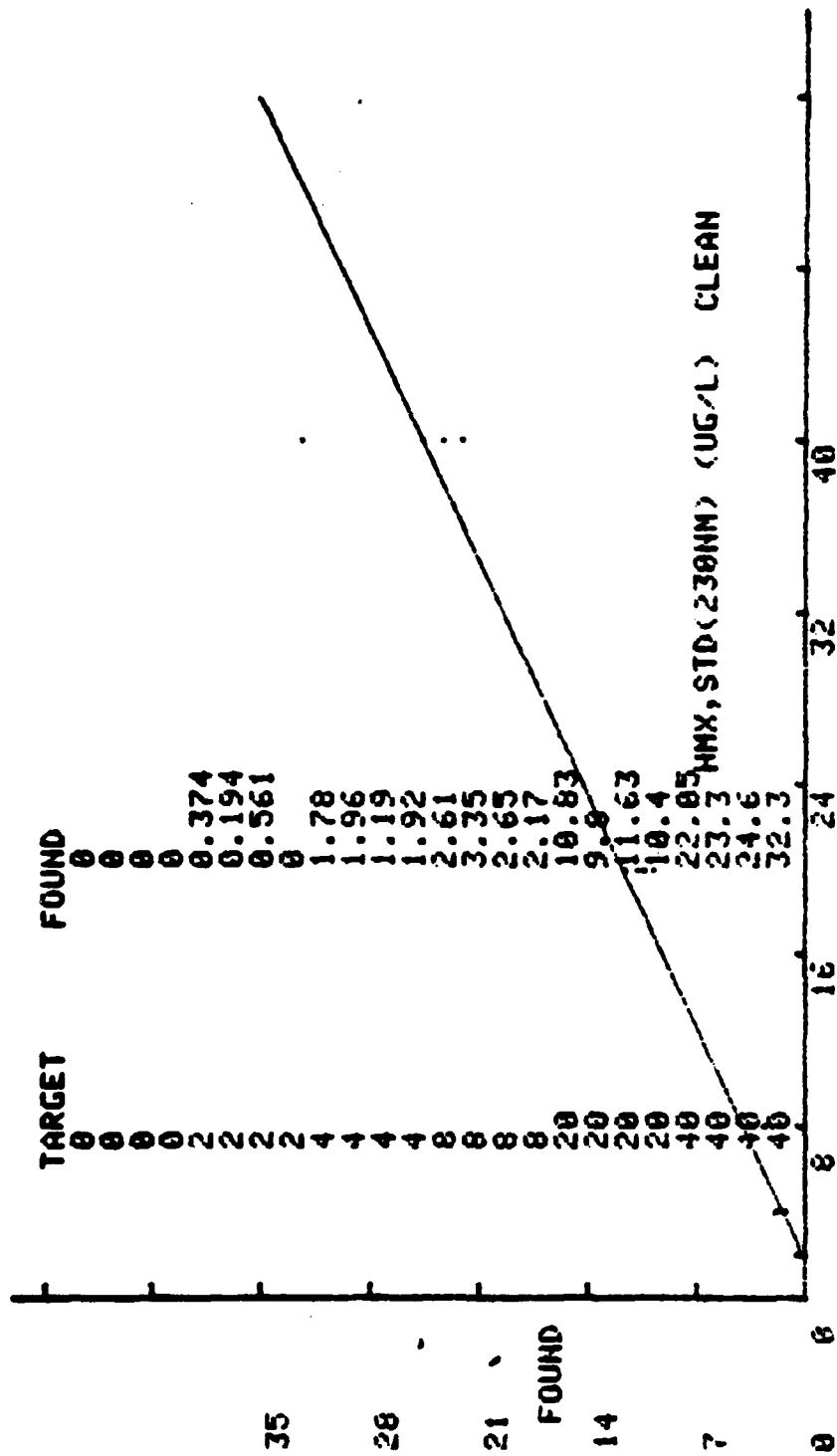


CORR. COEFF. = 0.9825 FOUND = -0.4612+ 0.563152*TARGET
 DETECTION LIMIT = 9.6653

HMX,STD(230NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.374	0.194	0.561	0.0000
4.00	1.78	1.96	1.19	1.92
8.00	2.61	3.35	2.65	2.17
20.0	10.8	9.80	11.6	10.4
40.0	22.0	23.3	24.6	32.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.282	0.241	85.2	-85.9
4.00	1.71	0.357	20.8	-57.2
8.00	2.69	0.488	18.1	-66.3
20.0	10.7	0.770	7.22	-46.7
40.0	25.6	4.61	18.0	-36.1

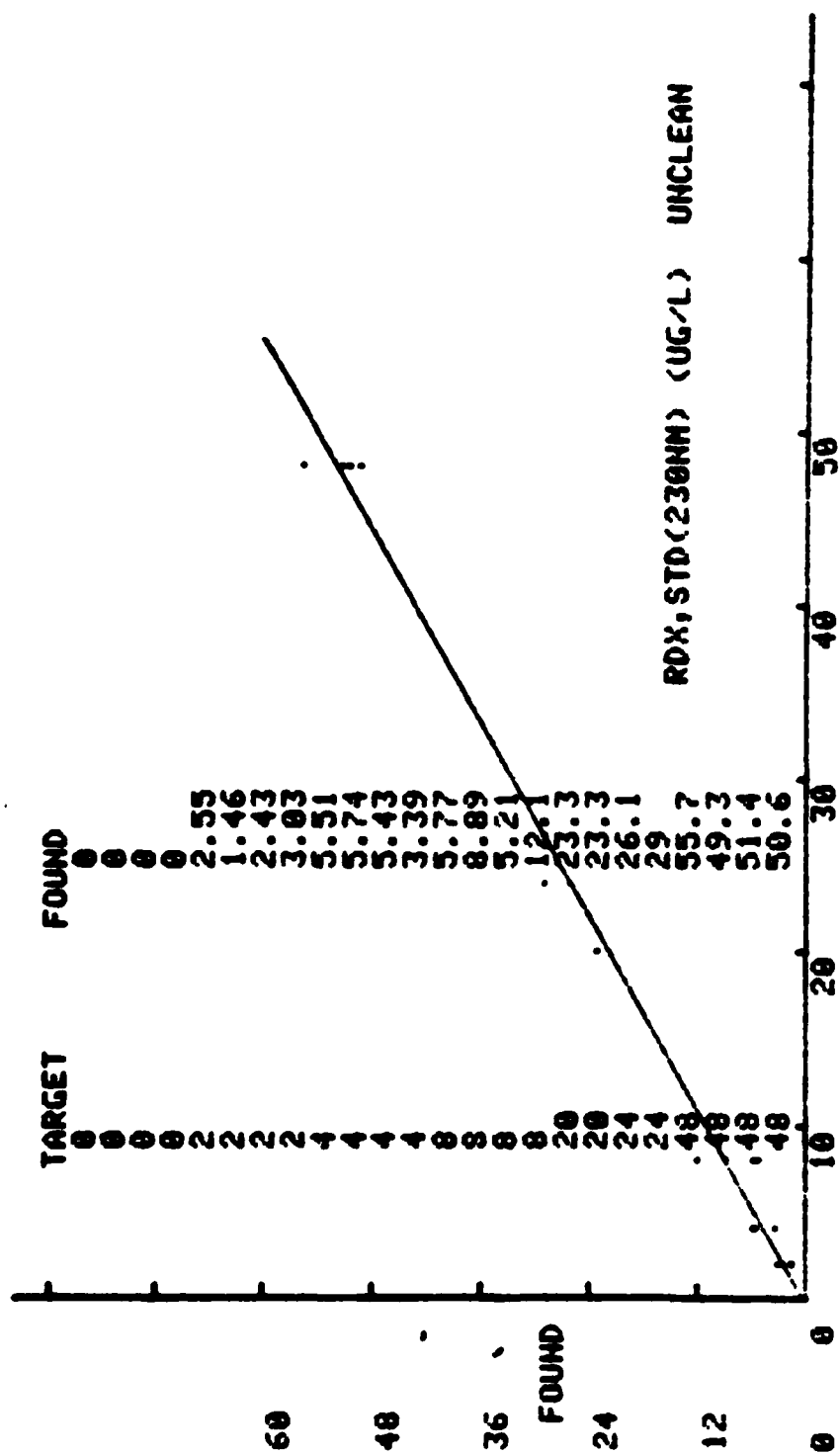


TARGET
 CORR. COEFF. = 0.9786 FOUND = -1.1841+ 0.648943*TARGET
 DETECTION LIMIT = 10.72038

RDX,STD(230NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.55	1.46	2.43	3.03
4.00	5.51	5.74	5.43	3.39
8.00	5.77	8.89	5.21	12.1
20.0	23.3	23.3	26.1	29.0
48.0	55.7	49.3	51.4	50.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.37	0.658	27.8	18.4
4.00	5.02	1.09	21.8	25.4
8.00	7.99	3.18	39.8	-0.0938
20.0	46.6	0.0000	0.0000	33.0
48.0	55.1	0.0000	0.0000	29.6

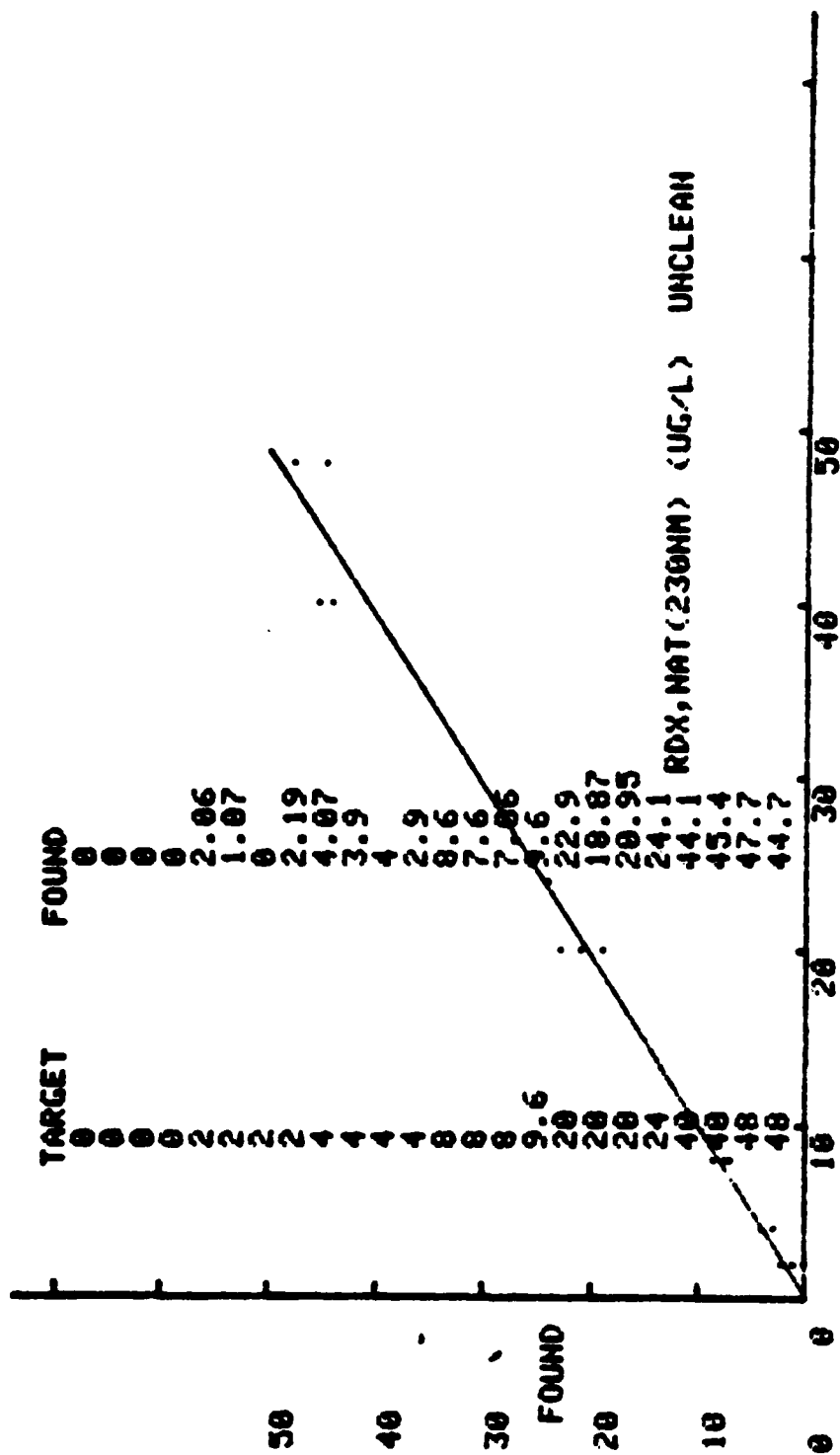


TARGET
CORR. COEFF. = 0.9953 FOUND = 0.2675+
DETECTION LIMIT = 5.94724
1.082712*TARGET

RDX,NAT(230NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.06	1.07	0.0000	2.19
4.00	4.07	3.90	4.00	2.90
8.00	8.60	7.60	7.06	9.60
20.0	22.9	18.9	20.9	24.1
40.0	44.1	45.4	47.7	44.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.33	1.02	76.5	-33.5
4.00	3.72	0.549	14.8	-7.06
8.00	7.75	0.781	10.1	-3.09
20.0	9.60	0.0000	0.0000	0.0000
40.0	20.9	2.02	9.64	4.53



CORR. COEFF. = 0.9945 FOUND = TARGET
 DETECTION LIMIT = 5.926 -0.2708+ 1.033596xTARGET

RDX,STD(230NM) (UG/L) CLEAN

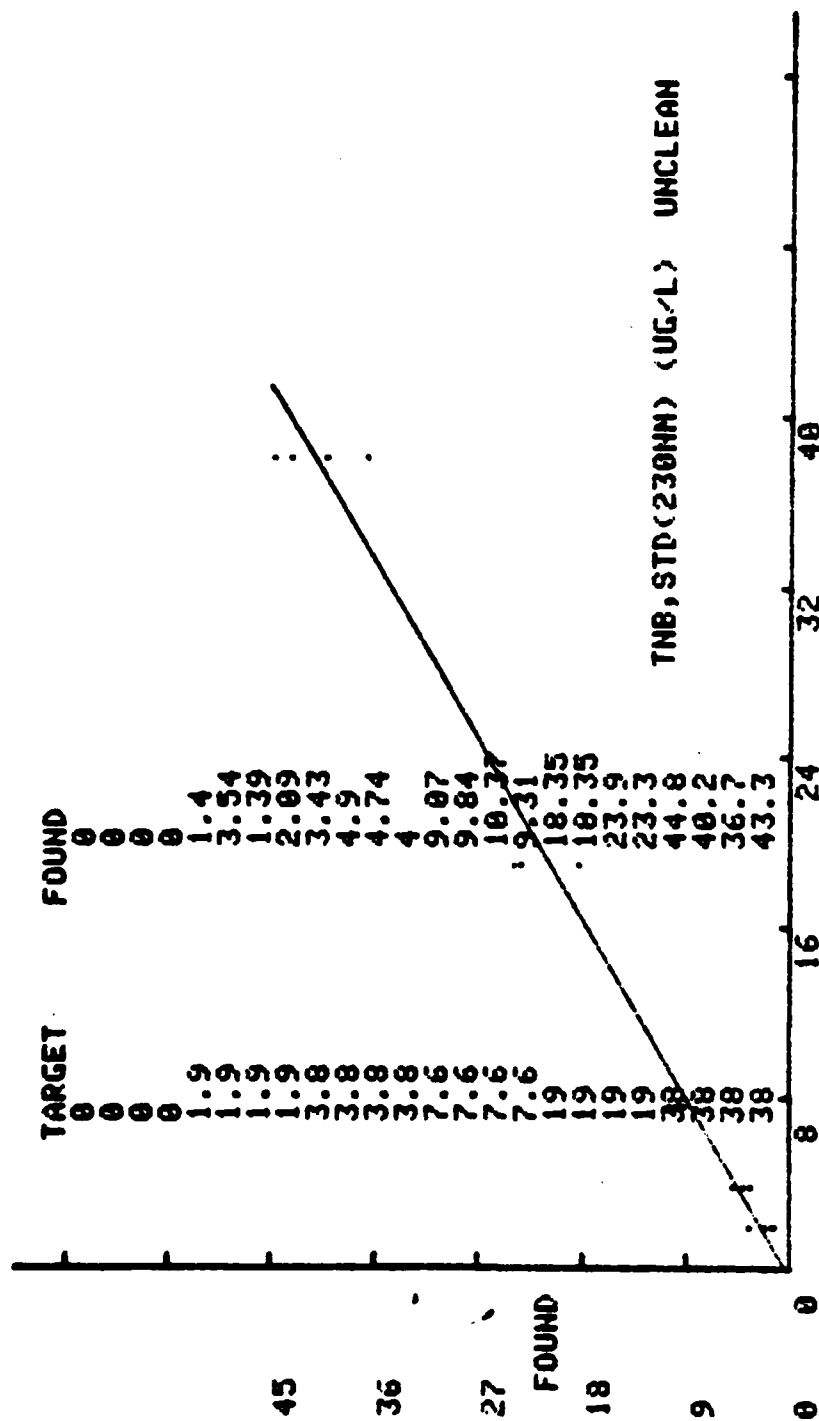
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	3.03	2.58	2.75	2.50
4.00	6.29	5.56	4.80	5.20
8.00	8.10	9.99	6.65	7.31
20.0	12.9	25.2	24.6	24.7
40.0	39.5	47.2	51.2	55.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.71	0.234	8.64	35.7
4.00	5.46	0.633	11.6	36.6
8.00	8.01	1.45	18.0	0.156
20.0	12.9	0.0000	0.0000	-35.4
40.0	24.8	0.311	1.25	3.44

TNB, STD(230NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.90	1.40	3.54	1.39	2.09
3.80	3.43	4.00	4.74	4.00
7.60	9.07	9.84	10.4	9.31
19.0	18.3	18.3	23.9	23.3
38.0	44.8	40.2	36.7	43.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.90	2.10	1.01	48.0	10.8
3.80	4.27	0.682	16.0	12.3
7.60	9.65	0.579	6.00	26.9
19.0	21.0	3.04	14.5	10.4
38.0	41.2	3.59	8.70	8.55

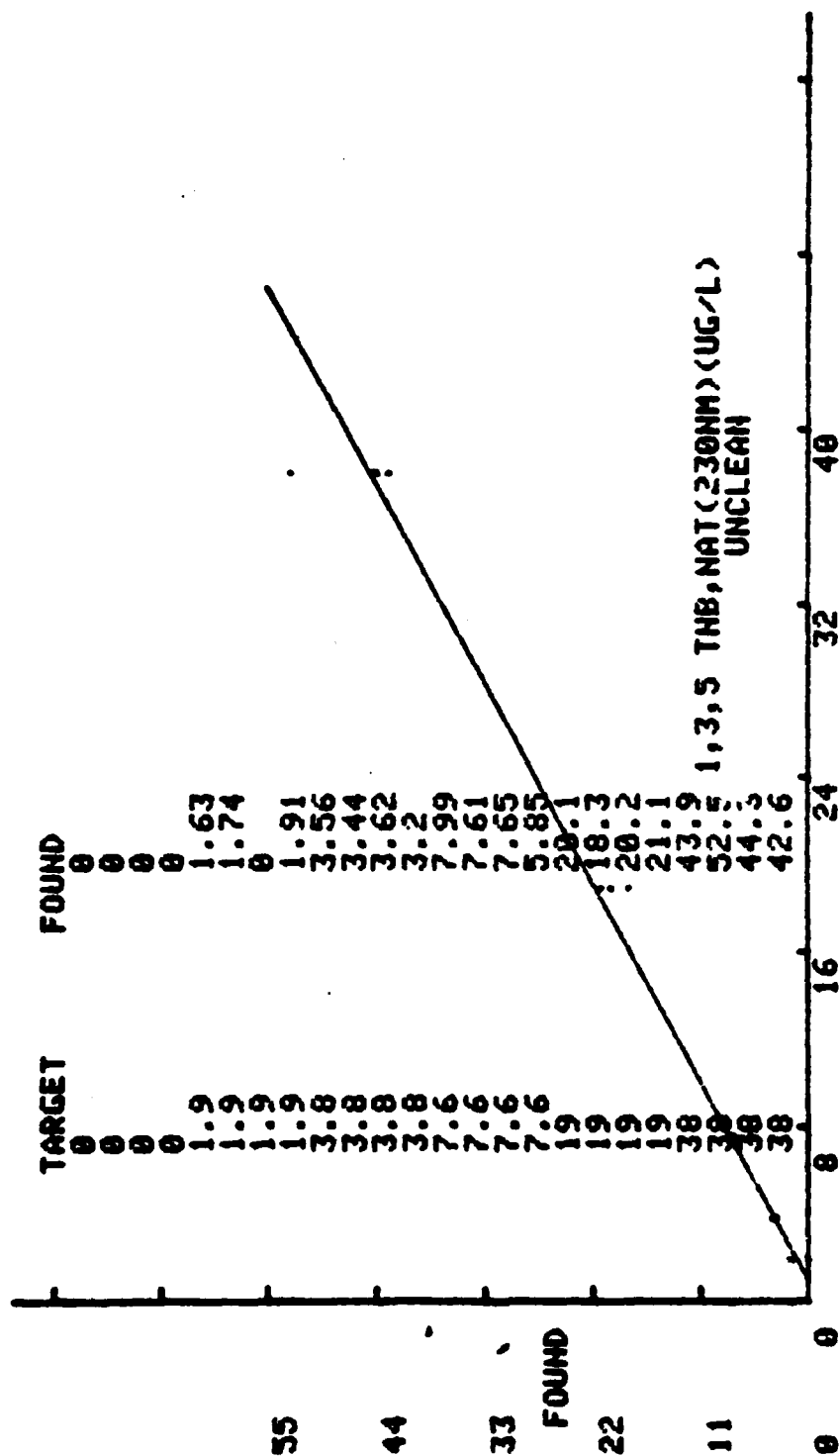


CORR. COEFF. = 0.9922 FOUND = TARGET
 DETECTION LIMIT = 6.06573

1.3,5 TNB,NAT(230NM)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.50	1.63	1.74	0.0000	1.91
3.20	3.55	3.44	3.62	3.20
7.60	7.99	7.61	7.65	5.85
19.0	20.1	18.3	20.2	21.1
38.0	43.9	52.5	44.3	42.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.50	1.32	0.888	67.2	-30.5
3.20	3.45	0.186	5.38	-9.38
7.6	7.27	0.965	13.3	-4.28
19.0	19.9	1.17	5.89	4.87
38.0	45.8	4.51	9.84	20.6

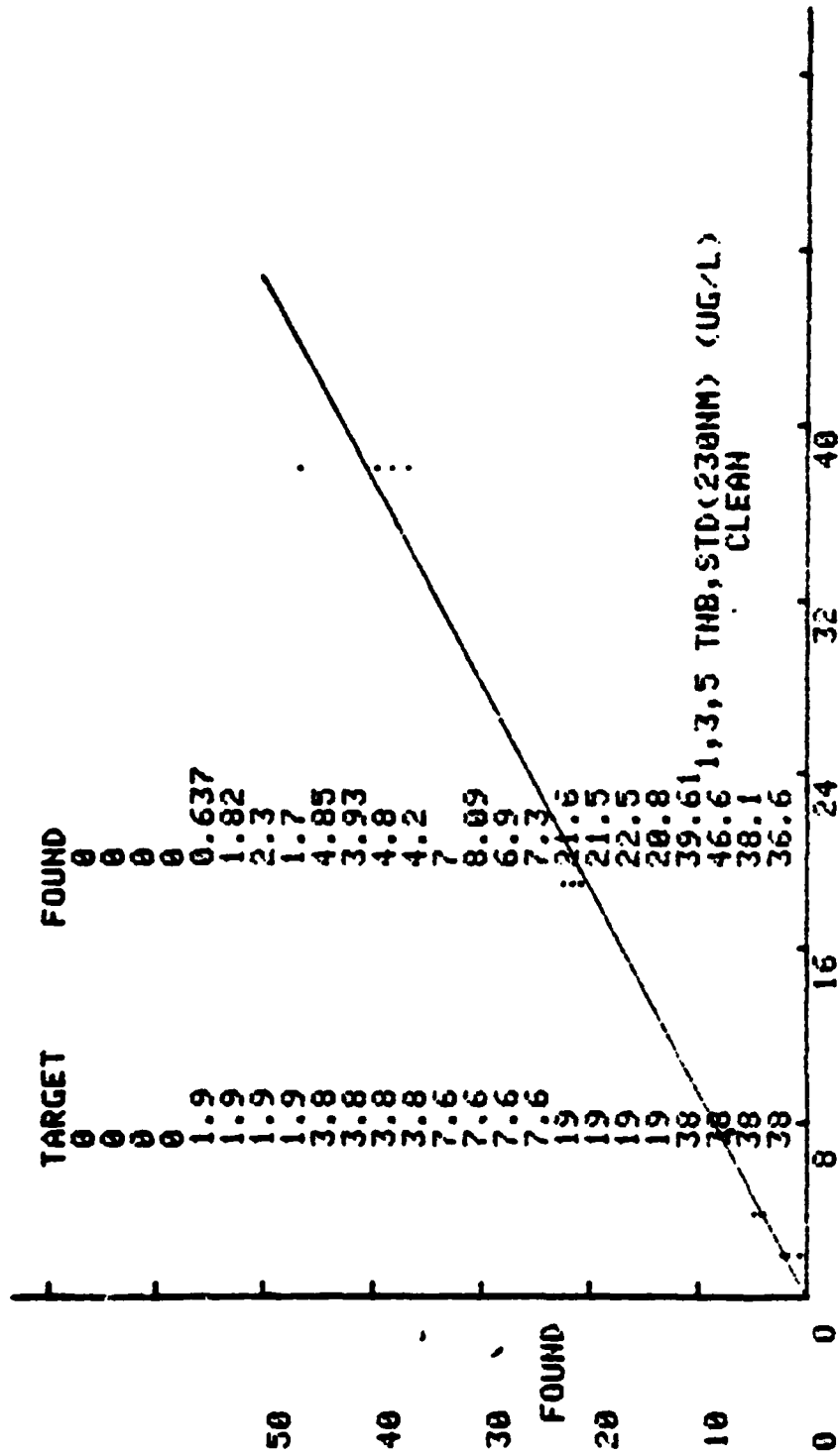


TARGET
 CORR. COEFF. = 0.9923 FOUND = -1.2124+ 1.210161 * TARGET
 DETECTION LIMIT = 6.04721

1,3,5 TNB, STD(230NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.90	0.637	1.82	2.30	1.70
3.80	4.85	3.93	4.80	4.20
7.60	7.00	8.09	6.90	7.30
19.0	21.6	21.5	22.5	20.8
38.0	39.6	46.6	38.1	36.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.90	1.61	0.701	43.4	-15.0
3.80	4.44	0.453	10.2	17.0
7.60	7.32	0.539	7.36	-3.65
19.0	21.6	0.698	3.23	13.7
38.0	40.2	4.42	11.0	5.86



CORR. COEFF. = 0.9925
 DETECTION LIMIT = 5.97772
 TARGET FOUND = -0.0277+
 1.072199*TARGET

DNB,STD(254NM) (UG/L) UNCLEAN

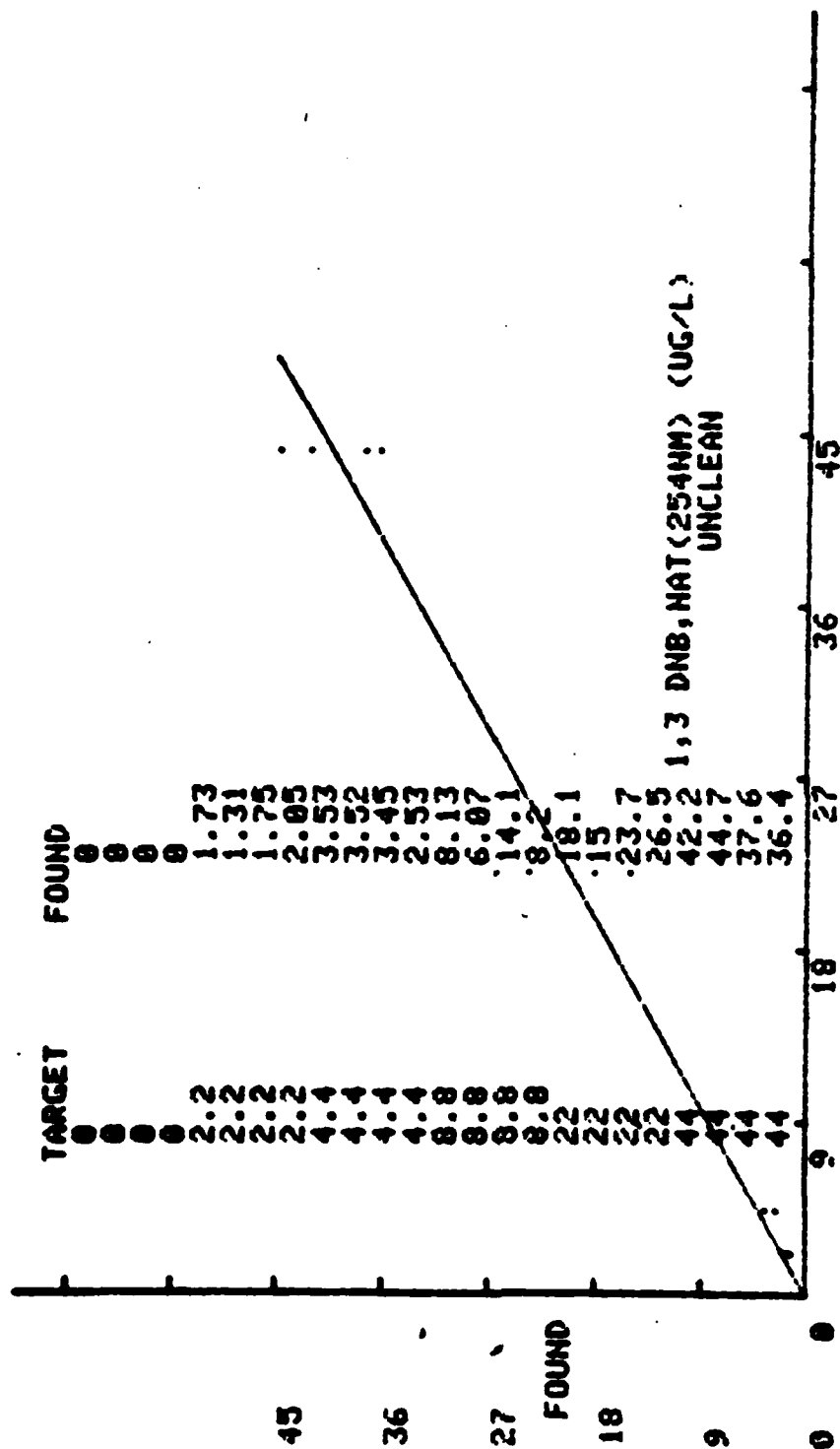
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	1.49	1.78	3.40	3.38
4.40	3.99	3.93	3.42	3.42
8.80	7.11	8.89	8.17	8.69
22.0	24.1	18.6	18.3	18.7
44.0	38.3	39.9	34.9	36.4

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	2.51	1.02	40.6	14.2
4.40	3.69	0.313	8.48	-16.1
8.80	8.21	0.797	9.70	-6.65
22.0	19.9	2.79	14.0	-9.43
44.0	37.4	2.18	5.84	-15.1

1,3 DNB,NAT(254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	1.73	1.31	1.75	2.05
4.40	3.53	3.52	3.45	2.53
8.80	8.13	6.07	14.1	8.20
22.0	18.1	15.0	23.7	26.5
44.0	42.2	44.7	37.6	36.4

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	1.71	0.304	17.8	-22.3
4.40	3.26	0.486	14.9	-26.0
8.80	9.12	3.46	37.9	3.69
22.0	20.8	5.22	25.1	-5.34
44.0	40.2	3.89	9.68	-8.58



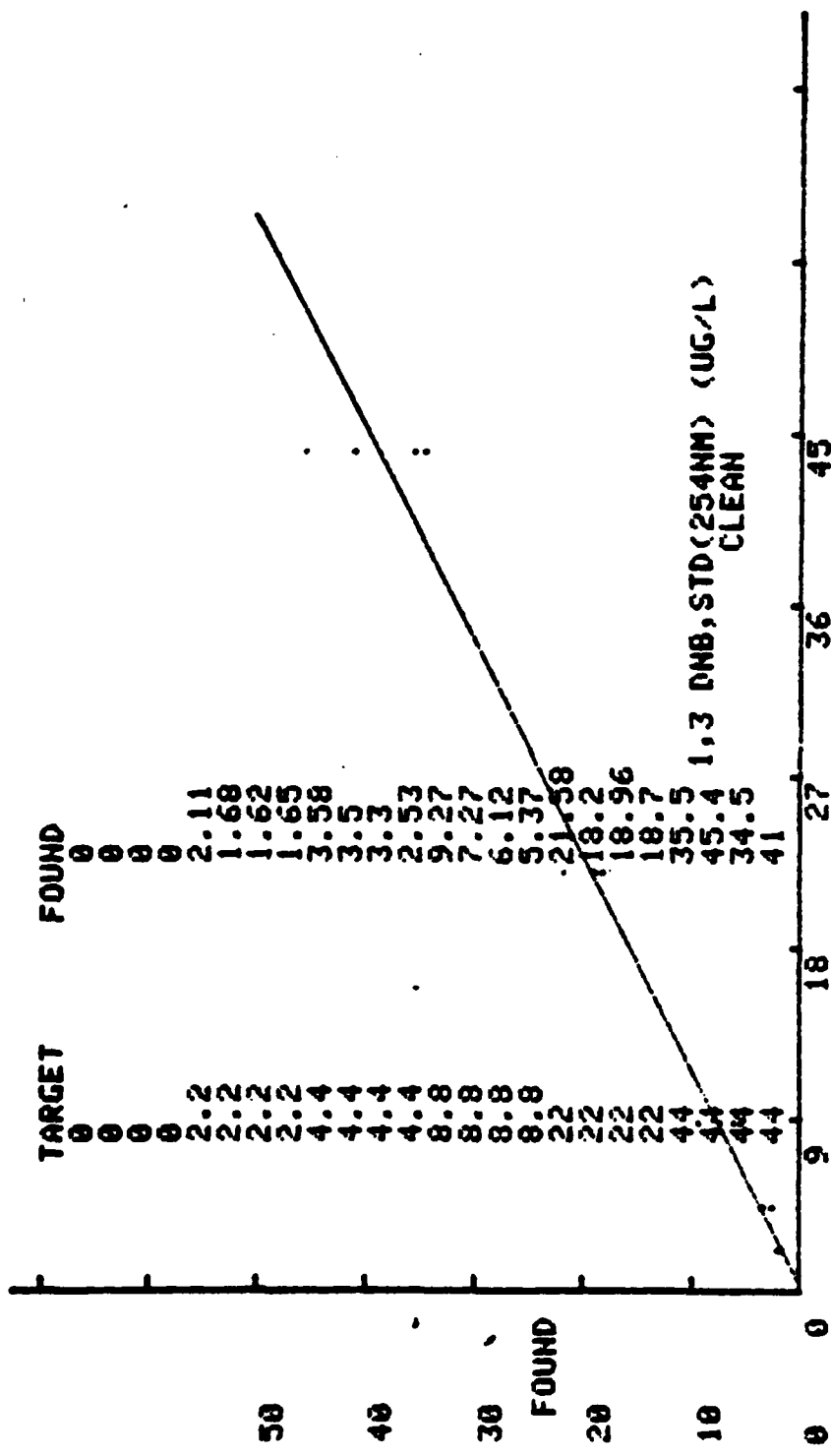
CORR. COEFF. = 0.9825 FOUND = TARGET
DETECTION LIMIT = 10.61057 0.0129+ 0.922176+TARGET

1.3 DNB, STD(254NM) (UG/L)

CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	2.11	1.68	1.62	1.65
4.40	3.58	3.50	3.30	2.53
8.80	9.27	7.27	6.12	5.37
22.0	21.6	18.2	19.0	18.7
44.0	35.5	45.4	34.5	41.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	1.76	0.231	13.1	-19.8
4.40	3.23	0.480	14.9	-26.6
8.80	7.01	1.70	24.2	-20.4
22.0	19.4	1.51	7.82	-12.0
44.0	39.1	5.08	13.0	-11.1



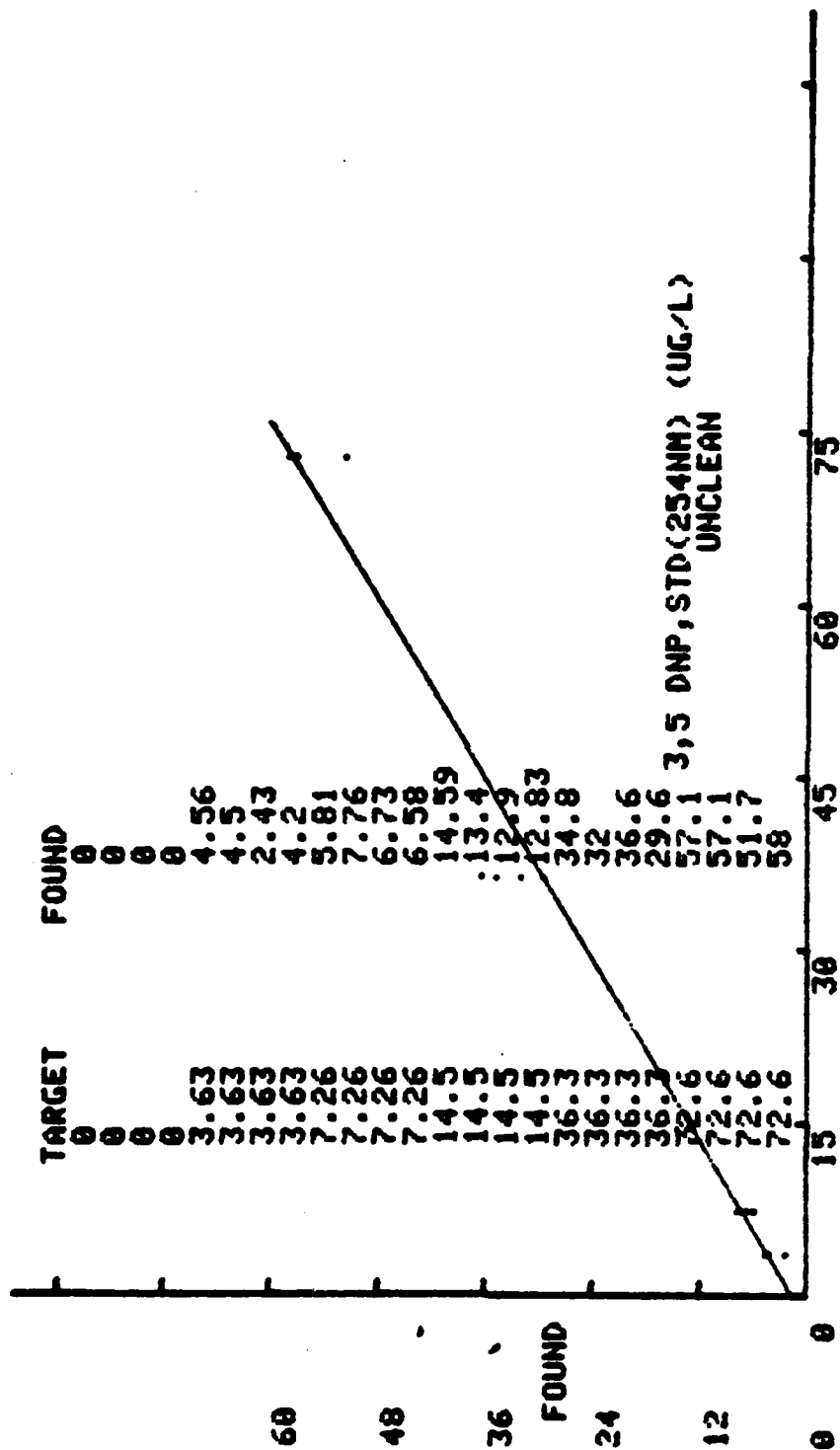
1,3 DNB, STD(254NM) (UG/L)
CLEAN

TARGET
CORR. COEFF. = 0.9896 FOUND = -0.4148+ 0.896176xTARGET
DETECTION LIMIT = 8.133

3,5 DNP,STD(254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	4.56	4.50	2.43	4.20
7.26	5.81	7.76	6.73	6.58
14.5	14.6	13.4	12.9	12.8
36.3	34.8	32.0	36.6	29.6
72.6	57.1	57.1	51.7	58.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	3.92	1.01	25.7	8.06
7.26	6.72	0.802	11.9	-7.44
14.5	13.4	0.814	6.06	-7.38
36.3	33.2	3.08	9.27	-8.40
72.6	56.0	2.88	5.15	-22.9

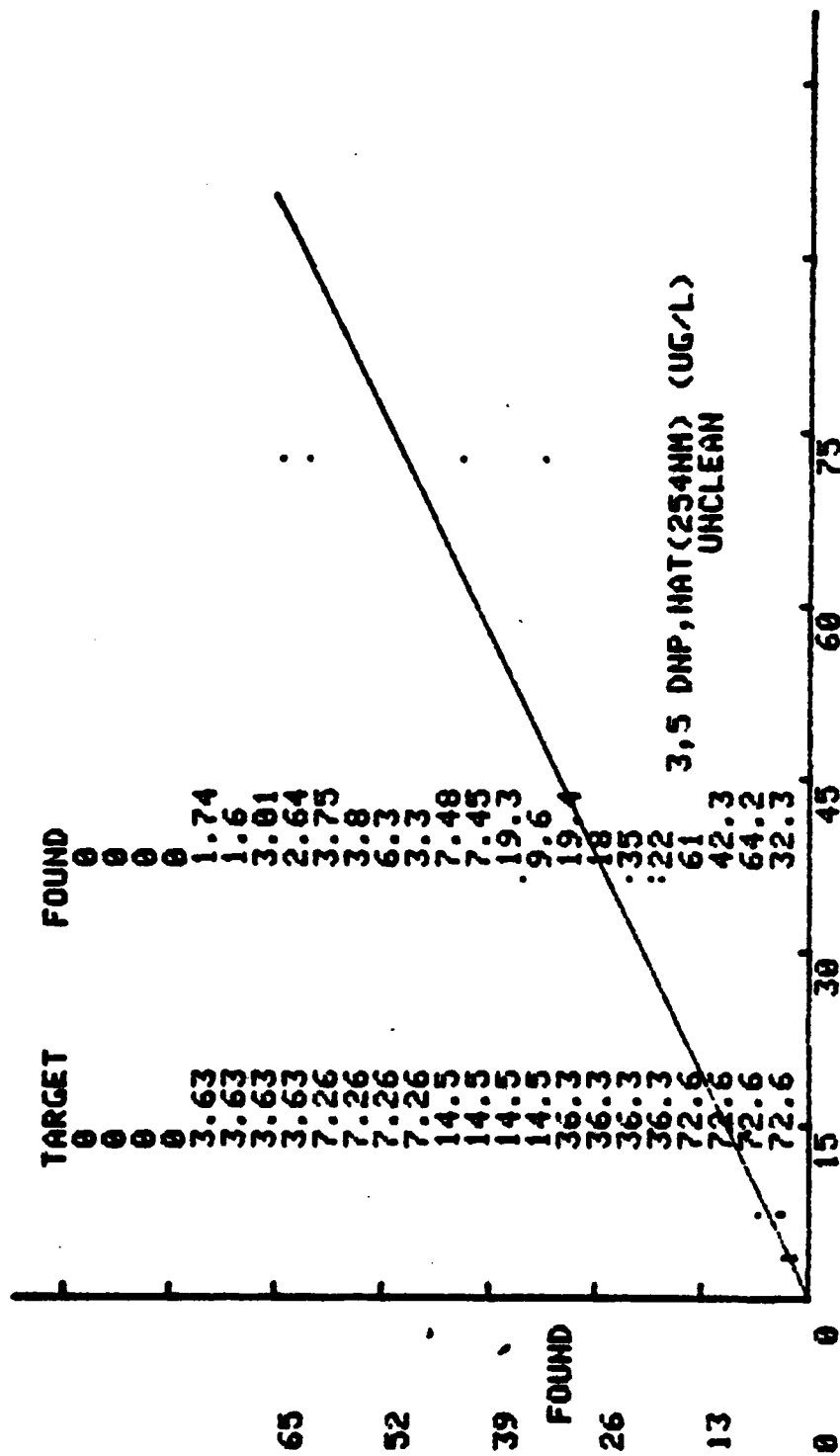


CORR. COEFF. = 0.9927
 DETECTION LIMIT = 11.25991
 TARGET
 FOUND = 1.5253+
 0.775531 * TARGET

3,5 DNP,NAT(254NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	1.74	1.60	3.31	2.64
7.26	3.75	3.80	6.30	3.30
14.5	7.48	7.45	19.3	9.60
36.3	19.4	18.0	35.0	22.0
72.6	61.0	42.3	64.2	32.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	2.25	0.686	30.5	-38.1
7.26	4.29	1.36	31.7	-40.9
14.5	11.0	5.65	51.6	-24.4
36.3	23.6	7.78	33.0	-35.0
72.6	49.9	15.2	30.5	-31.2

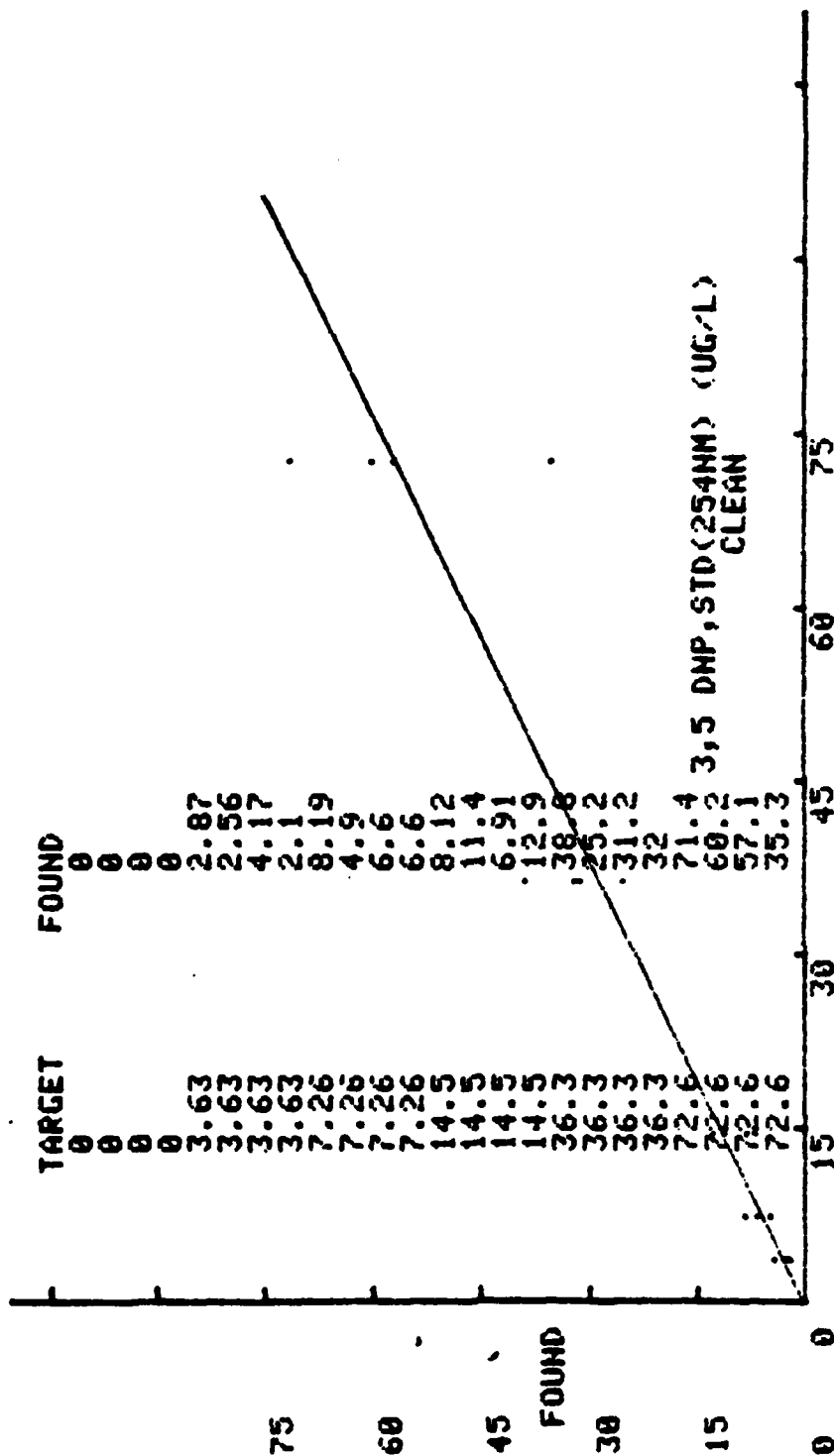


TARGET
CORR. COEFF. = 0.9379 FOUND = -0.1586+ 0.685040+TARGET
DETECTION LIMIT = 34.21232

3,5 DNP,STD(254NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	2.87	2.56	4.17	2.10
7.26	8.19	4.90	6.60	6.60
14.5	8.12	11.4	6.91	12.9
36.3	38.8	25.2	31.2	32.0
72.6	71.4	60.2	57.1	35.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	2.92	0.888	30.4	-19.4
7.26	6.57	1.34	20.4	-9.47
14.5	9.83	2.79	28.4	-32.2
36.3	31.8	5.57	17.5	-12.4
72.6	56.0	15.1	27.0	-22.0

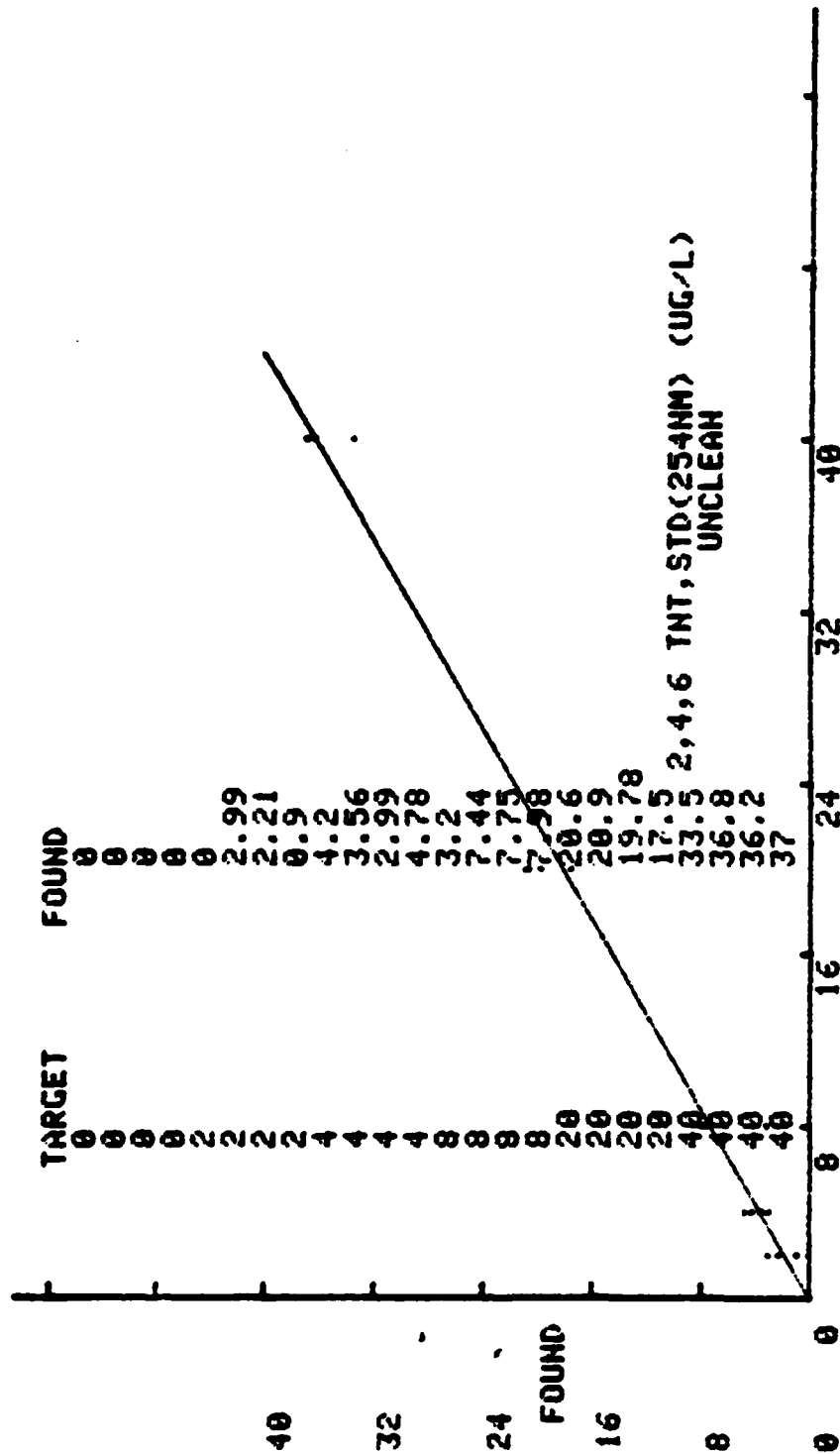


TARGET
CORR. COEFF. = 0.9572 FOUND = 0.3212+ 0.783399*TARGET
DETECTION LIMIT = 27.96878

2,4,6 TNT,STD(254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.0000	2.99	2.21	0.900
4.00	4.20	3.56	2.99	4.78
8.00	3.20	7.44	7.75	7.98
20.0	20.6	20.9	19.8	17.5
40.0	33.5	36.8	36.2	37.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.52	1.33	87.4	-23.8
4.00	3.88	0.776	20.0	-2.94
8.00	6.59	2.27	34.5	-17.6
20.0	19.7	1.54	7.81	-1.53
40.0	35.9	1.62	4.51	-10.3



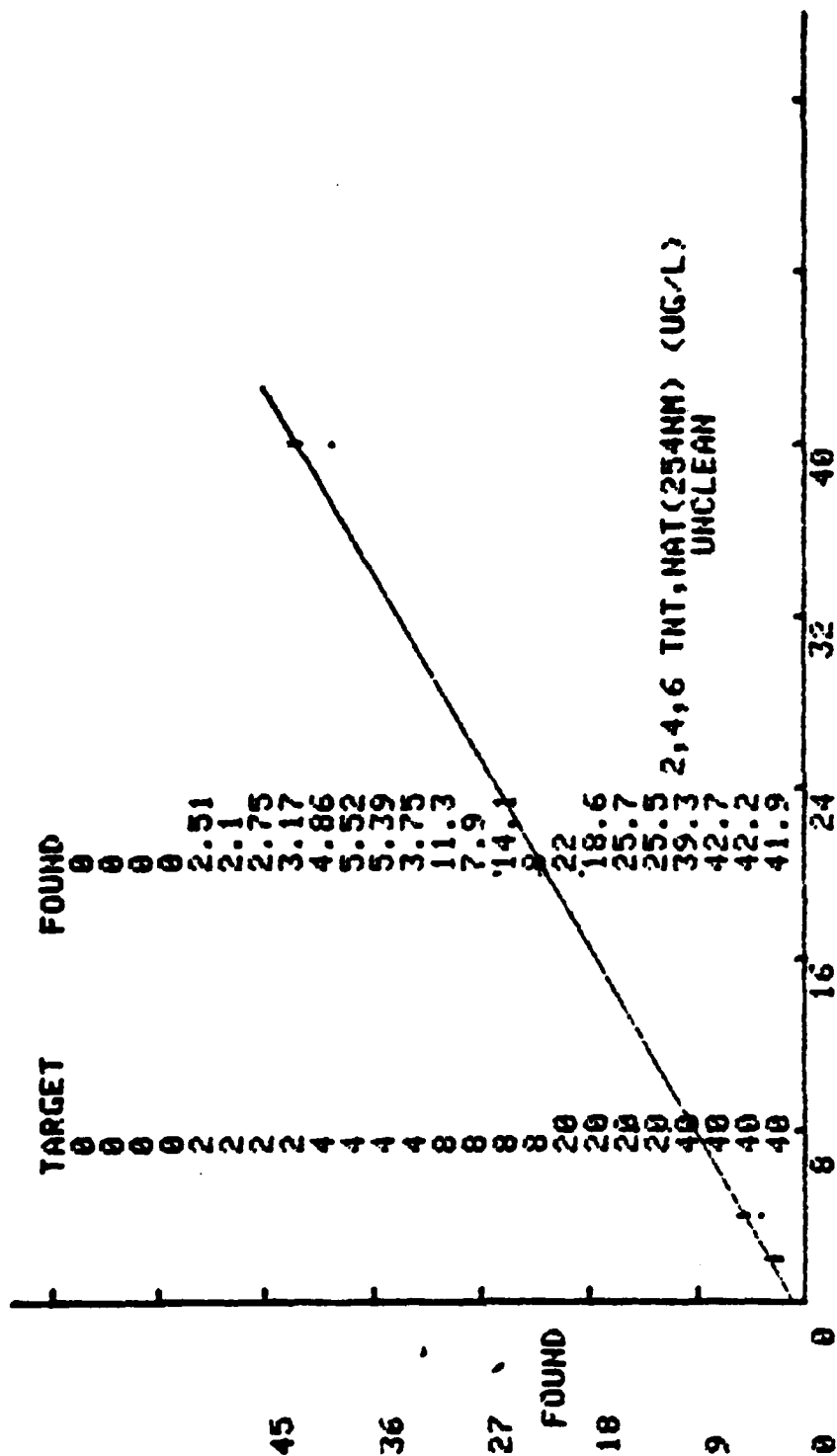
CORR. COEFF. = 0.9936
DETECTION LIMIT = 5.78173

TARGET
0.910805 * TARGET

2,4,6 TNT,NAT(254NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.51	2.10	2.75	3.17
4.00	4.86	5.52	5.39	3.75
8.00	11.3	7.90	14.1	8.00
20.0	22.0	18.6	25.7	25.5
40.0	39.3	42.7	42.2	41.9

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.53	0.448	17.0	31.4
4.00	4.88	0.806	16.5	22.0
8.00	10.3	2.97	28.8	29.1
20.0	22.9	3.36	14.6	14.7
40.0	41.5	1.52	3.66	3.81

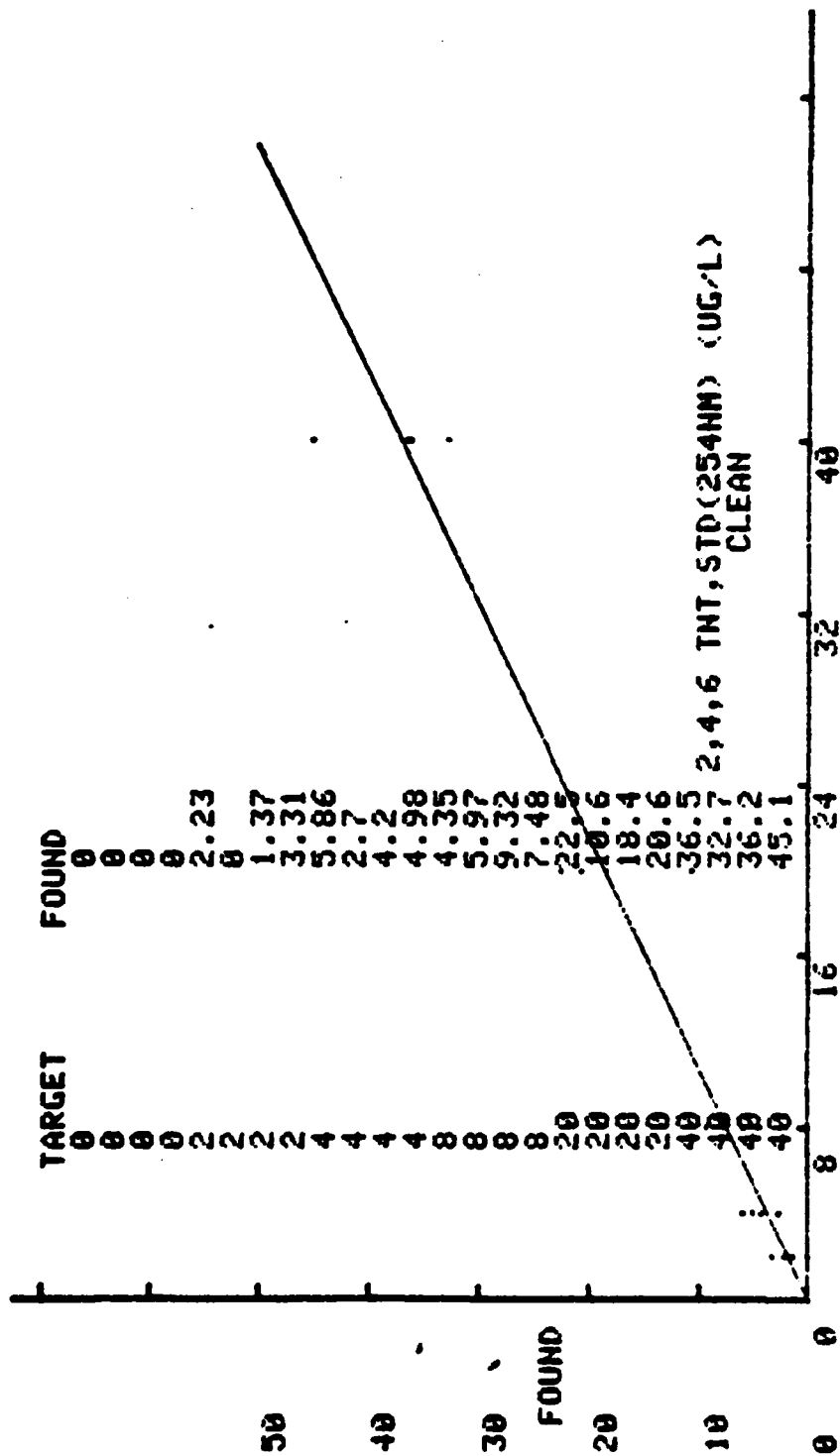


TARGET
CORR. COEFF. = 0.9913 FOUND = 0.9551+ 1.034887XTARGET
DETECTION LIMIT = 6.76562

2,4,6 TNT, STD (254NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.23	0.0000	1.37	3.31
4.00	5.86	2.70	4.20	4.98
8.00	4.35	5.97	9.32	7.48
20.0	22.5	10.6	18.4	20.6
40.0	36.5	32.7	36.2	45.1

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.73	1.40	81.0	-13.6
4.00	4.43	1.34	30.2	10.9
8.00	6.78	2.12	31.3	-15.3
20.0	18.0	5.23	29.0	-9.88
40.0	37.6	5.27	14.0	-5.94



CORR. COEFF. = 0.9769
 DETECTION LIMIT = 11.14182
 TARGET
 FOUND = -0.0971+
 TARGET = 0.934799*TARGET

2,6 DNT,STD(230NM) (UG/L)

UNCLEAN

TARGET
CONCENTRATION

1

DAY
2

3

4

0.0000

0.0000

0.0000

0.0000

0.0000

2.28

1.46

2.10

0.630

1.22

4.56

1.68

2.93

3.94

3.24

9.12

3.94

8.16

8.17

8.40

22.8

17.8

17.1

20.9

23.3

45.6

34.8

43.9

36.0

37.3

TARGET
CONCENTRATION

AVERAGE
FOUND VALUE

STANDARD
DEVIATION

PERCENT
IMPRECISION

PERCENT
INACCURACY

0.0000

0.0000

0.0000

0.0000

0.0000

2.28

1.35

0.608

45.0

-40.7

4.56

2.95

0.945

32.1

-35.4

9.12

7.17

2.15

30.1

-21.4

22.8

19.8

2.87

14.5

-13.3

45.6

38.0

4.06

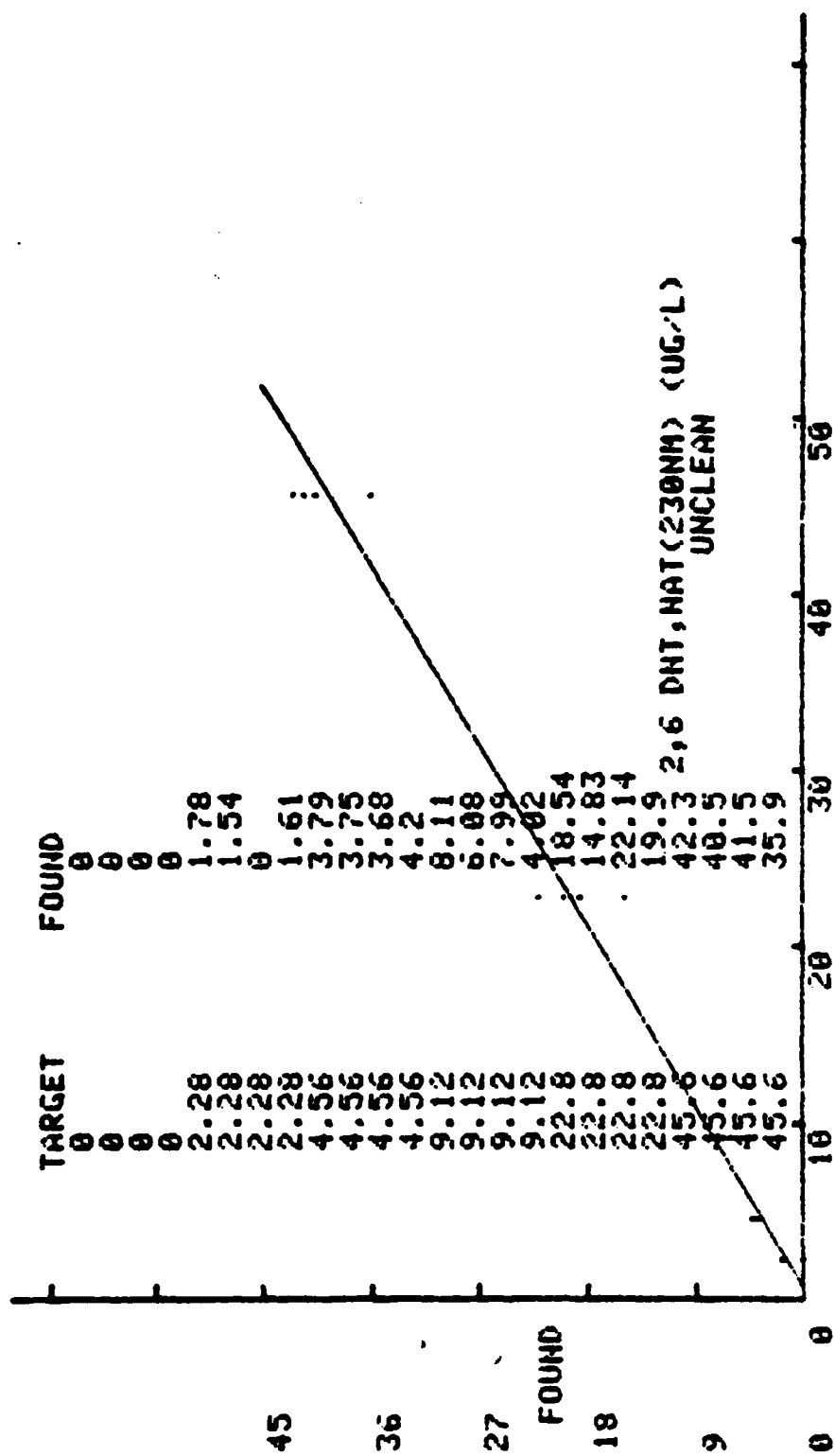
10.7

-16.7

2,6 DNT,NAT(23CNH) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.28	1.78	1.54	0.0000	1.61
4.56	3.79	3.75	3.68	4.20
9.12	8.11	6.08	7.99	4.02
22.8	18.5	14.8	22.1	19.9
45.6	42.3	40.5	41.5	35.9

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.28	1.23	0.828	67.2	-45.9
4.56	3.85	0.234	6.02	-15.5
9.12	6.55	1.93	29.4	-28.2
22.8	18.9	3.07	16.3	-17.3
45.6	40.0	2.86	7.15	-12.2

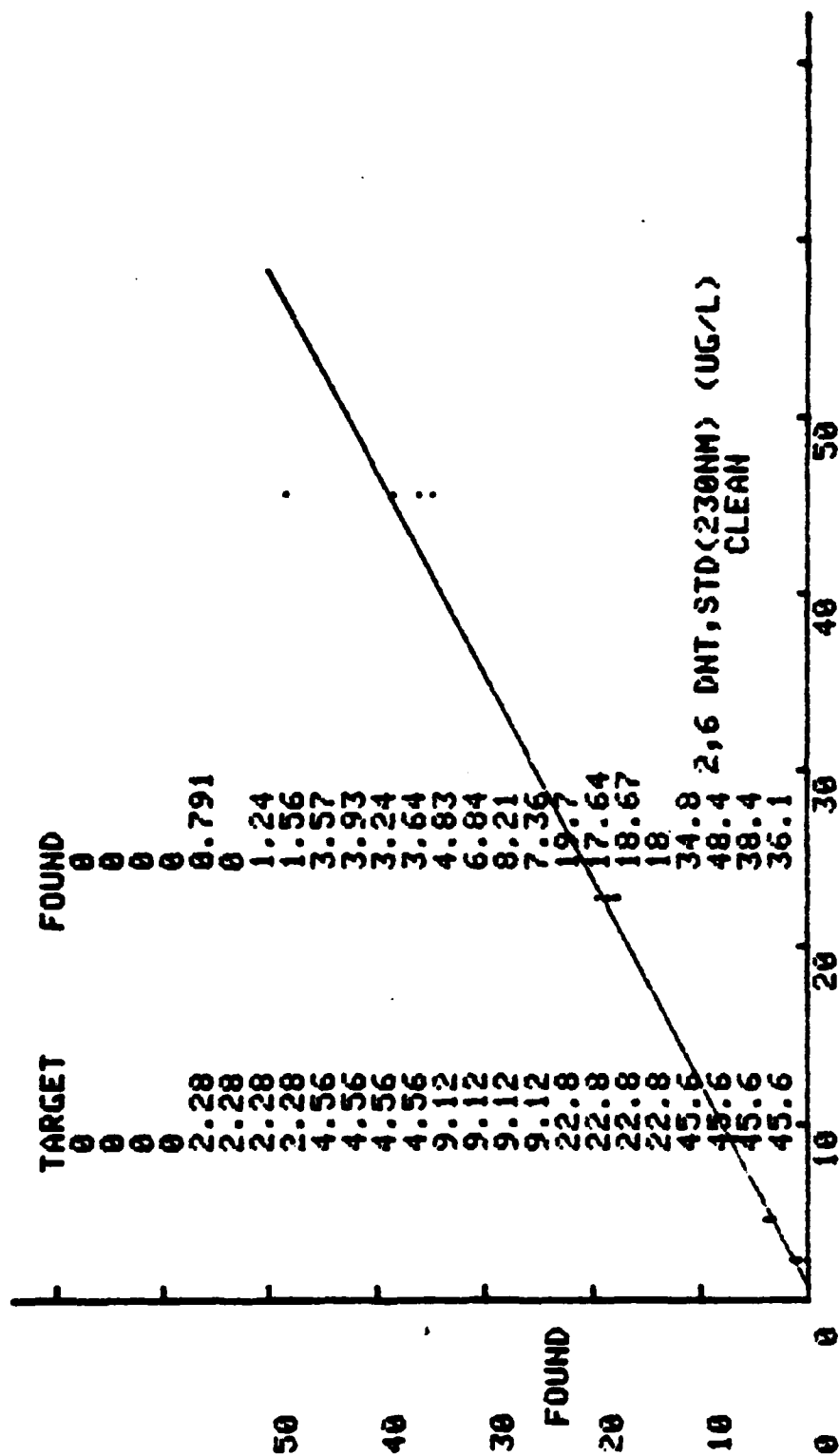


TARGET
CORR. COEFF. = 0.9923 FOUND = -0.6610+ 0.883190*TARGET
DETECTION LIMIT = 7.2509

2.6 DNT, STD(230NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	0.791	0.000	1.24	1.56
4.56	3.57	3.93	3.24	3.64
9.12	4.83	6.84	8.21	7.36
22.0	19.7	17.6	18.7	18.0
45.6	34.8	48.4	38.4	36.1

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.20	0.898	0.677	75.4	-60.6
4.56	3.59	0.283	7.88	-21.2
9.12	6.81	1.44	21.1	-25.3
22.0	18.5	0.905	4.85	-18.8
45.6	39.4	6.17	15.6	-13.5

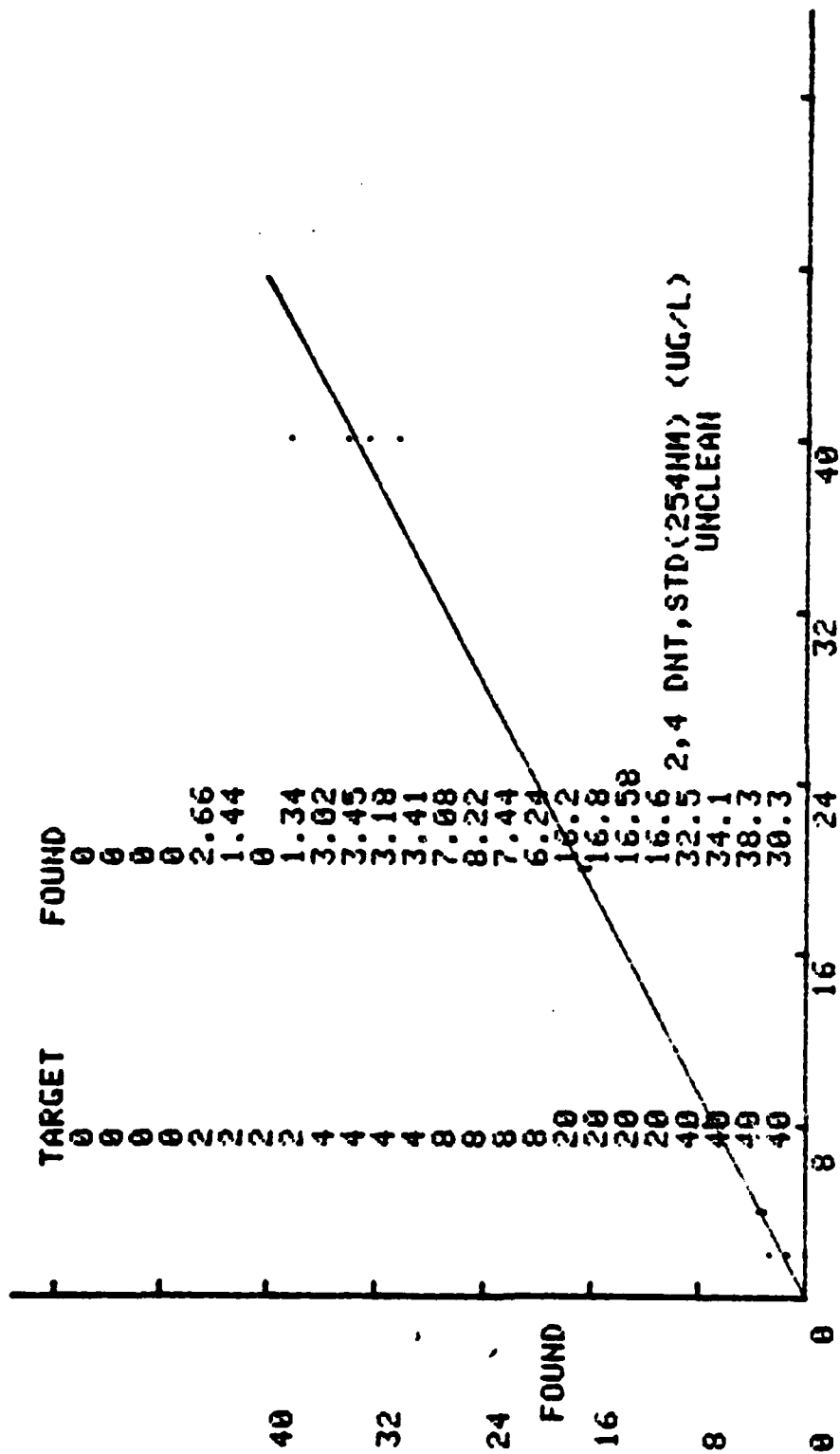


TARGET
CORR. COEFF. = 0.9862 FOUND = -0.7163+ 0.871601*TARGET
DETECTION LIMIT = 9.75064

2,4 DNT,STD(254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	2.66	1.44	0.0000	1.34
4.00	3.02	3.45	3.18	3.41
8.00	7.08	8.22	7.44	6.24
20.0	16.2	16.8	16.6	16.6
40.0	32.5	34.1	38.3	30.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.36	1.09	79.9	-32.0
4.00	3.26	0.202	6.19	-18.4
8.00	7.24	0.822	11.3	-9.44
20.0	16.5	0.251	1.52	-17.3
40.0	33.8	3.38	10.0	-15.5

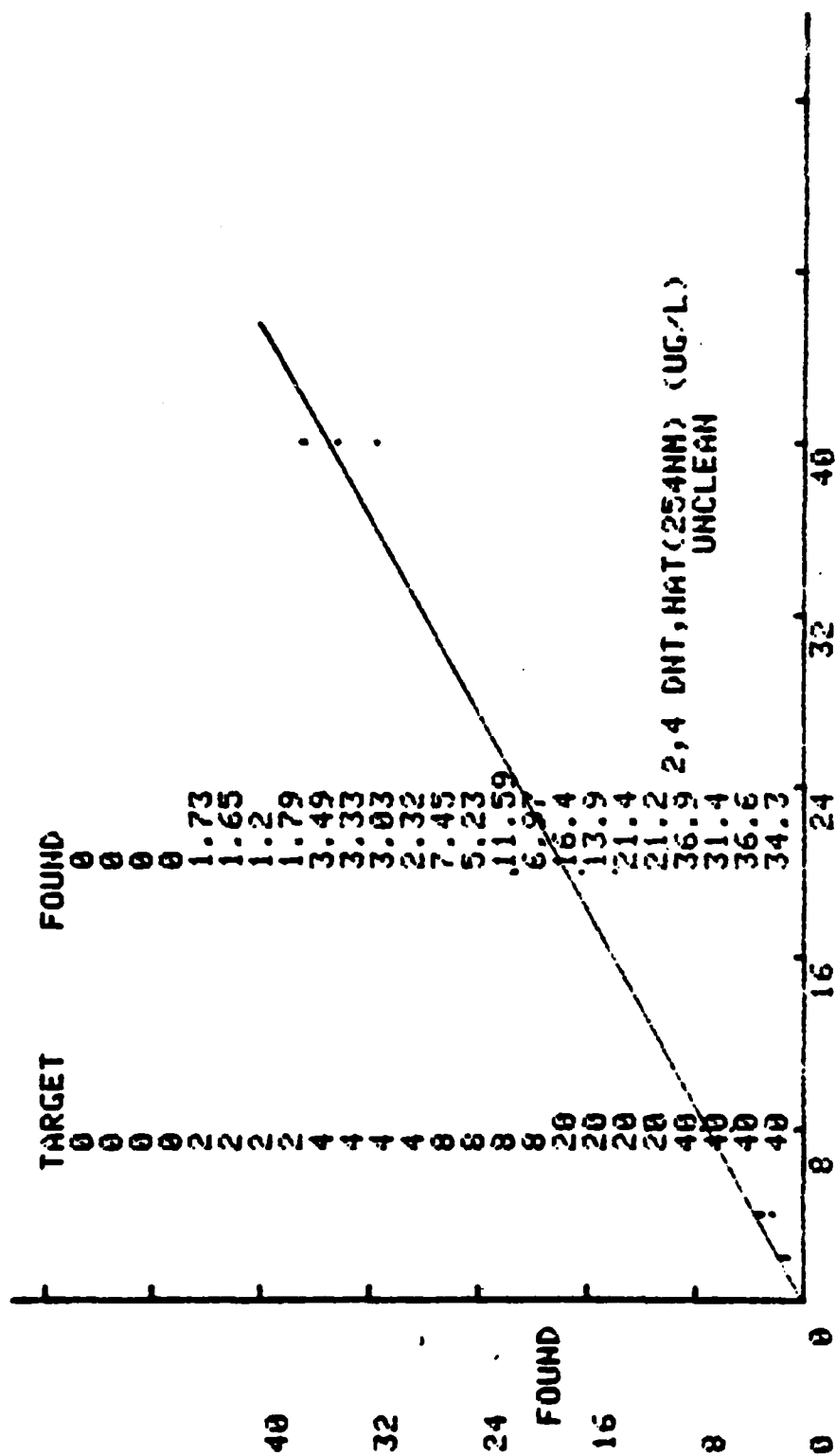


TARGET
 CORR. COEFF. = 0.9938 FOUND = -0.0477+ 0.844612*TARGET
 DETECTION LIMIT = 5.71221

2,4 DNT,NAT(254NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.73	1.65	1.20	1.79
4.00	3.49	3.33	3.03	2.32
8.00	7.45	5.23	11.6	6.97
20.0	16.4	13.9	21.4	21.2
40.0	36.9	31.4	36.6	34.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.59	0.268	16.8	-20.4
4.00	3.04	0.518	17.0	-23.9
8.00	7.81	2.69	34.5	-2.30
20.0	18.2	3.70	20.3	-8.88
40.0	34.8	2.55	7.32	-13.0



TARGET
 CORR. COEFF. = 0.9879 FOUND = 0.0994+ 0.876669*TARGET
 DETECTION LIMIT = 7.99442

2,4 DNT, STD(254NM) (UG/L) CLEAN

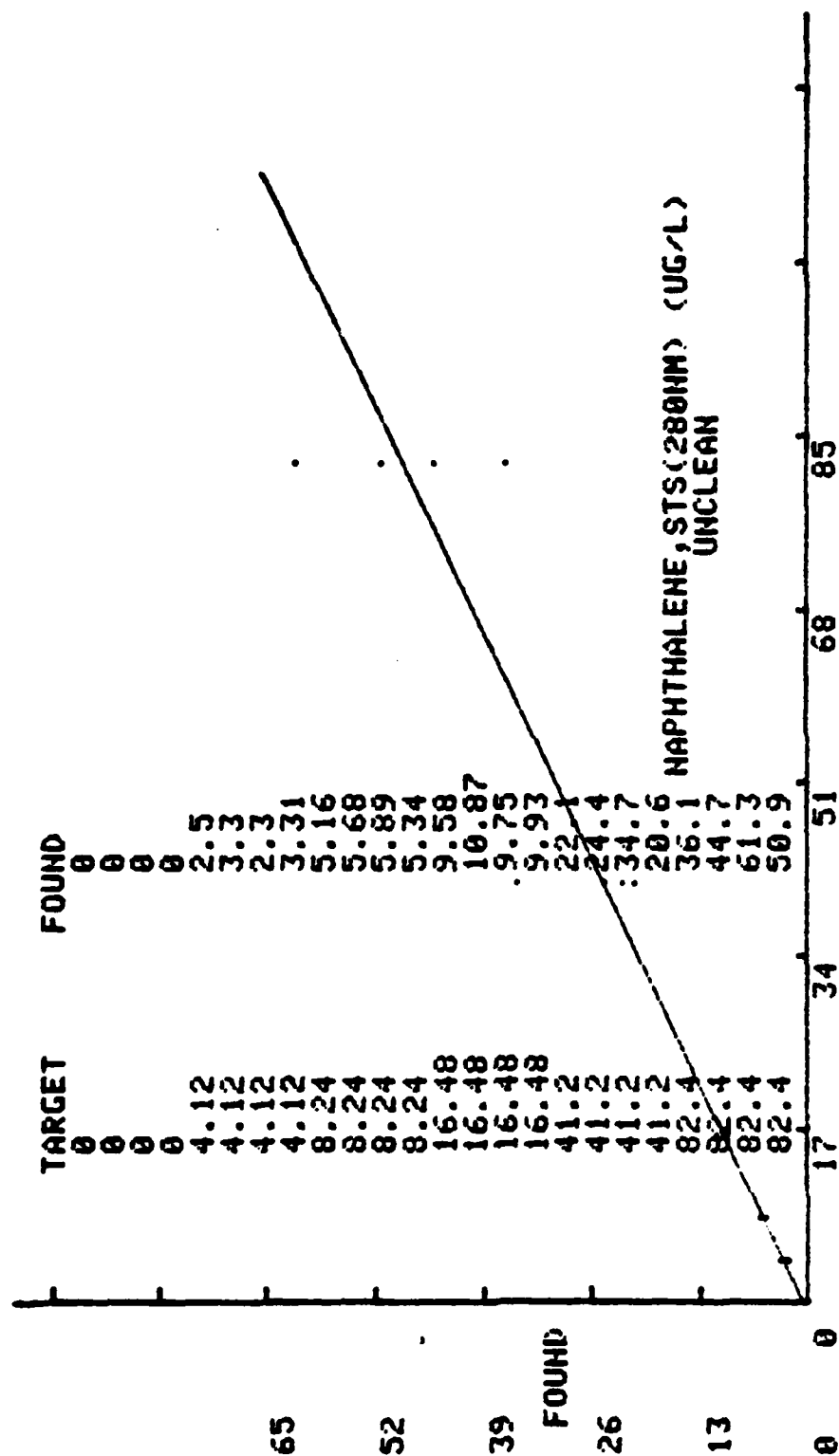
TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	1.86	0.0000	0.0000	1.62
4.00	3.13	2.91	3.00	1.81
8.00	7.51	6.31	5.46	5.92
20.0	18.5	16.5	16.6	16.2
40.0	31.2	38.7	30.2	35.4

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.870	1.01	116	-56.5
4.00	2.71	0.608	22.4	-32.2
8.00	6.30	0.878	13.9	-21.3
20.0	17.0	1.04	6.13	-15.2
40.0	33.9	3.93	11.6	-15.3

NAPHTHALENE,STD(280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	2.50	3.30	2.30	3.31
8.24	5.16	5.68	5.89	5.34
16.5	9.58	10.9	9.75	9.93
41.2	22.1	24.4	34.7	20.6
82.4	36.1	44.7	61.3	50.9

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	2.85	0.529	18.5	-30.8
8.24	5.52	0.329	5.96	-33.0
16.5	10.0	0.576	5.74	-39.1
41.2	25.4	6.36	25.0	-38.2
82.4	48.2	10.6	22.0	-41.4

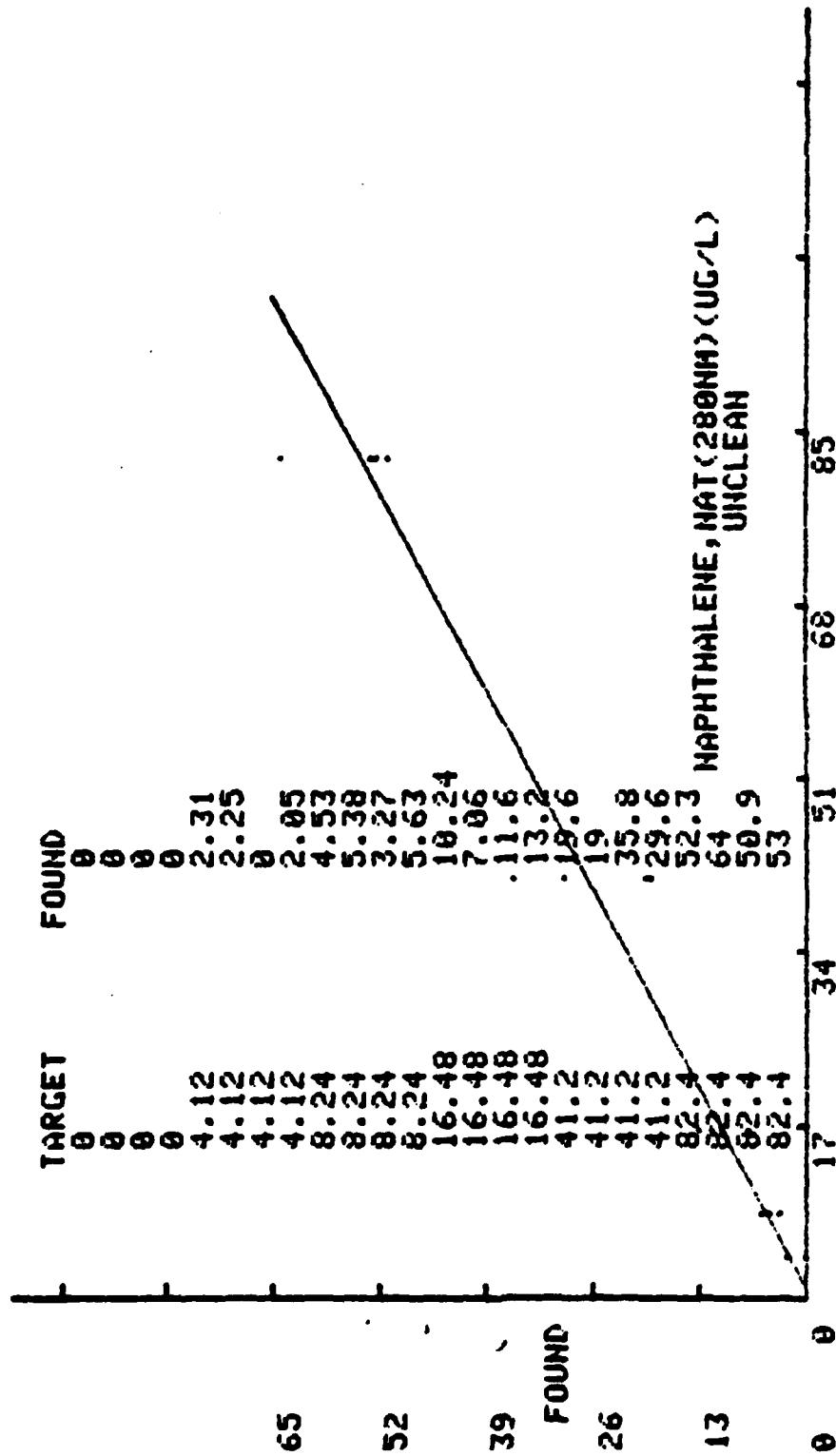


CORR. COEFF. = 0.9674
 DETECTION LIMIT = 27.47117
 TARGET FOUND = 0.4925+
 0.584805 * TARGET

NAPHTHALENE,NAT(280NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	2.31	2.25	0.0000	2.05
8.24	4.53	5.38	3.27	5.63
16.5	10.2	7.06	11.6	13.2
41.2	19.6	19.0	35.8	29.6
82.4	52.3	64.0	50.9	53.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	1.65	1.11	67.0	-59.9
8.24	4.70	1.06	22.6	-42.9
16.5	10.5	2.61	24.8	-36.1
41.2	26.0	8.14	31.3	-36.9
82.4	55.0	6.03	11.0	-33.2

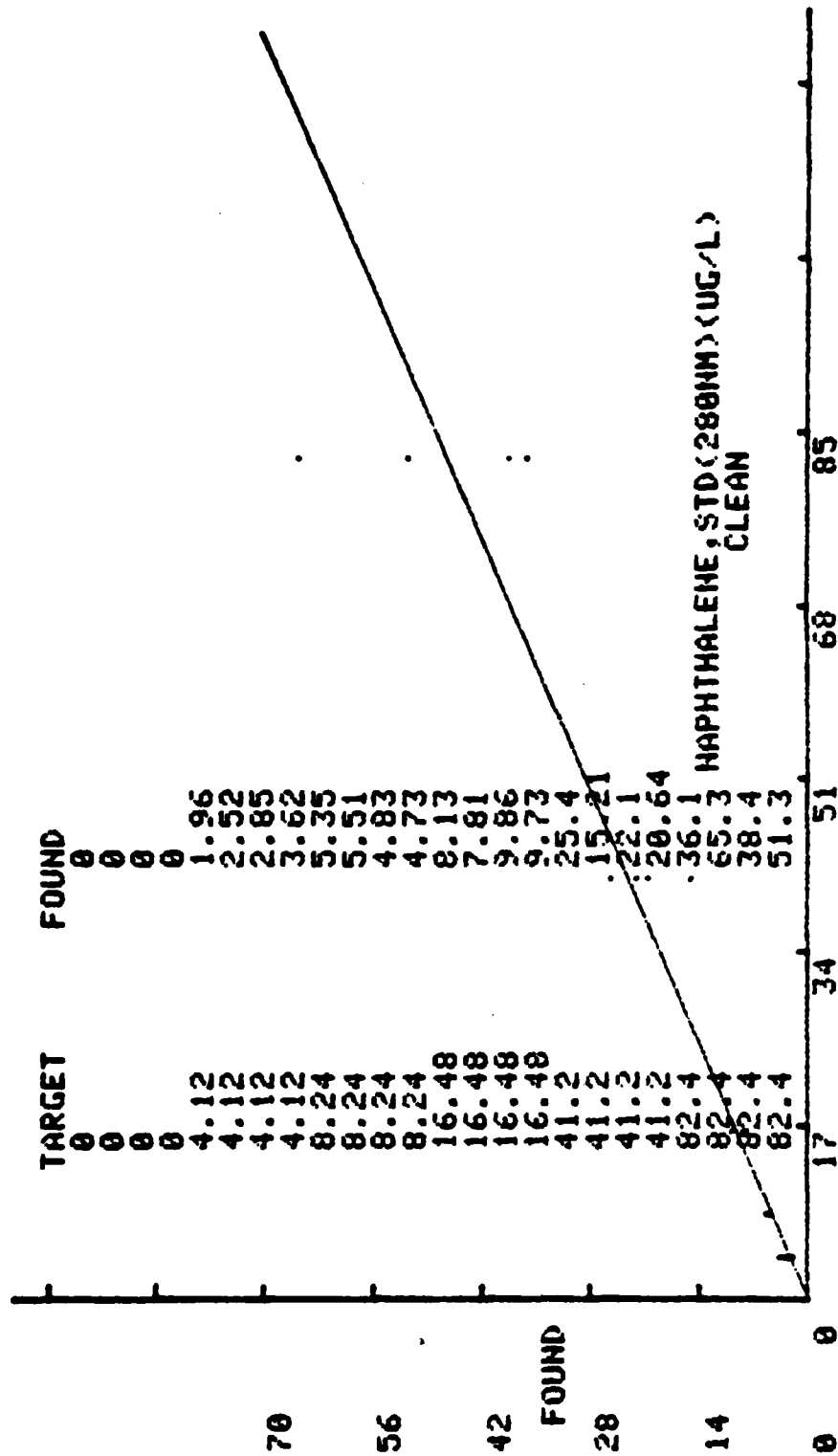


CORR. COEFF. = 0.9814
 DETECTION LIMIT = 20.51429
 TARGET
 FOUND = -0.7412+ 0.671590*TARGET

NAPHTHALENE, STD (280NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	1.96	2.52	2.85	3.62
8.24	5.35	5.51	4.83	4.73
16.5	8.13	7.81	9.86	9.73
41.2	25.4	15.2	22.1	20.6
82.4	36.1	65.3	38.4	51.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
4.12	2.74	0.694	25.3	-33.6
8.24	5.10	0.383	7.50	-38.0
16.5	8.88	1.06	12.0	-46.1
41.2	20.8	4.25	20.4	-49.4
82.4	47.8	13.5	28.2	-42.0

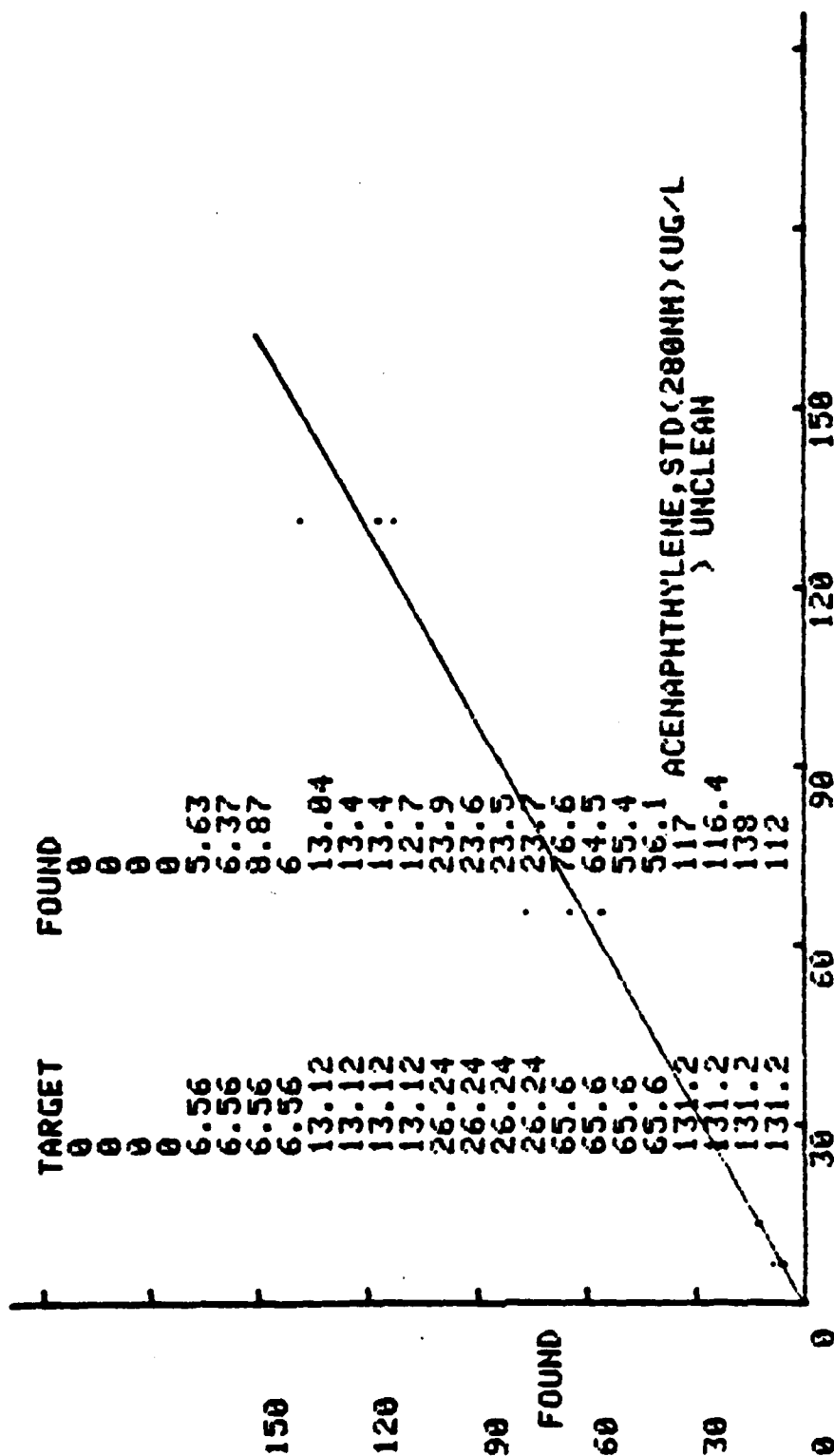


TARGET
CORR. COEFF. = 0.9540 FOUND = -0.2255+ 0.568686*TARGET
DETECTION LIMIT = 32.96101

ACENAPHTHYLENE, STD (280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	5.63	6.37	8.87	6.00
13.1	13.0	13.4	13.4	12.7
26.2	23.9	23.6	23.5	23.7
65.6	76.6	64.5	55.4	56.1
131	117	116	138	112

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	6.72	1.47	21.8	2.40
13.1	13.1	0.336	2.56	0.114
26.2	23.7	0.171	0.723	-9.78
65.6	63.1	9.87	15.6	-3.73
131	121	11.6	9.64	-7.89

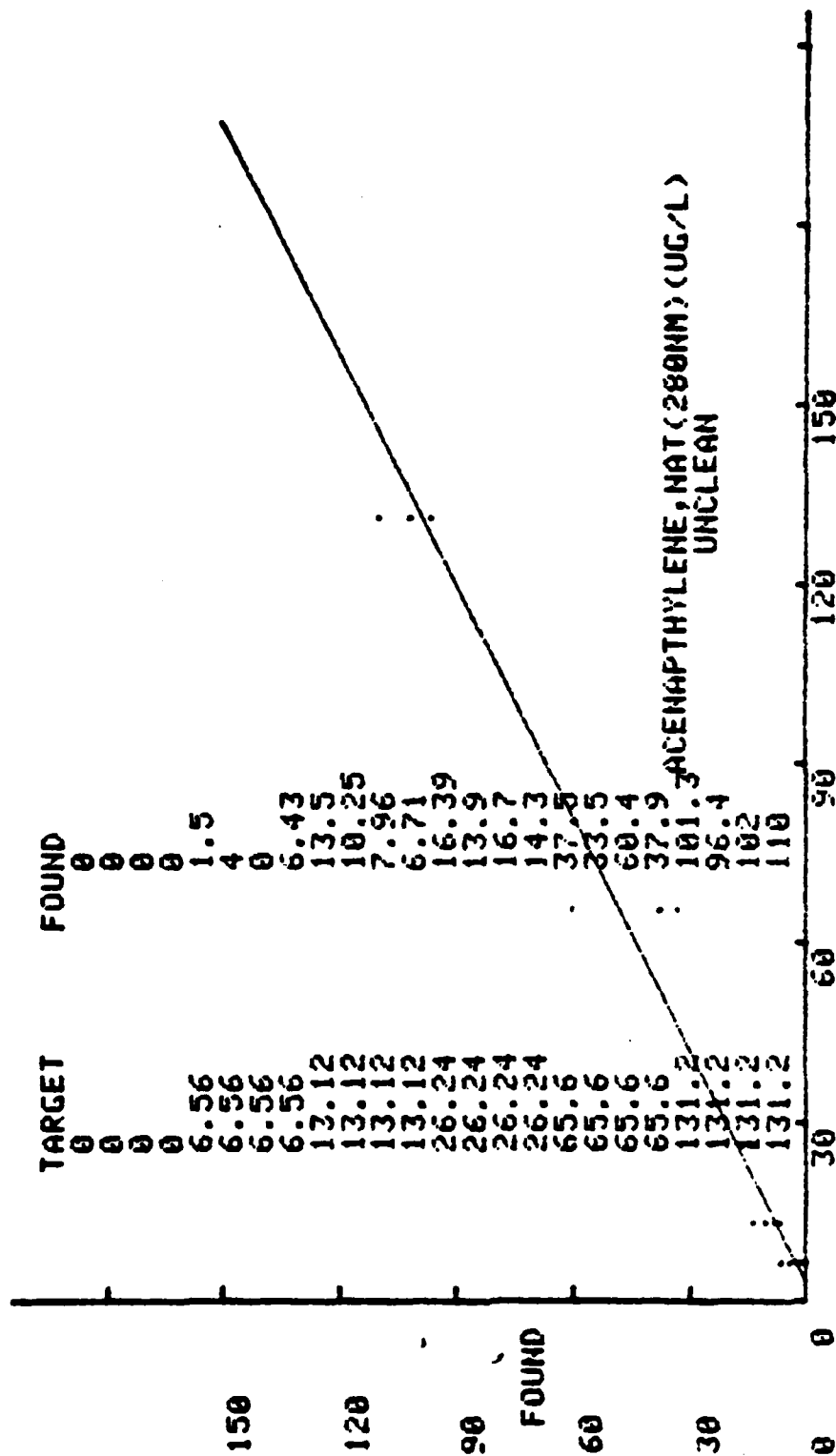


CORR. COEFF. = 0.9916
 DETECTION LIMIT = 21.82465
 TARGET
 FOUND = 0.923016 * TARGET
 0.5822+

ACENAPHTHYLENE, NAT (280NM) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	1.50	4.00	0.0000	6.43
13.1	13.5	10.3	7.96	6.71
26.2	16.4	13.9	16.7	14.3
65.6	37.5	33.5	60.4	37.9
131	101	96.4	102	110

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	2.98	2.83	94.9	-54.5
13.1	9.60	2.98	31.0	-26.8
26.2	15.3	1.43	9.31	-41.6
65.6	42.3	12.2	28.9	-35.5
131	102	5.63	5.50	-21.9

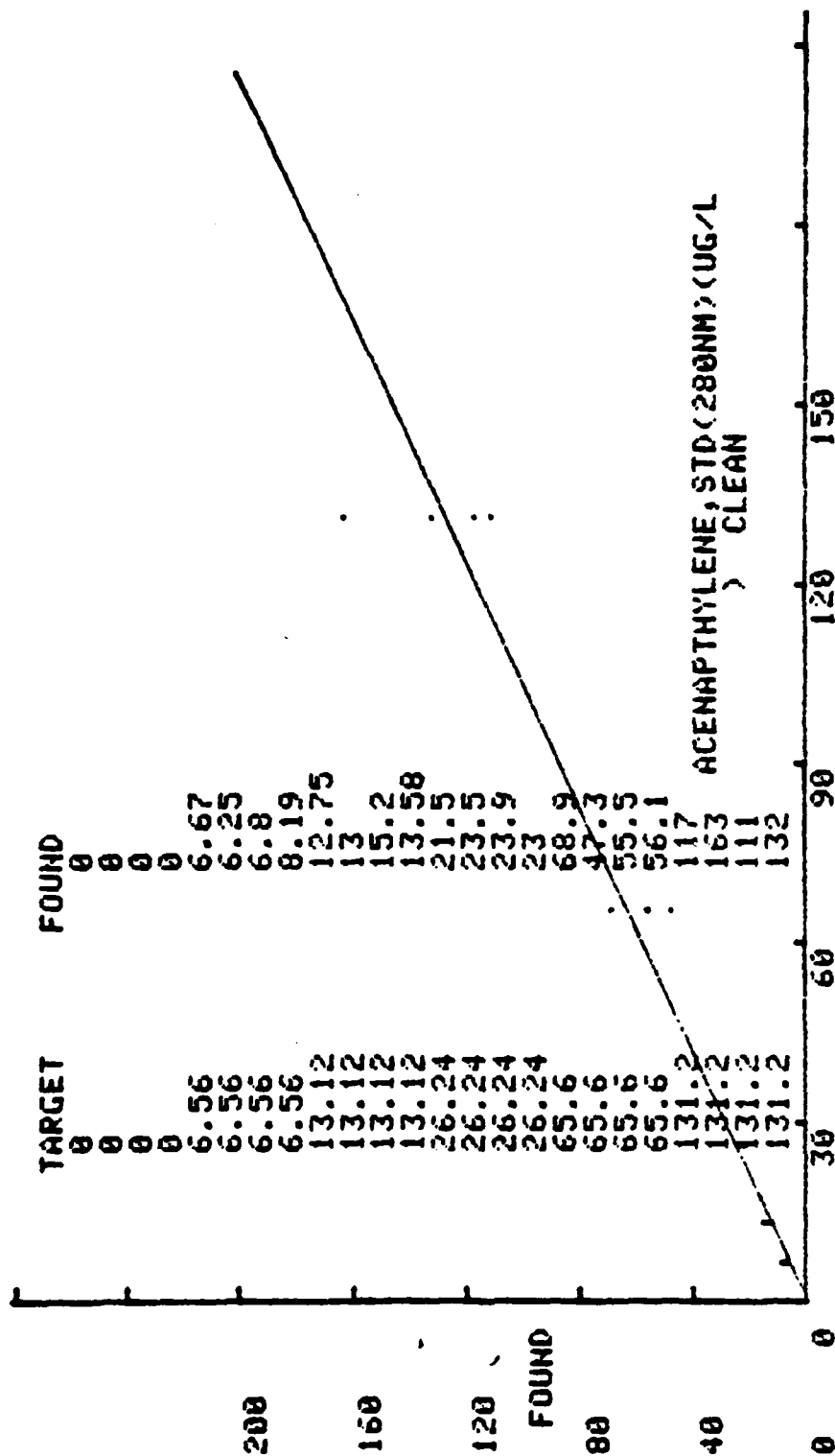


CORR. COEFF. = 0.9863 FOUND = TARGET
DETECTION LIMIT = 27.99501 TARGET = 0.775901 * TARGET

ACENAPHTHYLENE, STD (286NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	6.67	6.25	6.80	6.19
13.1	12.8	13.0	15.2	13.6
26.2	21.5	23.5	23.9	23.0
65.6	68.9	47.3	55.5	56.1
131	117	163	111	132

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
6.56	6.58	0.842	12.1	6.36
13.1	13.6	1.10	8.38	3.91
26.2	23.0	1.05	4.57	-12.4
65.6	56.7	8.92	15.7	-13.2
131	131	23.2	17.8	-0.343

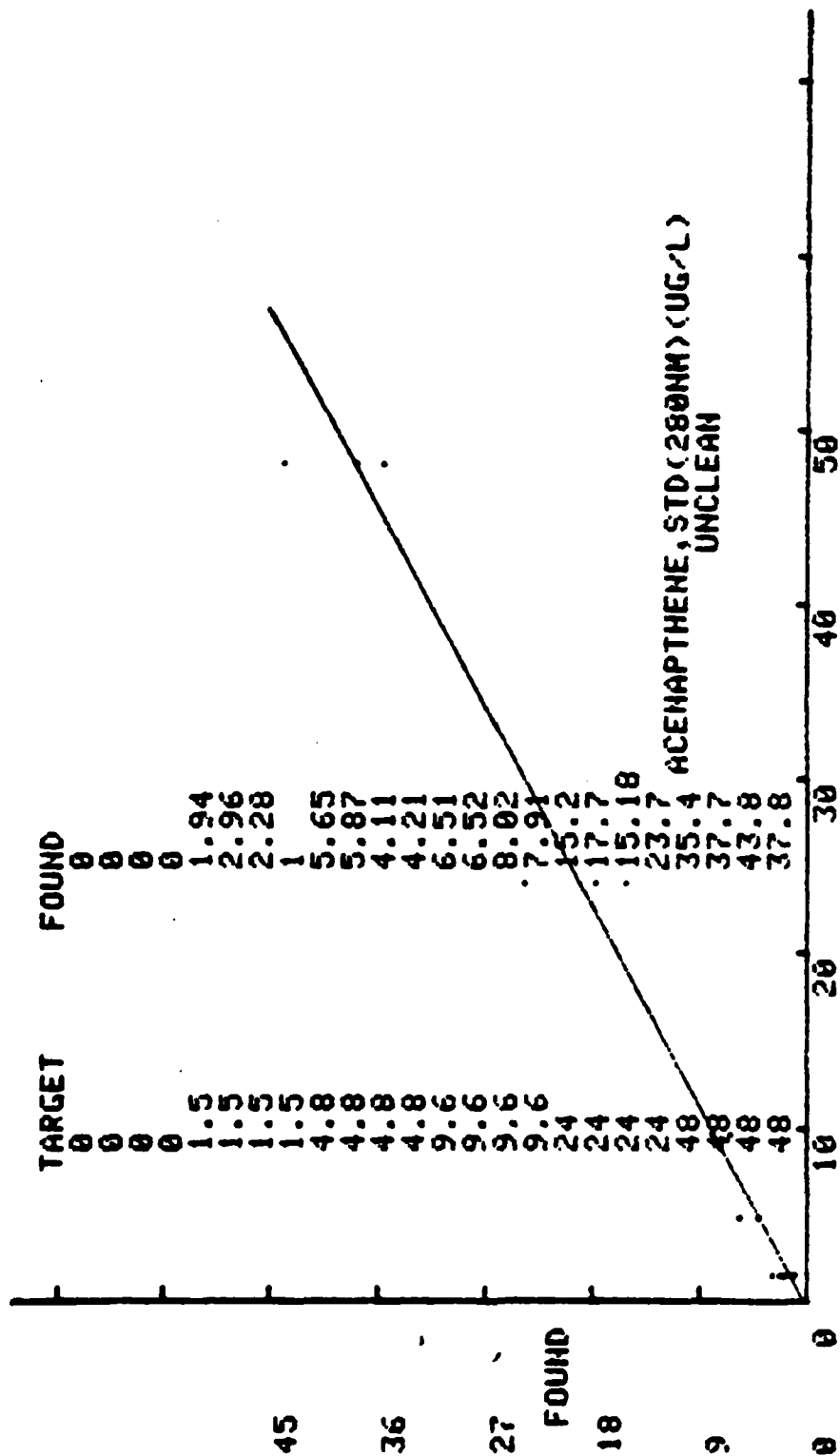


TARGET
CORR. COEFF. = 0.9790 FOUND = -1.1351+ 0.980946*TARGET
DETECTION LIMIT = 34.82787

ACENAPTHENE, STD(280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.50	1.94	2.96	2.28	1.000
4.80	5.65	5.87	4.11	4.21
9.60	6.51	6.52	8.02	7.91
24.0	15.2	17.7	15.2	23.7
48.0	35.4	37.7	43.8	37.8

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.50	2.04	0.816	39.9	36.3
4.80	4.96	0.929	18.7	3.33
9.60	7.24	0.838	11.6	-24.6
24.0	17.9	4.01	22.4	-25.2
48.0	38.7	3.59	9.29	-19.4

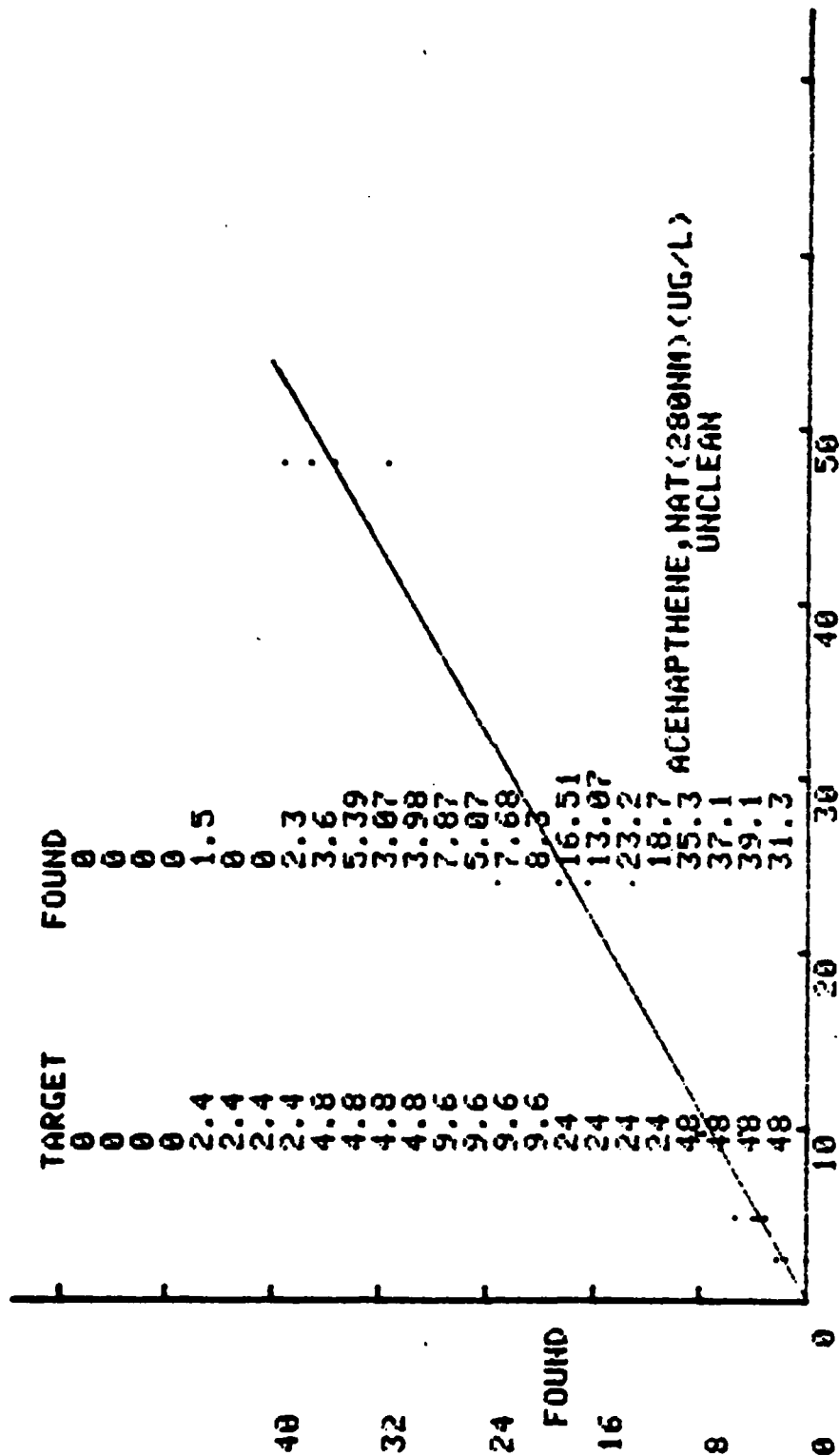


TARGET
CORR. COEFF. = 0.9875 FOUND = 0.2835+ 0.786849*TARGET
DETECTION LIMIT = 9.80686

ACENAPTHENE, NAT(2P0NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.40	1.50	0.0000	0.0000	2.30
4.80	3.60	5.39	3.07	3.42
9.60	7.87	5.17	7.68	8.30
24.0	16.5	13.1	23.2	18.7
48.0	35.3	37.1	39.1	31.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.40	0.950	1.14	120	-60.4
4.80	4.01	0.993	24.8	-16.5
9.60	7.23	1.46	20.2	-24.7
24.0	17.9	4.24	23.7	-25.5
48.0	35.7	3.32	9.30	-25.6

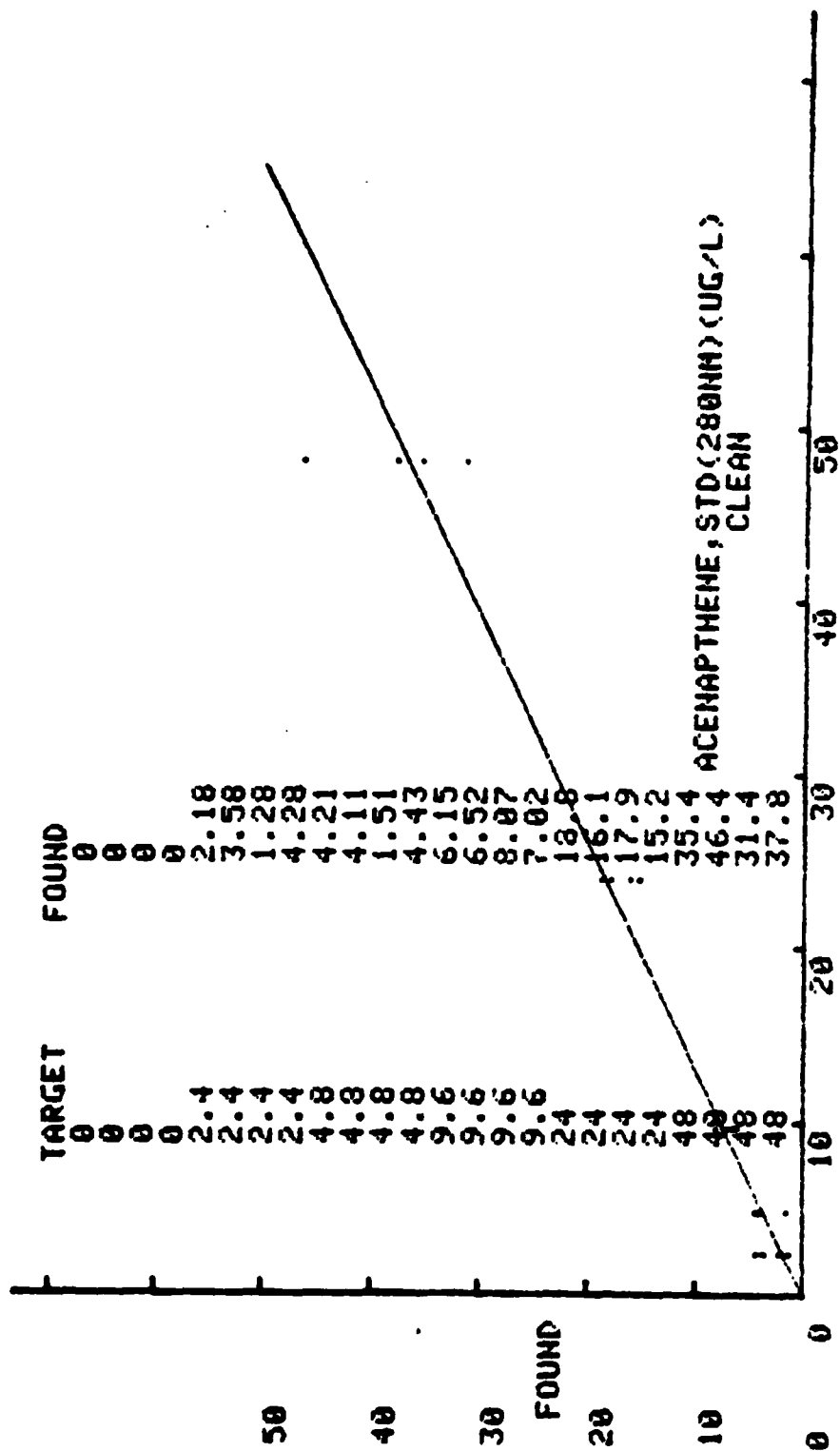


CORR. COEFF. = 0.9865 FOUND = TARGET
 DETECTION LIMIT = 10.14851
 -0.0973+ 0.747112*TARGET

ACENAPTHENE, STD (280NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
2.40	2.18	3.58	1.28	4.28
4.80	4.21	4.11	1.51	4.43
9.60	6.15	6.52	8.07	7.02
24.0	18.8	16.1	17.9	15.2
48.0	35.4	46.4	31.4	37.8

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.40	2.83	1.35	47.8	17.9
4.80	3.56	1.38	38.6	-25.7
9.60	6.94	0.833	12.0	-27.7
24.0	17.0	1.64	9.67	-29.2
48.0	37.7	6.34	16.8	-21.4

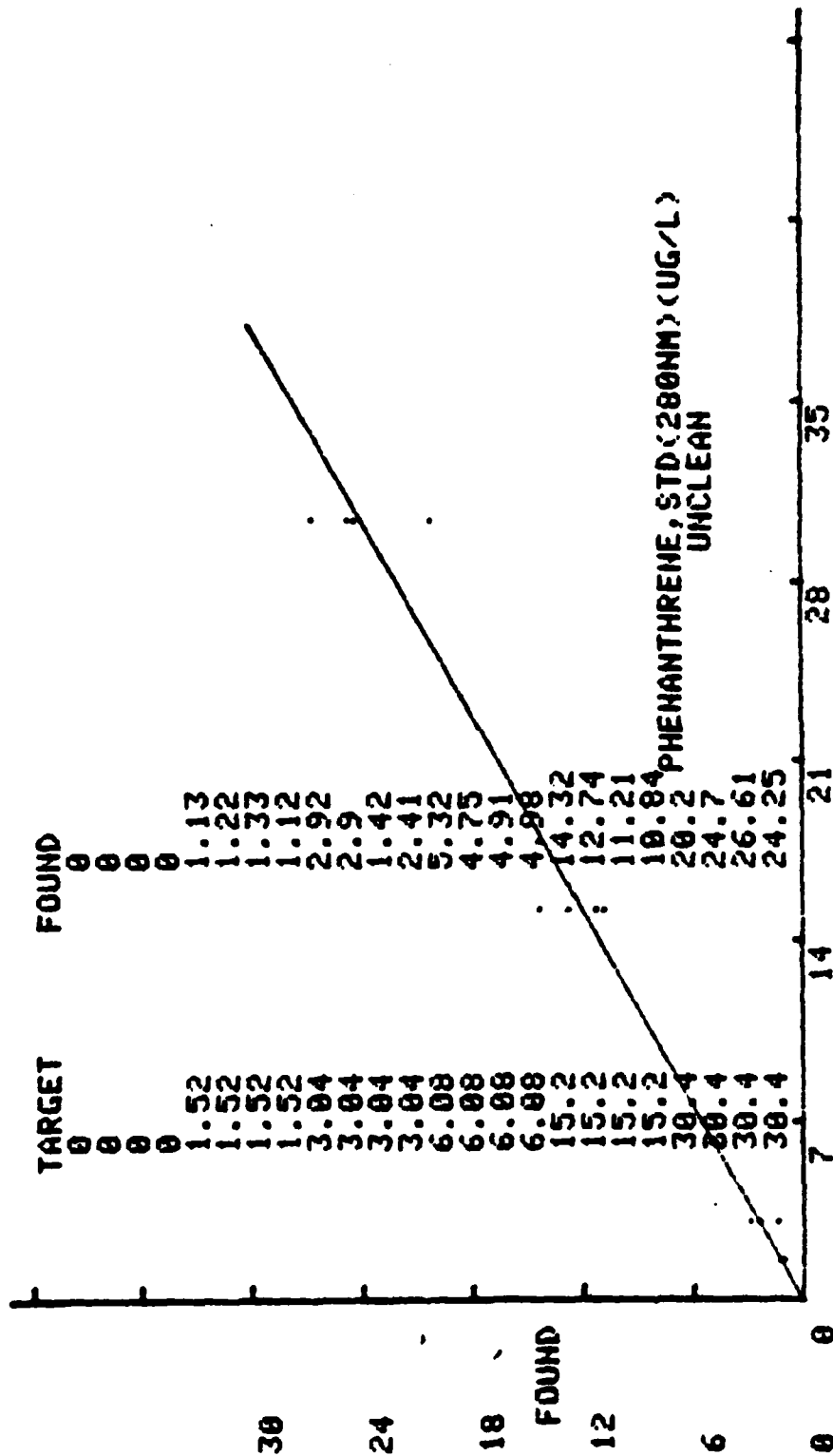


CORR. COEFF. = 0.9809 FOUND = TARGET
DETECTION LIMIT = 12.10759
-0.0844+ 0.772428*TARGET

PHENANTHRENE, STD (280NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.13	1.22	1.33	1.12
3.04	2.92	2.90	1.42	2.41
6.08	5.32	4.75	4.91	4.98
15.2	14.3	12.7	11.2	10.8
30.4	20.2	24.7	26.6	24.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.20	0.0976	8.14	-21.1
3.04	2.41	0.702	29.1	-20.6
6.08	4.99	0.240	4.81	-17.9
15.2	12.3	1.59	13.0	-19.2
30.4	23.9	2.70	11.3	-21.3

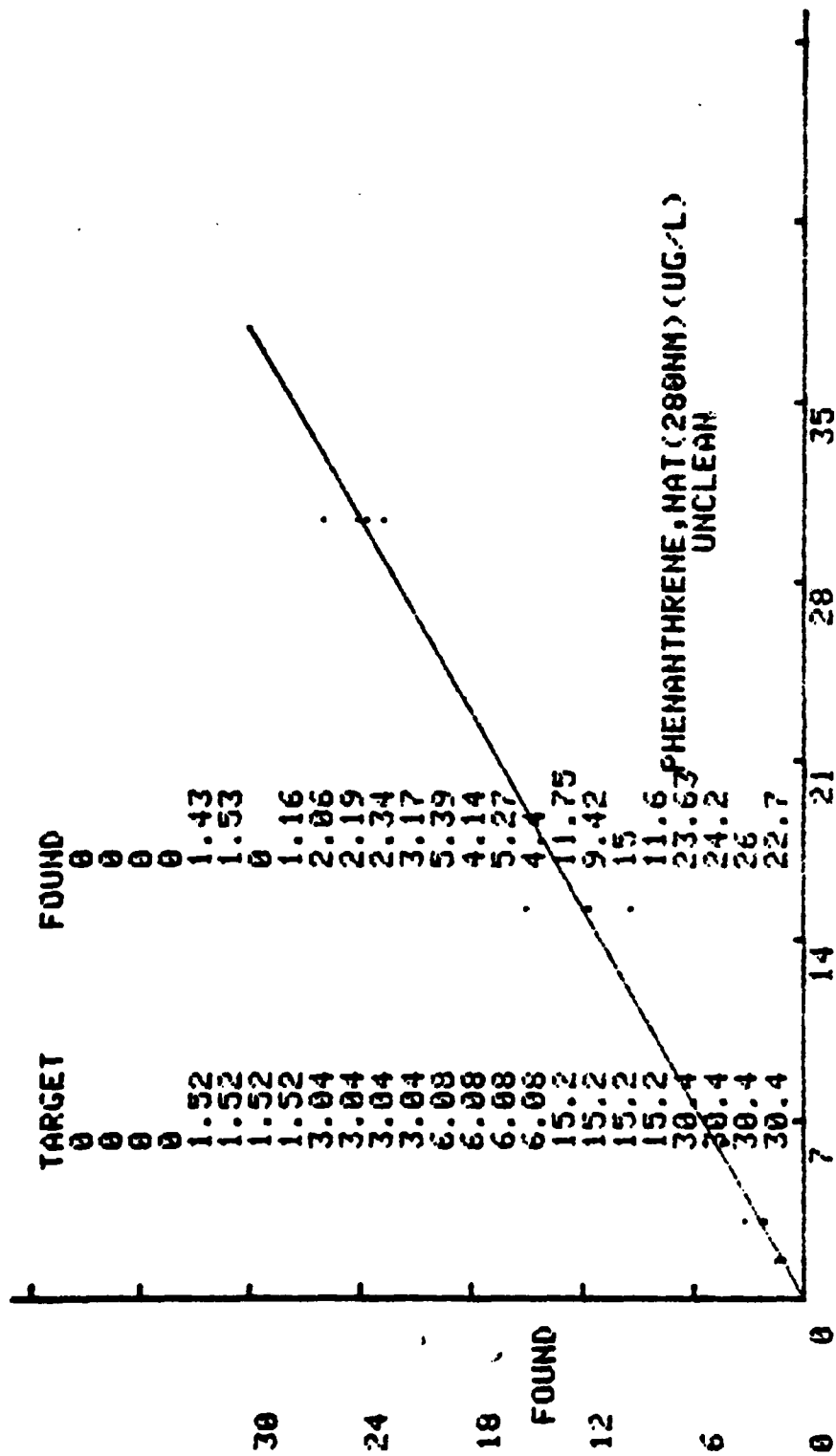


CORR. COEFF. = 0.9908 FOUND = TARGET
DETECTION LIMIT = 5.28666 0.0748+ 0.788957*TARGET

PHENANTHRENE, NAT (280NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.43	1.53	0.0000	1.16
3.04	2.86	2.19	2.34	3.17
6.08	5.39	4.14	5.27	4.40
15.2	11.8	9.42	15.0	11.6
30.4	23.6	24.2	26.0	22.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.03	0.704	68.4	-32.2
3.04	2.44	0.500	20.5	-19.7
6.08	4.80	0.623	13.0	-21.1
15.2	11.9	2.30	19.3	-21.4
30.4	24.1	1.39	5.76	-20.6

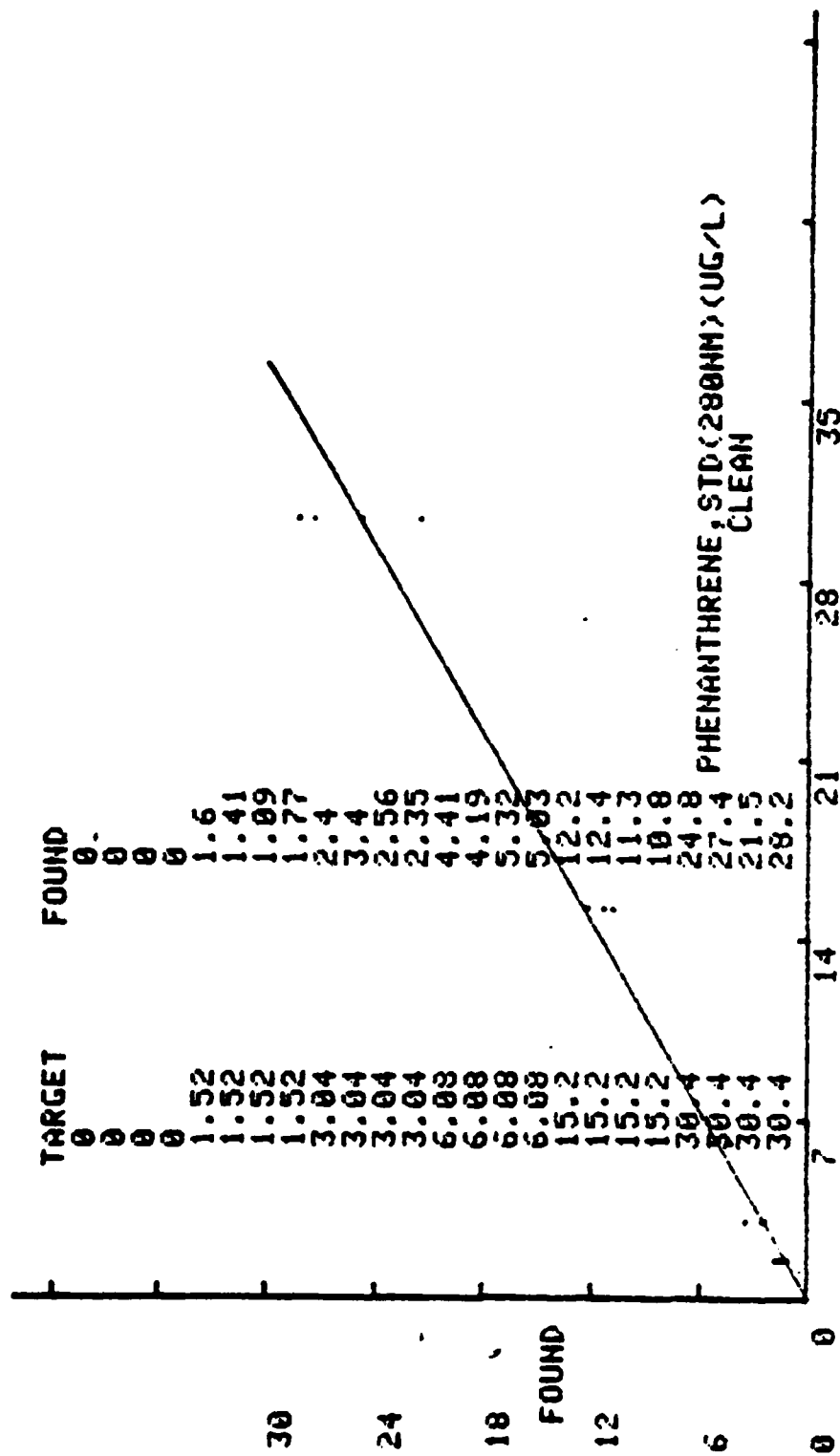


CORR. COEFF. = 0.9927
 DETECTION LIMIT = 4.70033
 TARGET
 FOUND = -0.0581+
 0.794695*TARGET

PHENANTHRENE,STD(280NM)(UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.60	1.41	1.09	1.77
3.04	2.40	3.40	2.56	2.35
6.08	4.41	4.19	5.32	5.03
15.2	12.2	12.4	11.3	10.8
30.4	24.8	27.4	21.5	28.2

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.52	1.47	0.291	19.9	-3.45
3.04	2.68	0.490	18.3	-11.9
6.08	4.74	0.527	11.1	-22.1
15.2	11.7	0.754	6.46	-23.2
30.4	25.5	3.02	11.9	-16.2

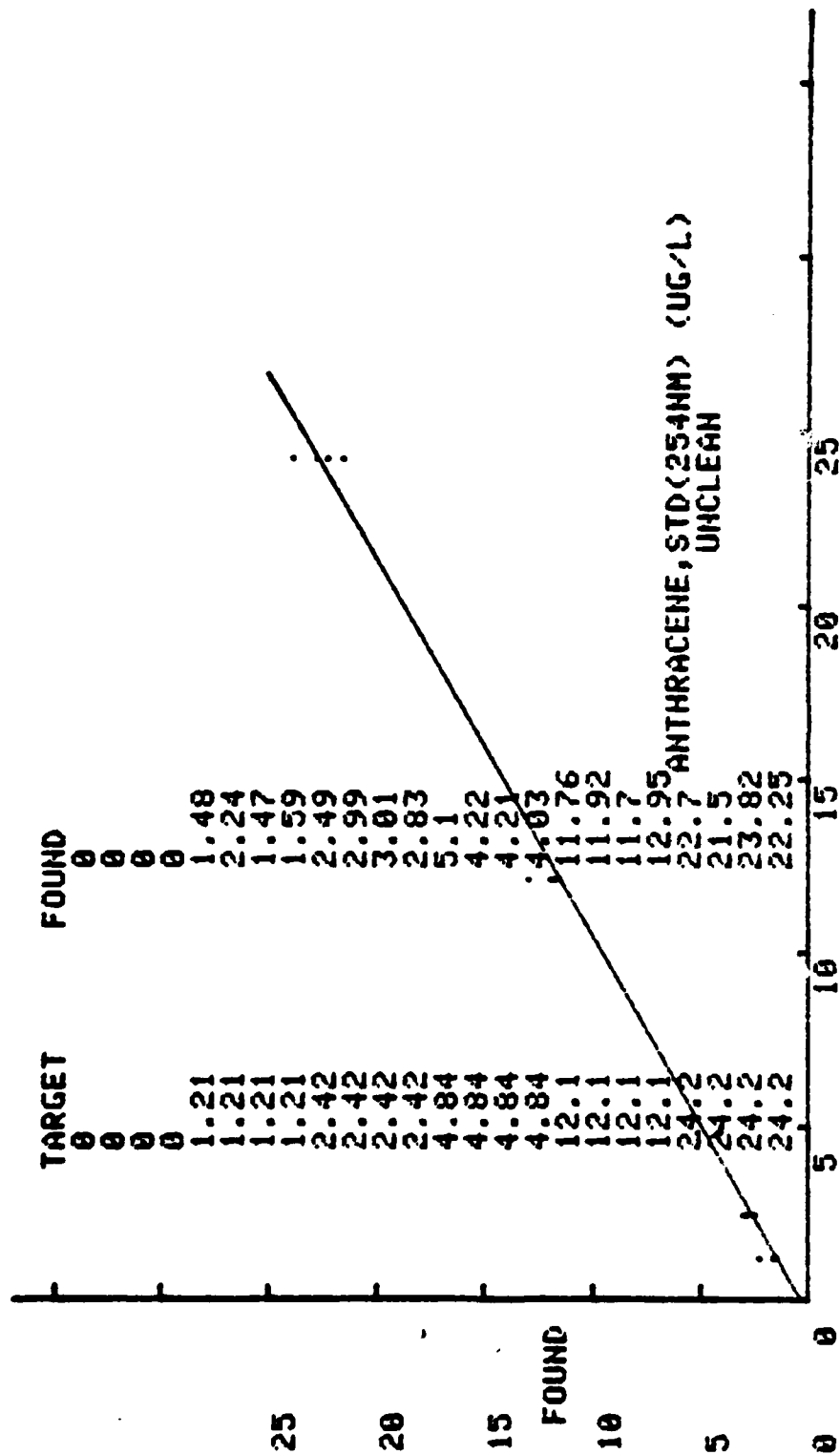


CORR. COEFF. = 0.9907
 DETECTION LIMIT = 5.32481
 TARGET FOUND = -0.0003+
 0.827117*TARGET

ANTHRACENE,STD(254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	1.48	2.24	1.47	1.59
2.42	2.49	2.99	3.01	2.83
4.84	5.10	4.22	4.21	4.03
12.1	11.8	11.9	11.7	12.9
24.2	22.7	21.5	23.8	22.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	1.69	0.367	21.7	40.1
2.42	2.83	0.241	8.50	16.9
4.84	4.39	0.481	11.0	-9.30
12.1	12.1	0.586	4.85	-0.145
24.2	22.6	0.971	4.30	-6.75

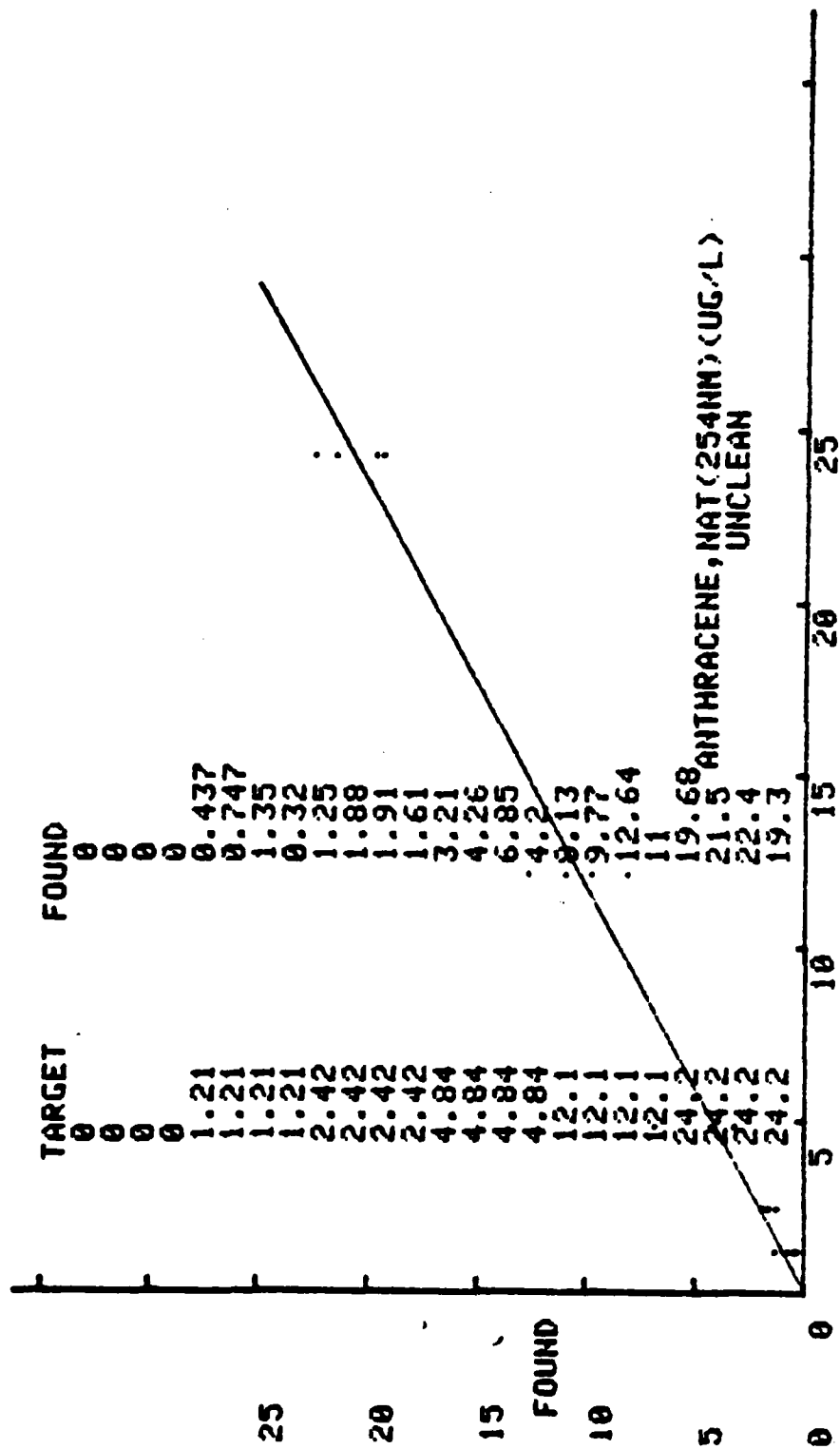


TARGET
CORR. COEFF. = 0.9973 FOUND = 0.3444+ 0.926935*TARGET
DETECTION LIMIT = 2.28363

ANTHRACENE, NAT (254NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	1.437	0.747	1.35	0.320
2.42	1.25	1.08	1.91	1.61
4.84	3.21	4.26	4.65	4.20
12.1	0.13	9.77	12.6	11.0
24.2	19.7	21.5	22.4	10.3

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	0.713	0.461	64.6	-41.3
2.42	1.66	0.306	18.4	-31.3
4.84	4.63	1.56	33.6	-4.34
12.1	10.4	1.91	18.4	-14.2
24.2	20.7	1.48	7.12	-14.4

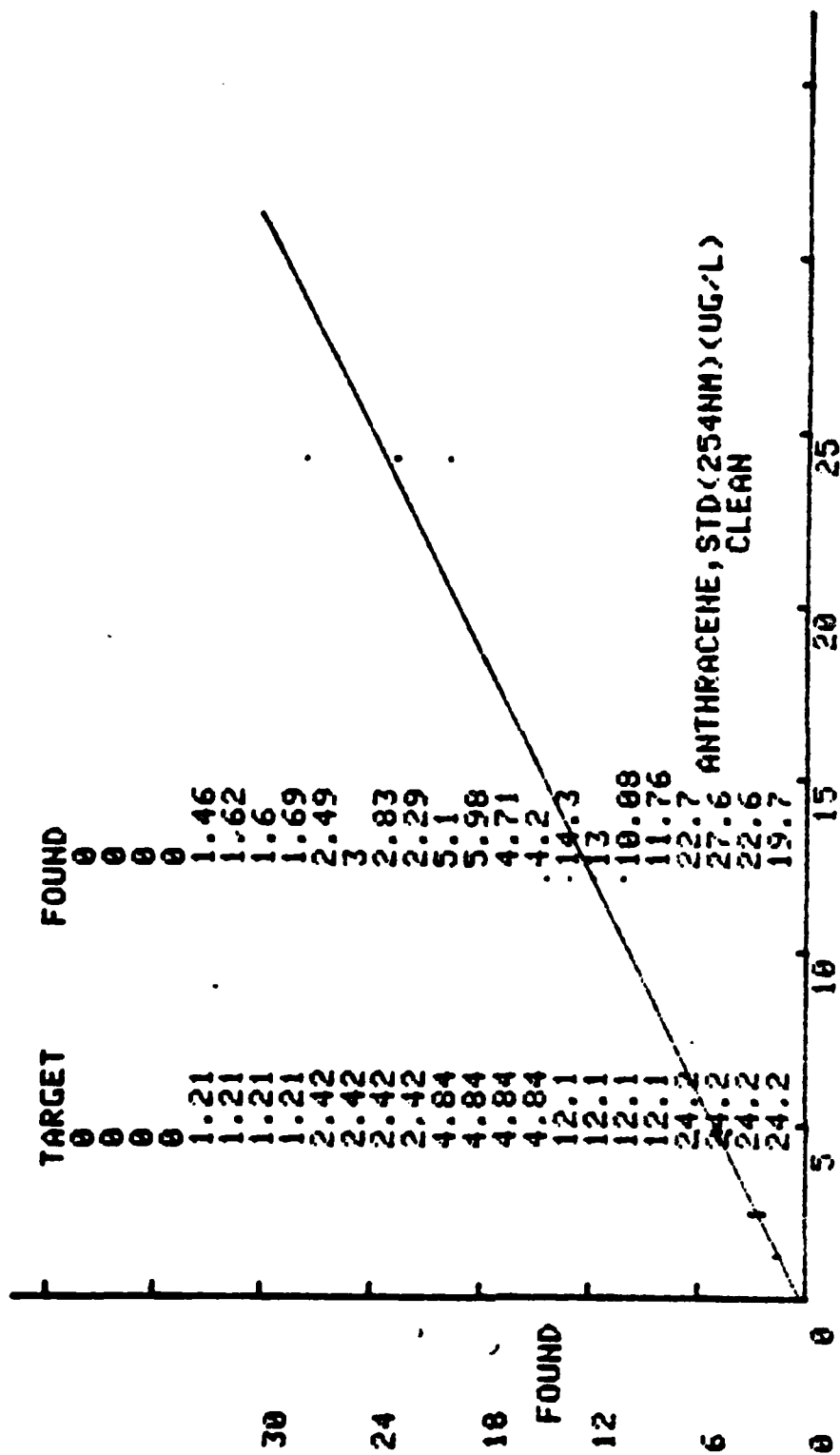


TARGET
CORR. COEFF. = 0.9894 FOUND = -0.0877+ 0.863015*TARGET
DETECTION LIMIT = 4.52715

ANTHRACENE, STD (284NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	1.46	1.62	1.63	1.69
2.42	2.49	3.00	2.83	2.29
4.84	5.10	5.98	4.71	4.20
12.1	14.3	13.0	10.1	11.8
24.2	22.7	27.6	22.6	19.7

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.21	1.59	0.0964	6.05	31.6
2.42	2.65	0.321	12.1	9.61
4.84	5.03	0.752	15.0	3.25
12.1	12.3	1.80	14.6	1.53
24.2	23.1	3.28	14.2	-4.34

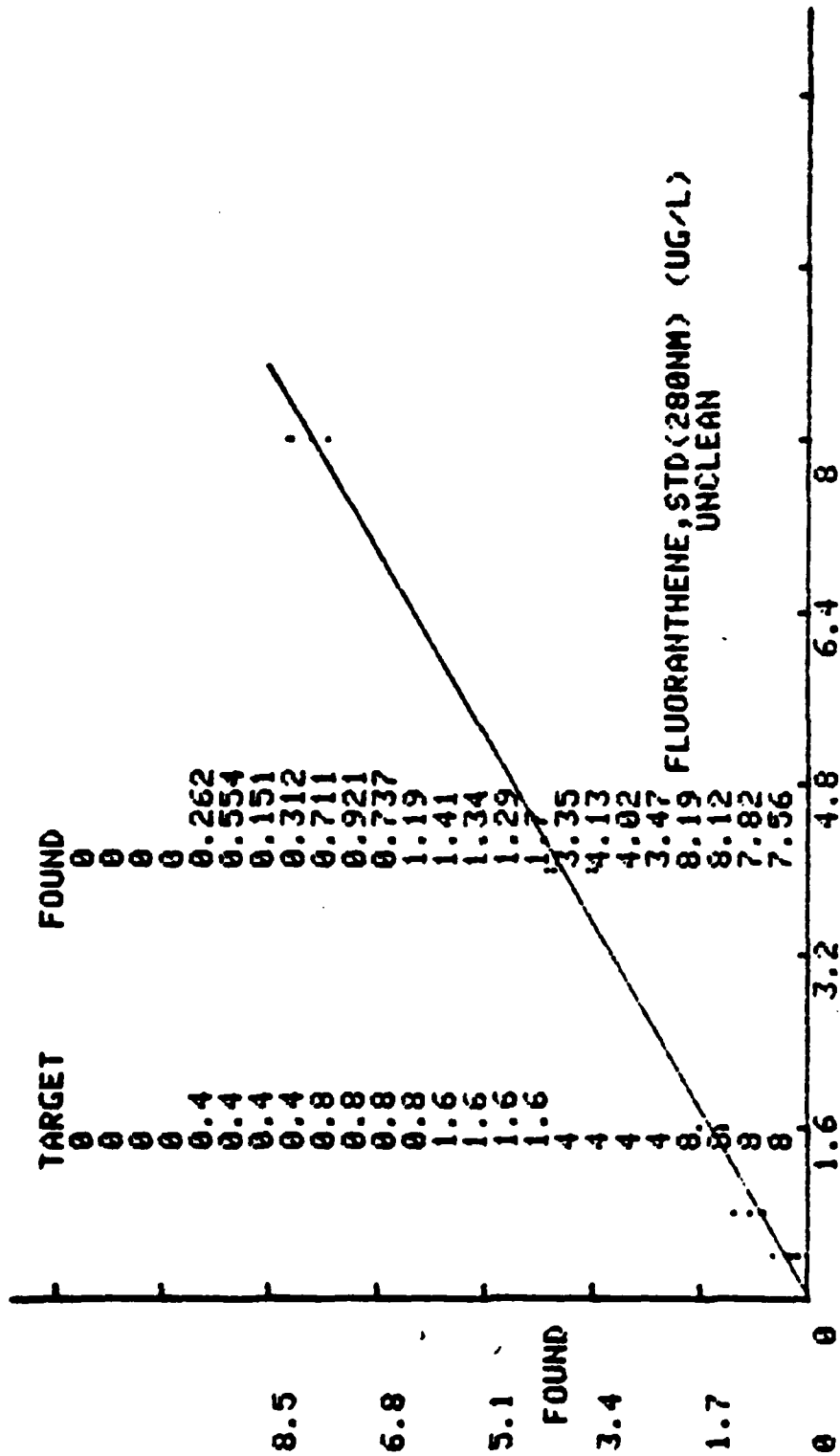


TARGET
CORR. COEFF. = 0.9857 FOUND = 0.3447+ 0.951734*TARGET
DETECTION LIMIT = 5.26848

FLUORANTHENE,STD(280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.262	0.554	0.151	0.312
0.800	0.711	0.921	0.737	1.19
1.60	1.41	1.34	1.29	1.70
4.00	3.35	4.13	4.02	3.47
8.00	8.19	8.12	7.82	7.56

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.320	0.170	53.2	-20.1
0.800	0.890	0.221	24.8	11.2
1.60	1.43	0.183	12.8	-10.3
4.00	3.74	0.390	10.4	-6.44
8.00	7.92	0.290	3.66	-0.969



TARGET
CORR. COEFF. = 0.9965 FOUND = 0.985820 * TARGET

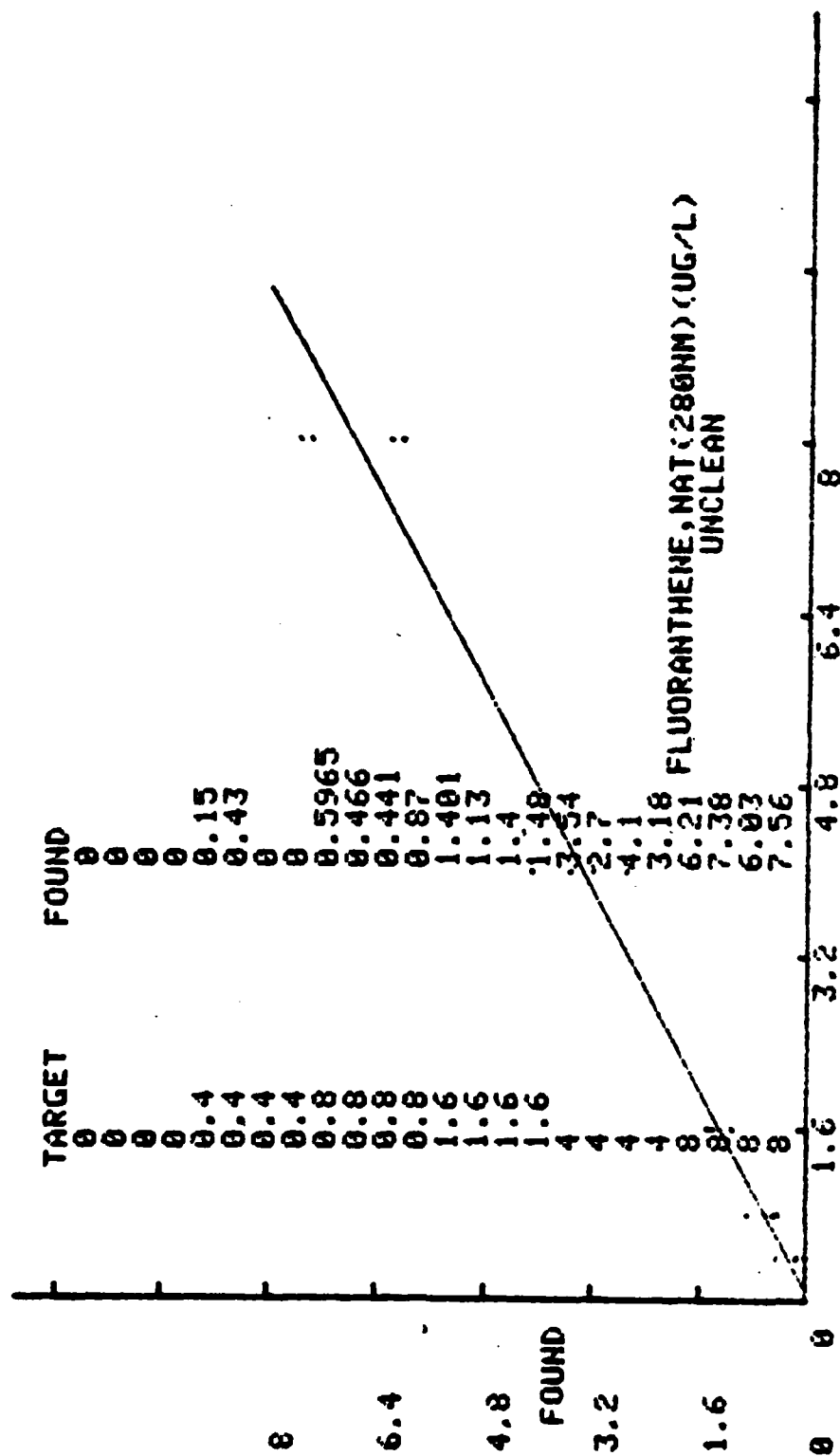
-0.0468 +

DETECTION LIMIT = 0.86872

FLUORANTHENE, NAT (280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.150	0.430	0.0000	0.0000
0.800	0.596	0.466	0.441	0.870
1.60	1.40	1.13	1.40	1.48
4.00	3.54	2.70	4.10	3.18
8.00	6.21	7.38	6.03	7.56

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.145	0.203	140	-63.8
0.800	0.593	0.197	33.1	-25.8
1.60	1.35	0.153	11.3	-15.5
4.00	3.38	0.591	17.5	-15.5
8.00	6.79	0.786	11.6	-15.1

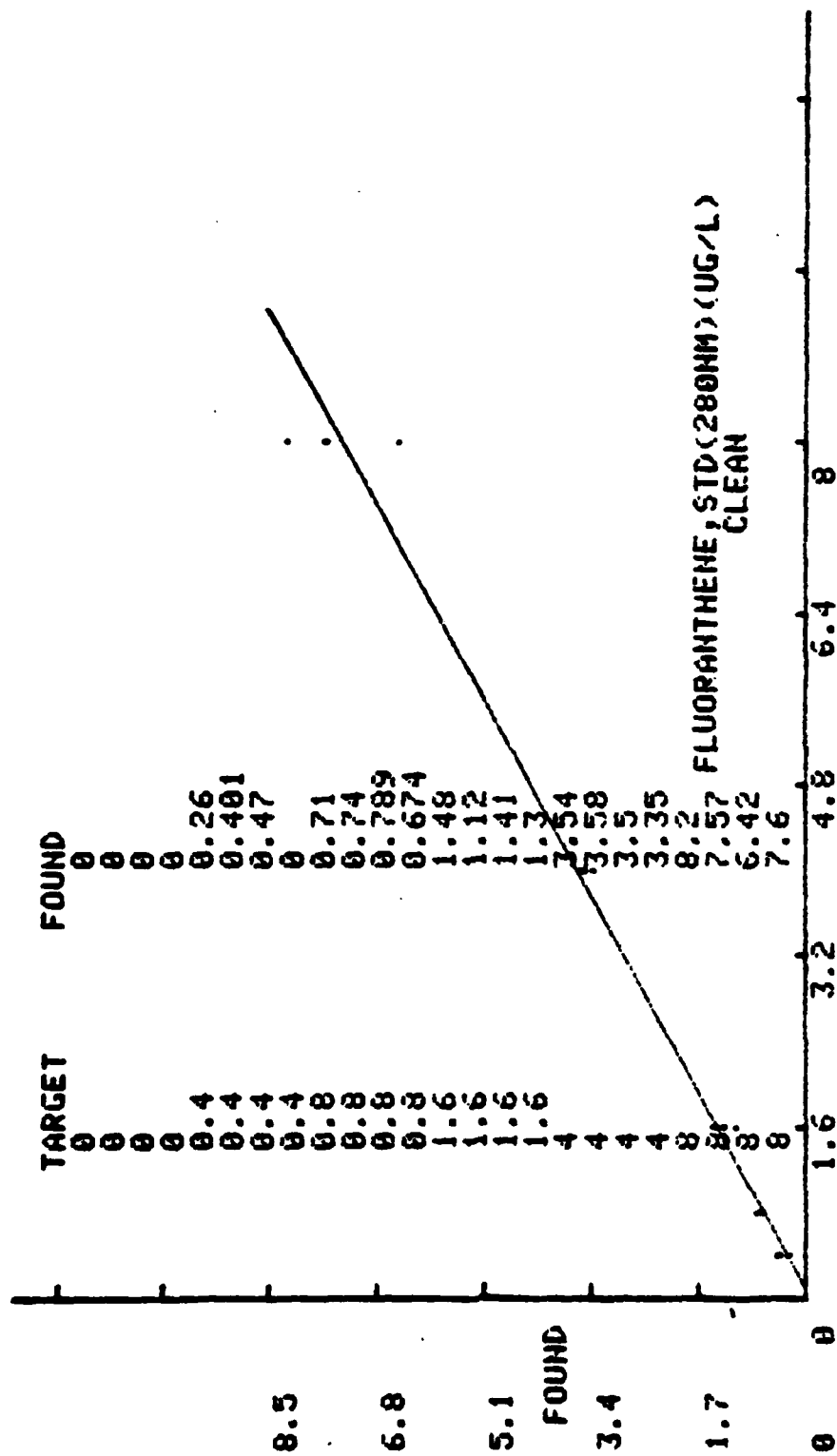


TARGET
 CORR. COEFF. = 0.9883 FOUND = -0.0784+ 0.860572*TARGET
 DETECTION LIMIT = 1.5729

F LORANTHENE, STD (280NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.260	0.401	0.470	0.0000
0.800	0.710	0.740	0.789	0.674
1.60	1.48	1.12	1.41	1.30
4.00	3.54	3.58	3.50	3.35
8.00	8.20	7.57	6.42	7.60

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	0.283	0.208	73.5	-29.3
0.800	0.728	0.0487	6.68	-8.97
1.60	1.33	0.157	11.8	-17.0
4.00	3.49	0.100	2.88	-12.7
8.00	7.45	0.744	9.99	-6.91



CORR. COEFF. = 0.9936
 DETECTION LIMIT = 1.15485
 TARGET FOUND = -0.0032+ 0.930906*TARGET

PYRENE STD (280NM) (UG/L)

UNCLEAN

TARGET
CONCENTRATION

1

DAY
2

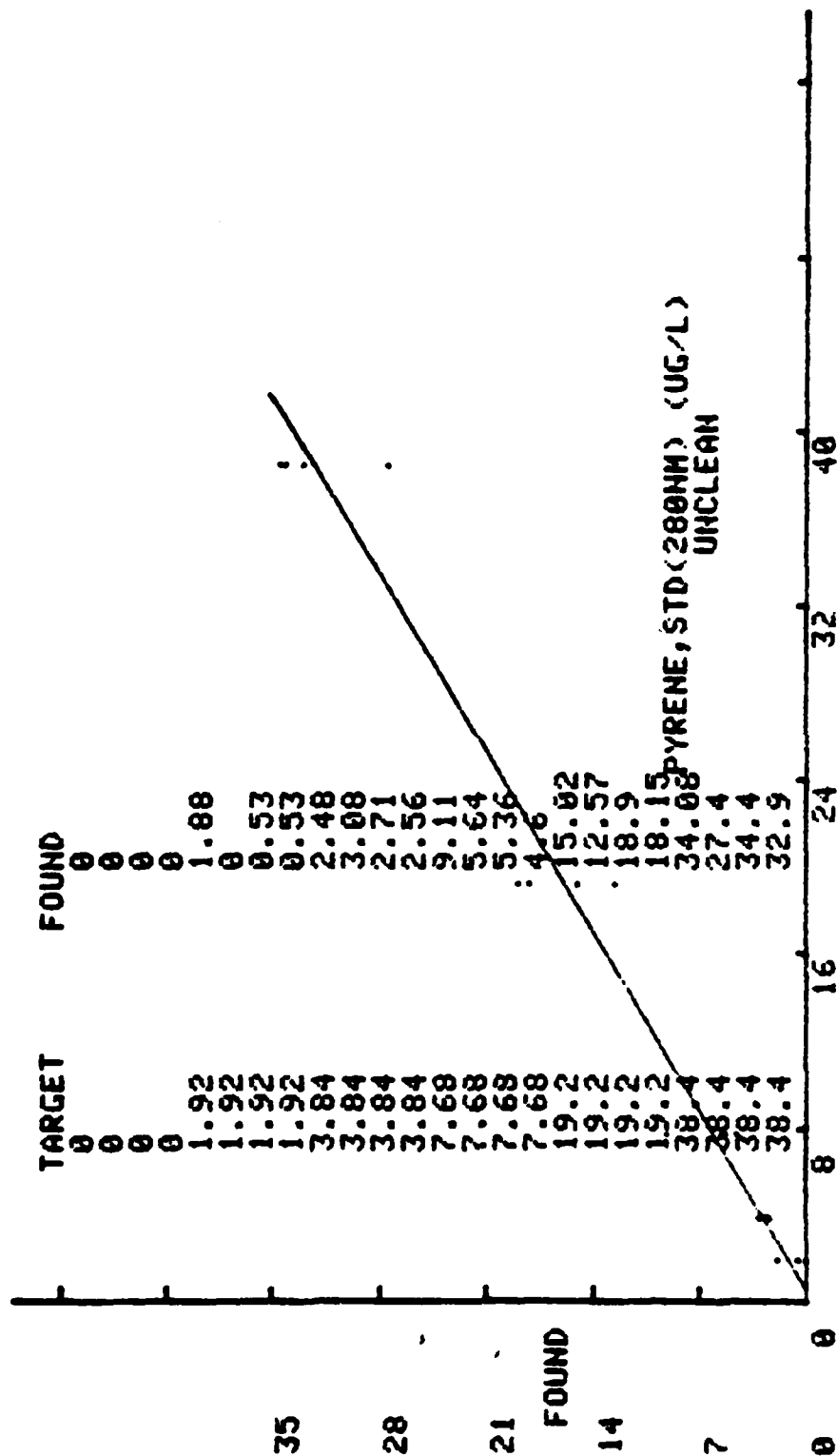
3

4

0.0000	0.0000	0.0000	0.0000	0.0000
1.92	1.88	0.0000	0.530	0.530
3.84	2.48	3.08	2.71	2.56
7.68	9.11	5.64	5.36	4.60
19.2	15.0	12.6	18.9	18.1
38.4	34.1	27.4	34.4	32.9

TARGET
CONCENTRATIONAVERAGE
FOUND VALUESTANDARD
DEVIATIONPERCENT
IMPRECISIONPERCENT
INACCURACY

0.0000	0.0000	0.0000	0.0000	0.0000
1.92	0.735	0.803	109	-61.7
3.84	2.71	0.266	9.82	-29.5
7.68	6.18	2.00	32.4	-19.6
19.2	16.2	2.92	18.1	-15.8
38.4	32.2	3.26	10.1	-16.2

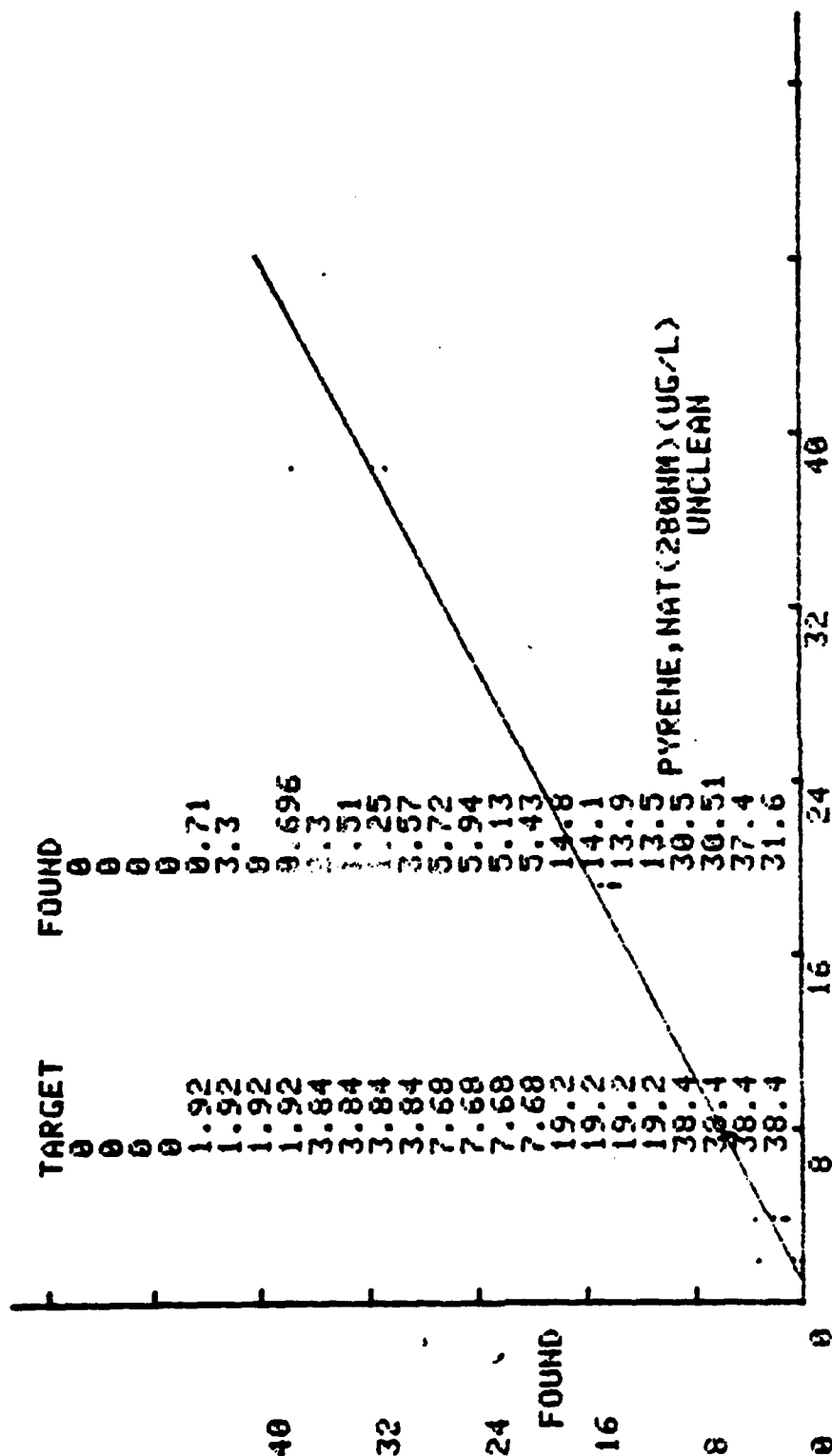


TARGET
CORR. COEFF. = 0.9884 FOUND = -0.4214+ 0.851678*TARGET
DETECTION LIMIT = 7.50211

PYRENE, NAT (280NM) (UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.92	0.710	3.30	0.0000	0.656
3.84	2.30	1.51	1.25	3.57
7.68	5.72	5.94	5.13	5.43
19.2	14.2	14.1	13.9	13.5
38.4	30.5	30.3	37.4	31.6

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.92	1.18	1.45	124	-38.7
3.84	2.16	1.04	48.3	-43.8
7.68	5.55	0.352	6.34	-27.7
19.2	14.1	0.544	3.86	-26.7
38.4	32.5	3.31	10.2	-15.4



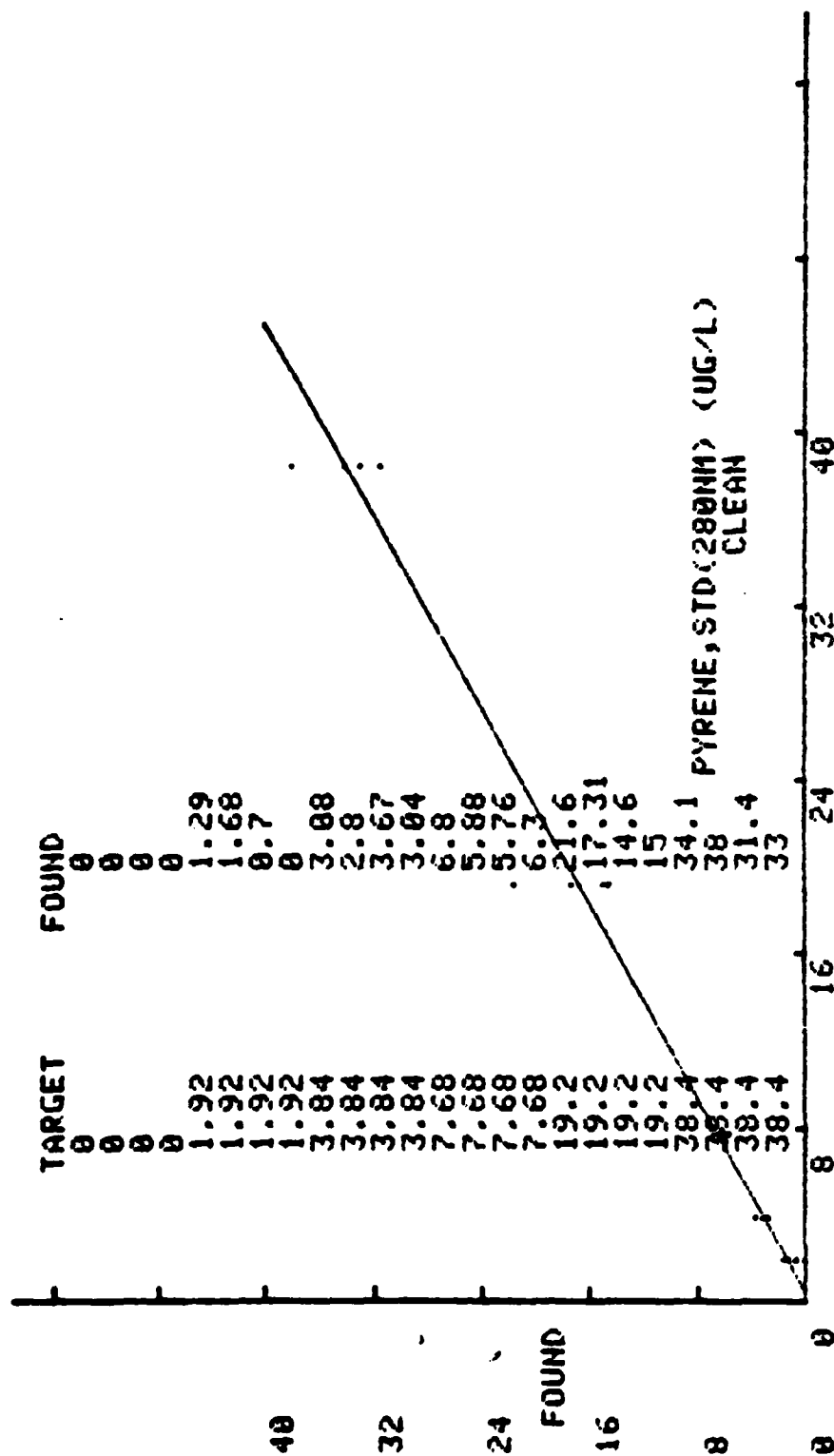
TARGET
CORR. COEFF. = 0.9909 FOUND = -0.7893+ 0.847446*TARGET
DETECTION LIMIT = 6.64384

PYRENE STD(280NM) (UG/L)

CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
1.92	1.29	1.68	0.700	0.0000
3.84	3.08	2.90	3.67	3.04
7.68	6.80	5.88	5.76	6.30
19.2	21.6	17.3	14.6	15.0
38.4	34.1	38.0	31.4	33.0

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
1.92	0.917	0.732	79.8	-52.2
3.84	3.15	0.370	11.7	-18.0
7.68	6.18	0.471	7.61	-19.5
19.2	17.1	3.21	16.8	-10.8
38.4	34.1	2.81	8.24	-11.1



CORR. COEFF. = 0.9916 TARGET
 FOUND = -0.4150+ 0.980793*TARGET
 DETECTION LIMIT = 6.36532

CHRYSENE, STD (280NM) (UG/L)

UNCLEAN

TARGET
CONCENTRATION

1

DAY
2

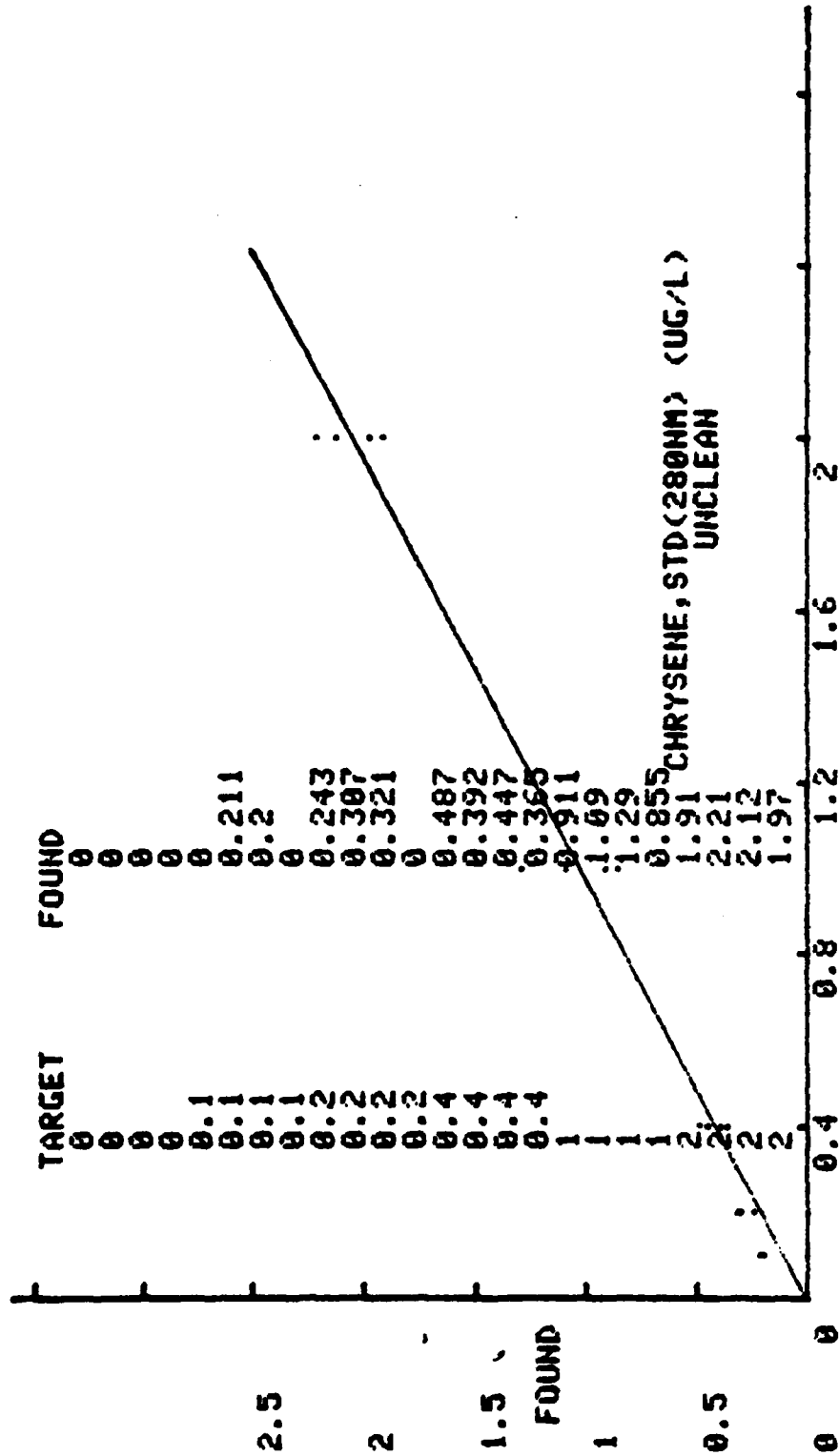
3

4

0.0000	0.0000	0.0000	0.0000	0.0000
0.1000	0.0000	0.211	0.200	0.0000
0.200	0.243	0.307	0.321	0.0000
0.400	0.487	0.392	0.447	0.365
1.000	0.911	1.09	1.29	0.855
2.00	1.91	2.21	2.12	1.97

TARGET
CONCENTRATIONAVERAGE
FOUND VALUESTANDARD
DEVIATIONPERCENT
IMPRECISIONPERCENT
INACCURACY

0.0000	0.0000	0.0000	0.0000	0.0000
0.100	0.103	0.119	116	2.75
0.200	0.218	0.149	68.5	0.87
0.400	0.423	0.0548	13.0	5.69
1.00	1.04	0.196	19.0	3.65
2.00	2.05	0.137	6.68	2.62

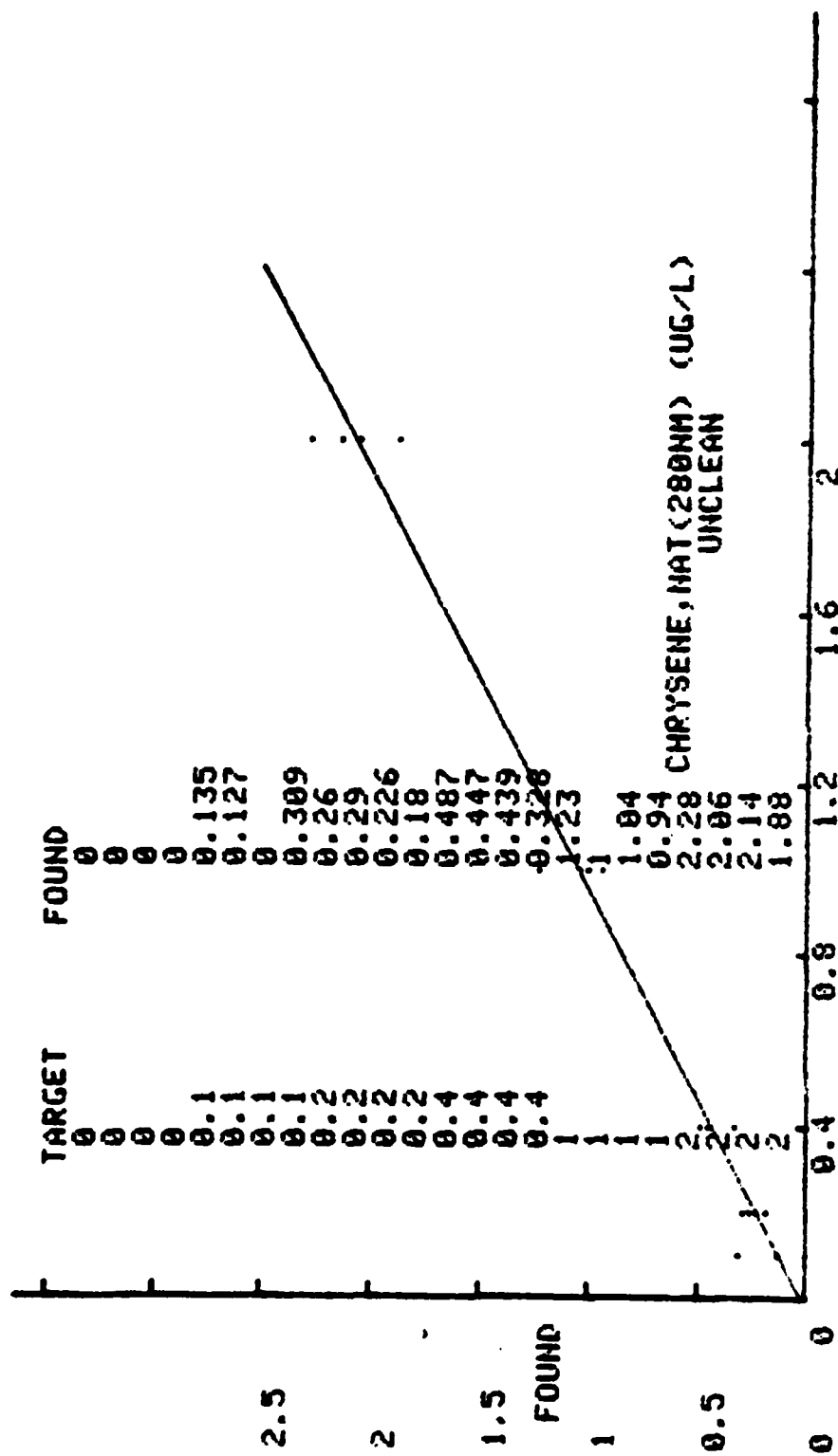


TARGET
CORR. COEFF. = 0.9884 FOUND = 0.0067+ 1.024885*TARGET
DETECTION LIMIT = 0.39174

CHRYSENE, NAT(280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.1000	0.135	0.127	0.1000	0.308
0.200	0.260	0.290	0.226	0.180
0.400	0.487	0.447	0.439	0.328
1.000	1.23	1.000	1.04	0.940
2.00	2.28	2.06	2.14	1.88

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.100	0.143	0.127	88.9	42.7
0.200	0.239	0.0472	19.8	19.5
0.400	0.425	0.0681	16.0	6.31
1.00	1.05	0.125	11.9	5.25
2.00	2.09	0.167	7.99	4.50



TARGET
CORR. COEFF. = 0.9920 FOUND = 0.0204+ 1.034428*TARGET
DETECTION LIMIT = 0.32462

CHRYSENE, STD (280NM) (UG/L)

CLEAN

TARGET
CONCENTRATION

1

DAY
2

3

0.0000	0.0000	0.0000	0.0000	0.0000
0.1000	0.0000	0.0000	0.1000	0.0000
0.200	0.244	0.187	0.0000	0.0000
0.400	0.463	0.388	0.487	1.09
1.000	0.968	0.905	0.919	1.50
2.00	2.21	1.79	1.80	0.0000

TARGET
CONCENTRATION

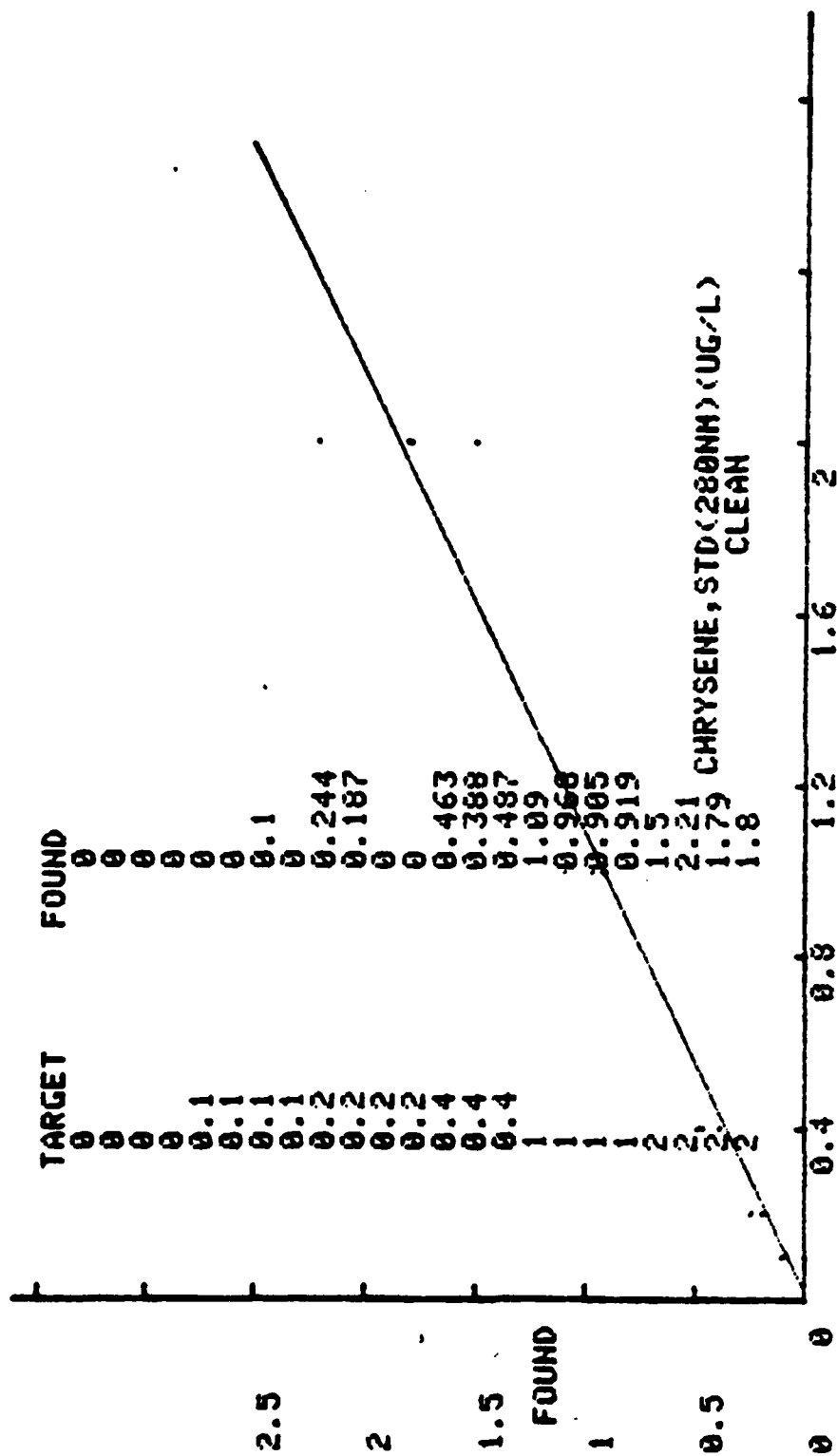
AVERAGE
FOUND VALUE

STANDARD
DEVIATION

PERCENT
IMPRECISION

PERCENT
INACCURACY

0.0000	0.0000	0.0000	0.0000	0.0000
0.100	0.0250	0.0500	200	-75.0
0.200	0.108	0.127	117	-46.1
0.400	0.446	0.0516	11.6	11.5
1.00	0.970	0.0841	8.67	-2.95
2.00	1.82	0.292	16.0	-8.75

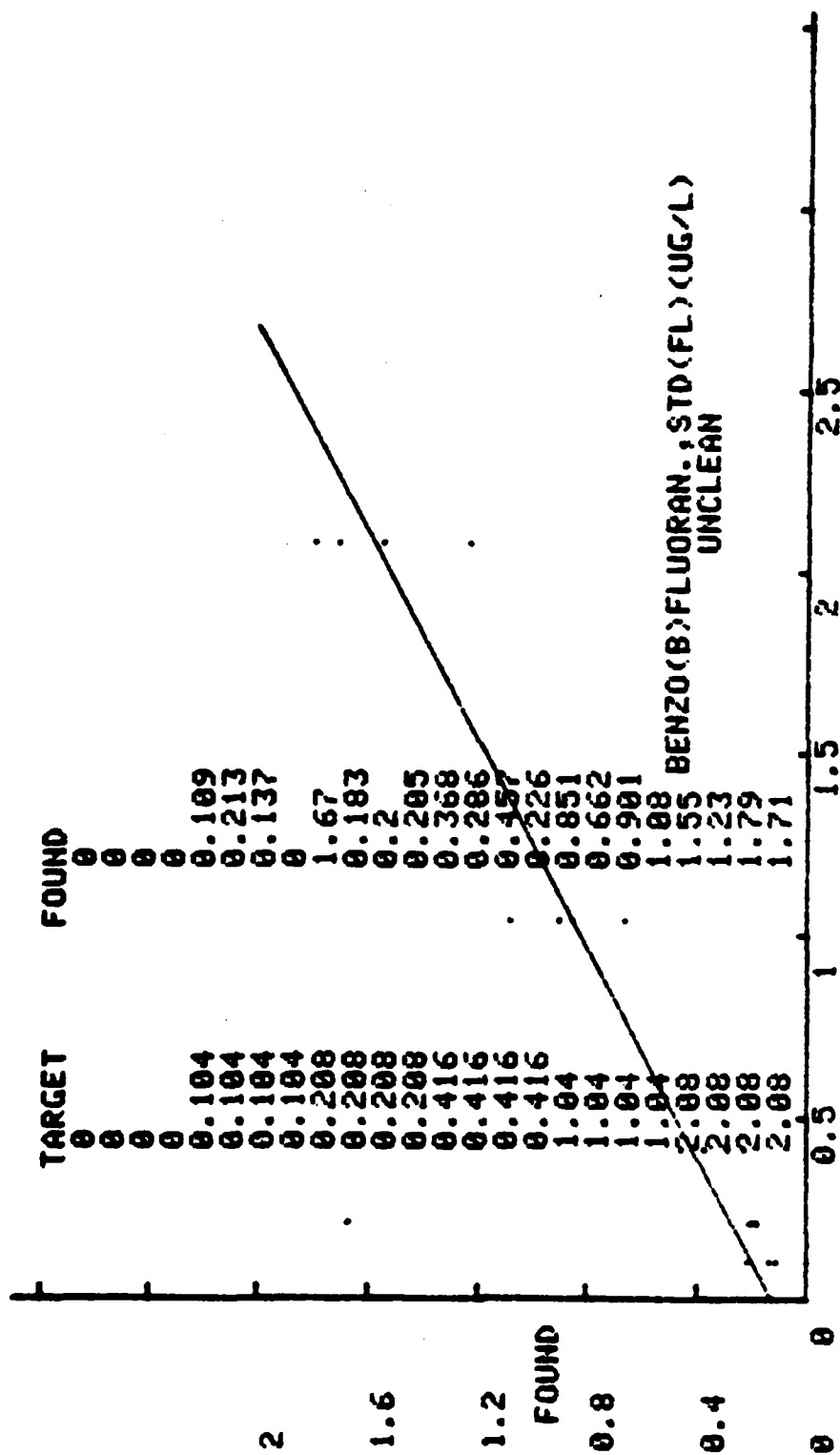


CORR. COEFF. = 0.9889 FOUND = TARGET
DETECTION LIMIT = 0.51674
-0.0199+ 0.938101xTARGET

BENZO(B)FLUORAN.,STD(FL)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.109	0.213	0.137	0.0000
0.208	1.67	0.183	0.210	0.205
0.416	0.368	0.286	0.457	0.226
1.04	0.851	0.662	0.901	1.08
2.08	1.55	1.23	1.79	1.71

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.115	0.0882	76.9	10.3
0.208	0.564	0.737	131	171
0.416	0.334	0.100	30.0	-19.7
1.04	0.873	0.172	19.7	-16.0
2.08	1.57	0.248	15.8	-24.5

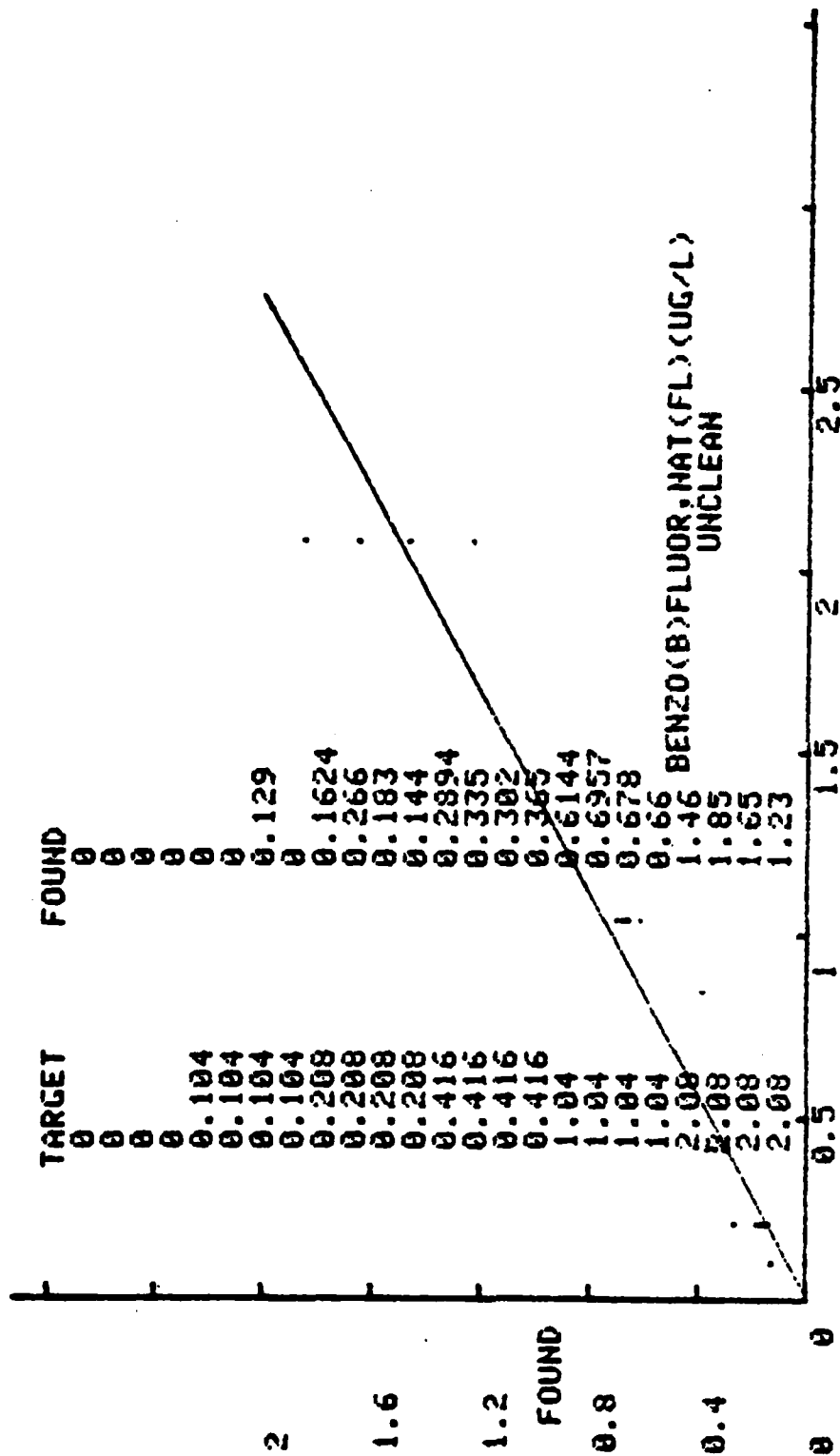


TARGET
CORR. COEFF. = 0.8491 FOUND = 0.1256+ 0.702604 * TARGET
DETECTION LIMIT = 1.64785

BENZG(B)FLUOR,NAT(FL)(UG/L) UNCLEA

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.0000	.0000	0.129	0.0000
0.208	0.162	0.266	0.183	0.144
0.416	0.289	0.335	0.302	0.365
1.04	0.614	0.696	0.678	0.660
2.08	1.46	1.85	1.65	1.23

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.0322	0.0645	200	-69.0
0.208	0.189	0.0538	28.5	-9.21
0.416	0.323	0.0340	10.5	-22.4
1.04	0.662	0.1349	5.29	-36.3
2.08	1.55	0.265	17.1	-25.6

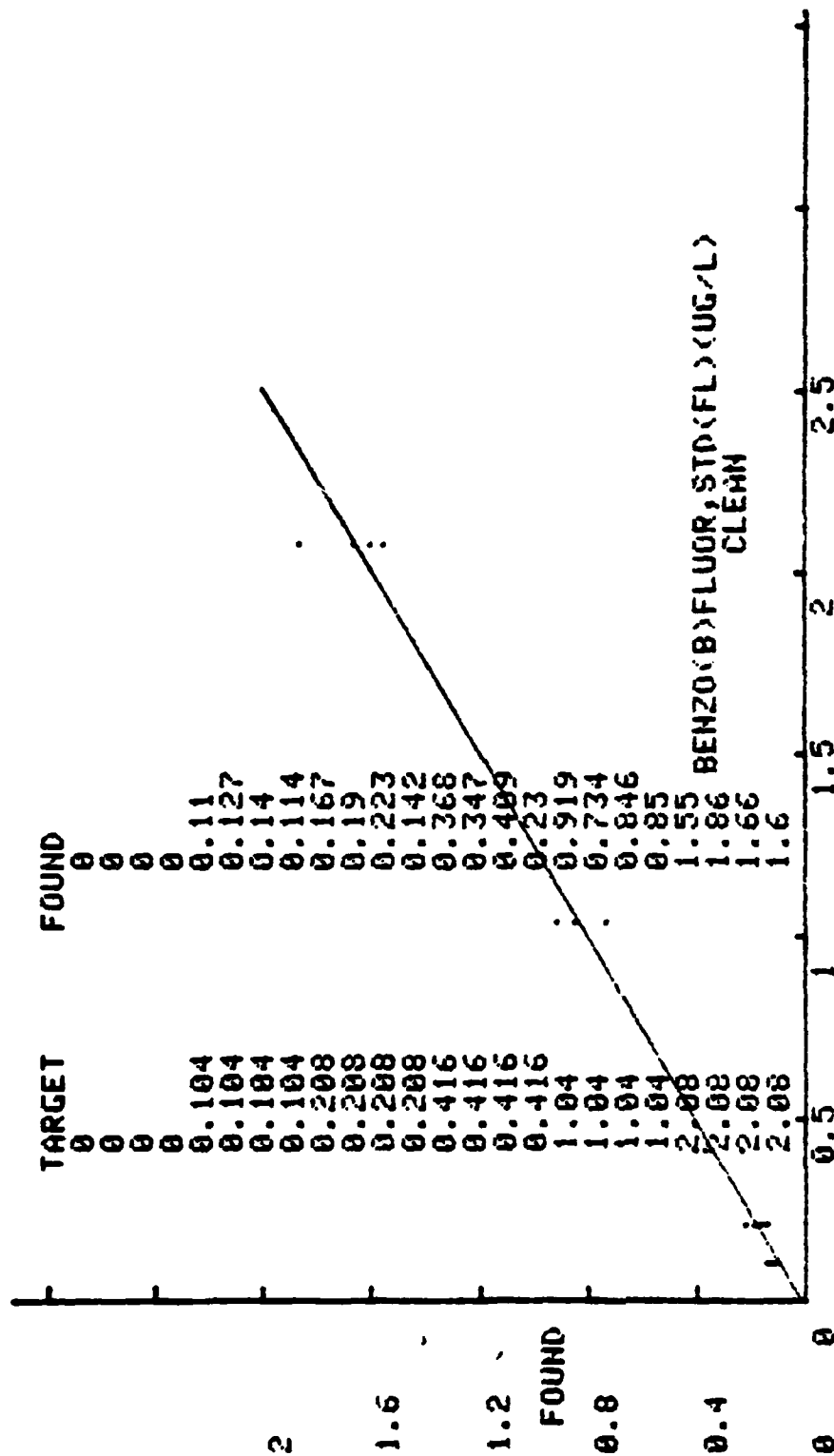


TARGET
CORR. COEFF. = 0.9791 FOUND = -0.0105+ 0.731968#TARGET
DETECTION LIMIT = 0.54978

BENZO(B)FLUOR, STD (FL) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.110	0.127	0.140	0.114
0.208	0.167	0.190	0.223	0.142
0.416	0.368	0.347	0.409	0.230
1.04	0.919	0.734	0.846	0.850
2.08	1.55	1.86	1.66	1.60

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.104	0.123	0.0136	11.1	18.5
0.208	0.180	0.0345	19.1	-13.2
0.416	0.338	0.0768	22.7	-18.6
1.04	0.837	0.0766	9.14	-19.5
2.08	1.67	0.136	8.15	-19.8

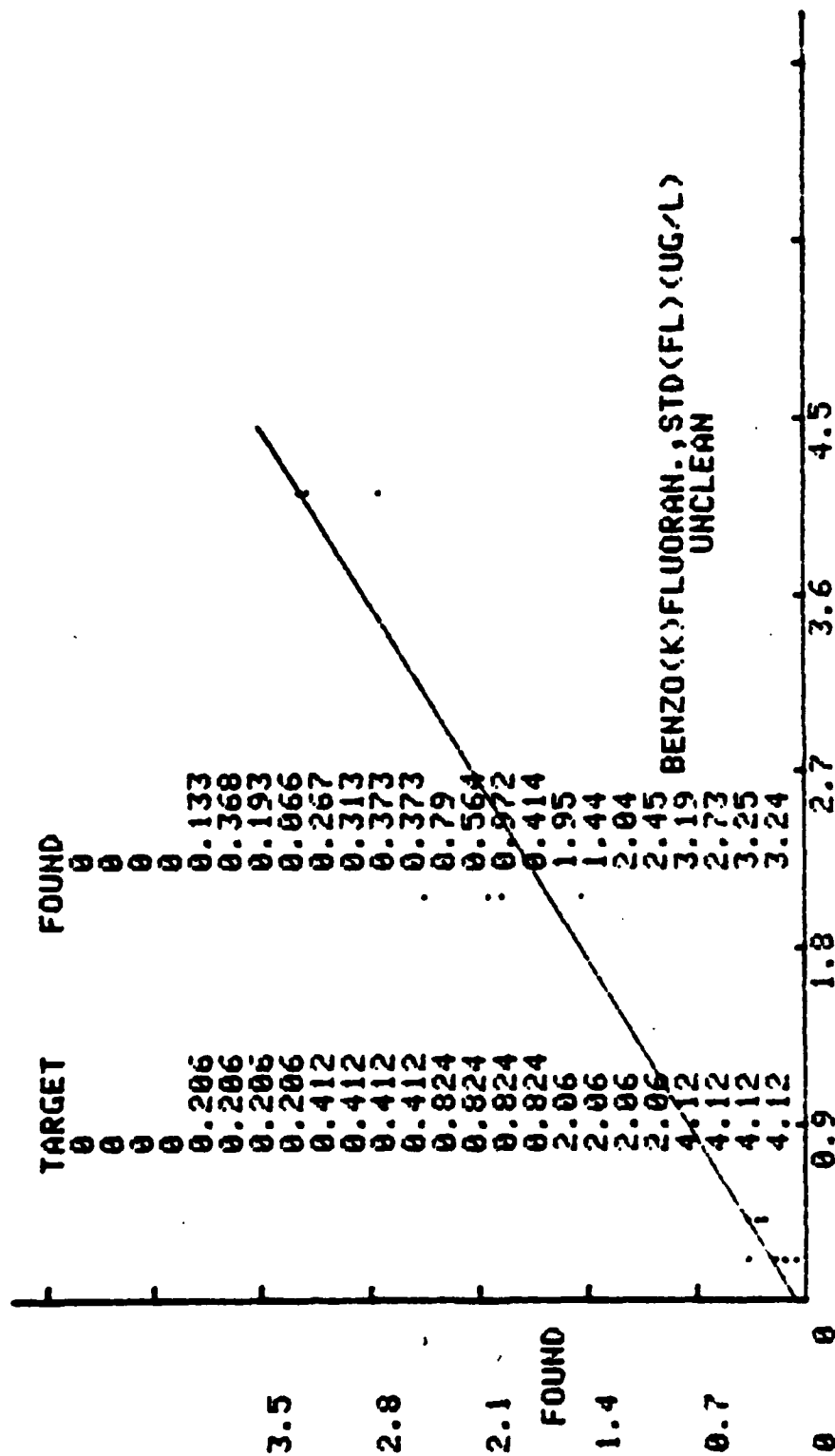


CORR. COEFF. = 0.9939
 DETECTION LIMIT = 0.29449
 TARGET
 FOUND = 0.0157+
 0.793206*TARGET

BENZO(K)FLUORAN.,STD(FL)(UG/L) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.133	0.368	0.193	0.0640
0.412	0.267	0.313	0.373	0.373
0.824	0.790	0.564	0.972	0.414
2.06	1.95	1.44	2.04	2.45
4.12	3.19	2.73	3.25	3.24

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.190	0.130	68.2	-7.77
0.412	0.331	0.0515	15.5	-19.5
0.824	0.685	0.246	35.9	-16.9
2.06	1.97	0.415	21.1	-4.37
4.12	3.10	0.250	8.05	-24.7

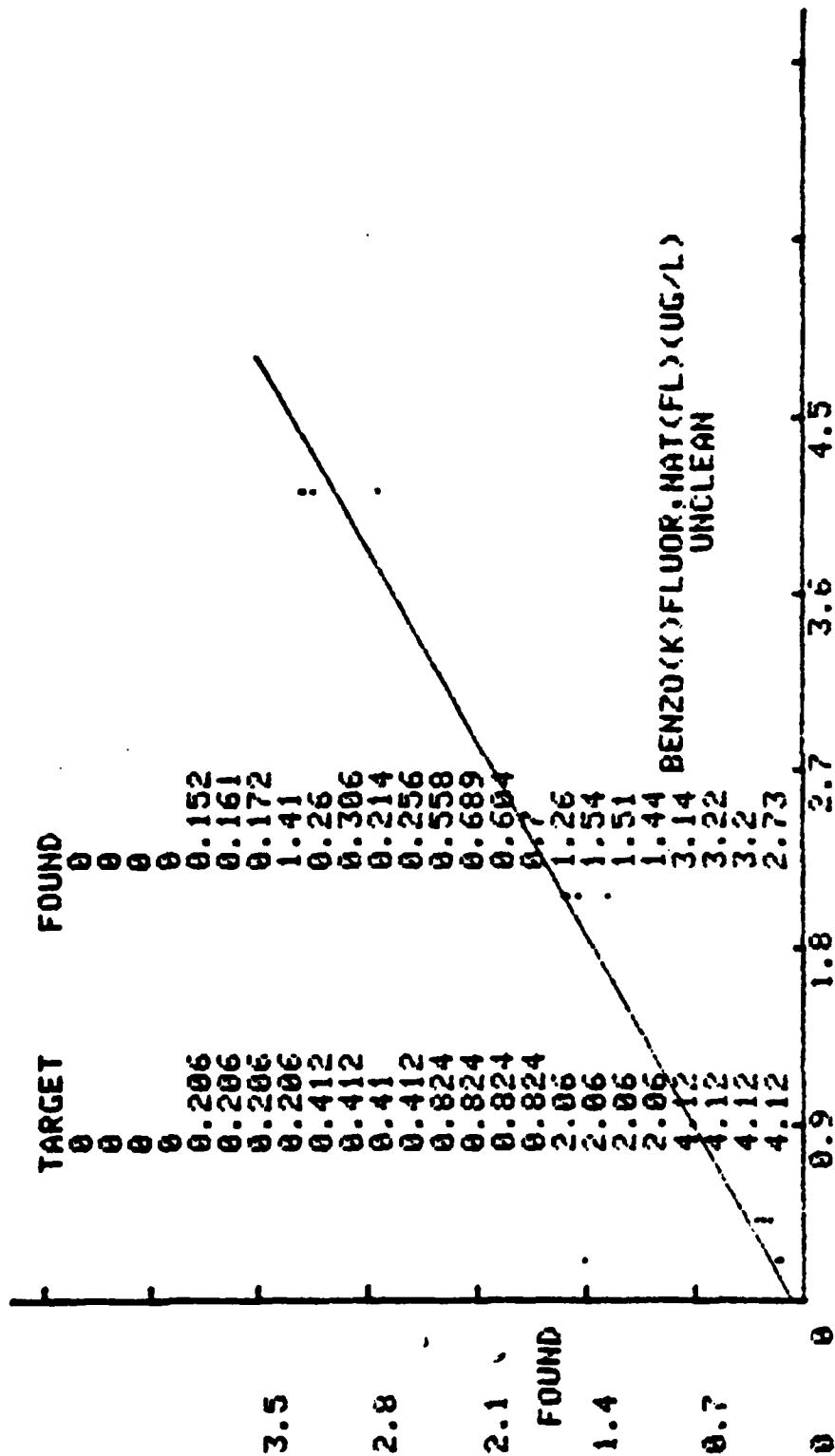


CORR. COEFF. = 0.9764 FOUND = TARGET
DETECTION LIMIT = 1.16131 0.0647+ 0.772872*TARGET

BENZO(K)FLUOR,NAT(FL)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.152	0.161	0.172	1.41
0.412	0.263	0.306	0.214	0.256
0.824	0.558	0.689	0.604	0.700
2.06	1.26	1.54	1.51	1.44
4.12	3.14	3.22	3.20	2.73

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.474	0.624	132	130
0.412	0.566	0.161	0.0000	-62.6
0.824	0.214	0.0458	0.0000	-47.8
2.06	0.256	0.0655	0.0000	-37.9
4.12	0.638	0.0683	10.7	-22.6



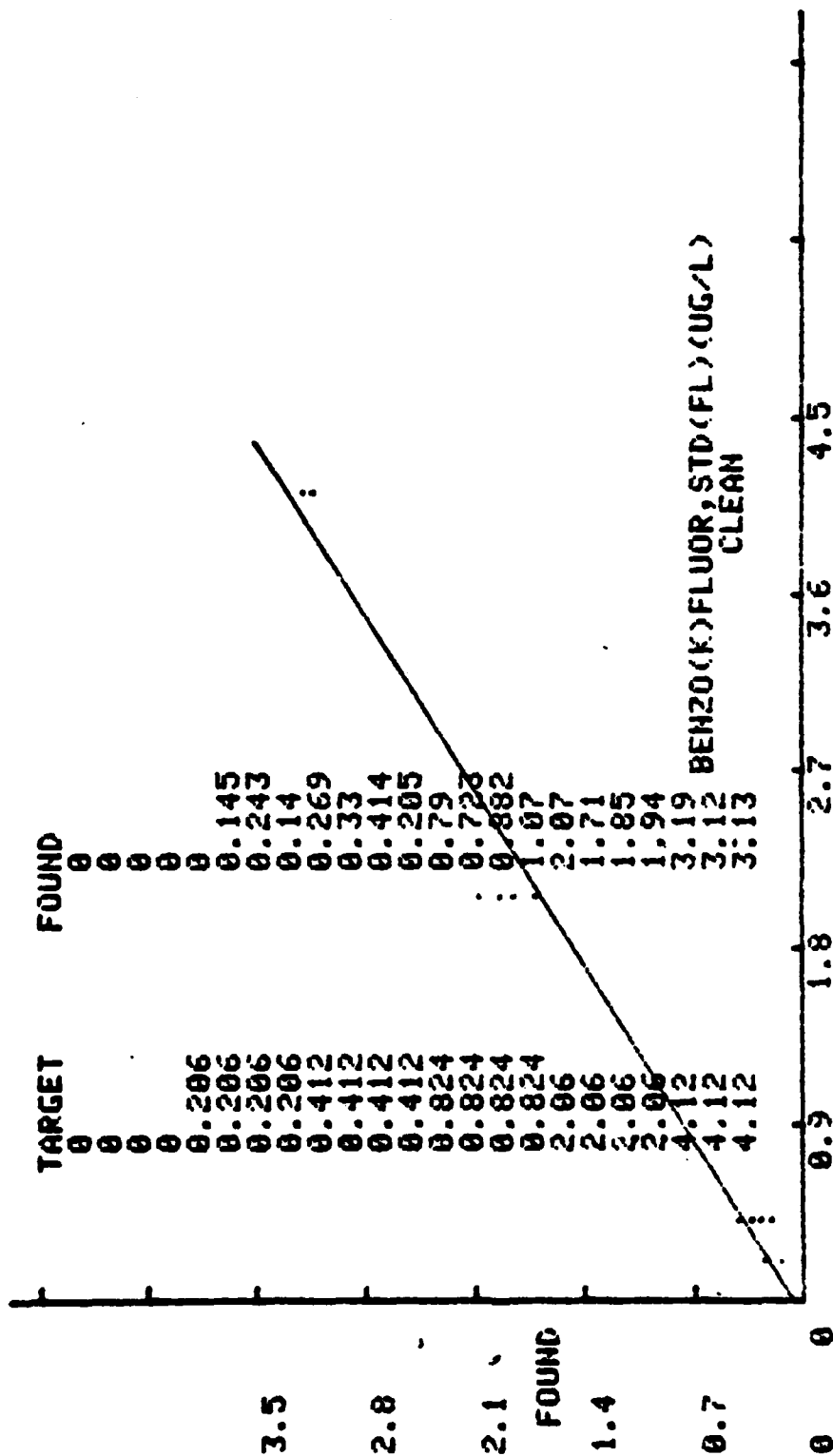
CORR. COEFF. = 0.9667 FOUND = 0.0724+ TARGET 0.714538*TARGET

DETECTION LIMIT = 1.38961

BENZOC(K)FLUOR, STD (FL) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.0000	0.145	0.243	0.140
0.412	0.269	0.330	0.414	0.205
0.824	0.790	0.723	0.892	1.07
2.06	2.07	1.71	1.85	1.94
4.12	3.19	3.12	3.13	0.0000

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.206	0.132	0.1000	75.7	-35.9
0.412	0.304	0.0891	29.3	-26.1
0.824	0.866	0.151	17.4	5.13
2.06	1.89	0.152	8.71	-8.13
4.12	3.15	0.0378	1.20	-23.6

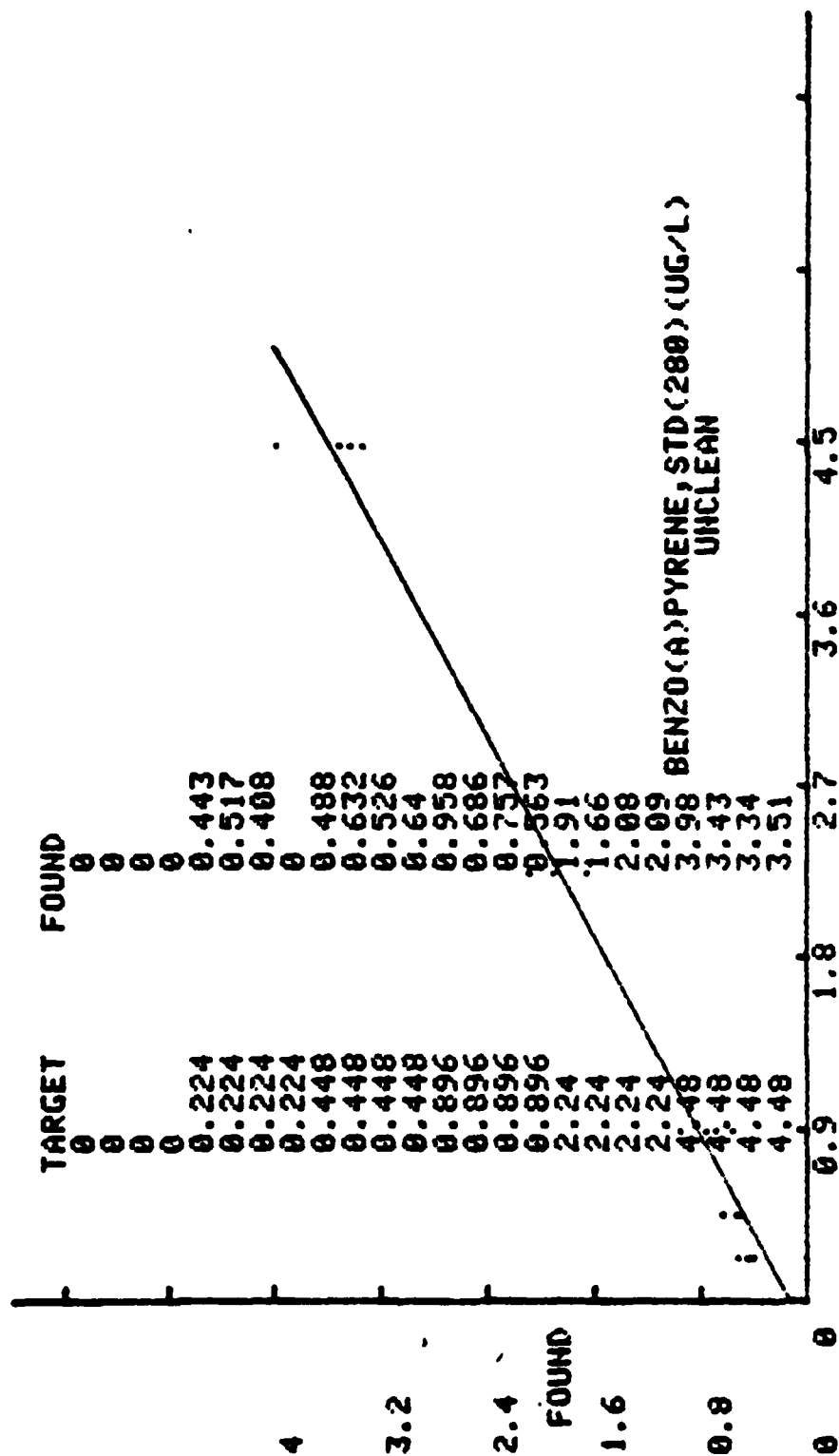


TARGET
CORR. COEFF. = 0.9882 FOUND = 0.0631+ 0.787665 * TARGET
DETECTION LIMIT = 0.76066

BENZO(A)PYRENE,STD(280)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	0.443	0.517	0.408	0.0000
0.448	0.488	0.632	0.526	0.640
0.896	0.958	0.686	0.757	0.563
2.24	1.91	1.66	2.08	2.09
4.48	3.98	3.43	3.34	3.51

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	0.342	0.232	68.0	52.7
0.448	0.571	0.0761	13.3	27.6
0.896	0.741	0.165	22.3	-17.3
2.24	1.93	0.201	10.4	-13.6
4.48	3.56	0.285	8.00	-20.4

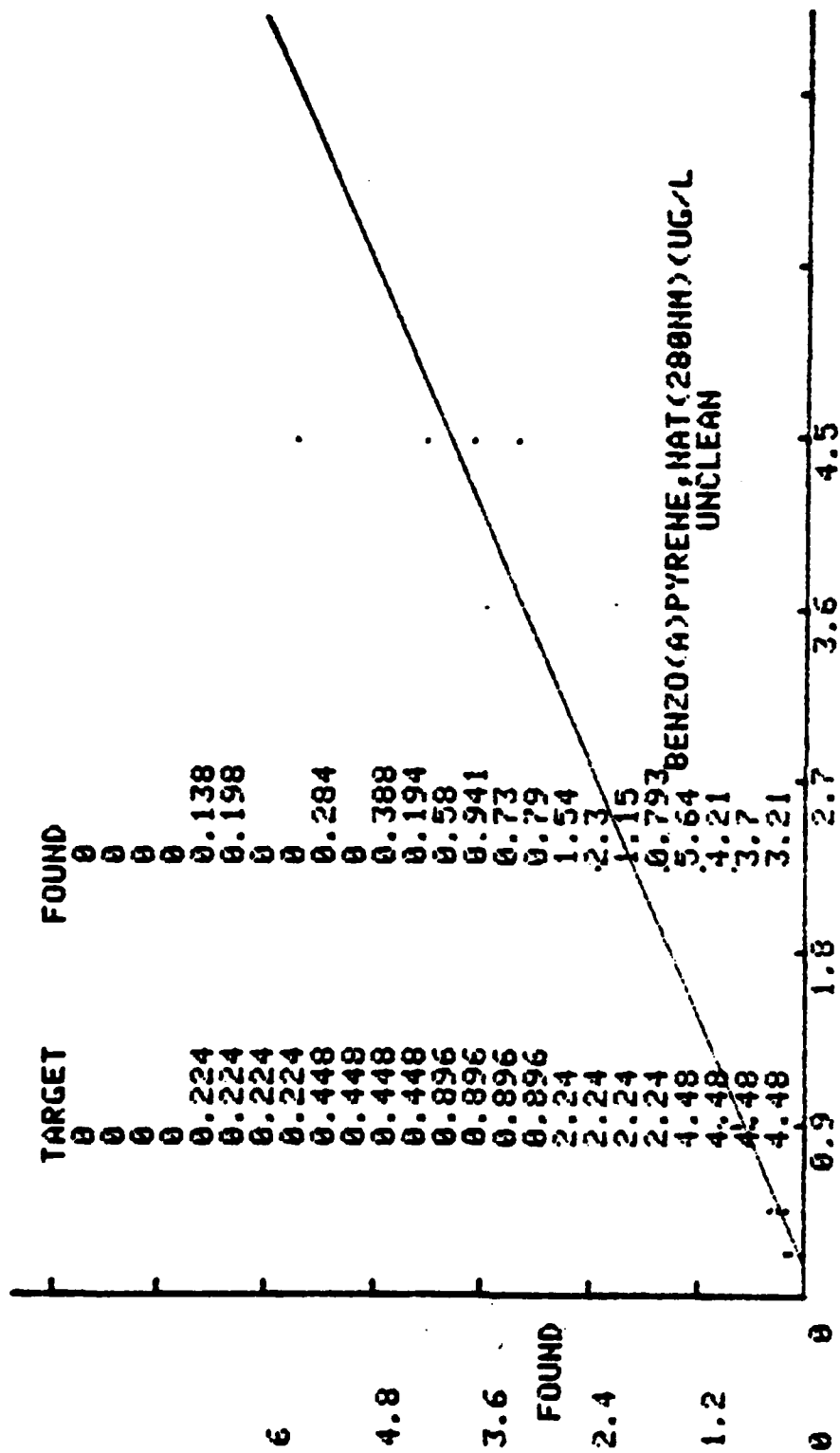


TARGET
CORR. COEFF. = 0.9891 FOUND = 0.1189+ 0.777191*TARGET
DETECTION LIMIT = 0.84921

BENZO(A)PYRENE,NAT(280NM) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	0.138	0.198	0.0000	.0000
0.448	0.284	0.0000	0.308	0.194
0.896	0.580	0.941	0.730	0.790
2.24	1.54	2.30	1.15	0.793
4.48	5.64	4.21	3.70	3.21

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	.0840	0.100	119	-62.5
0.448	0.216	0.165	76.1	-51.7
0.896	0.760	0.149	19.7	-15.2
2.24	1.45	0.646	44.7	-35.5
4.48	4.19	1.05	25.0	-6.47

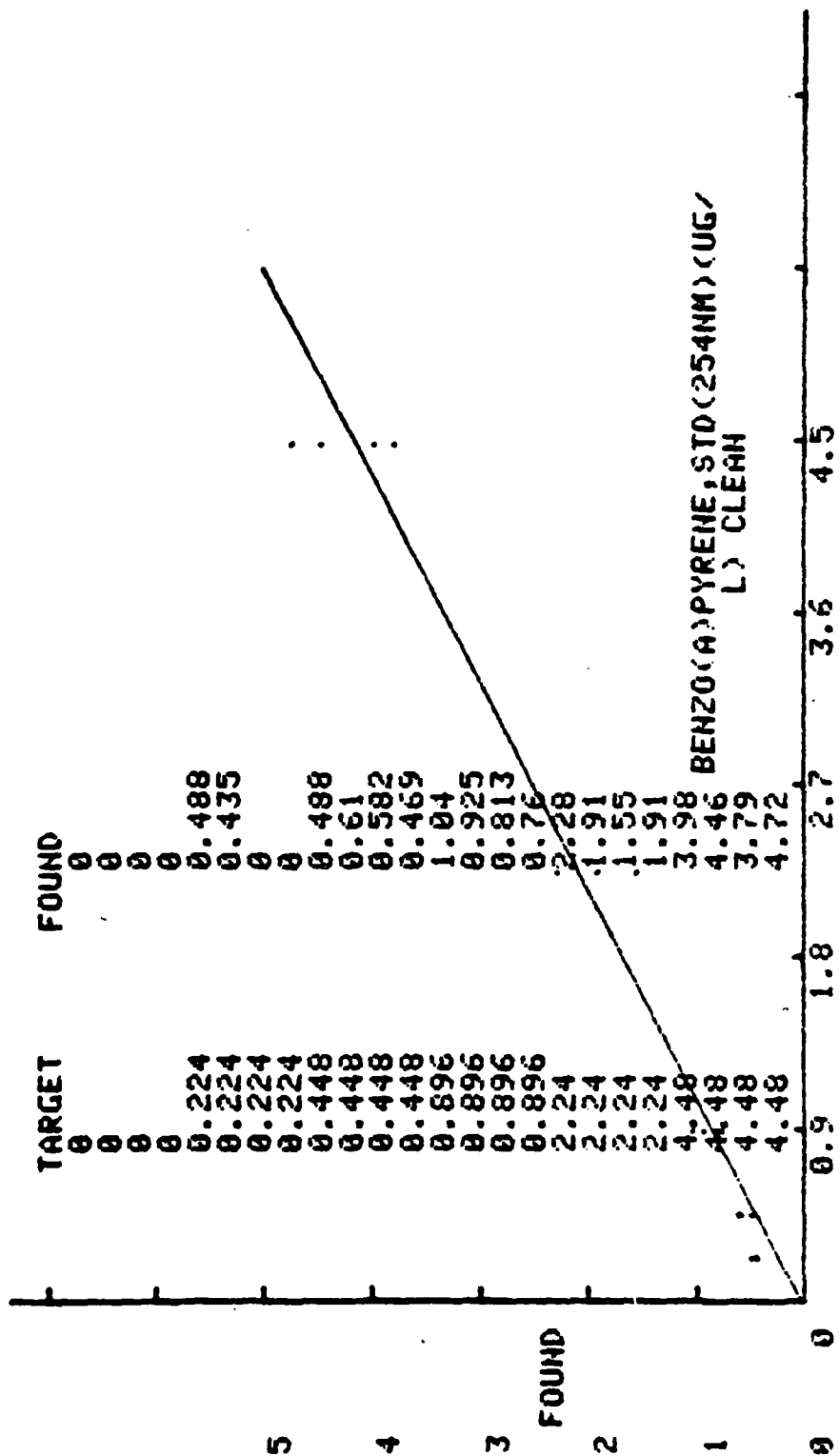


CORR. COEFF. = 0.9456 FOUND = TARGET
DETECTION LIMIT = 1.96246

BENZO(A)PYRENE,STD(254NM)(UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	0.488	0.435	0.0000	0.0000
0.448	0.488	0.610	0.582	0.469
0.896	1.04	0.925	0.813	0.760
2.24	2.28	1.91	1.55	1.91
4.48	3.98	4.46	3.79	4.72

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.224	0.231	0.267	116	3.01
0.448	0.537	0.0692	12.9	19.9
0.896	0.884	0.124	14.1	-1.28
2.24	1.91	0.298	15.6	-14.6
4.48	4.24	0.428	10.1	-5.41

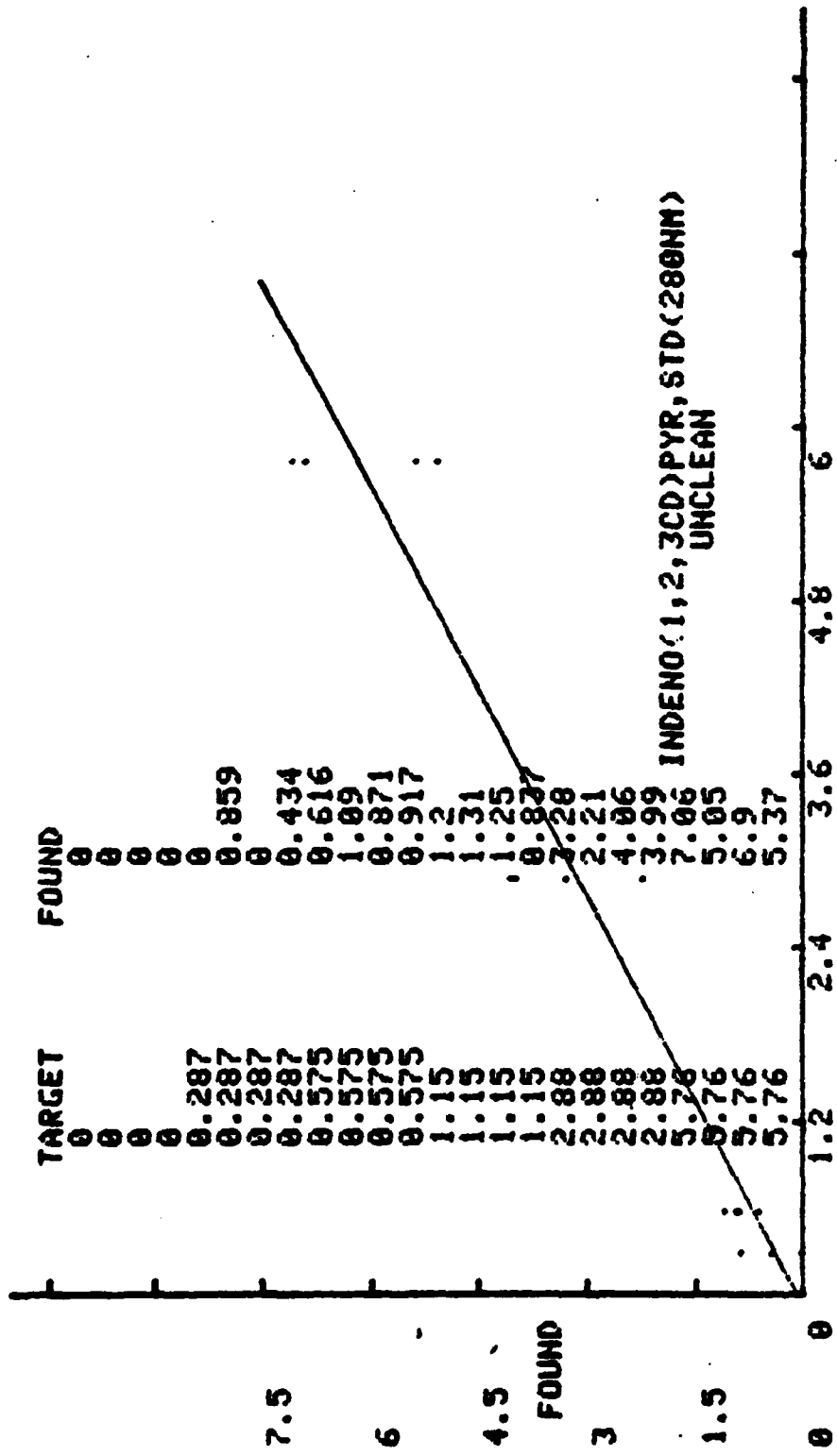


CORR. COEFF. = 0.9874
 DETECTION LIMIT = 0.9148
 TARGET FOUND = 0.0242+
 TARGET 0.923899

INDENO(1,2,3CD)PYR.,STD(280NM) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.287	0.0000	0.859	0.0000	0.434
0.575	0.616	1.09	0.871	0.917
1.15	1.20	1.31	1.25	0.837
2.88	3.28	2.21	4.06	3.99
5.76	7.06	5.05	6.99	5.37

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.287	0.323	0.412	127	12.6
0.575	0.873	0.196	22.4	51.9
1.15	1.15	0.213	18.5	-0.0652
2.88	3.38	0.859	25.4	17.5
5.76	6.09	1.03	16.9	5.82

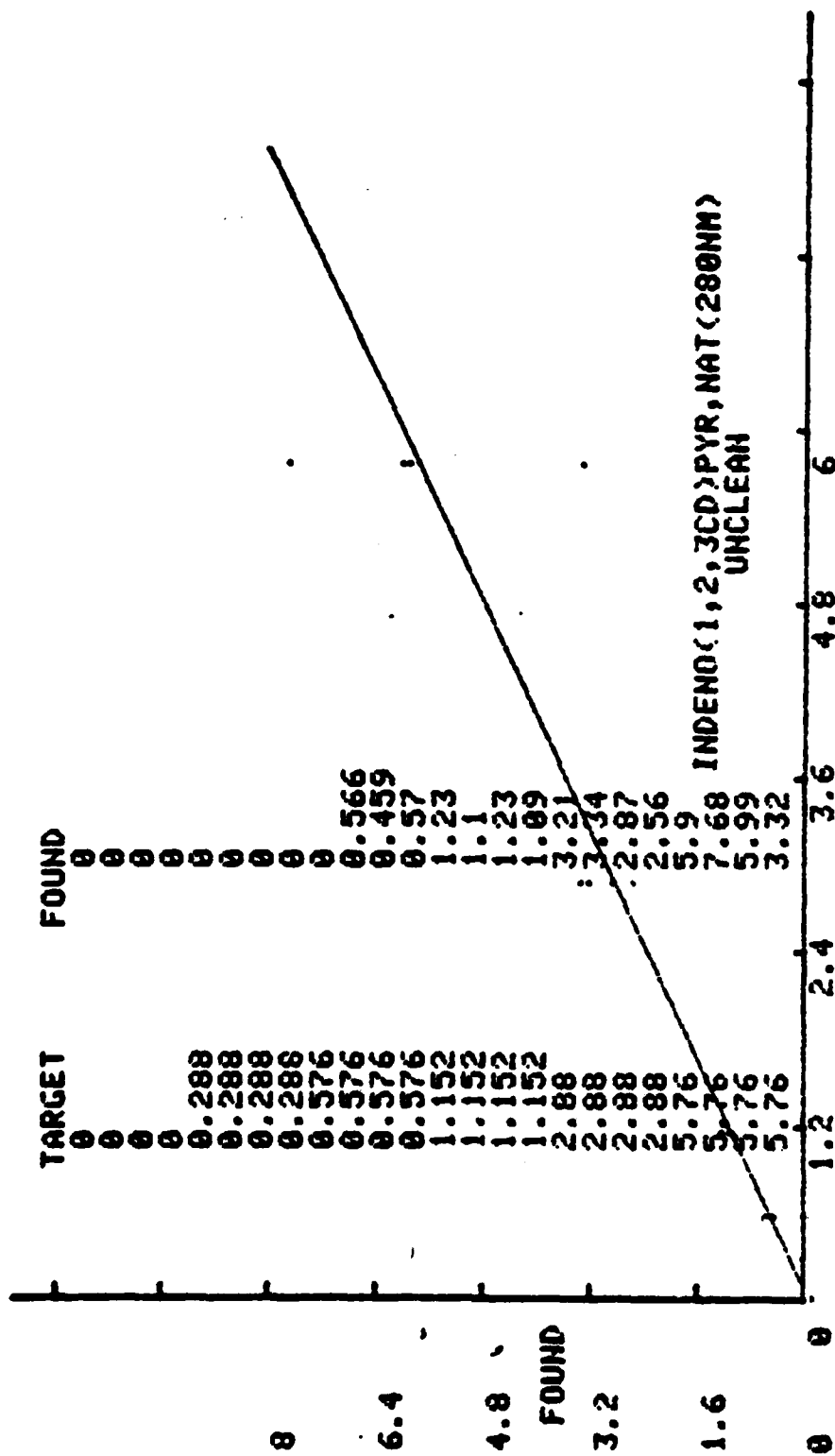


TARGET
CORR. COEFF. = 0.9706 FOUND = 0.0875+ 1.060952*TARGET
DETECTION LIMIT = 1.01834

INDENO(1,2,3CD)PYR,NAT(280NM) UNCLEAR

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.288	0.0000	0.0000	0.0000	0.0000
0.576	0.0000	0.566	0.459	0.570
1.15	1.23	1.10	1.23	1.00
2.88	3.21	3.34	2.87	2.56
5.76	5.9	7.68	5.99	3.32

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.288	0.0000	0.0000	0.0000	-100
0.576	0.399	0.271	67.9	-30.8
1.15	1.16	0.0780	6.71	0.911
2.88	2.99	0.351	11.7	3.99
5.76	5.72	1.80	31.4	-0.651

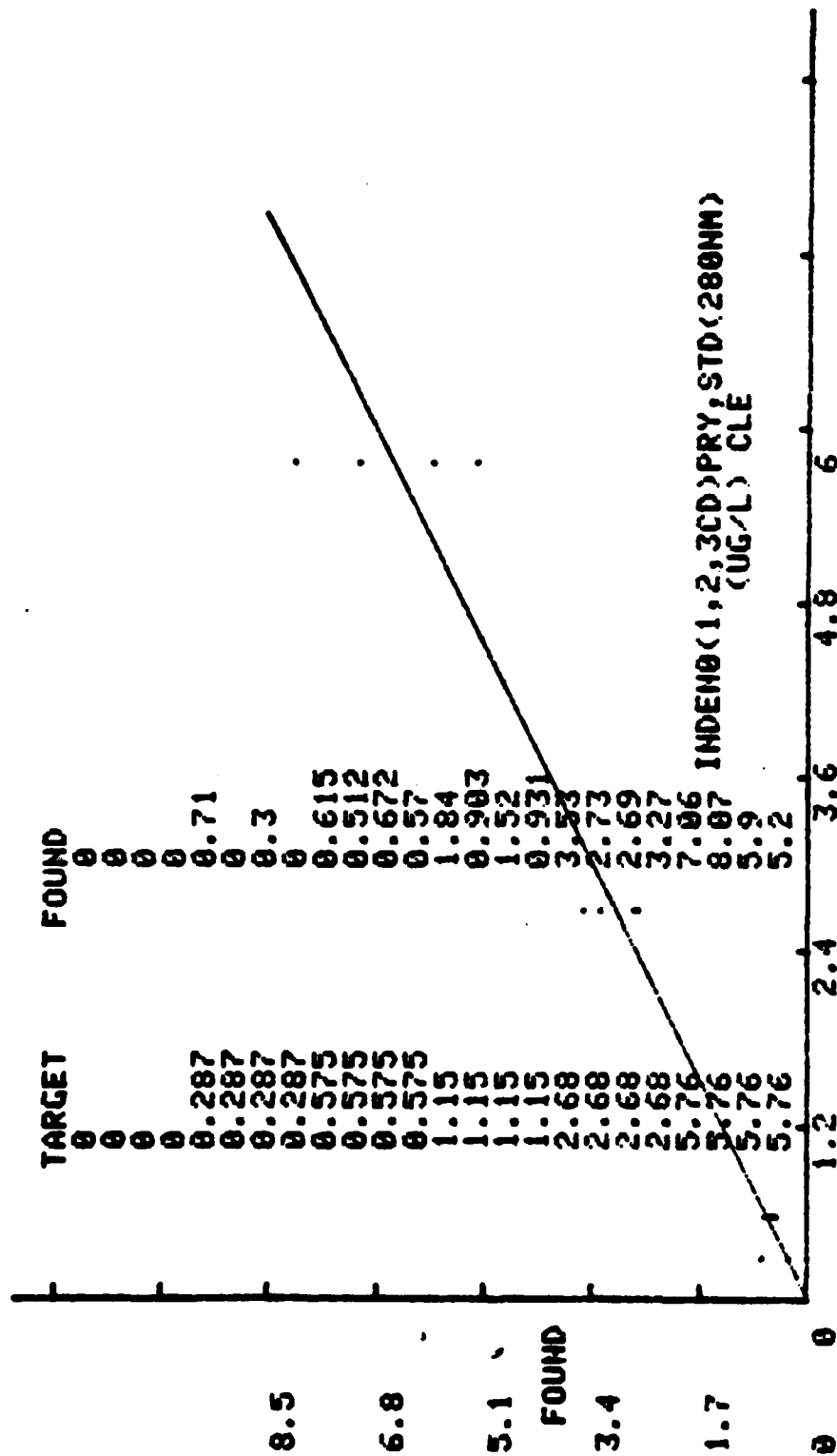


CORR. COEFF. = 0.9515 FOUND = TARGET
 DETECTION LIMIT = 2.37166
 -0.1076+ 1.025207*TARGET

INDEN(1,2,3CD)PRY,STD(280NM)(UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4
0.0000	0.0000	0.0000	0.0000	0.0000
0.287	0.710	0.0000	0.300	.0000
0.575	0.615	0.512	0.672	0.570
1.15	1.84	0.903	1.52	0.931
2.68	3.53	2.73	2.69	3.27
5.76	7.86	8.07	5.90	5.20

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.287	0.252	0.336	133	-12.0
0.575	0.592	0.0679	11.5	3.00
1.15	1.30	0.460	35.4	12.9
2.68	3.05	0.413	13.5	14.0
5.76	6.56	1.27	19.3	13.8



CORR. COEFF. = 0.9757 FOUND = TARGET
DETECTION LIMIT = 1.63272 1.14644 * TARGET