DTIC FILE COPY

1

ESL-TR-87-28
VOL III

THERMAL DESORPTION/ULTRAVIOLET PHOTOLYSIS PROCESS TECHNOLOGY RESEARCH, TEST, AND EVALUATION PERFORMED AT THE NAVAL CONSTRUCTION BATTALION CENTER, GULFPORT, MS, FOR THE USAF INSTALLATION RESTORATION PROGRAM, VOLUME III

AD-A202 266

R.W. HELSEL, R.W. THOMAS

EG&G IDAHO, INC.
P.O. BOX 1625
IDAHO FALLS ID 83415

DECEMBER 1987

FINAL REPORT

MAY 1985 - JULY 1985

DTIC
ELECTE
DEC 1 6 1988
S D
E

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED

AFESC

ENGINEERING & SERVICES LABORATORY
AIR FORCE ENGINEERING & SERVICES CENTER
TYNDALL AIR FORCE BASE, FLORIDA 32403

88

NOTICE

PLEASE DO NOT REQUEST COPIES OF THIS REPORT FROM HQ AFESC/RD (ENGINEERING AND SERVICES LABORATORY). ADDITIONAL COPIES MAY BE PURCHASED FROM:

NATIONAL TECHNICAL INFORMATION SERVICE
5285 PORT ROYAL ROAD
SPRINGFIELD, VIRGINIA 22161

FEDERAL GOVERNMENT AGENCIES AND THEIR CONTRACTORS REGISTERED WITH DEFENSE TECHNICAL INFORMATION CENTER SHOULD DIRECT REQUESTS FOR COPIES OF THIS REPORT TO:

DEFENSE TECHNICAL INFORMATION CENTER
CAMERON STATION
ALEXANDRIA, VIRGINIA 22314

DISCLAIMER NOTICE

THIS DOCUMENT IS BEST QUALITY PRACTICABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION

1b. RESTRICTIVE MARKINGS

2a. SECURITY CLASSIFICATION AUTHORITY

2b. DECLASSIFICATION / DOWNGRADING SCHEDULE

3. DISTRIBUTION / AVAILABILITY OF REPORT
Approved for public release
Distribution Unlimited

4. PERFORMING ORGANIZATION REPORT NUMBER(S)

5. MONITORING ORGANIZATION REPORT NUMBER(S)
ESL-TR-87-28

6a. NAME OF PERFORMING ORGANIZATION
EG&G Idaho

6b. OFFICE SYMBOL *(If applicable)*

7a. NAME OF MONITORING ORGANIZATION

6c. ADDRESS *(City, State, and ZIP Code)*
P.O. Box 1625
Idaho Falls, ID 83415

7b. ADDRESS *(City, State, and ZIP Code)*

8a. NAME OF FUNDING / SPONSORING ORGANIZATION Air Force Engineering & Services Center

8b. OFFICE SYMBOL *(If applicable)*
RDVW

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER

8c. ADDRESS *(City, State, and ZIP Code)*
HQ AFESC/RDVW
Tyndall AFB, Fl 32403-6001

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO	WORK UNIT ACCESSION NO.

11. TITLE *(Include Security Classification)*
Thermal Desorption/Ultraviolet Photolysis Process Technology Research, Test, and Evvaluatio
performed at the NCBC, Gulfport, MS, for the USAF Installation Restoration Program

12. PERSONAL AUTHOR(S)
R.W. Helsel and R.W. Thomas

13a. TYPE OF REPORT
FINAL

13b. TIME COVERED
FROM May 85 TO July 85

14. DATE OF REPORT *(Year, Month, Day)*
December 1987

15. PAGE COUNT
920

16. SUPPLEMENTARY NOTATION
Availability of this report is specified on reverse of front cover.

17. COSATI CODES

FIELD	GROUP	SUB-GROUP
07	01	
14	01	

18. SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*

Herbicide Orange	Thermal Treatment	2,4-D
Dioxin	Incineration	2,4,5, T
Analytical Methods		2,3,7,8-TCDD

19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

The objective of this effort was to examine the feasibility of using a thermal desorption/ultraviolet (TD/UV) destruction technology to treat Herbicide Orange (HO)-contaminated soil at the Naval Construction Battalion Center (NCBC), Gulfport, Mississippi. The IT Corporation pilot -scale TD/UV apparatus was used to successfully treat 1700 pounds of sandy-loam, cement stabilized, soil that had been contaminated with HO and 2,3,7,8 tetrachlorobenzo-p-dioxin (TCDD). The TD/UV process volatilizes organic compounds from the soil matrix; collects the desorbed organics in a solvent; and, destroys the contaminants with high-intensity ultraviolet light. The desorption process occurs between 850 to 1150 degrees F. in a nitrogen atmosphere to prevent combustion of the organics. Analysis of feedstock showed TCDD levels ranged from 233-272 parts per billion (ppb). Concentration in the treated soil, measured as the sum of all dioxin/furan congeners, was less than 1ppb, the USAF criterion. The TD/UV process demonstrated the capability to treat dioxin-contaminated soil

(cont'd. on reverse side)

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS

21. ABSTRACT SECURITY CLASSIFICATION
Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL
Terry L. Stoddart, Maj., USAF, BSC

22b. TELEPHONE *(Include Area Code)*
(904) 283-2942

22c. OFFICE SYMBOL
RDVW

DD Form 1473, JUN 86

Previous editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE
UNCLASSIFIED

No. 19 continued

and a scaled up version could be considered as a bulk reduction process for restoration of sites contaminated with chlorinated organic compounds including other DOD Herbicide Orange contaminated sites. Sensitivity analyses of six variables (geographic) location, soil quantity, electrical power prices, labor, capital equipment use charge, and transportation) were performed to estimate the cost for conditions other than those found at NCBC. The cost to treat one ton of contaminated soil using a scaled up system, based on treatment of 20,000 tons at NCBC, is $402/ton. The process may have application for treatment of other chlorinated organic compounds. The process may have unique application in geographical areas where incineration would not be accepted.

One negative aspect is that the photolysed solvent remains a hazardous waste and must be handled appropriately. Additional R&D is required to establish an alternate photolysis unit to overcome the problem.

Sent

This report is organized into four volumes: Volume I presents the final report on the performance of the Thermal Desorption/Ultraviolet Photolysis process for use in decontaminating soil containing Herbicide Orange/Dioxin. Volume II contains appendices A through O. Volume III contains appendix P. Volume IV contains appendices Q through V.

See pg V

NTIS GRA&I ☒
DTIC TAB ☐
Unannounced ☐
Justification
By
Distribution/
Availability Codes
Dist A-1 | Avail and/or Special pm 23

ALITY INSPECTED

PREFACE

This report was prepared for the Air Force Engineering and Services Center, Engineering and Services Laboratory, Tyndall AFB, Florida, under Job Order Number (JON) 2103 9027. The principal contractor, EG&G Idaho, Inc., is the prime contractor for the Department of Energy, Idaho National Engineering Laboratory. The major subcontractor for the project is the International Technologies Corporation, Knoxville, Tennessee.

This report is organized into four volumes: Volume I presents the final report on the performance of the Thermal Desorption/Ultraviolet Photolysis process for use in decontaminating soil containing Herbicide Orange/dioxin. Volume II contains appendices A through O. Volume III contains appendix P. Volume IV contains appendices Q through V.

Other contributors to this report include: E. Alperin, W.A. Prop, A.E. Grey, D.L. Miller, H.J. Welland, D.J. Harvego, H.D. Williams, and G. Peterson.

This report has been reviewed by the Public Affairs Office (PAO) and is releasable to the National Technical Information Services (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This report has been reviewed and approved for publication.

Terry L Stoddart
TERRY L. STODDART, Maj, USAF, BSC
Chief, Environmental Restoration R&D

Thomas J Walker
THOMAS J. WALKER, Lt Col, USAF, BSC
Chief, Environics Division

F. THOMAS LUBOZYNSKI, Maj, USAF, BSC
Chief, Environmental Engineering Branch

LAWRENCE D. HOKANSON, Colonel, USAF
Director, Engineering and Services Laboratory

TABLE OF CONTENTS

Section Title Page

Cont'd

P INVESTIGATION OF THE APPLICABILITY OF THE EPA MOBILE INCINERATOR SYSTEM FOR TREATING SOILS CONTAMINATED WITH HERBICIDE ORANGE 1

Contents of this appendix include; See pg 5

(The reverse of this page is blank.)

APPENDIX P

INVESTIGATION OF THE APPLICABILITY OF THE EPA MOBILE INCINERATOR SYSTEM FOR TREATING SOILS CONTAMINATED WITH HERBICIDE ORANGE

The documents contained in this appendix were published according to their own internal style, which deviates from ESL format. They have, therefore, been published without editing.

(The reverse of this page is blank.)

INVESTIGATION OF THE APPLICABILITY OF THE EPA MOBILE INCINERATOR SYSTEM FOR TREATING SOILS CONTAMINATED WITH HERBICIDE ORANGE

By

R. Helsel, J. Fleming, E. Alperin, A. Groen,

For

Air Force Engineering and Services Center
Tyndall AFB, Florida

July 3, 1985

(The reverse of this page is blank.)

TABLE OF CONTENTS

Section Title Page

I INTRODUCTION 11

A. OBJECTIVE 11
B. BACKGROUND 11
C. SCOPE 12

Cont'd

II SOIL CHARACTERIZATION; 13

A. SOIL PREPARATION PROCEDURES 13
B. ANALYSIS OF PHYSICAL-CHEMICAL PROPERTIES 14
C. ANALYSIS OF HERBICIDE CONTAMINATION 18

III LABORATORY TREATABILITY TESTS; 26

A. EXPERIMENTAL PLAN 26
B. THERMAL TREATMENT TEST EQUIPMENT 27
C. STANDARD TEST PROCEDURE 27
D. TREATABILITY TEST RESULTS AND DISCUSSION 29

1. Treatability Data 30
2. Physical Effects of Treatment 38

IV BATCH PILOT KILN TESTS; 43

A. EXPERIMENTAL PROCEDURES AND EQUIPMENT 43
B. TEST RESULTS AND DISCUSSION 47

1. Visual Observations 47
2. Test Data 48

V HEAT TRANSFER EVALUATION; 61

A. OBJECTIVES 61
B. PREVIOUS HEAT TRANSFER STUDY RESULTS 62
C. HEAT TRANSFER COMPUTER SIMULATION RESULTS 63

Mobile Incineration System

VI EVALUATION OF (MIS) TREATMENT CAPABILITIES (R.W) 68

VII CONCLUSIONS 77

VIII RECOMMENDATIONS 79

IX REFERENCES 80

LIST OF ANNEXES

Page

1. Battelle Methods Employed in the Physical-Chemical Analyses of JI, Eglin, and NCBC Soils 82

2. Battelle Final Report on Physical-Chemical Characterization of Uncontaminated Soils from JI, Eglin, and NCBC 115

3. Battelle Data Table for Physical-Chemical Characterization of Contaminated Soils from JI, Eglin, and NCBC 122

4. Allis-Chalmers Report on Small Batch Kiln Test Sieve Analysis and Bulk Density Measurements for JI, Eglin, and NCBC Soils 124

5. Analytical Methods for 2,3,7,8-TCDD 199

6. Summary of QA/QC Results for 2,3,7,8-TCDD Analyses 223

7. Analytical Data Sheets 227

8. Experimental Data—Laboratory [illegible] Tests 244

9. Particle Entrainment Theory 261

10. Sample Computer Heat Transfer and Heat and Material Balance Calculations 266

11. Statistical Data 276

LIST OF FIGURES

Figure	Title	Page
1	Schematic of Oven Interior..................................	28
2	Typical Time-Temperature Profiles for Laboratory Desorption Tests..	31
3	Effect of Time and Temperature on Removal of 2,3,7,8-TCDD from JI Soil..	33
4	Effect of Time and Temperature on Removal of 2,3,7,8-TCDD from Eglin Soil...	34
5	Effect of Time and Temperature on Removal of 2,3,7,8-TCDD from NCBC Soil..	35
6	Effect of Treatment Time at 481°C on Removal of 2,3,7,8-TCDD.	36
7	Schematic Diagram of the Pilot Batch Kiln..................	44
8	Photograph of the Allis-Chalmers Pilot Kiln System..........	45
9	Comparison of Entrainment With Superfical Gas Velocity in Batch Pilot Kiln...	53
10	Particle Size Distribution of JI Soil Before and After Batch Kiln Test 4...	55
11	Particle Size Distribution of Eglin Soil Before and After Batch Kiln Test 6...	56
12	Particle Size Distribution of NCBC Soil Before and After Batch Kiln Test 10..	57
13	Time-Temperature History for Three Batch Pilot Kiln Tests Using Approximately Equal Heat Inputs.........................	58
14	Projected Vapor Pressure of 2,3,7,8-TCDD at High Temperatures..	70
15	Plot of LNPD vs. IVP.......................................	71
16	Predicted Time-Temperature Required to Achieve 1 ppb 2,3,7,8-TCDD for Soils Initially Containing 100 ppb and 500 ppb..	72
17	Temperature vs. Time for Heat Transfer Run Number 2.........	74
18	Vapor Pressure vs. Time for Heat Transfer Run Number 2.......	75

LIST OF TABLES

Table	Title	Page
1	Soil Characterization Methods	15
2	Physical-Chemical Analysis of Uncontaminated Soil Samples	16
3	Physical-Chemical Analysis of Contaminated Soil Samples	17
4	Summary of Thermal Analysis Results	19
5	Analytical Methods Used for Determination of Herbicides	21
6	Analysis of Baseline Soil Samples for Compounds of Interest	22
7	Analysis of Contaminated Soil Samples for Compounds of Interest	23
8	Evaluation of Relative Concentrations of Herbicide Constituents for Contaminated Test Soils	25
9	Summary of Treatability Results - Effect of Time and Temperature on Final Concentration of 2,3,7,8-TCDD	32
10	Calculated Removal Efficiencies For 2,3,7,8-TCDD	37
11	Analysis of Test Soil Samples Treated at 558°C for Compounds of Interest	39
12	Calculated Removal Efficiencies for 2,4-DBE and 2,4,5-TBE After Treatment at 558°C	40
13	Summary of Weight Loss Data From Laboratory Treatability Tests	41
14	Test Conditions - Batch Kiln Testing	46
15	Weight Loss During Pilot Kiln Tests	49
16	Soil Bulk Densities Before and After Pilot Kiln Tests	50
17	Summary of Soil Entrainment Results During Batch Kiln Tests	52
18	Summary of Dynamic Angle-of-Repose Measurements	60
19	Parameters for Heat Transfer Runs	64

LIST OF TABLES

Table	Title	Page
20	Predicted Temperatures from Computer Models.............	65
21	Secondary Combustion Chamber Burner Fuel Requirements for Heat Transfer Runs..................................	67
22	Predicted Treatment Efficiency for Heat Transfer Runs..	76

LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

NCBC	Naval Construction Battalion Center, Gulfport, Mississippi
JI	Johnston Island
CDD	Chlorodibenzodioxin
CDF	Chlorodibenzofuran
TCDD	Tetrachlorodibenzo-p-dioxin
PCDD	Total pentachlordibenzo dioxin isomers
H_xCDD	Total hexachlorodibenzo dioxin isomers
H_pCDD	Total pentachlorodibenzo dioxin isomers
OCDD	Total octachlorodibenzodioxin isomers
2,4-DBE	Butyl ester of 2,4-dichlorophenoxyacetic acid ($C_{12}H_{14}Cl_2O_3$)
2,4,5-TBE	Butyl ester of 2,4,5-trichlorophenoxyacetic acid ($C_{12}H_{13}O_3Cl_3$)
2,4-D	2,4-dichlorophenoxyacetic acid ($C_8H_6Cl_2O_3$)
2,4,5-T	2,4,5-trichlorophenoxyacetic acid ($C_8H_5Cl_3O_3$)
HO	Herbicide Orange, 50:50 (by volume) mixture of n-butyl esters of 2,4-D and 2,4,5-T
MIS	EPA Mobile Incinerator System
TGA	Thermal Gravimetric Analysis
DSC	Differential scanning calorimeter
SCC	Secondary combustion chamber

SECTION I

INTRODUCTION

A. OBJECTIVE

This study was done to determine the applicability of the EPA's mobile incineration system (MIS) for treating soil contaminated with Herbicide Orange at three Department of Defense sites - Eglin Air Force Base, the Naval Construction Battalion Center (NCBC), Gulfport, Mississippi, and Johnston Island. Beyond demonstrating the technical feasibility of thermal decontamination, the study was to evaluate the treatment characteristics of the soils to identify potential processing problems and to estimate operating rates and corresponding operating conditions which would achieve treatment ("clean-up") criteria. The results of this study would provide some technical basis for supporting and planning a possible demonstration by the Air Force of the MIS at Johnston Island.

B. BACKGROUND

The U.S. Air Force is engaged in a multitask program to investigate three Department of Defense sites known to be contaminated with residual Herbicide Orange (Reference 1), and to identify, evaluate, and demonstrate selected technologies that could be used to decontaminate and restore these sites. Herbicide Orange is an equal mixture of the butyl esters of 2,4-dichlorophenoxyacetic acid (2,4-DBE) and 2,4,5-trichlorophenoxyacetic acid (2,4,5-TBE), containing low or trace concentrations of various related chemical compounds originating from raw materials or byproduct reactions (Reference 2). One of these compounds has been determined to be 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), a highly toxic and stable compound which is the subject of much scientific and regulatory attention today. Previous field investigations at all three sites have ascertained the approximate location and concentration of the herbicide constituents and have enabled preliminary estimates to be made of the quantities of soil that could require decontamination. Additional, more extensive site surveys are currently being completed.

New federal regulations enacted under RCRA, which will be effective after July 15, 1985, stipulate stringent requirements for treating or disposing of dioxin waste materials, including contaminated soil. Incineration has been demonstrated to be effective in destroying Herbicide Orange (References 3 and 4). Each incineration system and dioxin waste matrix must be tested to establish adequate treatment efficiency before being permitted to operate on a "production basis." Decontamination of soil containing dioxin by using incineration had not been demonstrated.

A mobile incineration system (MIS) has been constructed by EPA for use in treating hazardous waste materials at various sites, and in evaluating the technology for specific wastes. This process unit has recently completed a trial burn involving dioxin contaminated waste liquids, sludges, and soil in Missouri. A longer demonstration run is planned by EPA which will involve processing quantities of various dioxin contaminated soils from Missouri sites.

In support of EPA's mobilization of the MIS for that trial burn and demonstration, IT Corporation conducted a study to investigate the capability of the MIS to treat soil contaminated with 2,3,7,8-TCDD. The study consisted of two parts:

- A series of laboratory thermal desorption experiments to determine the effect of key process variables and soil (feed) characteristics on removal of 2,3,7,8-TCDD
- A series of pilot-scale batch kiln tests and computer simulations to define the material processing and heat transfer characteristics of the kiln

This research was completed in late 1984. A final report describing the results has been submitted to EPA for review.

C. SCOPE

This study, as an extension of the previous EPA study, consisted of four activities - soil characterization, laboratory treatability tests, pilot-scale kiln processing tests, and engineering assessment of projected MIS performance. Soil characterization measured chemical properties, including Herbicide Orange concentration, and physical properties which could affect processing. Bench-scale treatability tests determined the relationship between residence time, temperature, and treatment efficiency. Samples of contaminated soil were exposed to different conditions, and the final concentration of 2,3,7,8-TCDD was analyzed. The pilot kiln tests utilized uncontaminated soil to simulate the approximate MIS conditions of gas flow, temperature, and agitation to investigate particulate entrainment, heat transfer and changes in physical form (agglomeration/slagging or attrition). Engineering assessment included performing heat and material balance calculations and heat transfer calculations using computer simulations and determining the various limitations of the MIS which would establish processing rate capabilities.

SECTION II

SOIL CHARACTERIZATION

The chemical and physical properties of soils subjected to incineration temperatures are expected to influence the treatability and processing performance. Volatilization of organic pollutants from soil at ambient conditions has been shown to be affected by the exposed surface area (corresponding to particle size) and composition of the soil (References 5, 6 and 7). Interaction (adsorption/ absorption) between organic compounds and a soil matrix can reduce the apparent vapor pressure. Thermochemical transitions of natural organic matter and minerals contained in soils can occur at temperatures as low as 400°C (References 8 through 12). Both oxidation and pyrolysis of organic materials can occur, depending particularly on the availability of oxygen. The thermal stability of specific organic compounds which might be present in contaminated soils varies greatly within the operating range of the MIS (References 13 and 14). Physical alteration of the soil, such as attrition from thermal stresses and agitation/abrasion, and slagging or agglomeration caused by mineralogical transformations, will affect solids flow characteristics, heat transfer, and particulate generation (entrainment).

Previous studies of thermal treatment of contaminated soils included analysis of the untreated soil for primary chemical and physical parameters. These same analyses were performed for this study on samples from each of the three sites. The soil samples were collected from designated locations and shipped to ITC's Environmental Research Laboratory in Knoxville, Tennessee. Samples of both uncontaminated and contaminated soils were packaged in sealed metal paint cans. Each sample weighed approximately 4 kilograms. Both the contaminated and uncontaminated samples used for laboratory treatability tests were prepared for analysis and testing according to procedures described below. Samples of uncontaminated soil were shipped to Allis-Chalmers' test facility and were used for batch kiln tests without any preparation.

The initial contaminated NCBC soil sample had a very low concentration of 2,3,7,8-TCDD. Three additional samples were taken at the site, shipped to ITC, prepared, and analyzed. Based on the results, one of the three was selected as the test sample. It was intended that all three contaminated test soils would have greater than 400 ppb 2,3,7,8-TCDD.

A. SOIL PREPARATION PROCEDURES

The samples of soil received from the Air Force were pretreated for use before analysis and thermal treatment tests. This was necessary to achieve a uniform test soil of known 2,3,7,8-TCDD concentration, from which representative aliquots could be taken for each test. Previous ITC research activities with soil containing trace levels of contamination had established the importance of such soil preparation, particularly for small-scale tests.

1. A portion of soil was transferred within the hood from the container to a metal foil tray, separating larger organic matter (e.g., roots, grass) and breaking up large soil agglomerates.

2. The soil was spread out uniformly on a metal tray and allowed to air dry within the hood for 2-3 days with occasional stirring and gentle breaking of larger soil agglomerates using a spatula.

3. The dried soil was screened in several batches, using a standard wire mesh screen with 2 mm openings. Oversize soil was returned to the drying tray and manually broken up using a spatula. Final oversize soil was weighed and transferred to the original soil container. Grinding or milling of the soil was not done.

4. The soil fractions less than 2 mm from each batch were combined in a widemouth, 1-gallon glass jar, blended for 1 hour, using a jar roller, and weighed.

5. Three separate aliquots of soil from random locations within the 1-gallon jar were transferred to individual 250 cc, amber, widemouth jars with Teflon®-lined caps. These sample jars were coded, labeled, and submitted for analysis of 2,3,7,8-TCDD to verify uniformity.

B. ANALYSIS OF PHYSICAL-CHEMICAL PROPERTIES

Battelle-Columbus Laboratories was contracted by ITC to perform selected analyses, including pH, conductivity, organic matter, cation exchange capacity (CEC), moisture content, specific surface area, oil and grease, and particle size. Table 1 lists the parameters and corresponding methods used by Battelle. These procedures are described in Annex 1. Samples of uncontaminated soil from each site were initially analyzed by Battelle; samples of contaminated soil from each site were analyzed later for selected parameters to investigate the potential effect of the contamination on the measurements.

Table 2, as reproduced from Battelle's final test report Annex 2, summarizes the characterization data for the uncontaminated samples, and Table 3 summarizes the data from the contaminated samples. A comparison between the data from each site and between uncontaminated and contaminated samples can be summarized by the following major points:

- The pH of the Eglin soil is acidic whereas the JI and NCBC soils are slightly alkaline.

- The conductivity of the JI soil is more than one order of magnitude higher than the other two soils.

- The organic matter measured for all the contaminated soil samples was much higher than for the uncontaminated samples. This difference was most noticeable for the JI soil samples; the difference in NCBC samples was relatively small.

TABLE 1. SOIL CHARACTERIZATION METHODS

Parameter	Method references[a]	Method
Organic matter	Schultz, 1980 Watson, 1978	Potassium dichromate oxidation using colorimetric determination of Cr^{3+}
pH	McLean, 1980	Measurement of 1:1 (weight: volume mixture of soil water)
Electrical conductivity	Watson, 1978	Measurement of 1:2 (soil:water) slurry using conductivity meter
Surface area	Carter, et al., 1965 Heilman, et al., 1965 Cihacek and Bremner, 1979	Adsorption of monolayer of ethylene glycol monoethyl ether
Particle size	ASTM, 1972 ASTM, 1978	Combined sieving (particles >75 µm and sedimentation particles less than 75 µm) measured by hydrometer
Cation exchange capacity (CEC)	Allen, et al., 1974	Saturation of exchange sites with NH_4^+ followed by washing of excess NH_4^+ reagent and displacement of adsorbed NH_4^+ with KCl; NH_4^+ measured by ion specific electrode
Oil and grease	ASTM D4281-83	Extraction with methyl tert-butyl ether after soil is conditioned with aqueous sodium chloride and potassium hydrogen phosphate; gravimetric determination of filtered and evaporated extract
Moisture	ASTM Part II-82216	Oven drying at 110°C to constant weight

[a] Complete references given in Battelle-Columbus procedures, Annex 1.

TABLE 2. PHYSICAL-CHEMICAL ANALYSIS OF UNCONTAMINATED SOIL

Physical-chemical parameters[a]	JI	Eglin	NCBC
pH	8.24	5.21	8.29
Conductivity (millimhos/cm)	4.99	0.0418	0.279
Organic matter (percent)	<0.50	<0.50	1.5
Cation exchange capacity (milliequivalents/100)	0.12	0.15	3.9
Moisture content (percent)	1.86	0.12	0.48
Surface area (m^2/g)	6.74	2.46	12.3
Oil and grease content (micrograms/g)	664.9	116.0	1759
Particle size distribution (percent)			
Medium sand (between 425 microns and 2.00 mm)	41.3	41.3	25.7
Fine sand (between 75 and 425 microns)	36.2	51.9	59.4
Silt (between 5 and 75 microns)	19.2	4.77	12.3
Clay (smaller than 5 microns, including colloids)[b]	3.31	2.03	2.70
Colloids (less than 1 micron)	2.94	1.67	2.25

[a] All analyses were done in duplicate with the exception of surface area, which was done in replicates of five. Values reported represent averages. Annex 2 gives deviation values.

[b] Note that the clay fraction includes the colloid fraction, so that the percentages total greater than 100 percent.

TABLE 3. PHYSICAL-CHEMICAL ANALYSIS OF CONTAMINATED SOIL

Physical-chemical parameters[a]	JI	Eglin	NCBC
pH	8.45	3.83	8.55
Conductivity (millimhos/cm)	5.02	0.146	0.205
Organic matter (percent)	4.22	1.22	2.34
Cation exchange capacity (milliequivalents/100 g)	0.73	0.77	2.45
Moisture content (percent)	0.34	0.55	0.38
Oil and grease content (micrograms/g)	1884	4069	3386

[a] All analyses were done in duplicate. Values given are averages. Annex 3 gives deviation values.

- The oil and grease content of the contaminated soil samples was higher than the uncontaminated samples. This difference was most noticeable (factor of 40) for Eglin soil.

- The cation exchange capacity of all contaminated soils was higher than the corresponding uncontaminated samples. NCBC soil had much higher values than the other two soils.

- The surface area did not vary greatly between soils. Likewise, the particle size distributions of the three soils were not greatly different. The clay fraction varied only between 3.5 and 7 percent.

Visual inspection of each sample received resulted in the following observations:

JI -	White/gray; sandstone-like, uniform; contaminated sample slightly darker, probably higher moisture; large stones and chunks (agglomerates) present in both.
Eglin -	Tan; sandy with some agglomerates which easily broke up, twigs and grass noticeable; uncontaminated sample appeared drier and dustier.
NCBC -	Light to dark brown; sandy loam with pebbles and broken shells; some grass and twigs evident; variation in relative amounts of moisture and shell/pebble fraction between the five samples received.

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis of the three samples of uncontaminated soil were performed by Allis-Chalmers as part of the pilot batch kiln tests. Table 4 summarizes the results of these analyses; the actual test data are given in Annex 2. It is obvious that the JI soil, which is principally calcium carbonate, exhibits substantial weight loss as a result of calcination (decarboxylation). The theoretical weight loss attributed to conversion of $CaCO_3$ to CaO (with the release of CO_2) is 44 percent. Calcination occurs at temperatures above about 625°C, with a corresponding large endotherm. The Eglin soil showed a moderate and extended exotherm between about 270 and 550°C, where a sharp endotherm occurred; only a small weight loss occurred. The NCBC soil showed a major and extended endotherm between about 225 and 550°C, where a sharp endotherm occurred. A marked weight loss occurred above that temperature, starting at 630°C.

Allis-Chalmers also performed sieve analyses and bulk density measurements on soil before and after pilot batch kiln processing. These results are discussed in Section IV and the data are presented in Annex 4.

C. ANALYSIS OF HERBICIDE CONTAMINATION

The prepared uncontaminated and contaminated soils were sampled and analyzed by ITC for the primary chemical constituents of Herbicide Orange and

TABLE 4. SUMMARY OF THERMAL ANALYSIS RESULTS[a]

Soil identification	Weight loss (%)[b]	Corresponding temperature range (°C)	Initiation of exotherm or endotherm (°C)[c]
JI	1.4	25 - 350	130 (-)
	42.5	350 - 1050	310 (-)
	43.9		
Eglin	2.4	25 - 1050	270 (+)
			550 (-)
NCBC	0.3	25 - 200	225 (+)
	1.4	200 - 520	550 (-)
	3.8	520 - 1090	
	5.5		

[a] Refer to Annex 4 for DSC and TGA data.

[b] Percent of initial gross weight.

[c] Exotherm = (+), Endotherm = (-)

for CDD and CDF compounds, including 2,3,7,8-TCDD. Arsenic was also analyzed to establish if any Herbicide Blue contamination was present. Herbicide Blue is composed principally of sodium dimethyl arsenate. The "uncontaminated" samples were analyzed to establish a baseline concentration of all constituents. Standard published EPA methods listed in Table 5 were used for quantitative determination of each parameter, except for the sample preparation and analysis of 2,3,7,8-TCDD and other CDDs and CDFs, which are described in Annex 5.

Two pretreatment/extraction procedures were used for the untreated soil samples to evaluate potential differences in the accuracy of analytical results. Previous studies with thermally treated Missouri soil samples demonstrated significantly lower 2,3,7,8-TCDD results using the standard jar extraction procedure with hexane/methanol (specified by EPA - CLP) than the more rigorous procedure involving Soxhlet extraction with benzene after acid treatment of the sample. Therefore, the Soxhlet procedure was used for all thermally treated samples. Both procedures were used for untreated soil samples for comparative purposes, and a comparison of results is given in Annex 6.

The method used for determining 2,4-DBE and 2,4,5-TBE actually measures the free acids, 2,4-D and 2,4,5-T, after chemically converting any esters present to the respective acid. Therefore, the reported values represent the combined ester and acid concentration present in the soil.

Three aliquots of soil were taken from each prepared soil sample and submitted for 2,3,7,8-TCDD analysis. Other parameters were determined on single aliquots. The results of the triplicate analyses, using both sample preparation procedures, are given in Annex 6. Tables 6 and 7 summarize the analytical results for the baseline ("uncontaminated") and contaminated soil samples. Annex 7 gives the specific analytical data. The baseline soils showed the presence of very low (<1 ppm) levels of herbicide constituents. No 2,3,7,8-TCDD was detected at analytical detection limits of 0.2 to 0.8 ppb, although total tetra isomers were detected in the Eglin and NCBC samples. Hepta CDD was detected in both JI and NCBC samples at less than 1 ppb and octa CDD was found at 1 to 3 ppb in all three baseline soil samples. The only CDF detected was for the total octa isomers in the NCBC sample. Arsenic was not detected in any sample at the normal detection limit of 1 ppm for the analytical method used.

The contaminated test soils contained approximately 0.1 to 0.2 weight percent Herbicide Orange based on the combined values for 2,4-D (including 2,4-DBE) and 2,4,5-T (including 2,4,5-TBE). Three phenols were detected, with 2,4,5-trichlorophenol the most significant at concentrations of about 50 ppm. The concentrations of 2,3,7,8-TCDD were approximately 100 ppb for JI and Eglin and 500 ppb for NCBC. These values were considered appropriate for conducting treatability tests since they represented the higher range of values determined in site surveys. No other CDDs were detected except hepta and octa isomers in the JI soil and octa isomers in Eglin. TCDF was detected above 1 ppb in all samples, and OCDF was detected in JI soil.

TABLE 5. ANALYTICAL METHODS USED FOR DETERMINATION OF HERBICIDES

Parameter	Sample preparation	Analytical method
2,4-DBE/2,4-D and 2,4,5-TBE/2,4,5-T	8150[a]	8150[a]
Chlorophenols	3540[a]	8040[a]
2,3,7,8-TCDD	EPA-CLP[b] and Acid/Soxhlet[c]	EPA-CLP[b]
Arsenic	3020[a]	7060[a]

[a] Test Methods for Evaluating Solid Wastes, SW-846, 2nd Edition, U.S. EPA, July 1982.

[b] Described in EPA-EMSL IFB Solication WA-84-A002. Detailed procedure given in Annex 5.

[c] Detailed procedure given in Annex 5.

TABLE 6. ANALYSIS OF BASELINE SOIL SAMPLES FOR COMPOUNDS OF INTEREST[a]

Compound	Concentration[b] (ppb)		
	JI	Eglin	NCBC
2,4-D	160	160	470
2,4,5-T	210	240	150
2,4-dichlorophenol	ND(1000)	ND(1000)	ND(1000)
2,4,6-trichlorophenol	ND(1000)	ND(1000)	ND(1000)
2,4,5-trichlorophenol	ND(1000)	ND(1000)	ND(1000)
Arsenic	ND(10,000)	ND(10,000)	ND(10,000)
2,3,7,8-TCDD	ND(0.5)	ND(0.8)	ND(0.2)
TCDD	ND(0.52)	0.31	0.13
TCDF	ND(0.17)	ND(0.52)	ND(0.11)
PCDD	ND(0.41)	ND(0.81)	ND(0.42)
PCDF	ND(0.33)	ND(0.96)	ND(0.76)
H_xCDD	ND(0.84)	ND(0.37)	ND(0.60)
H_xCDF	ND(0.21)	ND(0.27)	ND(0.18)
H_pCDD	0.63	ND(0.17)	0.73
H_pCDF	ND(1.0)	ND(1.0)	ND(1.0)
OCDD	1.73	2.6	2.0
OCDF	ND(0.11)	ND(0.06)	0.26

[a] Soils were analyzed after preparation according to protocol described in Section III (e.g., <2 mm, air dried).

[b] ND = not detected using the analytical procedure identified in Table 5. Detection levels given in parentheses.

TABLE 7. ANALYSIS OF CONTAMINATED SOIL SAMPLES FOR COMPOUNDS OF INTEREST[a]

Compound	Concentration[b] (ppb)		
	JI	Eglin	NCBC
2,4-D	900,000	1,200,000	370,000
2,4,5-T	890,000	1,700,000	710,000
2,4-dichlorophenol	1,900	2,400	1,100
2,4,6-trichlorophenol	1,100	ND	ND
2,4,5-trichlorophenol	38,000	20,000	53,000
Arsenic	ND(10,000)	ND(10,000)	ND(10,000)
2,3,7,8-TCDD	106[c]	101[c]	494[c]
TCDD	126	127	604
TCDF	6.6	2.1	2.3
PCDD	ND(1.09)	ND(1.5)	ND(0.48)
PCDF	ND(1.7)	ND(1.6)	ND(1.9)
H_xCDD	ND(3.7)	ND(1.2)	ND(0.59)
H_xCDF	ND(0.67)	ND(0.73)	ND(3.6)
H_pCDD	29.0	ND(0.72)	ND(1.5)
H_pCDF	ND(3.5)	ND(2.1)	ND(1.7)
OCDD	32.0	2.6	ND(2.4)
OCDF	1.3	ND(0.33)	ND(0.98)

[a] Soils were analyzed after preparation according to protocol (e.g., <2 mm, air dried).

[b] ND = not detected. Detection levels given in parentheses.

[c] Average of analyses of triplicate aliquots of prepared test soil. Values for other compounds are for a single aliquot.

Arsenic was not detected in any sample at the 1 ppm detection limit. No additional arsenic analyses were performed for any test samples.

Table 8 compares the relative concentrations of 2,3,7,8-TCDD, the three chlorophenols, and 2,4-DBE to the principal Herbicide Orange constituent, 2,4,5-TBE. These ratios can be evaluated relative to the original composition of Herbicide Orange reported in previous studies by the Air Force (Reference 2). The chlorophenols are present at approximately the same relative concentrations as the original material, whereas the 2,3,7,8-TCDD levels are approximately three orders of magnitude higher than levels determined in the original material. This could be a result of differences in volatilization rates and chemical decomposition occurring in the environment during the past decade.

TABLE 8. EVALUATION OF RELATIVE CONCENTRATIONS OF HERBICIDE CONSTITUENTS FOR CONTAMINATED TEST SOILS

Compound	Weight ratio of compound to 2,4,5-TBE		
	JI	Eglin	NCBC
2,4-DBE[a]	1.04	0.723	0.534
2,4,5-TBE[a]	1.00	1.00	1.00
2,4-dichlorophenol	1.75×10^{-3}	1.16×10^{-3}	1.27×10^{-3}
2,4,6-trichlorophenol	1.01×10^{-3}	b	b
2,4,5-trichlorophenol	3.5×10^{-2}	9.6×10^{-3}	6.12×10^{-2}
2,3,7,8-TCDD	9.76×10^{-5}	4.87×10^{-5}	5.70×10^{-4}

[a] 2,4-DBE = butyl ester of 2,4-D; 2,4,5-TBE = butyl ester of 2,4,5-T.

[b] 2,4,6-trichlorophenol was not detected for these samples.

SECTION III

LABORATORY TREATABILITY TESTS

A series of bench scale tests was performed to establish the effect of the key process variables, residence time and temperature, on the treatability (dioxin-removal efficiency) of the three test soils. After the soils were prepared by drying and screening as described in Section II, separate aliquots were placed in a small furnace for different time periods. The treated samples were analyzed for 2,3,7,8-TCDD and the results were evaluated to determine the relationship between treatment conditions and final concentration of 2,3,7,8-TCDD. These results were also compared to previous results in which Missouri soils were used. This section describes the experimental activities and test results.

A. EXPERIMENTAL PLAN

Experimental activities for this project were conducted at ITC's Environmental Research Laboratory in Knoxville, Tennessee. A specially designed "high-hazard" section of the laboratory permitted the dioxin-contaminated soils to be prepared, tested, and analyzed safely and efficiently. This entire section of the laboratory is designed for the safe handling and analysis of chemicals and samples having toxic or unknown properties. This laboratory has four isolated, negative-pressure cubicles that contain hoods and laboratory benches constructed of materials selected for easy decontamination. Total containment is assured, since all used water is collected and all air leaving the facility is filtered through HEPA and activated carbon filters on the high-velocity hoods. All dioxin-containing materials generated from test activities were packaged and stored for disposal.

The results developed from this study were obtained using a simple and consistent experimental method and apparatus designed to expose the study soils to a specified temperature for a specified time. Treatability was measured by comparing the analyses of 2,3,7,8-TCDD in the untreated and treated soil. The test equipment and procedures are described in this section. The features incorporated into the test system and procedures included (1) sufficient soil quantity to enable analytical sensitivity below 1 ppb and duplicate analysis as necessary, (2) a static but very thin layer of soil to minimize the potential effects of temperature gradients and gas-phase diffusion (in terms of both release of volatilized materials and exposure to the purge gas), and (3) quickly achieving and maintaining steady-state conditions. The experimental apparatus and procedures were the same as those used on previous treatability testing of Missouri soils. A QA/QC plan was prepared and followed during the testing.

B. THERMAL TREATMENT TEST EQUIPMENT

The principal test equipment was a Lindberg furnace, Model 51848, with an electronic temperature controller and 1600-watt heater system. The oven is double-shell construction with interior surfaces made of Moldatherm®, a molded aluminum-silicate insulation material. This oven is capable of operating up to 1100°C and has a relatively fast heat-up rate due to low mass. The interior space is approximately 10 cm wide by 11 cm high by 21 cm deep. A loose block (1.2 cm thick) of Moldatherm is placed on the bottom of the oven to provide additional separation between an object placed in the oven and the hot interior surface of the oven. A built-in thermocouple was used to control the interior oven temperature to the set point. The oven was placed inside a bench-top hood for all tests. Several modifications were made to the oven. Incoloy® (3/8 inch) tubing was inserted through the back wall and connected to an air cylinder to provide continuous purging of the interior space during each test. The purge gas was directed against the back wall to promote preheating and distribution. The purge gas flow rate, measured with a standard rotometer, was maintained at approximately 100 cc/minute, equivalent to about 5 percent turnover per minute. A separate thermocouple was used to measure the test temperature. This NBS traceable, Type K, sheathed thermocouple was placed approximately 3 centimeters above the soil surface at the center of the oven. The thermocouple, the temperature indicator, and the purge gas rotometer were calibrated before testing. A Keithley Model 871 Digital Temperature Indicator was used with both thermocouples.

A specially made tray was used to contain the soil within the oven. The tray, which weighed approximately 430 grams, was 9 cm wide by 3.2 cm high by 19.3 cm long and made of Incoloy to resist oxidation at the expected maximum test temperatures. A separate Incoloy lid was used to cover the tray when necessary. The soil was leveled within the tray to a fixed uniform thickness of approximately 2.5 mm. Figure 1 is a scale schematic drawing of the interior of the oven, with tray inserted.

C. STANDARD TEST PROCEDURE

The standard thermal desorption test consisted of the following steps:

1. Initiate a data log sheet (see Annex 8).

2. Weigh the empty, clean tray.

3. Transfer a representative aliquot (~30 grams) of prepared soil from the jar to the tray using a stainless steel scoop.

4. Weigh the soil and tray and adjust the soil quantity to achieve approximately 30 grams, which results in the approximate desired soil depth.

5. Distribute and level the soil within the tray to ~2.5 mm. Return excess to the sample jar.

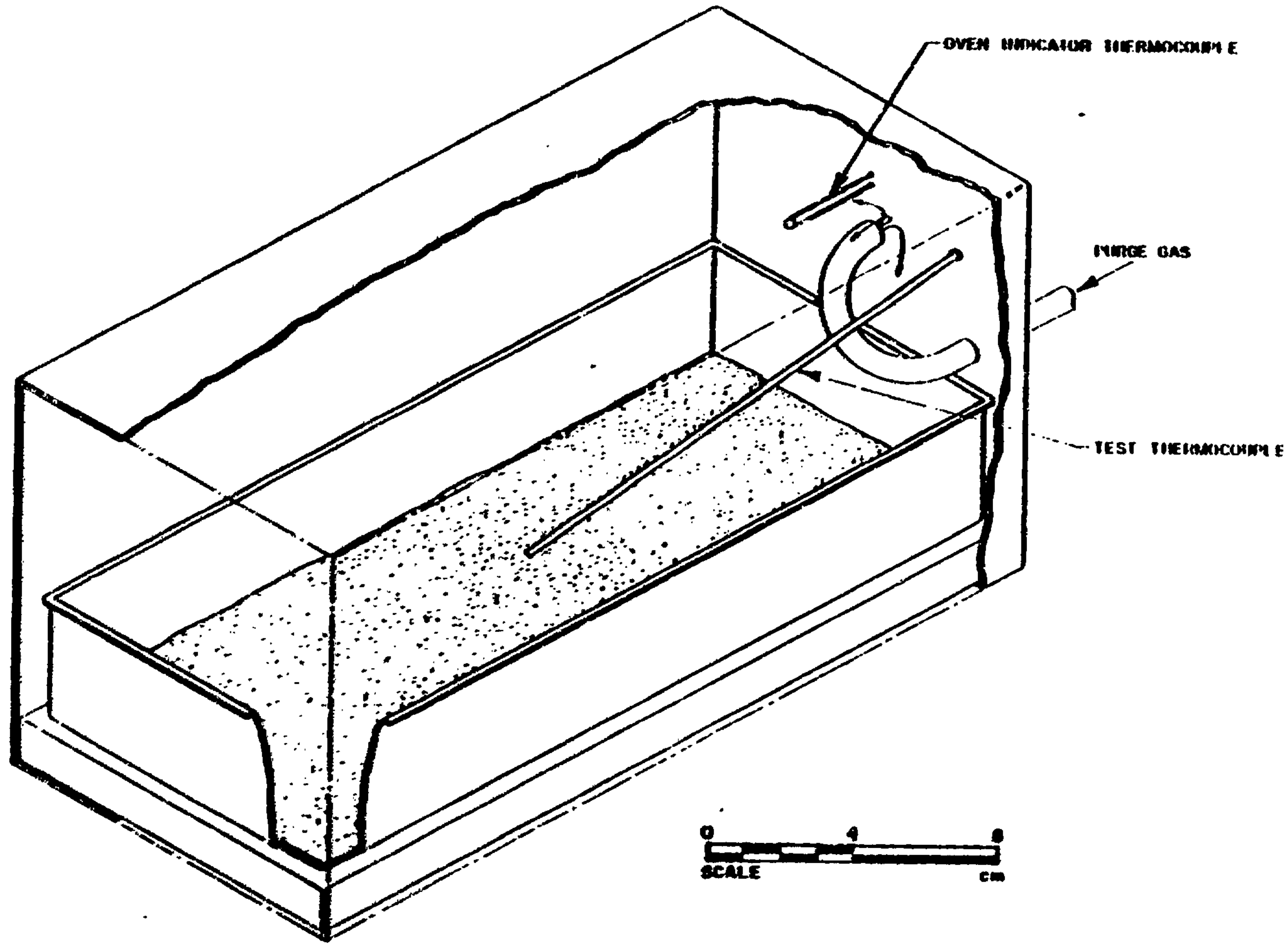

OVEN INDICATOR THERMOCOUPLE
PURGE GAS
TEST THERMOCOUPLE
0
4
8
SCALE
cm

Figure 1. Schematic of Oven Interior

6. Turn the purge gas flow on to the proper setting on the rotometer.

7. Place the tray with soil in the oven at ambient temperature and close the oven door.

8. Set the oven temperature controller set-point to the target test temperature and start the timer.

9. Monitor and record the times and temperatures, periodically, throughout the test period.

10. When the prescribed residence time at the target temperature is reached, shut off the oven heater and purge gas flow and open the oven door.

11. Cautiously withdraw the hot tray and soil using special tongs, place a cover on the tray, and place the covered tray in a separate hood for cooling for approximately 1 hour.

12. Close the oven door and set the temperature at 600°C for approximately 10 minutes. This step is to ensure that no residual materials from the sample remain in the oven.

13. Weigh the tray (without cover) plus treated soil.

14. Transfer an aliquot (typically about 10 grams) of treated soil from the tray to a tared, 250 cc, widemouth, amber bottle with Teflon-lined cap. Code, label, and submit this aliquot for 2,3,7,8-TCDD analysis. Transfer the remainder of the treated soil to an identical type bottle, label and store as a retainer.

15. Clean the tray, cover, and nondisposable implements using the following procedure:

    - Rinse with acetone and wipe clean
    - Scrub with detergent (Alconox®) solution and rinse with hot tap water followed by distilled water
    - Rinse with acetone and allow to dry
    - Rinse three times with methylene chloride (e.g., ~15-25 ml each rinse for the tray)
    - Air dry and store.

D. TREATABILITY TEST RESULTS AND DISCUSSION

A total of 16 separate thermal desorption tests were performed using three different test temperatures, ranging from about 430°C to 560°C, for times between 8 and 30 minutes. Two sets of duplicate tests were included for QA/QC purposes. All tests were performed using soil prepared as described previously, except for two tests which used oversize (>2 mm) pieces of JI and NCBC soil to investigate any differences in treatability due to gross differences in particle size. The test results are presented according to: (1) treatability or removal efficiency for the herbicide constituents and (2) effects of treatment on soil characteristics. Experimental data are summarized in Annex 8. Annex 7 presents all the analytical data while Annex 6 gives QA/QC results.

1. Treatability Data

Analysis and interpretation of the test results must consider the typical temperature profile (temperature vs residence time) for the small oven system used in testing. The soil samples were placed in the oven at ambient temperature, and the initial test period involved heating the oven and soil to the target test temperature. This unsteady state heat-up period lasted from about 5 to 9 minutes, with longer time corresponding to higher ultimate temperature. Figure 2 illustrates typical temperature profiles for the three target test temperatures. The target test time was initiated (at time zero) when the test temperature reached approximately the target test temperature.

Treatability or removal efficiency was determined by measuring the final concentration of 2,3,7,8-TCDD and comparing it to the initial concentration and the criteria or goal of 1 ppb. Test conditions (time and temperature combinations) were based on previous treatability tests to demonstrate the influence of these two parameters on treatment. Longer times were selected for certain tests using the NCBC soil which had considerably higher initial concentration than the other two test soil samples (i.e., ~500 ppb vs ~100 ppb). The residence times were considered representative of the normal operational capabilities of the MIS.

Table 9 summarizes the treatability test results, arranged by test temperature, soil type, and treatment time. The test number is listed for reference to data given in Appendix F. The results for each soil were plotted in Figures 3, 4, and 5 to show the effects of time and temperature. Data for all three soils at 481°C were plotted in Figure 6 to show the effect of soil type. The shape of the treatability curves in these figures was developed, in part, based on previous testing with Missouri soil which yielded more data points (times) for each particular target test temperature.

The data show that treatment of all three soils to less than 1 ppb 2,3,7,8-TCDD is feasible if temperatures of 500°C or more are achieved. The results are very similar to those obtained in previous tests with Missouri soils. Table 10 lists the calculated removal efficiencies for 2,3,7,8-TCDD. Nearly all tests above the minimum temperature resulted in at least 99 percent removal. The significant effect of temperature appears to be more pronounced for JI soil, which showed very low removal efficiency at the lowest temperature of 429°C. The coarse (>2 mm) particle size material had lower final 2,3,7,8-TCDD concentration than the prepared (<2 mm) soil used for most tests, despite the fact that analysis of an aliquot of coarse material from NCBC indicated a higher initial 2,3,7,8-TCDD concentration. Since there is considerable variation in the type of soil particles within

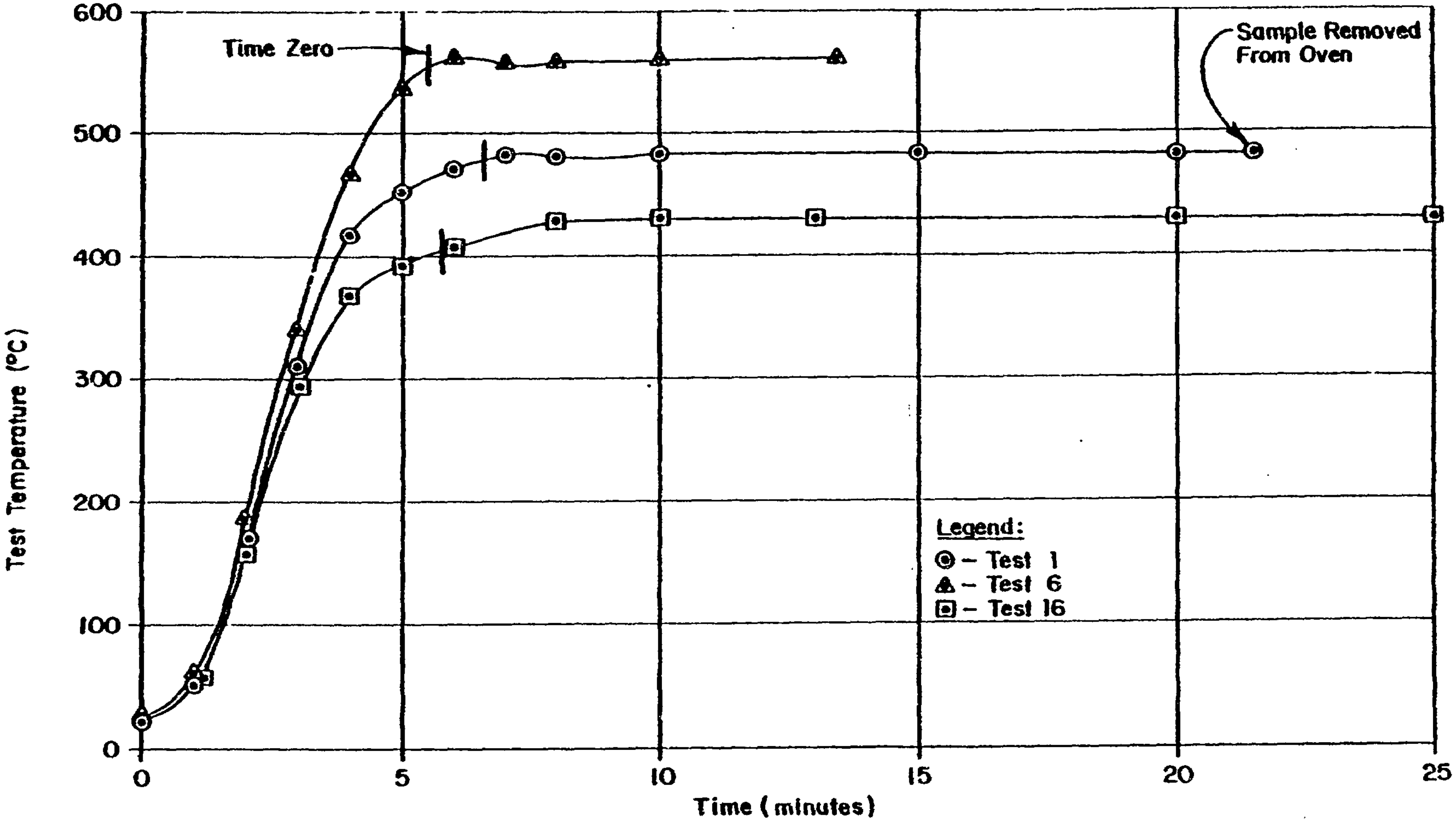


Figure 2. Typical Time-Temperature Profiles for Laboratory Desorption Tests

TABLE 9. SUMMARY OF TREATABILITY RESULTS - EFFECT OF TIME AND TEMPERATURE ON FINAL CONCENTRATION OF 2,3,7,8-TCDD

Nominal test temperature[a] (°C)	Soil identification	Time at test temperature[b] (min)	Final 2,3,7,8-TCDD concentration (ppb)	Test number
430	JI	20	38.5	15
	Eglin	20	4.4	16
	NCBC	30	27.4[c] 25.7[c]	14
481	JI	15	4.5	1
	JI	30	1.6	5
	JI (>2 mm)	30	0.44	12
	Eglin	15[d]	1.3	2[d]
	Eglin	15[d]	0.8	4[d]
	Eglin	30	0.45	3
	NCBC	15	10.1	9
	NCBC	30	4.6	10
	NCBC (>2 mm)	30	0.74	11
558	JI	8	0.81[c]	6
	JI	c	0.31[c]	c
	Eglin	8	0.71	7
	NCBC	15[d]	0.99	8[d]
	NCBC	15[d]	0.53	13[d]

---

[a] Refer to time-temperature data (Annex 8) for exact temperatures during entire test.

[b] This period begins at "zero time" when the target test temperature is reached; zero time is actually 5 to 9 minutes after start of heat-up.

[c] Analytical duplicate; separate aliquots of treated soil were analyzed.

[d] Experimental duplicates.

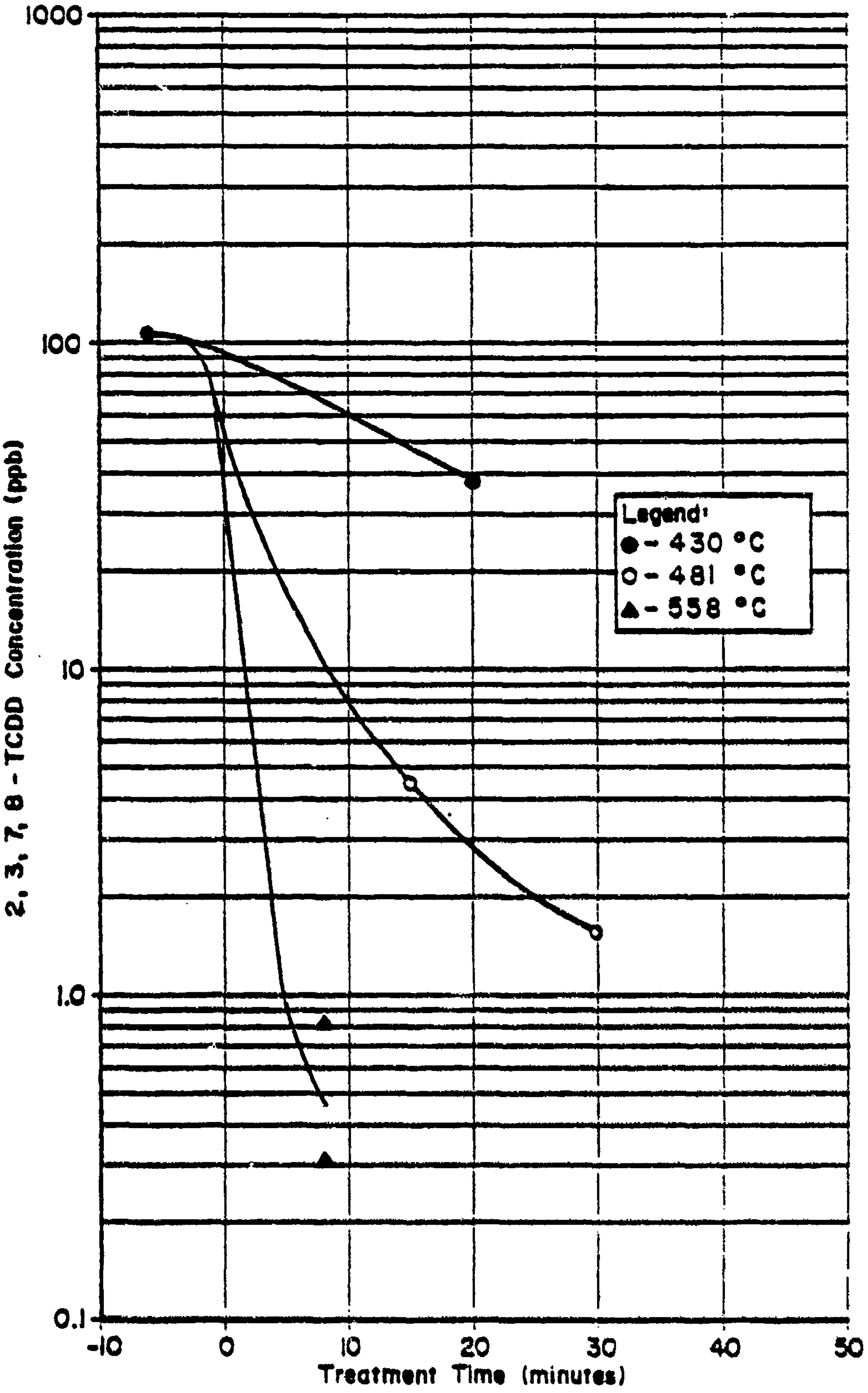


Figure 3. Effect of Time and Temperature on Removal of 2,3,7,8,-TCDD from JI Soil

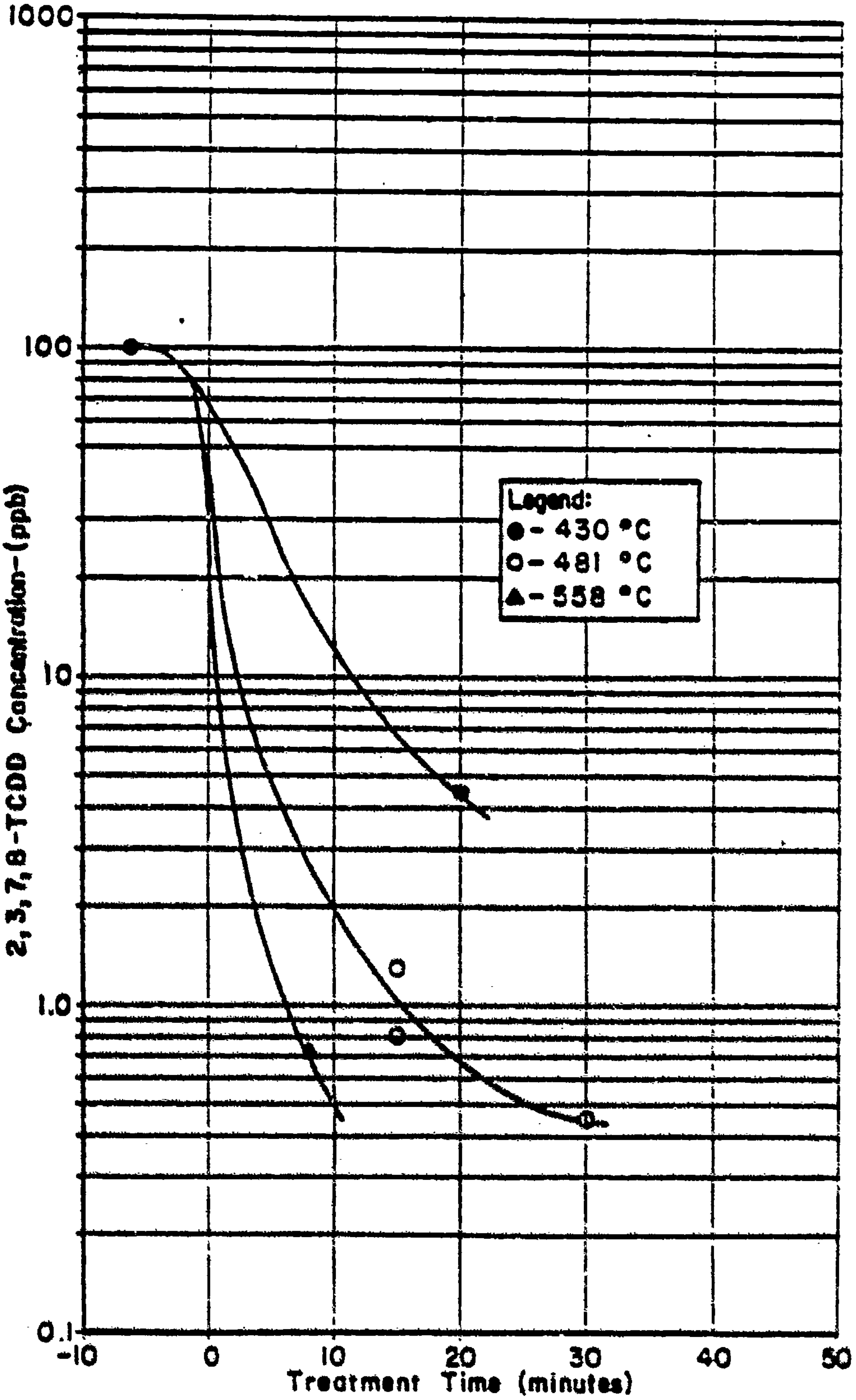


Figure 4. Effect of Time and Temperature on Removal of 2,3,7,8-TCDD from Eglin Soil

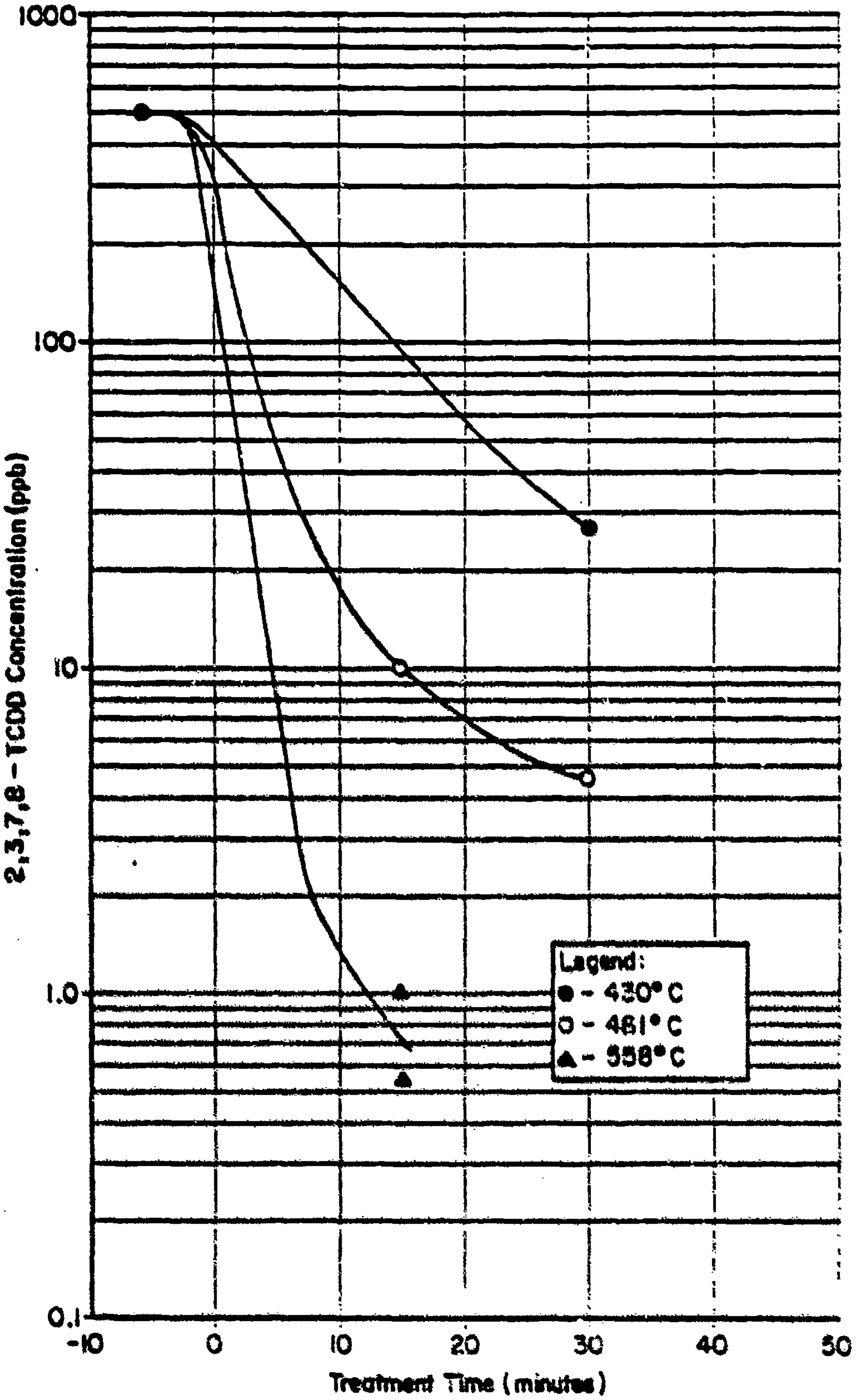


Figure 5. Effect of Time and Temperature on Removal of 2,3,7,8-TCDD from NCBC Soil

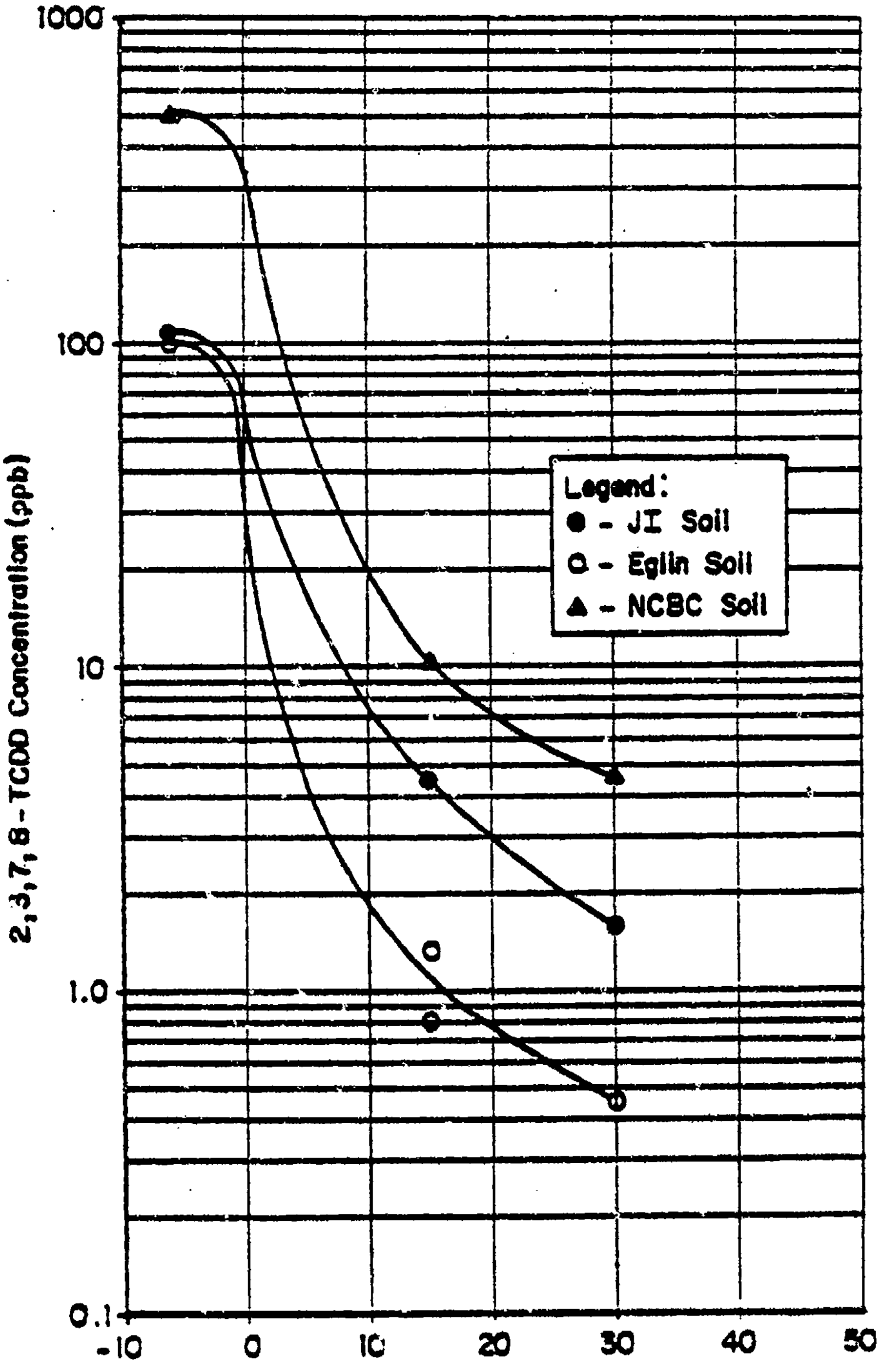


Figure 6. Effect of Treatment Time at 481°C on Removal of 2,3,7,8-TCDD

TABLE 10. CALCULATED REMOVAL EFFICIENCIES FOR 2,3,7,8-TCDD

Test number	Initial 2,3,7,8-TCDD concentration (ppb)	Final 2,3,7,8-TCDD concentration (ppb)	Removal efficiency[a] (%)
15	106	38.5	65.51
16	101	4.4	95.72
14	494	26.6[b]	94.75
1	106	4.5	96.00
5	106	1.6	98.58
12[c]	106	0.44	99.61
2[d]	101	1.3	98.74
4[d]	101	0.8	99.22
3	101	0.45	99.56
9	494	10.1	98.01
10	494	4.6	99.09
11[d]	148	0.74	99.51
6	106	0.56[b]	99.29
7	101	0.71	99.31
8[d]	494	0.99	99.81
13[d]	494	0.53	99.90

[a] Removal Efficiency =

$$100 \quad 1- \left[\frac{\text{(Final conc. X treated sample weight)}}{\text{(Initial conc. X untreated sample weight)}}\right].$$

[b] Average of analytical duplicates.

[c] Test soil was >2 mm fraction.

[d] Experimental duplicates.

the NCBC test soil sample, it is possible that the actual aliquot of oversize material used in the test had a much lower initial concentration. On the other hand, these results may be due to the distribution or location of herbicide on/within the soil matrix. Coarse material, being comprised more of impervious gravel and shells, may contain predominantly surficial deposits of herbicide, which vaporize more readily; heat and mass transfer are not expected to significantly affect desorption rates for surficial deposits.

For the tests performed at 558°C which gave final 2,3,7,8-TCDD values less than 1 ppb, analysis of the treated soil was also done for the herbicide constituents identified in the untreated test soil (see Section II). Table 11 lists these results. For all soils, the residual concentration of 2,4-D was nondetectable at less than 50 ppb detection level and 2,4,5-T was detected at less than 20 ppb. This represents a removal efficiency of greater than 99.99 percent as given in Table 12. The residual concentrations of the three chlorophenols could not be determined below the normal detection level (1 ppm) by the standard analytical protocol used.

Residual concentrations of all CDD and CDF cogeners above tetra were non-detectable at levels typically less than 0.5 ppb except for the JI samples. The hepta and octa CDD contained in the untreated JI soil were effectively removed, although a residual concentration of octa CDF represented 25 percent of the starting concentration. The detection levels of the thermally treated samples were lower by a factor of 2 to 8 times, apparently because of the absence of other chemical compounds and the reduced interaction of the CDDs and CDFs with the soil matrix (Section II). The residual concentration of TCDF compared to the untreated soil indicates a removal efficiency of only about 90 percent for JI soil and 80 percent for NCBC, although final concentrations were less than 0.7 ppb for all soils. This is contrasted to the removal efficiencies for 2,3,7,8-TCDD of greater than 99 percent.

2. Physical Effects of Treatment

Visual inspection and weight loss measurements were conducted to understand the changes in the soil matrix caused by the exposure to high temperatures. DTA analysis of each soil was discussed in Section II. The test temperatures used in the treatability study were lower than the DTA range and lower than the temperatures achieved in the batch pilot kiln tests. Therefore, transformations observed during the treatability tests are not fully indicative of expected behavior in the MIS.

Table 13 presents the data on the total weight loss which occurred as a result of thermal treatment, and compares this loss with the theoretical loss attributed to initial "free moisture." The final column of values represents the weight loss unaccounted for by initial moisture and is assumed to be a result of combined thermochemical transitions of the mineral and organic matter in each soil matrix. The data are arranged by soil type and in order of increasing temperature and time. All three soils show some increase in unaccounted-for weight loss as the temperature increases. This

TABLE 11. ANALYSIS OF TEST SOIL SAMPLES TREATED AT 558°C FOR COMPOUNDS OF INTEREST

Compound	Concentration[a] (ppb)		
	JI[b]	Eglin[b]	NCBC[c]
2,4-D	ND(47)	ND(12)	ND(31)
2,4,5-T	16	0.8	3
2,4-dichlorophenol	ND(1000)	ND(1000)	ND(1000)
2,4,6-trichlorophenol	ND(1000)	ND(1000)	ND(1000)
2,4,5-trichlorophenol	ND(1000)	ND(1000)	ND(1000)
Arsenic	NA[d]	NA[d]	NA[d]
2,3,7,8-TCDD	0.81/0.31[e]	0.71	0.99/0.55[f]
TCDD	0.27	0.42	1.6
TCDF	0.63	0.40	ND(0.26)
PCDD	ND(0.35)	ND(0.29)	ND(0.30)
PCDF	ND(0.44)	ND(0.39)	ND(0.79)
H_xCDD	ND(0.40)	ND(0.44)	ND(0.41)
H_xCDF	ND(0.82)	ND(0.31)	ND(0.41)
H_pCDD	ND(0.20)	ND(0.34)	ND(0.30)
H_pCDF	ND(0.52)	ND(0.78)	ND(0.49)
OCDD	ND(0.34)	ND(0.68)	ND(0.49)
OCDF	0.30	ND(0.40)	ND(0.38)

[a] ND = not detected using the analytical procedure identified in Section II. Detection levels are given in parentheses.

[b] Time at 550°C = 8 minutes.

[c] Time at 550°C = 15 minutes.

[d] Arsenic not analyzed on treated soils since it was not detected in initial test soils.

[e] Analytical duplicates.

[f] Experimental duplicates.

TABLE 12. CALCULATED REMOVAL EFFICIENCIES FOR 2,4-DBE AND 2,4,5-TBE AFTER TREATMENT AT 558°C

Test number	Soil identification	Removal efficiency[a] (%)	
		2,4-DBE	2,4,5-TBE
6	JI[b]	>99.995[c]	99.9982
7	Eglin[b]	>99.999[c]	99.9999
8	NCBC[d]	>99.991[c]	99.9996

[a] Removal Efficiency = 100 $1 - \left[\frac{\text{(Final conc. X treated sample weight)}}{\text{(Initial conc. X untreated sample weight)}}\right]$.

[b] Time of treatment = 8 minutes.

[c] Efficiency calculated based on detection level for the specific analysis.

[d] Time of treatment = 15 minutes.

TABLE 13. SUMMARY OF WEIGHT LOSS DATA FROM LABORATORY TREATABILITY TESTS

Test number	Soil identification	Nominal test temperature (°C)	Test time (min.)	Initial sample weight[a] (grams)	Final sample weight (grams)	Weight loss (grams)	Total weight loss (%)	Initial moisture (%)	Total weight loss less moisture loss (%)
15	JI	431	15	29.93	28.42	1.51	5.05	2.31	2.74
1	JI	481	15	30.80	29.00	1.80	5.84	2.31	3.53
5	JI	480	30	34.21	32.22	1.99	5.82	2.31	3.51
12	JI (>2 mm)	482	30	36.90	34.83	2.07	5.61	(2.31)[b]	3.30
6	JI	558	8	36.13	33.72	2.41	6.67	2.31	4.36
16	Eglin	431	15	30.71	30.16	0.55	1.79	0.79	1.00
2[b]	Eglin	481	15	31.89	31.32	0.57	1.79	0.79	1.00[c]
4[b]	Eglin	481	15	32.83	32.25	0.58	1.77	0.79	0.98[c]
3	Eglin	483	30	36.62	35.88	0.74	2.02	0.79	1.23
7	Eglin	558	8	31.04	30.37	0.67	2.16	0.79	1.37
14	NCBC	433	20	29.49	28.75	0.74	2.51	1.07	1.44
9	NCBC	483	15	31.38	30.54	0.84	2.68	1.07	1.61
10	NCBC	482	30	31.56	30.76	0.80	2.53	1.07	1.46
11	NCBC (>2 mm)	481	30	41.19	40.29	0.90	2.19	(1.07)[b]	1.12
8	NCBC	556	15	30.63	29.65	0.98	3.20	1.07	2.13
13	NCBC	560	8	30.19	29.26	0.93	3.08	1.07	2.01

[a] Initial moisture of air dried, screened test soil. Moisture of soil as received was higher.

[b] Moisture was not determined specifically for the coarse (>2 mm) fraction.

[c] Experimental duplicates.

weight loss, which is highest for JI soil, was expected, based on the DTA results. The weight loss for JI soil would be significantly higher at temperatures above those used for the treatability tests (see Section IV).

The visual changes which occur to constituents of soil exposed to high temperatures can indicate irreversible physical and chemical transitions which may influence processing characteristics in the MIS. For example, upon drying a moist clay can fragment, producing many very fine clay particles which show greatly different behavior in the kiln. Attrition and fusion or slagging are important changes that can be demonstrated in bench- and pilot-scale devices. Combustion or pyrolysis (charring) of organic matter can usually be detected through color changes. Color change may also indicate a mineralogical transition.

Inspection of the three test soils before and after the thermal desorption tests resulted in several observations. The JI soil became darker (gray) in color and had evidence of small black particles. The NCBC and Eglin soils changed from brown to reddish brown. No significant difference in particle size was noticed. Color photographs were taken of the soil samples before and after treatment and are on file at IT Corporation.

SECTION IV

BATCH KILN PILOT TESTS

A. EXPERIMENTAL PROCEDURES AND EQUIPMENT

Pilot-scale batch kiln tests were performed on uncontaminated samples from the three sites. Testing was conducted by Allis-Chalmers during the period of January 9-11, 1985 at its test center in Oak Creek, Wisconsin. The test unit and procedures have been used by Allis-Chalmers for studying the processing of many different materials other than soil. The test conditions were specified by ITC and the tests were witnessed by an ITC technical representative.

Tests were conducted in a 14-inch diameter batch kiln which is shown sche-matically in Figure 7. A photograph of the pilot kiln system (being used for a different project) is shown in Figure 8. A total of 11 tests were conducted, using the three soils. The test conditions are summarized in Table 14. Sample numbers used by Allis-Chalmers to identify the three soils are given for references. The soil samples used in the tests were not sieved; the particle size range was representative of the composite drum of soil as received.

All tests were performed so that the final kiln temperature was approximately 1038°C (1900°F) and the initial kiln loadings were 7 percent by volume. All tests were made with a kiln rotational speed of 2.8 rpm. These values were selected to be representative of the MIS; the temperature and loading are approximate upper limits of the operating range. Likewise, the ranges of total gas flow rates were chosen to reflect the ranges of superficial gas velocities that are used in operation of the MIS at 50 percent excess air (10 percent excess oxygen) in the kiln. Three gas flow rates were selected for each soil. In addition, one test using Eglin soil was performed at a lower gas flow rate to allow comparison with previous test data from Missouri soils. Another test using JI soil was made using an intermediate gas flow rate to further define the relationship between entrainment and gas flow rate. Higher rates of gas flow were not possible due to limitations of the batch kiln.

Each test was run for 60 minutes. The kiln was brought up to temperature and the initial gas flow rates were set before charging the kiln with a preweighed quantity of soil. The soil charges were placed into the kiln through the exit gas port, using a shovel made for this purpose. The kiln is heated using natural gas and air and/or oxygen to satisfy combustion parameters. The relative feed rates of these three gases were varied during each run to reach and maintain the desired operating temperature while keeping the total gas flow rate constant throughout the run. The gas feed rates given in Table 14 represent the equilibrium rates achieved after reaching steady-state.

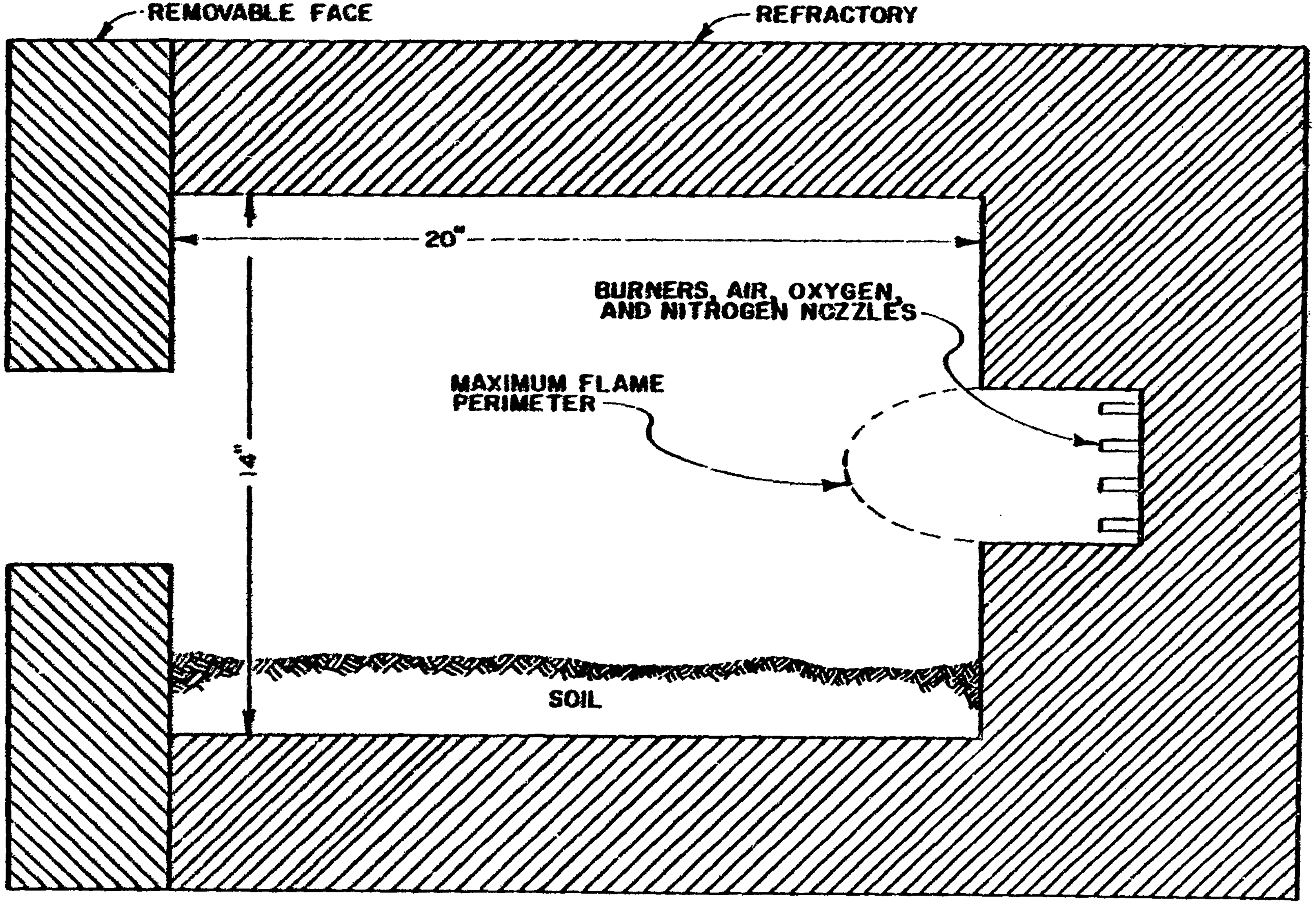

REMOVABLE FACE
REFRACTORY
20"
14"
BURNERS, AIR, OXYGEN, AND NITROGEN NOZZLES
MAXIMUM FLAME PERIMETER
SOIL

Figure 7. Schematic Diagram of Pilot Batch Kiln

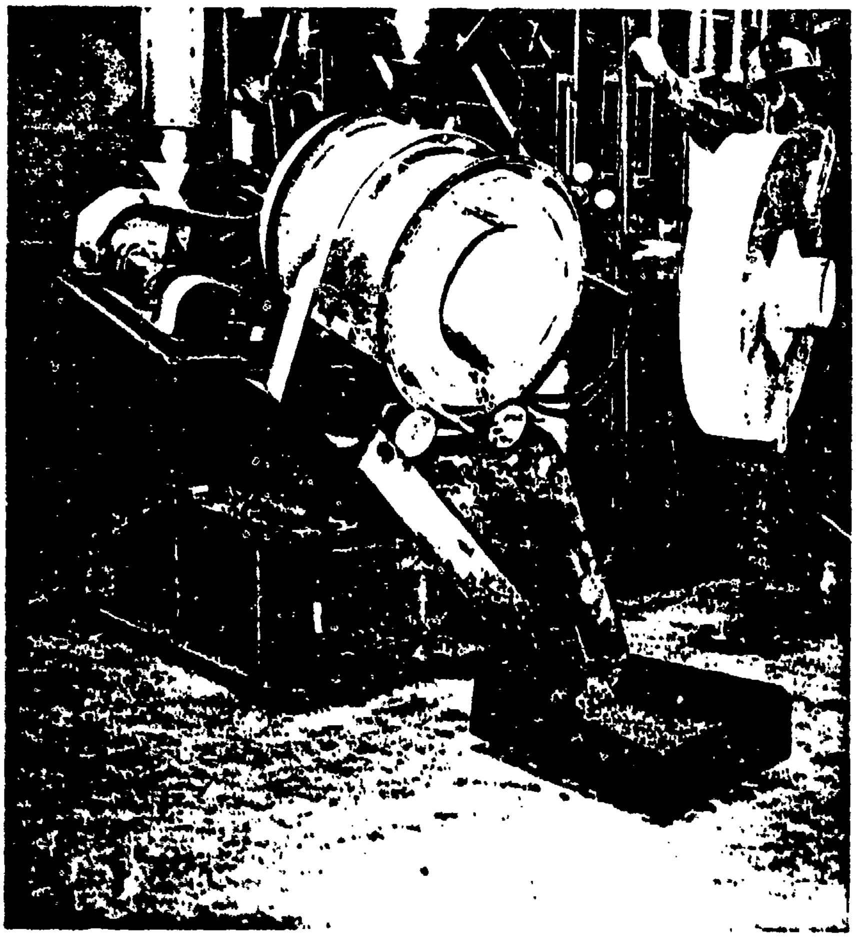


Figure 8. Photograph of the Allis-Chalmers Pilot Kiln System

TABLE 14. TEST CONDITIONS - BATCH KILN TESTING

Test number	Soil identification	Soil code number	Feed rates (SCFM)				Exit gas flow rate (ACFM)	Charge weight (kg)	Final temperatures (°C)	
			Air	Natural	Oxygen	Total			Gas	Soil
1	JI	4761	28.1	1.7	2.2	32	150	5.44	1041	1020
2[a]	JI	4761	39.1	2.8	1.6	45	200	5.44	1040	1039
3	Eglin	4760	27.3	2.4	2.3	32	150	4.75	1038	1028
4	JI	4761	91.0	6.0	2.9	100	450	5.44	1041	1029
5	Eglin	4760	58.5	4.4	3.1	66	300	4.75	1040	1021
6	Eglin	4760	91.0	6.0	2.9	100	450	4.75	1040	1021
7	JI	4761	59.0	4.4	3.1	66	300	5.44	1039	1035
8	NCBC	4768	29.0	1.8	2.0	32[b]	150[b]	4.68	1038	1014
9	NCBC	4768	59.2	4.2	2.6	66	300	4.68	1038	1024
10	NCBC	4768	92.8	5.8	2.3	100	450	4.68	1040	1037
11	Eglin	4760	20.9	2.0	2.1	25	110	4.75	1038	1033

[a] Test 2 is labeled as 4761-2 in Annex 4.

[b] Power failure; total gas flow varied from 32 to 24 scfm.

The gas and soil temperatures were taken by means of two stationary thermocouples inserted through the exit gas port of the kiln. Temperatures were recorded on a continuous chart recorder.

Particles escaping from the gas exit port were collected on three metal trays placed on the floor and weighed after each test. Particle size determinations were also performed on the collected material. For tests with the highest gas velocity, some entrained solids fell outside the area covered by the trays. When this occurred, the floor was swept after the test and the sweepings kept separate from the contents of the trays. Some entrained particles may have escaped through the ventilation hood above the gas exit port. This loss is considered to be negligible, based on observations made during the tests.

During Tests 3 and 5, the kiln was observed to leak at the beginning of each test. This leakage was stopped after several minutes. The spilled material from Test 5 was collected at 8 minutes into the test and found to weigh 0.66 kg. The material was allowed to remain in the tray during Test 3.

During Test 8, a power outage interfered with operation of the compressor that supplied combustion air to the kiln. The reduced air pressure did not provide the desired air flow. Therefore, this test was performed at a total gas feed rate of 32 scfm for 40 minutes and then at 24 scfm for the final 20 minutes of the test.

B. TEST RESULTS AND DISCUSSION

1. Visual Observations

During batch kiln testing, visual observations were made to assess the characteristics of the soils during processing. JI soil was observed to break apart during the run. The greatest apparent attrition occurred after 15 to 30 minutes. Bed temperatures during the maximum attrition period were about 850°C (1562°F). This behavior is considered to be a result of physical changes occurring during calcination. The color changed from light gray to white.

Eglin soil showed little visual change during the tests other than a slight color change. Small pieces of organic matter were observed to burn, but these particles amounted to only a very small percentage of the soil.

The NCBC soil was observed to have significant organic "burnout" during the tests. Burning was observed both from fine organic matter distributed evenly within the soil as well as a number of larger pieces such as small sticks. After heating in the kiln, this soil had a distinctly different appearance, changing from what appeared to be a relatively homogeneous mass of fine material with some shells intermixed to a mixture of shell, sand, and small gravel. Treated soil appeared to have a much higher percentage of shell than was apparent from inspecting the untreated sample, possibly as a result of attrition of nonshell material or burn-off of superficial material.

In contrast to previous testing on Missouri soil, these three test soils were observed to have significantly greater entrainment. This was apparent from the visible presence of particles in the air-stream exiting the kiln. Although most of the material was collected, some was observed as being in the air that entered the fume hood positioned over the exit gas port. Higher superficial gas velocities were used for these tests than for previous tests. Entrainment is discussed more fully below.

2. Test Data

The 11 test runs were performed at constant total gas feed rates. The relative rates of air, oxygen, and natural gas supplied to the kiln were adjusted throughout the tests to maintain the desired test conditions of 1038°C (1900°F) and 10 percent excess oxygen (~50 percent excess air). Data sheets given in Appendix B show the actual gas feed rates for each test, along with recorded temperatures of the gas and soil bed.

Feed moisture contents given in the data sheets represent the soil moisture content of the samples as received. Previous tests with Missouri soil demonstrated the influence of moisture content on heat-up rates, attrition and entrainment; the influence of moisture content was not studied during this test work.

a. Weight Loss Data

The data sheets in Annex 4 give the specific weights of product remaining in the kiln, exhaust dust collected in trays positioned under the exit air port of the kiln, and floor sweepings taken from around the trays. The floor sweepings were taken since entrained particles were observed to fall outside the area covered by the trays. The weight of entrained dust for each test was taken to be a combination of collected exhaust dust and floor sweepings. Charge weights were determined based on the bulk density of the soil and were calculated to be equivalent to a volume of soil equal to 7 percent of the volume of the batch kiln. Table 15 compares the total product-plus-entrainment weight with the charge weight and demonstrates the effect of differences in the thermal stability and composition of the three soils. All tests with the JI soil were observed to have a significant loss between the charge weight and the total product weight. Some of this loss is attributable to loss of moisture, but the greatest loss is attributed to calcination. In comparison, the Eglin soil had only a small weight loss that can be accounted for by loss of moisture and organic burnout. The Gulfport soil experienced a weight loss intermediate between the other two soils which was attributed to a combination of moisture loss, organic burnout, and calcination of the many shells observed in the soil sample. These results are in agreement with the DSC/TGA results reported in Section II.

Bulk densities for the three soils as received are given in Table 16, along with the densities of the kiln products after the tests. It is apparent that the JI soil underwent a significant decrease in density during the tests. This is attributed in part to the weight loss caused by calcination (conversion of calcium carbonate to calcium oxide). The density of

TABLE 15. WEIGHT LOSS DURING PILOT KILN TESTS

Soil identification	Weight (kg)					Weight loss[b] (%)	Test No.
	Initial charge	Product	Entrainment	Total collected[a]	Unaccounted for		
JI	5.44	2.43	0.23	2.66	2.78	51.1	1
JI	5.44	2.13	0.40	2.53	2.91	53.5	2
JI	5.44	2.00	0.51	2.51	2.93	53.9	7
JI	5.44	1.77	0.69	2.46	2.98	54.8	4
Eglin	4.75	3.73	0.89	4.62	0.13	2.7	11
Eglin	4.75	3.03	1.40	4.43	0.32	6.7	3
Eglin	4.75	3.23	1.08	4.31	0.44	9.3	5
Elgin	4.75	2.84	1.45	4.29	0.46	9.7	6
NCRC	4.68	3.36	0.14	3.50	1.18	25.2	8
NCRC	4.68	3.00	0.57	3.57	1.11	23.7	9
NCRC	4.68	2.60	0.94	3.54	1.14	24.4	10

[a] Includes floor sweepings, does not include any fine particulates which may have been drawn into exhaust hood above gas exit port

[b] Weight loss is assumed to be equivalent to unaccounted for material.

TABLE 16. SOIL BULK DENSITIES BEFORE AND AFTER KILN TESTS

Sample or test Number	Soil description	Density (lb/ft^3)
Sample 4760	Eglin As Received	80.04
Sample 4761	JI As Received	85.98
Sample 4768	NCBC As Received	78.92
Test 1	JI/After Test	55.56
Test 2	JI/After Test	54.31
Test 3	Eglin/After Test	94.28
Test 4	JI/After Test	51.19
Test 5	Eglin/After Test	92.40
Test 6	Eglin/After Test	92.40
Test 7	JI/After Test	47.45
Test 8	NCBC/After Test	84.29
Test 9	NCBC/After Test	87.41
Test 10	NCBC/After Test	118.63
Test 11	Elgin/After Test	93.03

Eglin soil appeared to increase slightly during the test. The density of NCBC soil increased slightly in two tests and increased a moderate amount in the third test.

b. Particle Entrainment Data

Based on the total combustion gas flow through the pilot kiln given in Table 14, the average superficial velocity within the kiln was calculated. These values are given in Table 17 and compared to the relative weight of entrained soil. Figure 9 shows the relationship between entrainment and kiln gas superficial velocity for the three soils. Data were taken at gas superficial velocities representative of MIS operation at 50 percent excess air. One test for Eglin soil at 2.3 feet per second superficial velocity was observed to have some leakage of soil from the front of the batch kiln. This accounts for the high value for that data point.

Entrainment of NCBC soil appears to have the greatest dependence on velocity while Eglin soil has the least. Eglin soil had high entrainment for the entire range of superficial velocities, while the other soils had lower (less than 10 percent of total charge after 60 minutes) entrainment, particularly at low gas velocities.

During the batch kiln tests, the soil bed was watched through the 5-inch diameter exit gas port. The nature of the air currents in the kiln was noted in all of the tests and could be observed particularly well during periods when flames were present in the kiln as the result of organic burnout. The air currents appeared to be turbulent in all of the tests, although tests at lower gas velocities (1.8 and 2.3 feet per second) had significantly slower "swirling" of flames.

The entrainment process appeared to be influenced by kiln rotation and flow characteristics of the particular soil. As particles were rolled over and subjected to the gas flow, they were caught by swirling currents (eddies) near the soil surface. The eddies appeared to lift soil particles into the exit airstream.

A direct relationship between entrainment in the pilot kiln and the MIS cannot be made, so the values given in Figure 9 are not representative of the MIS, nor can simple correlations be used to predict entrainment rate. A limited study of entrainment theory was carried out as part of this project to understand the implications of the batch kiln results. Although the literature reports empirical relationships which are useful for evaluating simple flow situations, the accurate modeling of a direct-fired rotary kiln processing a diverse range of solids requires considerable additional technical study and testing. A brief summary of literature findings is presented in Annex 9.

c. Particle Size

Screen analyses were run on the soil samples as received, and for each test on kiln products and entrained dust collected on the trays. These data

TABLE 17. SUMMARY OF SOIL ENTRAINMENT RESULTS DURING BATCH KILN TESTS

Soil identification	Superficial gas velocity in kiln (ft/sec.)	Entrainment Weight (kg)	Entrainment % of charge	Test No.
JI	2.34	0.23	4.2	1
JI	3.12	0.40	7.4	2
JI	4.67	0.51	9.4	7
JI	7.01	0.69	12.7	4
Eglin	1.71	0.89	18.7	11
Eglin	2.34	1.40	29.5	3
Eglin	4.67	1.08	22.7	5
Eglin	7.01	1.45	30.5	6
NCBC	2.34	0.14	3.0	8
NCBC	4.67	0.57	12.2	9
NCBC	7.01	0.94	20.1	10

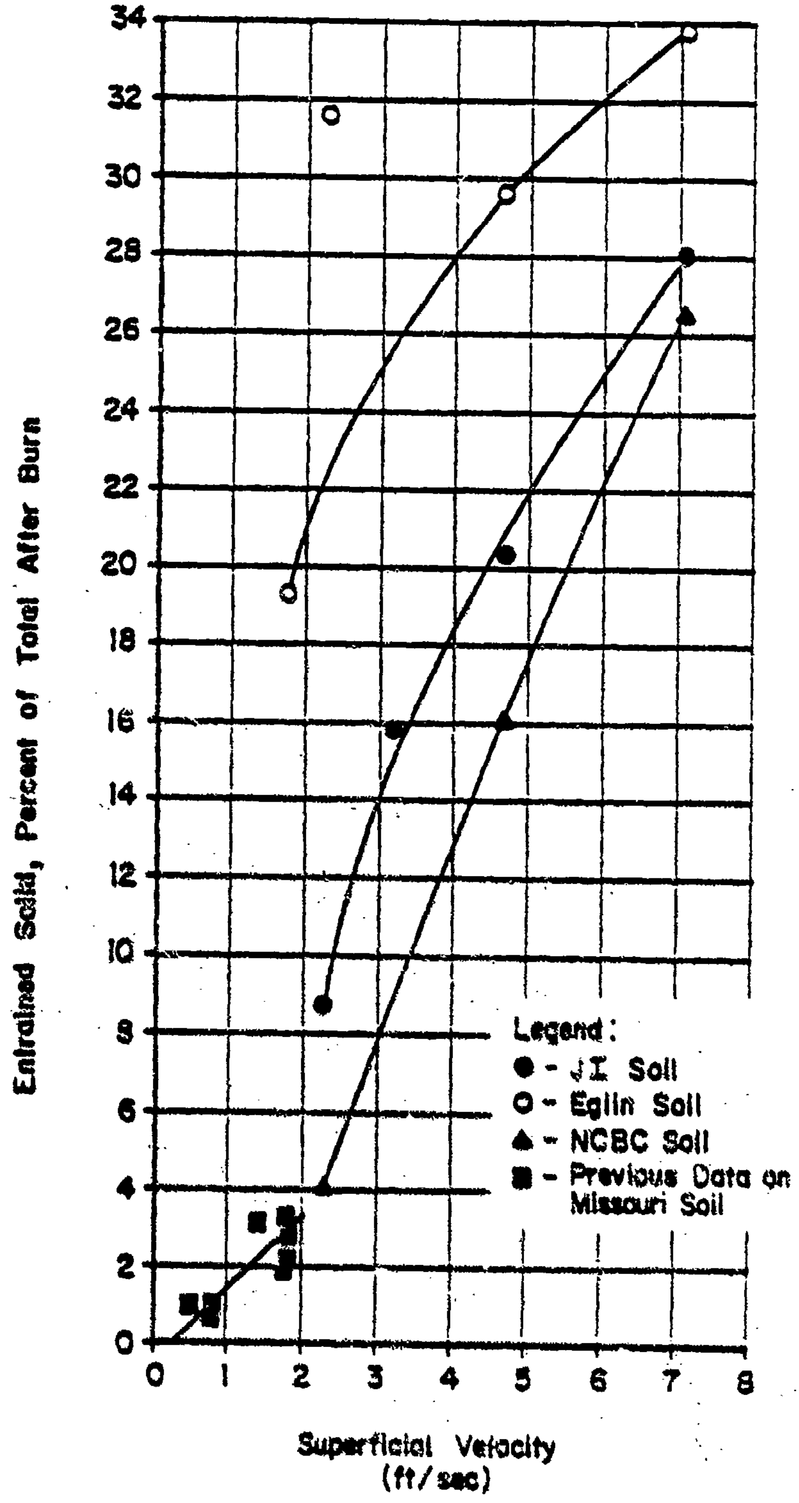


Figure 9. Comparison of Entrainment with Superficial Gas Velocity in Batch Pilot Kiln

are presented in Annex 4. All screen fractions are given as a percentage of the initial sample weight. Both Tyler and ASTM micron sieve sizes are given.

The results of selected screen analyses are summarized in Figures 10, 11, and 12. Each figure gives the particle size distribution of the soil fractions that apply to the test conducted on each soil with a gas superficial velocity of 7.1 feet per second. This superficial velocity was selected since it produced the maximum entrainment during the tests, giving a larger sample of exhaust dust for screen analyses. The figures plot the screen size versus the weight percent of the total sample retained on that sieve. The total of the kiln product and exhaust dust equals 100 percent of the total soil recovered after the test.

Figure 10 gives the results from Test 4 with JI soil, showing a shift in the particle size representing attrition during the test. It is also apparent that the smaller particles were selectively entrained.

Figure 11 gives the results from Test 6 with Eglin soil. The screen analysis of the sample as received reveals that the soil has a fairly uniform size of about 28 mesh. No separation of finer particles from coarser particles was observed in the exhaust dust and kiln product. The size distribution after batch testing closely matched that of the sample as received.

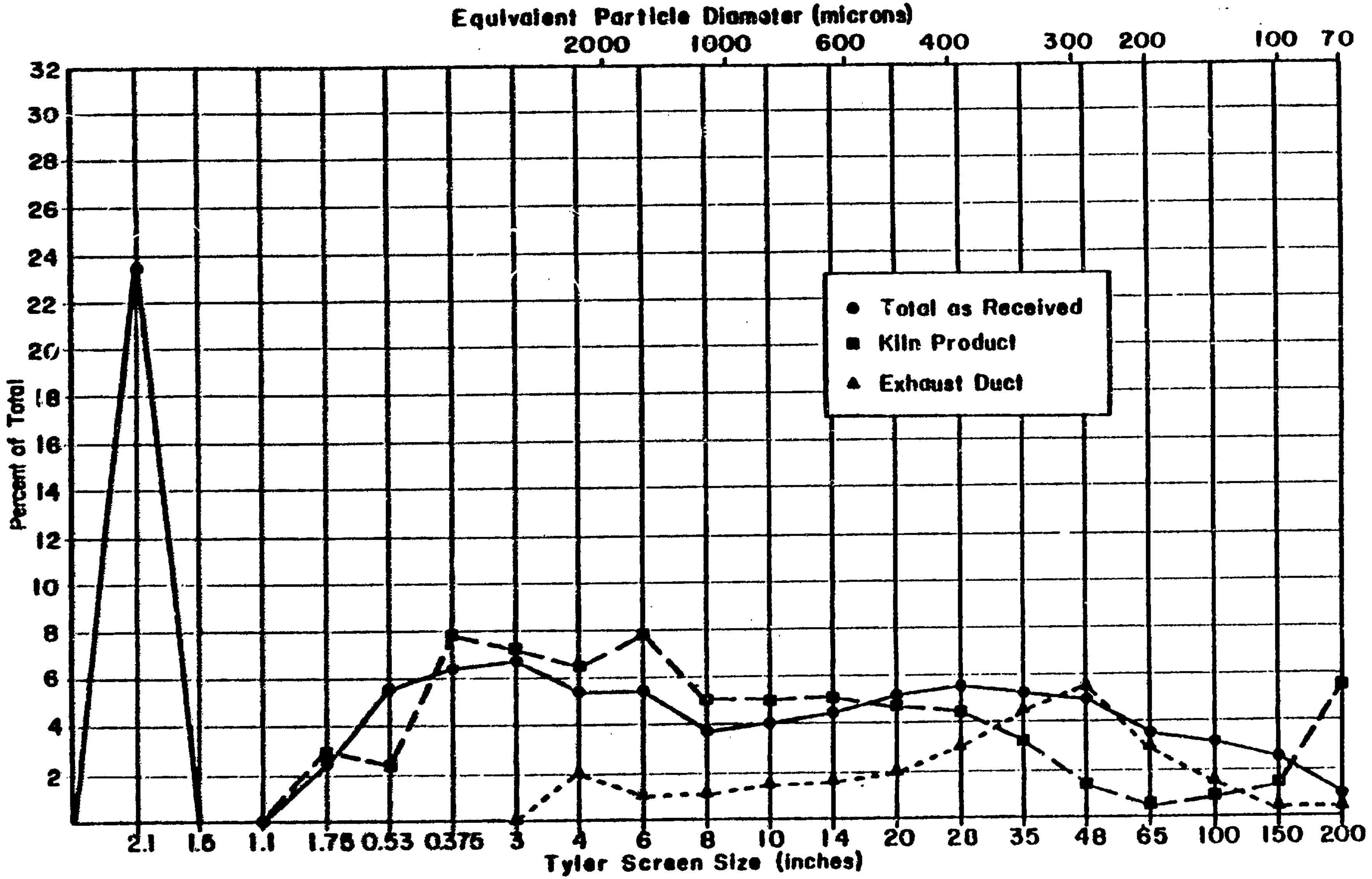
Figure 12 gives the results from Test 10 with NCBC soil. The screen analyses of the kiln product and exhaust dust show the selective entrainment of the smaller particles, starting at about 28 mesh. The combination of the kiln product and exhaust dust closely matches the screen sizes found in the sample as received, indicating little or no attrition or agglomeration. The reduction of particles in the 0.75-inch range is attributed to the burning of sticks that were observed in the sample.

d. Temperature Data

During batch kiln tests, temperature data for the soil and exhaust gas were continuously recorded; these data are given in Annex 4. Final temperatures (at steady-state) are listed in Table 14. Since all the tests were conducted to achieve the same final temperatures, the only differences between tests were during the heat-up period, which lasted from about 6 to 30 minutes depending on heat input, charge weight, moisture content, and soil type. Figure 13 compares temperature profiles for the three soils at the same heat input. For JI soil, a plateau at about 850°C was obvious, corresponding to calcination. The heat-up rates for JI and NCBC soil were influenced by the higher initial moisture content. Also, the charge weight for JI soil was greater than the other two.

e. Dynamic Angle of Repose

The dynamic angle of repose for the three soils was measured using a 38-inch diameter drum, rotating at 0.82 rpm. This property is important in



Fiugre 10. Particle Size Distribution of JI Soil Before and After Batch Kiln Test 4

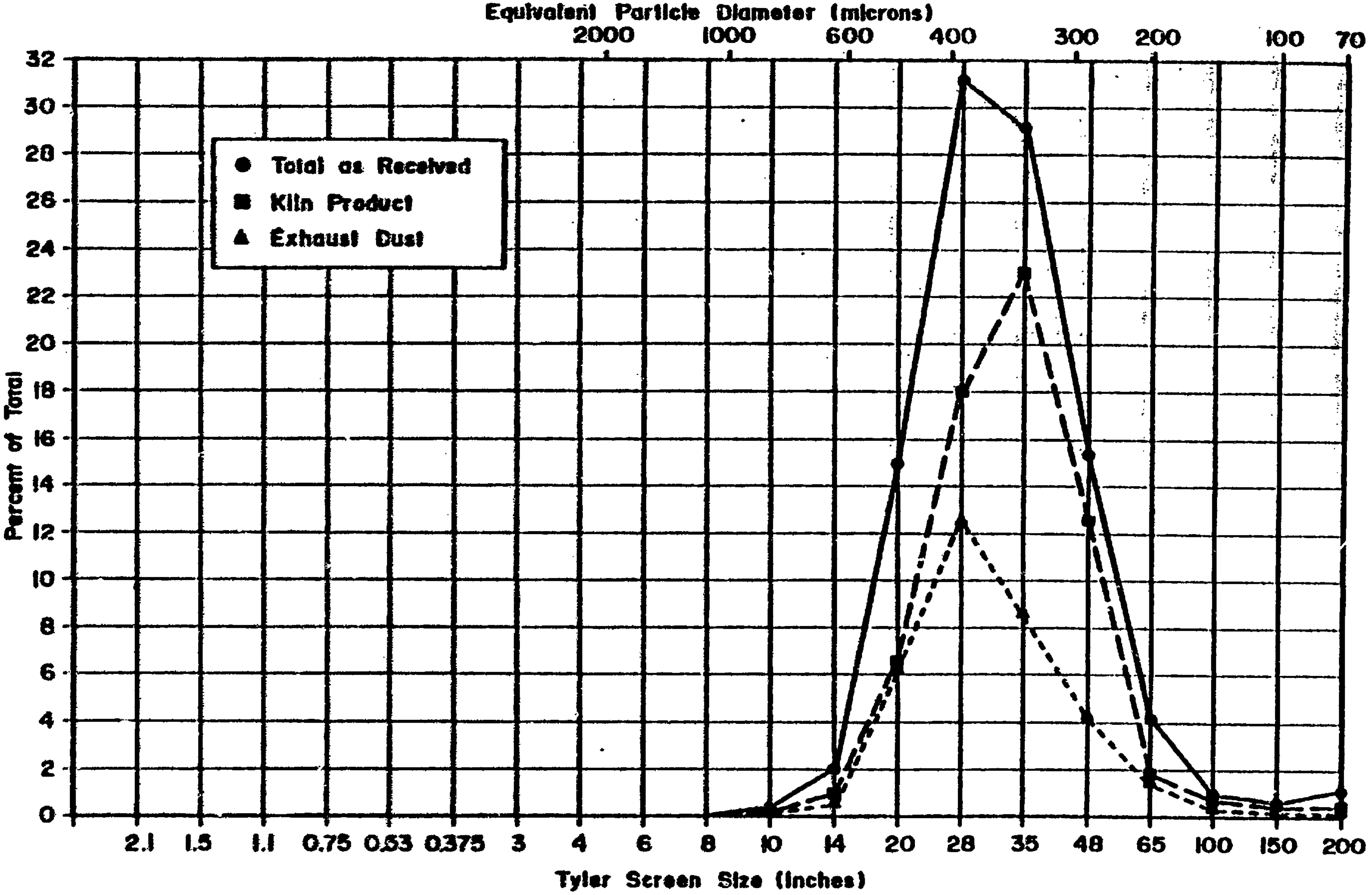


Figure 11. Particle Size Distribution of Elgin Soil
Before and After Batch Kiln Test 6

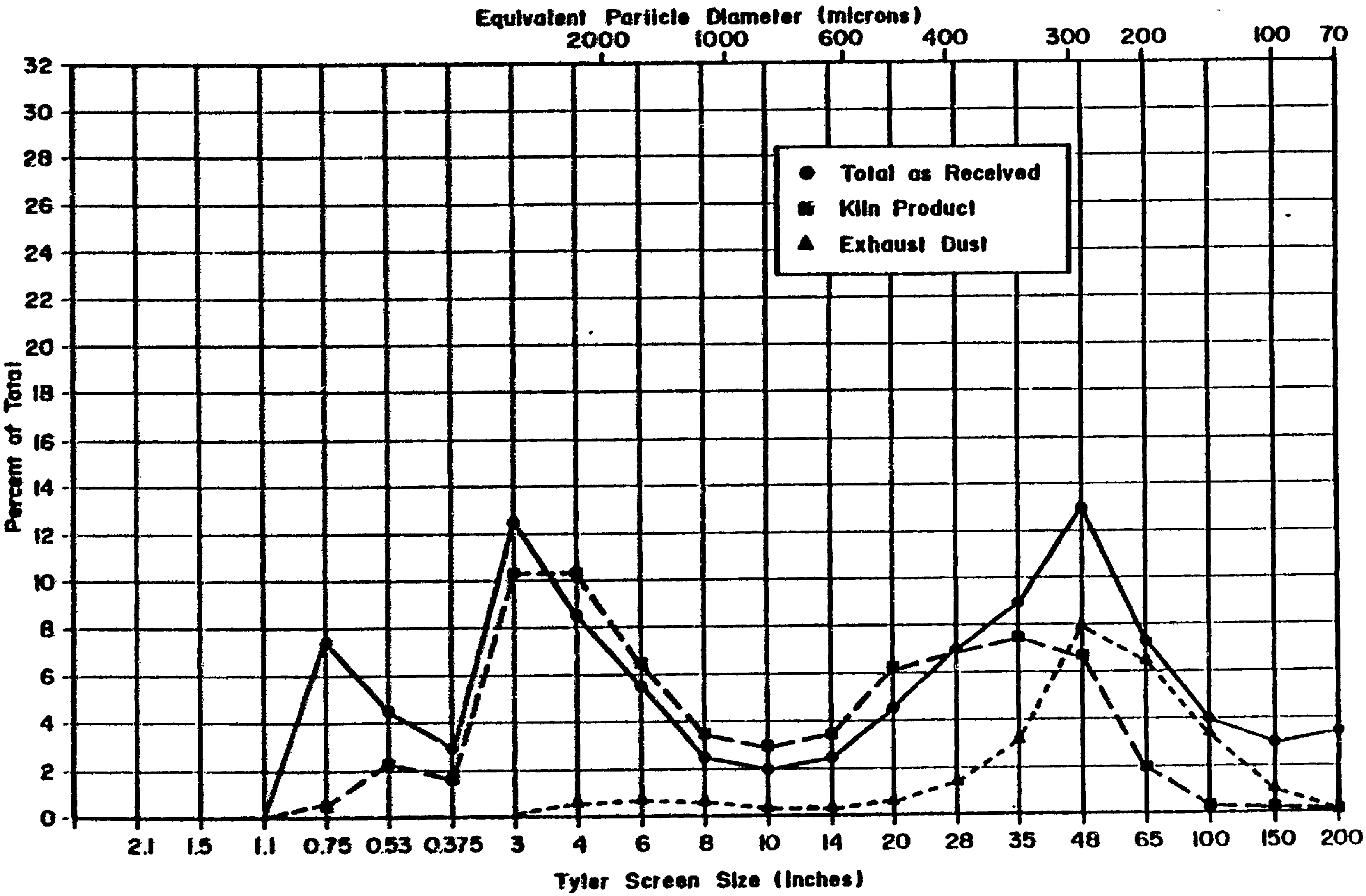


Figure 12. Particle Size Distribution of NCBC Soil Before and After Batch Kiln Test 10

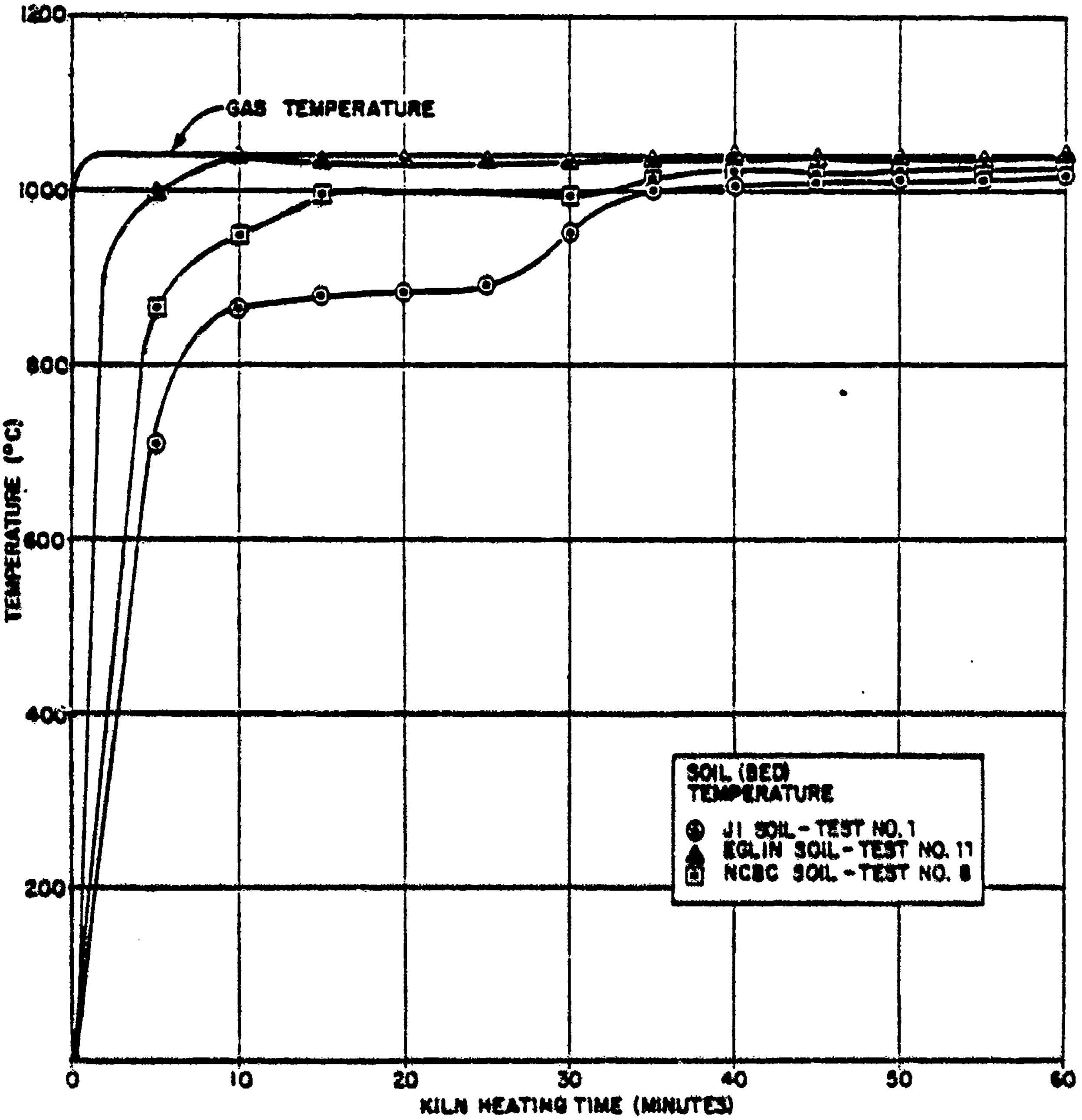


Figure 13. Time-Temperature History for Three Batch Pilot Kiln Tests Using Approximately Equal Heat Inputs

assessing the soil flow characteristics and resulting effect of kiln inclination on residence time. Tests were conducted on all three soils as received and on NCBC and Eglin samples after firing in the kiln. Data from these tests are given in Annex 4 and summarized in Table 18.

During this testing, JI soil was observed to roll over in a continuous fashion, with the larger particles generally staying on the outside of the sample, covering smaller particles underneath. The observed angle of repose was slightly greater than that for Missouri soil (clay-loam) studied previously, but close enough to allow data measurements of flow of Missouri soil through the MIS kiln to be applied to predictions for JI soil. A kiln loading of approximately 6 percent will be used in calculations related to heat transfer.

Eglin soil was observed to have a relatively low angle of repose, as would be expected from sand. After firing, the observed angle decreased significantly and the sand was observed to flow in a smooth motion. It is expected that this soil will flow quickly through the MIS. The Allis-Chalmers computerized mass flow model has not been verified with soils and, therefore, it is not possible to accurately predict soil flow rates through the MIS. An assumption of a kiln loading of 4 percent will be used in heat transfer analyses, based on previous MIS trials using sand.

NCBC soil was observed not to have the rolling motion characteristic of the other two soils. Instead, this soil stayed in a mostly stable mass which slid down the side of the test apparatus without much blending. After firing, the soil was observed to exhibit some rolling motion, although the sliding was still apparent. The sliding motion is thought to result from the presence of a large number of shells in the soil. This soil will have a high loading in the MIS, even though the measured angle of repose is low. No previous specific MIS test data or validated flow models are available to confirm this. The loading of this soil will be taken to be 7 percent for the purpose of heat transfer calculations.

TABLE 18. SUMMARY OF DYNAMIC ANGLE-OF-REPOSE MEASUREMENTS

Soil identification	Initial moisture (%)	Bed loading (%)	Angle of repose Upper section (%)	Angle of repose Lower section (%)
JI	6.5	7	49	39
Elgin	1.2	7	44	35
Eglin[b]	~0	4.4[a]	32	32
NCBC	6.4	7	40	40
NCBC[b]	~0	2.5[a]	35	33

[a] Insufficient sample remaining to have desired soil charge.

[b] After firing in kiln.

SECTION V

HEAT TRANSFER EVALUATION

A special computer program was used to model heat transfer in the MIS kiln for the three test soils. The program had been developed and was applied to this study by Allis-Chalmers personnel. Previous results ITC obtained from a similar study of Missouri soil were used as a basis to select the input conditions for computer runs. The program models the overall heat transfer in a kiln by dividing the kiln into three or four regions and modeling each region separately. In the first region, the wet solid is heated from ambient temperature to the boiling point of water. In the second region, water is evaporated at a constant solid temperature. In the third region, the dry solid achieves its final temperature. If calcination is known to occur, a fourth region is modeled for its heat duty at constant temperature. This region is important for studying lime kiln performance.

The heat transfer constants used in the computer calculations were derived from a proprietary Allis-Chalmers data base developed from studies using other feed materials typically processed in rotary kilns, such as limestone. The constants are adjusted according to the characteristics and configuration of the kiln which is to be modeled. A difficulty in accurately modeling the heat transfer characteristics of the MIS kiln is presented by the flights within the kiln toward the feed end. The heat transfer constants used in this study were the same as those used for the study of the treatment of Missouri soil in the MIS. The program does not account for differences in the heat transfer characteristics (other than heat capacity) of various soil types or particle sizes. The constants are listed with the sample computer printouts in Annex 10.

The runs were selected to demonstrate the differences in the predicted soil time-temperature profiles due to differences in overall heat duty and soil heat capacities, as obtained from TGA and DSC analyses described in Section II.

A. OBJECTIVES

Definition of heat transfer in the MIS kiln enables prediction of the operating limits for maximum soil processing rates. The maximum feed rate varies dramatically with the composition of the soil being treated. Differences in soil that affect the treatment rate include the moisture content, organic content, quantity of material subject to thermochemical transitions (e.g., calcination), heat capacity, and requirement for excess air (to meet RCRA/DRE requirements) during kiln operation.

Prediction of the maximum treatment rate is important in assessing the cost effectiveness of using the MIS or some larger rotary kiln incinerator. The projected maximum processing rate will also be useful in planning and conducting effective field demonstrations. Operating the MIS near its maximum capacity will provide valuable data with which to evaluate/design larger systems. This will allow accurate cost analyses to be done on larger units to aid in comparison with other treatment alternatives.

An additional objective of the heat transfer work was to help define possible improvements in the MIS, identify important data collection needs for field demonstrations, and reveal major design changes that could be incorporated in a larger-scale unit.

B. PREVIOUS HEAT TRANSFER STUDY RESULTS

The heat transfer characteristics of the MIS in treating soil from Missouri were studied extensively during previous work for the EPA. In that study, the effects of soil moisture content, heat capacity, and kiln excess air on the time-temperature profile of soil in the kiln were investigated. Also, a series of heat and material balances was performed using ITC's computer model. The results of that work, which are important to understanding of the results reported herein, will be documented in an EPA report in 1985. A synopsis of the conclusions and observations of the previous work is given here.

Previous work demonstrated that the kiln would not normally be limited by heat transfer within the range of operating conditions selected for the Missouri trial burn. Overall MIS capacity limitations include the rate of the solids feed system and the requirement for a 2-second residence time for gas in the secondary combustion chamber (SCC). The limit on the SCC results from the high percentage of kiln excess air used. It was determined that kiln excess air needed to be less than 100 percent and preferably less than 50 percent before the kiln could be operated at its heat transfer limit. The primary effect of soil moisture content was the increase in enthalpy due to vaporization, which reduces the heat available for quickly increasing the soil temperature. The reduction in available heat caused by soil moisture becomes very important above 10 percent. Soil moisture content had a greater effect on soil discharge temperature than the dry soil feed rate within the range studied. This limit is a result of the total fuel that can be fired, rather than of heat transfer. The heat transfer rate will affect capacity only when these other limitations are removed. Heat transfer is limiting when the feed rate increases to the point where the soil residence time in the kiln is low, and therefore the soil does not reach the time-temperature condition required for sufficient decontamination.

Several recommendations were presented to EPA based on this previous work. The first and foremost was to operate the incinerator at low excess air concentrations. Normal operation of hazardous waste incinerators calls for excess air concentrations of 100 to 150 percent. This gives a "cushion" to allow the system to handle sudden surges in the waste feed heating value, and prevents conditions where complete combustion is not obtained. When treating soil, which has a negligible heating value, this mode of operation is not necessary, eliminating the need for high excess air. This allows operation at lower (25 to 50 percent) excess air, reducing entrainment and enabling higher heat input and corresponding higher soil feed rates to achieve a given time-temperature condition.

Physical modifications recommended for the MIS included the addition of dams at the discharge end of the kiln and modifications in the exit air discharge ducting to permit greater adjustment in kiln slope. These modifications would increase the maximum kiln loading and therefore enable higher feed rates while maintaining the required soil residence times. These modifications would be particularly important in treating a free-flowing soil, such as sand.

C. HEAT TRANSFER COMPUTER SIMULATION RESULTS

Computer runs were selected to give a number of data points in the region of maximum kiln capacity for each of the three soils. The conditions were selected based on knowledge gained through the previous work and specific data gathered on the soils during the batch pilot kiln tests and laboratory treatibility tests.

Table 19 outlines the 15 heat transfer runs performed; five runs were performed for each soil. Example printouts of the computer runs are given in Annex 10, along with heat and material balance program runs performed at the same conditions. Three of the runs for each soil were performed at conditions of 12 percent moisture and 50 percent excess air, with varied feed rates, to allow definition of the heat transfer limit. Two additional runs were performed on each soil at 25 and 100 percent excess air to demonstrate the effect of excess air concentration on treatment. A constant fuel feed rate to the kiln equivalent to 5.5 million Btu/hour was used for all runs. Higher kiln heat duties would make it difficult to maintain the mandatory 2-second residence time in the SCC.

The results of the 15 heat transfer runs are summarized in Table 20. Comparison of the runs demonstrates the effects of soil feed rate and kiln excess air on soil discharge temperatures. The results of the heat and material balance program runs are also summarized in Table 20. This program assumes temperature equilibrium between the gas and solids. This temperature represents an average between the two temperatures given by the heat transfer program, weighted for the heat capacity of the total gas and solids leaving the kiln. Inspection of Table 20 reveals that the heat and material balance program gives a somewhat higher temperature than does the Allis-Chalmers heat transfer model. The magnitude of this difference is not considered significant since the lower temperature calculated by the heat transfer program will result in conservative estimates of the kiln capacity.

The soil temperature predicted by the heat transfer program assumes a uniform bed temperature. In an operating kiln, temperature gradients will be present in both individual (large) soil particles and in the bulk soil bed. To account for this, an allowance of approximately 50°C (90°F) should be made in the soil discharge temperatures. For soils that may have a higher temperature gradient, such as those with relatively large chunks or those that do not mix well in a moving bed, the allowance may need to be slightly greater.

TABLE 19. PARAMETERS FOR HEAT TRANSFER RUNS

Run No.	Soil identification	Soil dry weight (lb/hr)	H_2O (lb/hr)	H_2O (%)	Loading (%)	Kiln air (lb/hr)	Excess air (%)	Fuel (10^6 Btu/hr)	Specific heat of dry soil (Btu/lb·°F)	Δ H Calc.	Soil heat value (Btu/lb)
1	Eglin	3400	464	12	4	6270	50	5.5	0.23	14	9
2	Eglin	4000	545	12	4	6270	50	5.5	0.23	14	9
3	Eglin	3000	409	12	4	6270	50	5.5	0.23	14	9
4	Eglin	3400	464	12	4	8354	100	5.5	0.23	14	9
5	Eglin	3400	464	12	4	5220	25	5.5	0.23	14	9
6	JI	3400	464	12	6	6270	50	5.5	0.19	520	25
7	JI	4000	545	12	6	6270	50	5.5	0.19	520	25
8	JI	3000	409	12	6	6270	50	5.5	0.19	520	25
9	JI	3400	464	12	6	8354	100	5.5	0.19	520	25
10	JI	3400	464	12	6	5220	25	5.5	0.19	520	25
11	NCBC	3400	464	12	7	6270	50	5.5	0.20	65	90
12	NCBC	4000	545	12	7	6270	50	5.5	0.20	65	90
13	NCBC	3000	409	12	7	6270	50	5.5	0.20	65	90
14	NCBC	3400	464	12	7	8354	100	5.5	0.20	65	90
15	NCBC	3400	464	12	7	5220	25	5.5	0.20	65	90

TABLE 20. PREDICTED TEMPERATURES FROM COMPUTER MODELS

Run No.	Soil identification	Excess air (%)	Soil residence time (min.)	Soil dry feed rate (lb/hr)	Predicted temperature (°C)		
					Heat transfer program		HMB program[a]
					Soil discharge	Flue gas	
1	Eglin	50	15.5	3400	821	953	937
2	Eglin	50	13.1	4000	733	921	876
3	Eglin	50	17.5	3000	888	979	981
4	Eglin	100	15.5	3400	634	825	801
5	Eglin	25	15.5	3400	951	1030	1025
6	JI	50	20.0	3400	793	955	947
7	JI	50	17.0	4000	781	934	888
8	JI	50	22.6	3000	802	966	991
9	JI	100	20.0	3400	640	809	811
10	JI	25	20.0	3400	811[b]	998[b]	1038
11	NCBC	50	21.7	3400	828	949	990
12	NCBC	50	18.5	4000	792	920	936
13	NCBC	50	24.6	3000	884	982	1029
14	NCBC	100	21.7	3400	708	825	846
15	NCBC	25	21.7	3400	960	1020	1083

[a] From heat and material balance program; soil discharge and flue gas temperatures are considered equal.

[b] Data for soil heat value was input as negative; actual temperatures should be somewhat higher (~35°C).

Past comparisons of the heat transfer model with actual MIS operational data have indicated that the heat transfer coefficients used in the model may be slightly high, causing the model to predict higher soil discharge temperatures and lower exit gas temperatures than those experienced in the kiln. The magnitude of this effect is undefined and will require evaluation of additional operating data. However, preliminary indications are that the effect is less than 55°C (100°F) for soil discharge temperature when the kiln is operating at low mass flow (~1000 pounds/hour), low firing rate (~3.0 mm Btu/hour), and high excess air (~100 percent). The results presented in Table 20 should only be employed for approximate estimates of kiln capacity.

The data presented in Table 20 indicate the thermal and heat transfer limitations of the MIS kiln for treating the three soils of interest. At 50 percent excess air and 12 percent moisture, all three soils apparently can be treated at a rate of 4000 pounds per hour dry weight as determined by soil exit temperatures above 700°C (see Section VI). The Eglin soil may have a slightly lower maximum rate, due primarily to the free-flowing nature of the soil which reduces the kiln loading and associated soil residence time. At 100 percent excess air, the heat transfer limit appears to be approximately 3400 pounds/hour for the JI and NCBC soils and somewhat lower for the Eglin soil.

The heat and material balance program was used to check the results from the heat transfer program and to examine limits in the secondary combustion chamber (SCC). The heat and material balance evaluation results revealed an additional system limitation at 100 percent excess air. Table 21 gives the secondary burner fuel requirements for the 15 example runs. Examination of Runs 4, 9, and 14 in Table 21 shows that the burner requirements in the SCC exceed the burner limitations of 5.5 times 10^6 Btu/hour. This limit relates to both the actual burner feed rate limitations and the limit on combustion gas flow rate that may be tolerated in the SCC to meet the 2-second gas residence time requirement. Analysis of the runs performed at 3400 pounds/hour and 12 percent moisture yielded maximum excess air percentages of 61, 66, and 74 percent for treating Eglin, JI, and NCBC, respectively. However, the above analysis assumes that the solids discharge and exit gas temperatures in the kiln are equal. In the operating system, the gas temperature entering the secondary is significantly higher than that predicted by the heat and material balance program. Therefore, the heat load on the secondary burners would be expected to be less than calculated, allowing higher soil feed rates at the same excess air and moisture indicated by the values in Table 21. In summary, operation at 100 percent excess air, 3400 pounds/hour dry solids feed, and 12 percent moisture represents a point close to the operational limit of both the kiln and SCC.

The effect of soil moisture content was not included as part of this heat transfer study. For estimates of system performance at higher moisture contents, the total water feed rate as given in Table 19 should be used for comparison, rather than the dry soil feed rate. This should yield reasonably accurate results for soil having moisture contents up to 20 percent. Reports describing previous heat transfer studies should be referenced.

TABLE 21. SECONDARY COMBUSTION CHAMBER BURNER FUEL REQUIREMENTS FOR HEAT TRANSFER RUNS

Run No.	Soil identification	Excess air (%)	Soil dry feed rate (lb/hr)	SCC burner requirement (10^6 Btu/hr)
1	Eglin	50	3400	4.756
2	Eglin	50	4000	5.562
3	Eglin	50	3000	4.186
4	Eglin	100	3400	7.651
5	Eglin	25	3400	3.402
6	JI	50	3400	4.630
7	JI	50	4000	5.424
8	JI	50	3000	4.063
9	JI	100	3400	7.501
10	JI	25	3400	3.262
11	NCBC	50	3400	4.119
12	NCBC	50	4000	4.842
13	NCBC	50	3000	3.606
14	NCBC	100	3400	6.988
15	NCBC	25	3400	2.781

SECTION VI

EVALUATION OF MIS TREATMENT CAPABILITIES

In order to translate the laboratory treatability data and the calculated heat transfer rates of the MIS kiln to a projected overall soil treatment capacity, a statistical evaluation was performed and the results were combined with the predicted soil temperature profile in the kiln. Multiple linear regression analysis was performed using all the treatability data, considering a variety of different mathematical models relating time, temperature, and 2,3,7,8-TCDD concentration. This data analysis was done using the computer software package SAS (Statistical Analysis System) developed and maintained by the SAS Institute, Box 8000, Cary, North Carolina. Dr. Robert McLean, Professor of Statistics at the University of Tennessee, Knoxville, Tennessee carried out the statistical evaluation as a consultant to ITC. A similar statistical evaluation was previously performed on treatability studies of a single Missouri soil contaminated with 2,3,7,8-TCDD. Therefore, some of the models which were evaluated had already been identified and were simply confirmed using the new data. In addition, several new models were considered which utilized the initial 2,3,7,8-TCDD concentration as a variable, since each of the three soils had different initial concentrations.

Statistical evaluation of previous laboratory thermal desorption test data had shown that soil moisture content and purge gas type and flow rate had no significant effect on treatability, except the indirect effect of initial soil moisture on the temperature profile (heat-up time) during a specified treatment period Particle size was shown to have an effect only for gross differences (e.g., 5 cm cubes vs 2 mm material), and this was considered to be primarily due to heat transfer within the large particles.

The data base used for the statistical analysis is presented in Appendix I. For each test, different parameters such as time, temperature, vapor pressure, etc. were calculated for use in the regression analysis. Mathematical models were developed relating the concentration of 2,3,7,8-TCDD after treatment with one or more of the parameters.

The effect of soil type on treatability was determined to be minor if the variation in starting concentration was accounted for. There was no statistical method to ascertain the effect of soil type because only one starting concentration for each soil was available.

The three models which were found to best fit the 16 laboratory desorption test results from this study, and their R-square values are as follows:

$$\text{LNPD} = 1.170 - 4.164 \times 10^{-5}\ (\text{IVP}) \qquad (1)$$

$$\text{R - square} = 0.80$$

$$\text{LNPD} = -0.213 - 8.63 \times 10^{-5}\ (\text{IVP}) + 3.02 \times 10^{-10}\ (\text{IVP})^2 \qquad (2)$$

$$\text{R-square} = 0.91$$

$$\text{LD} = 0.204 + 0.908\ (\text{LINC}) + 0.226\ (\text{TIME}) + 1.91 \times 10^{-4}\ (\text{TEMP}) - 5.97 \times 10^{-6}\ (\text{TEMP})^2 - 5.24 \times 10^{-4}\ (\text{TITP}) \qquad (3)$$

$$\text{R-square} = 0.93$$

where:

LNPD = $\log_e$ of weight fraction of initial 2,3,7,8-TCDD remaining after treatment,
IVP = integral of vapor pressure over the treatment period (mmHg·minutes),
LD = $\log_{10}$ of final concentration (in ppb) of 2,3,7,8-TCDD,
LINC = $\log_{10}$ of initial concentration (in ppb) of 2,3,7,8-TCDD,
TIME = time at constant nominal treatment temperature (minutes),
TEMP = nominal treatment temperature (°C), and
TITP = TIME · TEMP.

The integral of vapor pressure is determined by calculating the vapor pressure of 2,3,7,8-TCDD for the average temperature during each 1-minute interval (including during the heat up period) and summing these values, which is essentially equivalent to the area under a plot of vapor pressure versus time. The predicted vapor pressure of 2,3,7,8-TCDD is shown in Figure 14 with the corresponding Antione equation (Reference 15).

These models are, to some extent, a function of the characteristic time-temperature profile for the experimental apparatus and procedures used to generate the data. This must be considered when attempting to predict treatment performance in the MIS, which will have a somewhat different time-temperature profile depending on a number of factors, such as initial soil moisture content, solids feed rate, and heat input.

Figure 15 is a plot of LNPN versus IVP, showing reasonably linear dependence. By using Equation (2), the approximate conditions of time and temperature to achieve certain treatment efficiencies can be calculated. Figure 16 shows the significance of temperature on reducing the concentration of 2,3,7,8-TCDD from 100 ppb and 500 ppb to 1 ppb. Assuming a constant solids temperature within the MIS kiln, the required residence time for the 100 ppb contamination level varies from less than 1 minute at 800°C to about 16 minutes at 500°C. Less residence time would be required for soils with lower contamination levels. However, the solids temperature in the kiln varies and complicates the prediction of residence time requirements.

The relationship found in this study between treatment efficiency and time-temperature, as represented by Equations (1) through (3), is comparable to the results developed previously for Missouri soil.

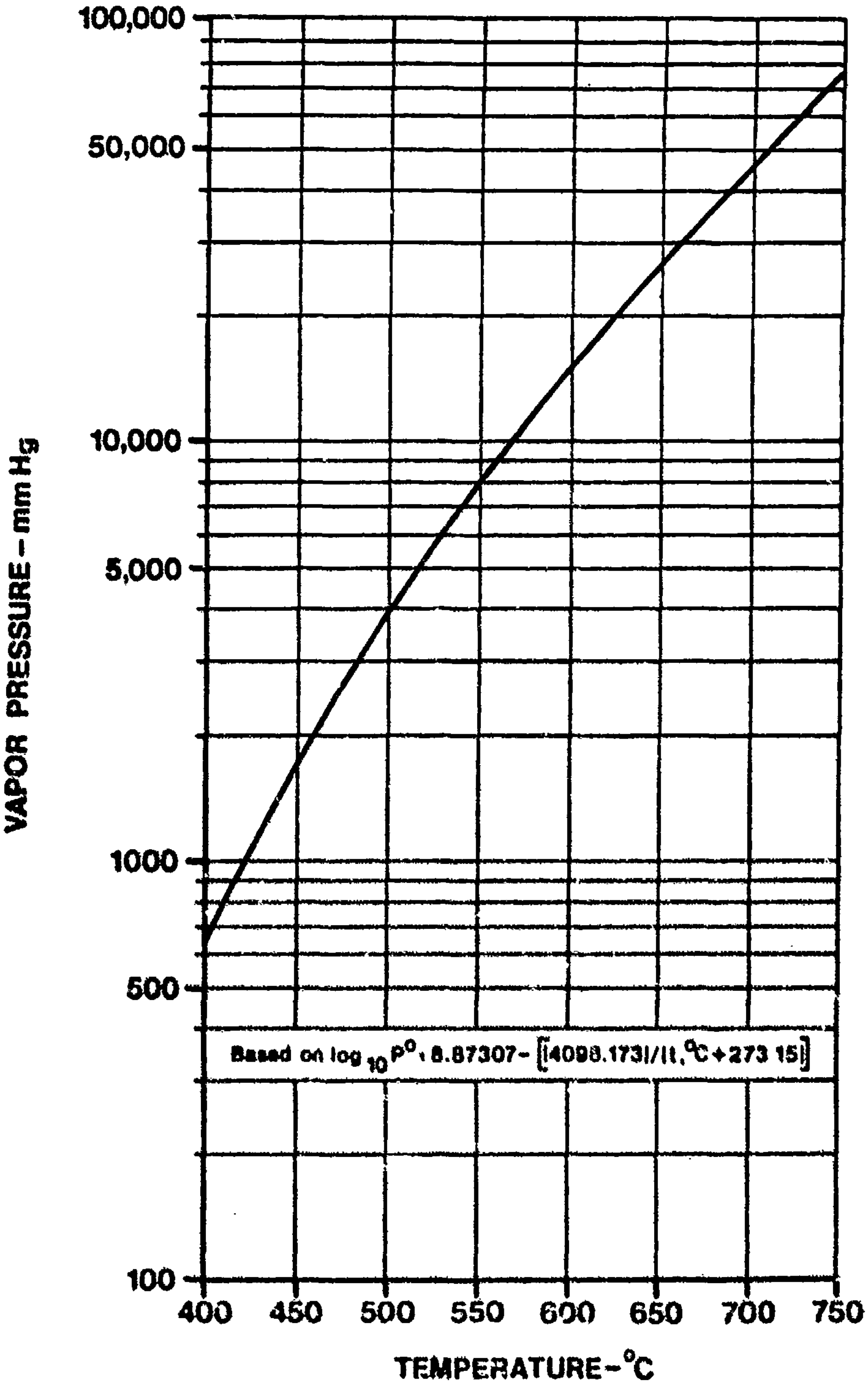


Figure 14. Projected Vapor Pressure of 2,3,7,8-TCDD at High Temperatures

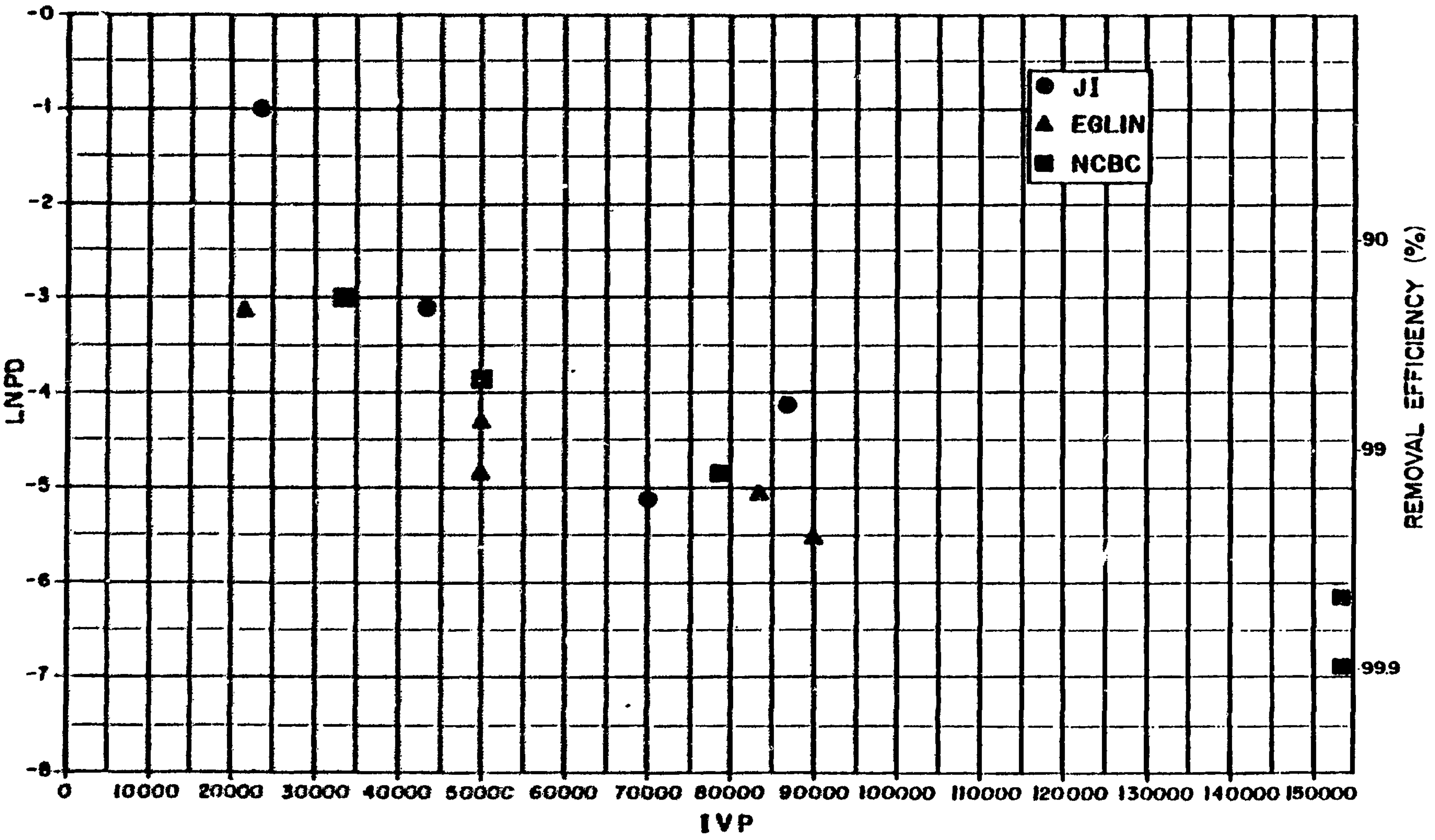


Figure 15. Plot of LNPD Vs. IVP

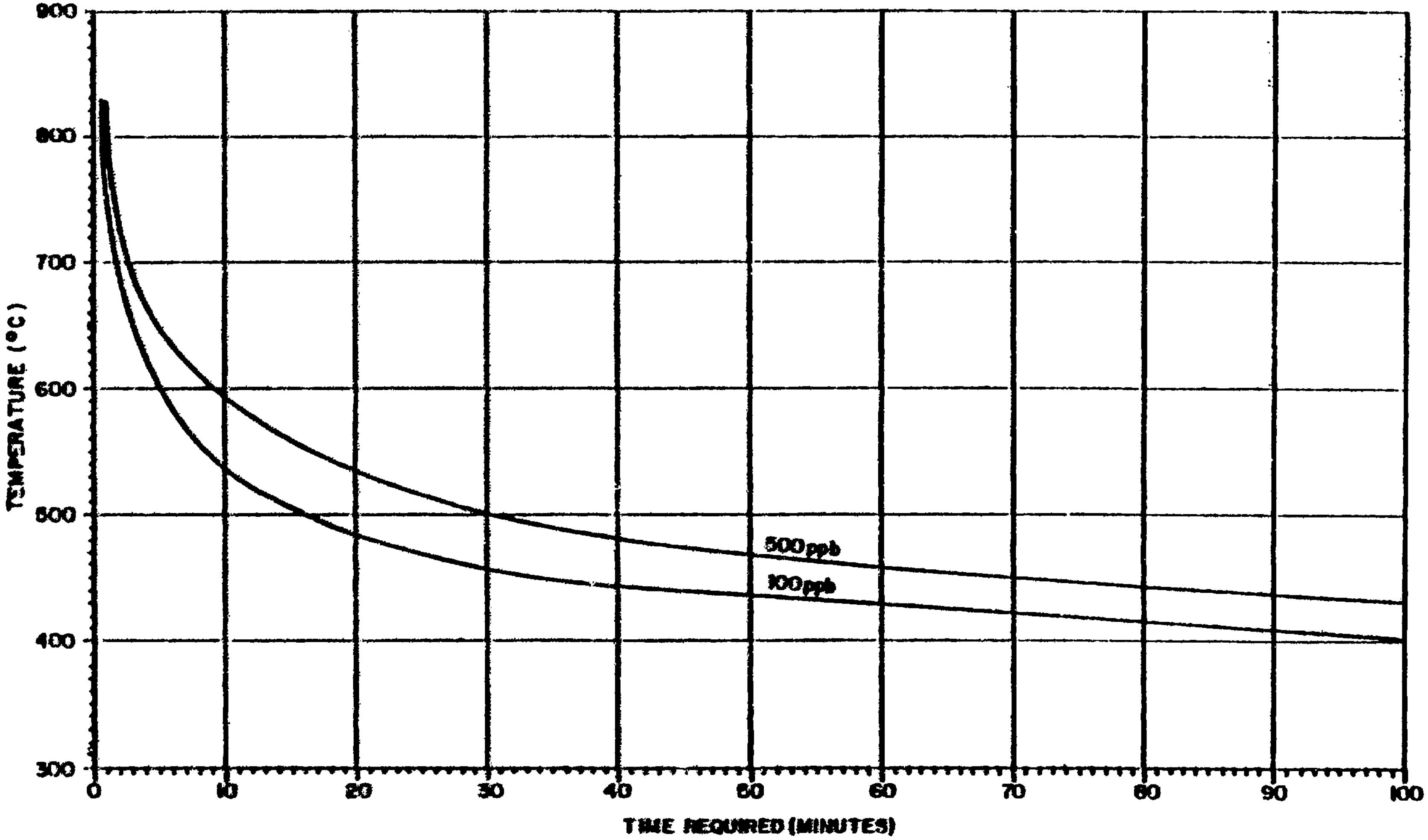


Figure 16. Predicted Time-Temperature Required to Achieve 1ppb 2,3,7,8-TCDD for Soils Initially Containing 100ppb and 500ppb

By employing the heat transfer program discussed in Section V, the soil time-temperature profile for any set of conditions can be estimated. Figure 17 shows the calculated soil residence time for each linear foot (indicated by a dot) of the kiln, considering the measured soil density, estimated loading, and selected soil feed rate of 4000 pounds per hour dry weight. The corresponding vapor pressure-versus-time profile shown in Figure 18 was developed from Figure 17, assuming that the soil temperature increases stepwise at 1-foot intervals. The average of the calculated vapor pressures at the front and rear of each 1-foot interval was used to develop the values for each point on the curve. The area under this time-vapor pressure curve was approximated by summing the area of the steps in the figure. By using the resulting IVP value of 141,000 mmHg•min in Equation (1), and assuming a soil feed containing 500 ppb, the estimated final 2,3,7,8-TCDD concentration was calculated to be 0.4 ppb. This value is near the lower limit of the treatability data used to generate Equation (1). Predicting values below 0.1 ppb would not be justified without further data taken at low dioxin concentrations (<0.1 ppb).

This same procedure was used to calculate the final 2,3,7,8-TCDD concentration for each of the heat transfer cases described in Section V. Table 22 reveals that the model predicts that 2,3,7,8-TCDD concentrations of less than 1 ppb will be met for all but two cases. Both of these cases assumed high excess air concentrations (100 percent). However, upon examination of the computer sheets for Run 9, it was discovered that the heat value was input as a negative number, reducing the discharge temperature by about 50°C (90°F). If this had been input correctly, the predicted concentration would fall below 1 ppb. Run 2, which had a high soil feed rate (4000 pounds/hour), and low kiln residence time, was the only other run that gave a final concentration above 0.1 ppb.

The predictive model is strongly a function of temperature. Using Run 2 as an example, predicted temperatures were reduced by 55°C (100°F) and the final 2,3,7,8-TCDD concentration recalculated. This method yielded a revised concentration of 7 ppb. The other runs were evaluated using the same reduced temperature. This did not significantly affect the results, for Run 14, which yielded a predicted dioxin concentration of 2 ppb.

The predictive model is also affected by the assumptions made on kiln loading given in Section IV and summarized in Table 19. As an example, the predicted 2,3,7,8-TCDD concentrations were recalculated for Run 4 assuming a kiln loading of 7 percent as opposed to 4 percent as used for Table 22. The resulting dioxin concentration for this case was 5 ppb, which represents close to the maximum effect possible for variations in kiln loadings.

To select a set of MIS operating conditions (time-temperature combinations) that will meet the required treatment efficiency (1 ppb), all parameters affecting heat transfer, heat duty, soil residence time, and starting 2,3,7,8-TCDD concentration must be specified, and the other limitations of the MIS discussed in Section V must be considered. Excessive moisture and soil feed rate will pose difficulty in achieving decontamination criteria if highly contaminated soil is processed.

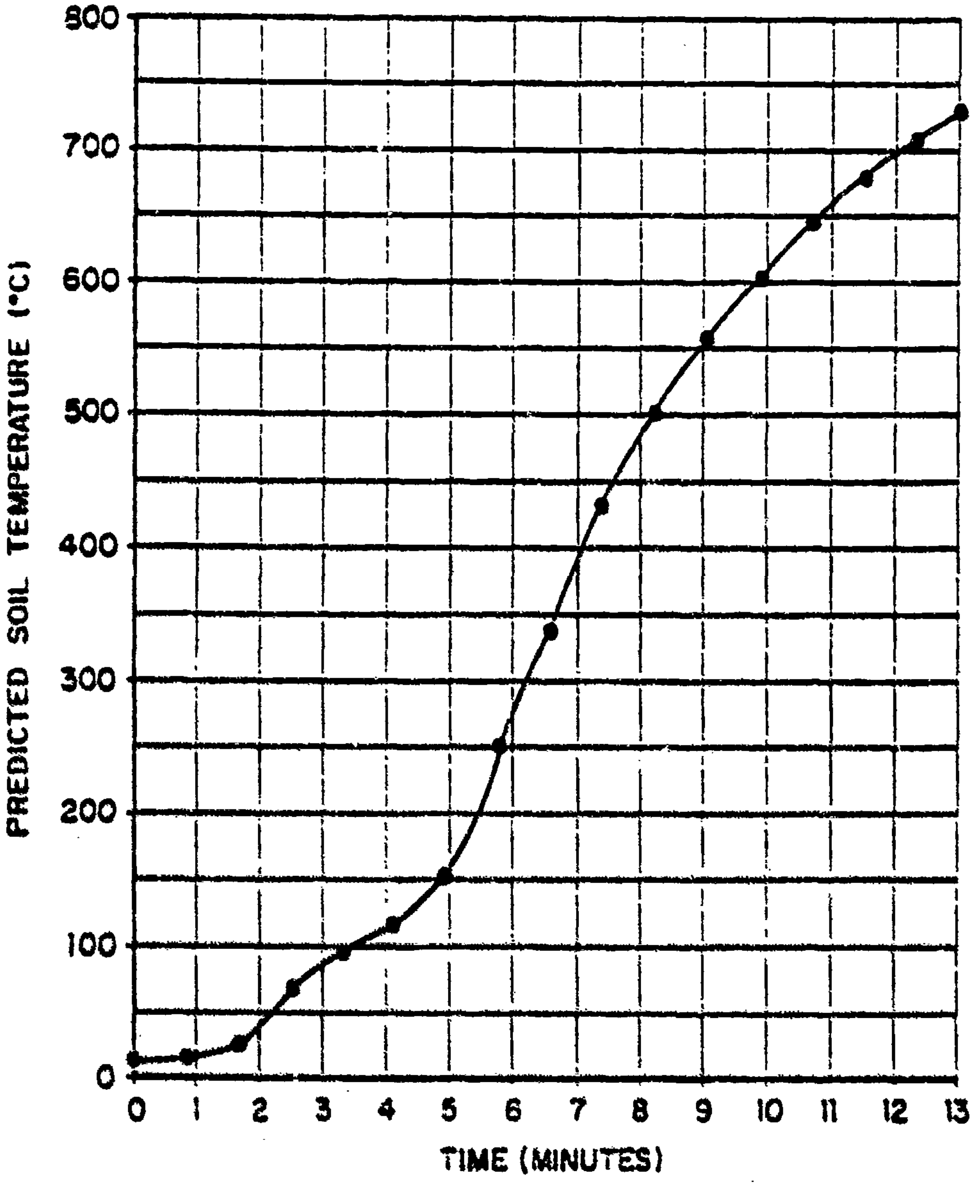


Figure 17. Temperature Vs. Time for Heat Transfer Run Number 2

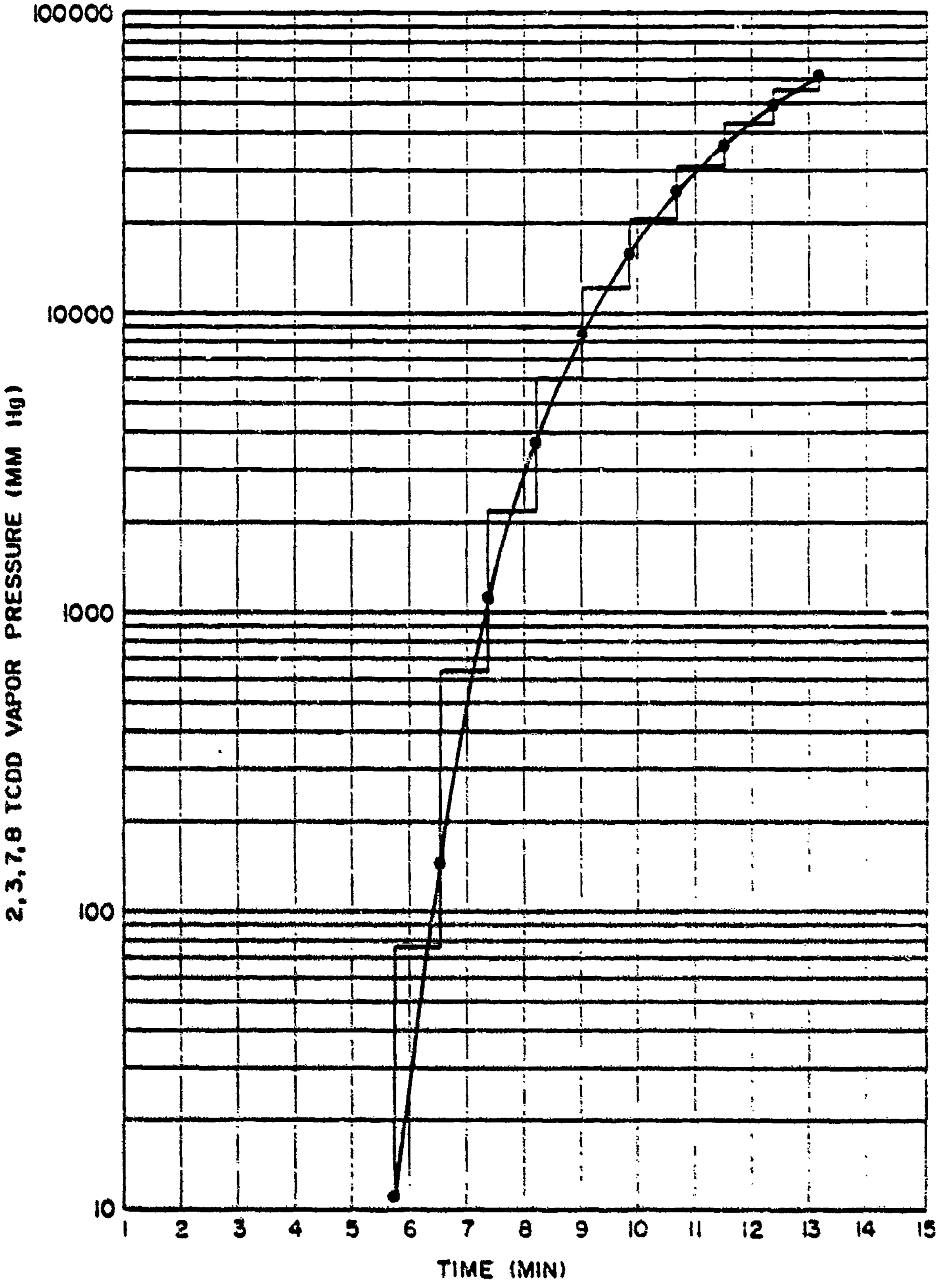


Figure 18. Vapor Pressure Vs. Time for Heat Transfer Run Number 2

TABLE 22. PREDICTED TREATMENT EFFICIENCY FOR HEAT TRANSFER RUNS

Run No.	Soil identification	Excess air (%)	Soil dry feed rate (lb/hr)	Calculated 2,3,7,8-TCDD concentration (ppb)[a]
1	Eglin	50	3400	<0.1
2	Eglin	50	4000	0.4
3	Eglin	50	3000	<0.1
4	Eglin	100	3400	20
5	Eglin	25	3400	<0.1
6	JI	50	3400	<0.1
7	JI	50	4000	<0.1
8	JI	50	3000	<0.1
9	JI	100	3400	15[b]
10	JI	25	3400	<0.1
11	NCBC	50	3400	<0.1
12	NCBC	50	4000	<0.1
13	NCBC	50	3000	<0.1
14	NCBC	100	3400	<0.1
15	NCBC	25	3400	<0.1

[a] Assumes initial concentration of 500 ppb 2,3,7,8-TCDD and 12% moisture.

[b] Data for soil heating value was input as negative due to calcination endotherm; actual concentration is expected to be somewhat lower (<1 ppb).

SECTION VII

CONCLUSIONS

The following conclusions, based on the results of the project, are supported by previous results of related studies.

1. Thermal treatment studies of Herbicide Orange-contaminated soils from Johnston Island, Eglin Air Force Base, and the Naval Construction Battalion Center at conditions representative of the MIS capabilities were successful in achieving less than 1 ppb residual concentrations of 2,3,7,8-TCDD and related isomers in the treated soils. Concentrations of 2,3-DBE and 2,4,5-TBE were reduced to less than 50 ppb under the same condition, equivalent to greater than 99.99 percent removal efficiency from the soils. This removal efficiency is not the same as the DRE determined by stack gas sampling on incinerators.

2. Treatment is greatly influenced by temperature and residence time. Statistical evaluation of the treatability data from all three test soils showed the 2,3,7,8-TCDD concentration after treatment to be directly proportional to the starting concentration and logarithmically proportional to the time and temperature.

3. The soil type had a minor influence on treatability. Eglin soil gave the highest removal efficiencies and Johnston Island soil gave the lowest. The differences in treatability appeared greater at lower temperatures.

4. The treatability of the three soils studied was comparable to that of a Missouri soil sample studied previously in the first trial/demonstration burn of the EPA MIS. The influence of treatment time and temperature on removal efficiency was very similar.

5. Chemical and physical changes occurred in each of the three soils studied, depending on their composition. None of these changes were determined to pose a serious operational difficulty, such as slagging. However, the JI soil showed substantial weight loss, presumably accompanied by the generation of carbon dioxide, and consumption of heat as a result of calcination.

6. Entrainment of soil particles was determined to represent a potential operating difficulty if high soil feed rates and high excess air are used. The amount of entrainment is dependent on soil type and corresponding particle size distribution.

7. Based on computer simulation, heat transfer rates in the kiln should not limit the MIS capacity to treat contaminated soil for any of the three soils if moisture content is below about 20 percent. Other physical

attributes of the MIS, including SCC burner feed rates, soil feed system, kiln loading, and operating protocol, such as using high excess air, represent kiln capacity limitations. Soil processing rates of 3500 to 4000 pounds per hour should be realized, even with highly contaminated soil at these sites.

SECTION VIII

RECOMMENDATIONS

To validate the heat transfer and heat and material balance computer simulations used in this study, a thorough review and interpretation of all available operating data from the recent MIS trial/demonstration burns should be performed. In addition, appropriate additions in data collection/instrumentation, particularly accurate measurement of solids temperature in the kiln, would provide a sounder data base for this validation. This would enable better predictions of operating capabilities when considering the variety of applications and operating conditions which could be involved in soil decontamination efforts.

Additional studies should be carried out to better define particulate entrainment from soils processed by the MIS (or other kiln systems). Preliminary review of pertinent theory and available data provides justification and a basis for comprehensive evaluation of this important process aspect.

SECTION IX

REFERENCES

1. Channell, R. E. and Stoddart, T. L., Herbicide Orange Monitoring Program, ESL-TR-83-56, Engineering and Services Laboratory, Air Force Engineering and Services Center, Tyndall AFB, FL, April 1984.

2. Hughes, B. M., et al., Analytical Methodology for Herbicide Orange. Volume I: Determination of Chemical Composition, NTIS ADA01579, Aerospace Research Laboratories, Wright-Patterson AFB, OH, May 1975.

3. Wong, T. S., "Dioxin Formation and Destruction in Combustion Processes," presented at 77th Annual Meeting of the Air Pollution Control Association, San Francisco, CA, 24-29 June 1984.

4. Ackerman, D. G., Fisher, H. J., Johnson, R. J., Maddalone, R. F., Matthews, B. J., Moon, E. L., Schever, K. H., Shih, C. C., Tobias R. F. (TRW, Inc.), Venezia, R. A., (EPA), At Sea Incineration of Herbicide Orange Onboard the M/T Vulcanus, EPA-600/2-78-086, U. S. Environmental Protection Agency, April 1978.

5. Spencer, W. F., Farmer, W. J., and Cliath, M. M., Pesticide Volatilization, Agricultural Reserach Service, U.S. Department of Agriculture, 1973.

6. Thibodeaux, L. J., Chemodynamics-Environmental Movement of Chemicals in Air, Water, and Soil, John Wiley & Sons, NY, 1979.

7. Khan, S. U., Pesticides in the Soil Environment, Elsevier Scientific Publishing Company, NY, 1980.

8. Schnitzer, M. and Khan, S. U., eds., Soil Organic Matter, Elsevier Scientific Publishing Company, NY, 1978.

9. Schnitzer, M. and Hoffman, I., "Pyrolysis of Soil Organic Matter," in Division 5-3--Soil Microbiology, Soil Science Proceedings, 1964.

10. Flaig, W., Beutelspacher, H., and Reitz, E., in Soil Components - Organic Components, Vol. 1, Gieseking, J. E., ed., Springer-Verla, New York, NY, 1975.

11. Earnest, C. M., "Thermal Analysis of Minerals in Coal and Coal Ash," presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectrocopy, Atlantic City, NJ, 1981.

12. Earnest, C. M., The Application of Differential Thermal Analysis and Thermogravimetry to the Study of Kaolinite Clay Minerals, Perkin-Elmer Thermal Analysis Application Study 30, 1980.

13. Cudahy, J. J., et al., IT Corporation, Incineration Characteristics of RCRA Listed Hazardous Wastes, Contract No. 68-03-2568, USEPA Industrial Research Laboratory, Cincinnati, OH, July 1981.

14. Dellinger, B., et al., Determination of the Thermal Stability of Selected Hazardous Organic Compounds, USEPA, Combustion Research Facility, Jefferson, AR, 1984.

15. Freeman, R. A. and Schroy, J. M., "Environmental Mobility of Dioxins," presented at 8th ASTM Aquatic Symposium for Toxic Substances, Soil Science, 137:6, pp. 457-463, June 1984.

BATTELLE METHODS EMPLOYED IN THE PHYSICAL-CHEMICAL ANALYSES OF JI, EGLIN, AND NCBC SOILS

1A STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL ORGANIC MATTER

1B STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL PH

1C STANDARD OPERATING PROCEDURE FOR DETERMINATION OF ELECTRICAL CONDUCTIVITY IN SOILS

1D STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL SURFACE AREA

1E STANDARD OPERATING PROCEDURE FOR PARTICLE SIZE ANALYSIS OF SOILS

1F STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL CATION EXCHANGE CAPABILITY

1G METHOD FOR DETERMINATION OF TOTAL SOLVENT-EXTRACTABLE CONTENT OF SOILS AND SEDIMENTS

SEF 0-08-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL ORGANIC MATTER

1.0 Scope

This method detects the organic matter content of soils based on the oxidation of organic carbon by potassium dichromate. A colorimetric determination of the Cr^{3+} ion produced is used to determine the organic matter present based on the comparisons with standard soils of known organic matter. By adjusting the ratios of reagents and soil, the organic matter content of a wide variety of soils can be determined by this method (Schulte, 1980; Watson, 1978).

2.0 Summary of Method

Soils of known organic matter content and test soils are treated with potassium dichromate and sulfuric acid to oxidize organic carbon. After treatment with barium chloride, all solutions are allowed to stand overnight. The absorbance for each solution is read at 611 nm on a spectrophotometer and organic matter is determined from the standard curve.

3.0 Interferences

Chlorides in soils can reduce $Cr_2O_7^{2-}$, leading to Cr^{3+} and thus unrealistically high results. Ferrous iron can also lead to unrealistically high results. During air drying of soils, however, Fe^{2+} is oxidized to Fe^{3+}, minimizing the interference caused by Fe^{2+}. Oxides of Mn can oxidize soil organic matter, giving unrealistically low results. These interferences are not usually serious (Schulte, 1980).

4.0 Apparatus

4.1 Colorimeter. A high quality spectrophotometer capable of measuring absorbance at 611 nm, for detecting the presence of Cr^{3+}.

4.2 Flasks. 125 ml Erlenmeyer flasks acid washed, for mixing soil and reagents.

4.3 Balance. A top-loading balance for weighing soil to the nearest 0.01g.

4.4 Stir Rods. Glass stirring rod, for mixing the soil and reagents.

4.5 Pipettes. Glass volumetric pipettes of 10.0 and 20.0 ml capacity.

4.6 Graduated cylinder. 100 ml capacity graduated cylinder, for adding $BaCl_2$ solution to soils.

5.0 Reagents

5.1 2N potassium dichromate. Dissolve 98.07g $K_2Cr_2O_7$ per 1.0 l Barnstead water.

5.2 Concentrated sulfuric acid (96% H_2SO_4)

5.3 0.5% (w/v) barium chloride. Dissolve 5 g $BaCl_2$ in 1.0 l Barnstead water.

5.4 Organic matter standard. A soil of known organic matter content, from which various weights are taken to prepare the standard curve.

6.0 Calibration

Calibration for this procedure, aside from routine calibration of balances, involves careful preparation of the standard curve. Using the standard soil, weigh out 0, 0.5, 1.0, 1.5, 2.0, and 2.5 g portions into

4.2 Flasks. 125 ml Erlenmeyer flasks acid washed, for mixing soil and reagents.

4.3 Balance. A top-loading balance for weighing soil to the nearest 0.01g.

4.4 Stir Rods. Glass stirring rod, for mixing the soil and reagents.

4.5 Pipettes. Glass volumeteric pipettes of 10.0 and 20.0 ml capacity.

4.6 Graduated cylinder. 100 ml capacity graduated cylinder, for adding $BaCl_2$ solution to soils.

5.0 Reagents

5.1 2N potassium dichromate. Dissolve 98.07g $K_2Cr_2O_7$ per 1.0 l Barnstead water.

5.2 Concentrated sulfuric acid (96% H_2SO_4)

5.3 0.5% (w/v) barium chloride. Dissolve 5 g $BaCl_2$ in 1.0 l Barnstead water.

5.4 Organic matter standard. A soil of known organic matter content, from which various weights are taken to prepare the standard curve.

6.0 Calibration

Calibration for this procedure, aside from routine calibration of balances, involves careful preparation of the standard curve. Using the standard soil, weigh out 0, 0.5, 1.0, 1.5, 2.0, and 2.5 g portions into separate 125 ml Erlenmeyer flasks that have been acid washed. Handle the standards exactly as described below in Section 7.0, Procedures.

7.0 Procedures

Weigh 1.0g of air-dry, 2 mm mesh sieved soil into a 125 ml Erlenmeyer flask. Depending on the organic matter content, the amount of soil may have to be reduced. For soils expected to contain greater than 10% organic matter, but which are not truly organic soils, use a 0.5 g sample. For an organic soil, use a 0.1 g portion. To each flask (standards and samples), add 10 ml of 2N $K_2Cr_2O_7$ and swirl to mix. Then add 20 ml concentrated H_2SO_4 and swirl to mix well. Leave each flask undisturbed for 60 minutes. Next, in the same sequence as the acid was added, add to each flask 100 ml of 0.5% $BaCl_2$ solution. Leave the flasks undisturbed overnight. Set the spectrophotometer to 611 nm, the slit width to 2.0 nm, the lamp switches to visible, and the mode to absorbance. Turn on the spectrophotometer, allow sufficient time for warm-up (approximately 2 hours), and determine the absorbance of each solution at 611 nm, including all standards, blanks, and samples.

8.0 Calculations

Plot the standard curve on graph paper or via linear regression as percent organic matter versus absorbance at 611 nm. From the absorbance of the samples, the corresponding percent organic matter can be determined from the standard curve.

9.0 References

Schulte, E.E., 1980. Recommended soil organic matter test, pp 28-31. In: Recommended Chemical Soil Test Procedures for the North Central Region, North Central Regional Publication No. 221 (Revised). North Dakota Central Agricultural Experiment Station, North Dakota State University, Fargo, North Dakota.

Watson, M.E. 1978. Soil testing procedures, Research Extension Analytical Laboratory. Ohio Agricultural Research and Development Center, Wooster, Ohio 20 pp.

Approved by:

Mick F. Roth 7/25/84
Section 326 SUP Representative

A. Barker

Remma Mayer 7/23/84
R. Mayer

EEF 3-08-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL pH

1.0 Scope

Soil pH is a measure of the hydrogen ion concentration in the soil solution. This method determines soil solution pH when soils are mixed in a 1:1 ratio (weight:volume) with water. It has been recommended as a standard method for the determination of soil pH by the North Central Region Soil Testing Committee, which includes representatives from Alaska, Illinois, Indiana, Iowa, Kansas, Michigan, Minnesota, Missouri, Nebraska, North Dakota, Ohio, Pennsylvania, South Dakota, and Wisconsin (McLean, 1980).

2.0 Summary of Method

A soil solution is made by mixing equal parts of soil and distilled water. The hydrogen ion concentration is determined by using a glass indicating electrode paired with a calomel reference electrode. The combination electrode is attached to a pH meter and the unit is calibrated with standardized buffer solutions prior to use. pH of the soil solution is measured to the nearest 0.1 pH unit.

3.0 Interferences

Soil pH measurements can be influenced by a number of factors, depending upon soil type and individual characteristics. The use of a standard protocol that has been used on many different soil types, such as the standard method described here, minimizes interferences.

4.0 Apparatus

4.1 pH meter. A high quality pH meter, such as a digital display ion analyzer/pH meter.

4.2 Electrodes. A combination glass indicating and calomel reference electrode for sensing hydrogen ion activity.

4.3 Balance. A top-loading digital display balance accurate to 0.01 g for weighing soil.

4.4 Beaker. A beaker of approximately 30 ml capacity.

4.5 Stirring apparatus. A magnetic stir plate with stir bar for the constant mixing of soil and water while determining pH.

4.6 Pipette. A pipette of 10.0 ml capacity.

5.0 Reagents

5.1 Distilled or Barnstead water. Required to form a soil/water slurry.

5.2 Standard buffers. Commercially standardized buffer of pH 4.0 and 7.0, for standardizing the pH meter and electrode.

6.0 Calibration

Prior to determining soil pH, the pH meter must be standardized. Using the pH 7.0 standard buffer in a small beaker, adjust the digital display to read 7.0 by turning the calibration knob and while constantly stirring the beaker. Return the function knob to standby, remove the electrode from the pH 7.0 buffer, wash the electrode with distilled water, and submerge the electrode in constantly swirled pH 4.0 standard buffer. If the meter does not read pH 4.0, use the temperature compensator knob to achieve a reading of pH 4.0, wash the electrode as before, and standardize again with pH 7.0 buffer. Repeat the standardization procedure until the two readings are consistent.

7.0 Procedure

Weigh 10.0 g of air-dry sieved soil (passing a 2.00 mm screen) into a small beaker. Add 10.0 ml of distilled water, mix thoroughly with a glass rod, and allow the slurry to stand undisturbed for 10 minutes. Stir the soil suspension again and immediately submerge the pH electrode while continuing to swirl the beaker. Turn the function knob from standby to pH and read the pH to the nearest 0.1 pH unit. Return the switch to standby, remove the electrode, and rinse it thoroughly with distilled water.

8.0 Calculations

No calculations are required.

9.0 References

McLean, E.O. 1980. Recommended pH and lime requirement tests, pp. 5-8. In: Recommended Chemical Soil Test Procedures for the North Central Region, North Central Regional Publication No. 221 (Revised). North Dakota Agricultural Experiment Station, North Dakota State University, Fargo, North Dakota.

Approved by:

7/25/84
Section 325 SOP Representative

R. Barker

7/25/84
R. Mayer

SOP 0-08-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF ELECTRICAL CONDUCTIVITY IN SOILS

1.0 Scope

This method is designed to determine the electrical conductivity in a soil-water slurry, an indication of the soluble salts content of the soil. The method described below is applicable to most soils, including those in the North Central Region (Alaska, Illinois, Indiana, Iowa, Kansas, Michigan, Minnesota, Missouri, Nebraska, North Dakota, Ohio, Pennsylvania, South Dakota, and Wisconsin.) Procedures are adapted from the soil testing procedures used at the Research Extension Analytical Laboratory at the Ohio Agricultural Research and Development Center (Watson, 1978).

2.0 Summary of Method

A soil-water slurry (1:2 soil:water ratio) is prepared, allowed to equilibrate, filtered, and pipetted into a calibrated conductivity cell connected to a conductivity meter. The meter display is in conductance and is multiplied by the cell constant to give conductivity in micro- or millimhos/cm.

3.0 Interferences

Accurate conductance readings require a very clean conductivity cell. This can be accomplished between readings by thorough washing with distilled or Barnstead water. Periodically, the cell should be washed with a solution containing 100 ml isopropyl alcohol, 100 ml ethyl ether, 50 ml concentrated HCl, and 50 ml distilled water. For storage between uses, the conductivity cell should be soaked in Barnstead water.

4.0 Apparatus

4.1 Conductance meter. A high quality digital display conductance meter, e.g., a YSI Model 32.

4.2 Conductivity cell. A conductivity cell compatible with the conductivity meter and having a known cell constant, such as a YSI 3401 platinized platinum - iridium electrode with a cell constant of K= 1.0/cm.

4.3 Balance. A top-loading digital display balance accurate to 0.01 g. for weighing soil.

4.4 Beaker. A 150 ml beaker for preparing the soil-water slurry.

4.5 Funnel and Filter Paper. A glass or plastic funnel and filter paper for filtering the soil-water solution.

4.6 Pipette. Pipette or small graduated cylinder, for adding 40 ml water to 20g soil.

4.7 Stir rods. Glass stir rod, for mixing the soil-water slurry.

5.0 Reagents

5.1 Distilled or Barnstead water. Required to form a soil-water slurry.

6.0 Calibration

The YSI 3401 conductivity cell has a cell constant of 1.0/cm. If the cell is placed in a solution in an upright manner, no correction of the cell constant is required. If, however, the cell is inverted and the testing solution is pipetted into the cell, a correction factor needs to be calculated. To determine the corrected cell constant, immerse the cell in room temperature tap water, record the conductance, and multiply by the cell constant (1.0/cm). Remove the cell from the tap water, rinse with distilled water, invert the cell, and seal the vent hole in the cell by wrapping it in parafilm. Fill the cell with the same tap water used previously, record the conductance, and multiply by 1.0/cm to get conductivity. Subtract the two conductivity determinations, and divide the answer by the second conductivity determination. This final answer is the percent variation of the cell constant and when added to 1.0/cm is the corrected cell constant. All subsequent conductance measurements are to be multiplied by the corrected cell constant to determine the conductivity.

7.0 Procedure

Weigh 20.0 grams of air-dry, 2mm mesh sieved soil into a 150 ml beaker. Add 40.0 ml distilled or Barnstead water, stir thoroughly with a glass rod, and allow the contents to stand undisturbed for 30 minutes. Prepare a funnel and filter paper (e.g., Whatman 1). Pour the supernatant through the filter funnel, being careful not to disturb the settled soil in the bottom of the beaker. After the filter has drained, fill a calibrated conductivity cell with the filtrate (or immerse the upright cell in the filtrate if the volume of filtrate is great enough to submerge the cell), turn the knob on the conductivity meter to the conductance mode, adjust the range switch until a digital display is obtained, and record the conductance.

8.0 Calculations

Multiply the conductance by the appropriate cell constant to obtain conductivity in milli- or micromhos.

9.0 References

Watson, M.E. 1978. Soil Testing Procedures, Research Extension Analytical Laboratory. Ohio Agricultural Research and Development Center, Wooster, Ohio 20 pp.

Approved by:

7/25/84
Section 920 SUP Representative

A. Barker

7/25/84
A. Mayer

Annex 1D

EEF 0-08-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL SURFACE AREA

1.0 Scope

This method determines the specific surface area of soils based on the adsorption by soil particles of a monolayer of ethylene glycol monoethyl ether (EGME). Adsorption is based on an equilibration of EGME and soil particles followed by vacuum desiccation of unsorbed EGME. Previous studies in the soil science community have shown that the method is applicable to widely different soil types (Carter et. al., 1965; Heilman, et. al., 1965; Cihacek and Bremner, 1979).

2.0 Summary of Method

Air-dried sieved soil is mixed with reagent grade EGME and equilibrated for 1 hour. The sample is then vacuum desiccated to constant weight over desiccant. Surface area is calculated by dividing the grams of adsorbate retained per gram of soil by 0.000286 g/m^2, which is the weight of EGME required to form a monolayer over $1m^2$ surface area.

3.0 Interferences

There are no inferences described in the literature using this procedure.

4.0 Apparatus

4.1 Balance. An electronic (digital read out) balance accurate to 0.01 g for weighing soil samples prior to the addition of EGME, and a balance accurate to 0.001 g for weighing desiccated soils.

4.2 Weighing pans. Shallow aluminum weighing pans for equilibrating soil and EGME.

4.3 Desiccator. A vacuum desiccator plus desiccant and vacuum pump for drying soils.

4.4 Pipettes. Glass or plastic pipettes capable of delivering 3 ml of EGME.

4.5 Soil sieve. A soil sieve with a mesh opening of 2.00 mm for sieving air-dry soil.

5.0 Reagents

5.1 Ethylene glycol monoethyl ether (2-ethoxyethanol). Reagent grade EGME for sorbing to soil surfaces; approximately 3.0 ml per soil sample.

5.2 Calcium sulfate. Several hundred grams of $CaSO_4$ (drierite) as a desiccant for drying EGME-equilibrated soil samples.

6.0 Calibration

The calibration procedures involved in this method include those associated with balances. These are performed on a periodic basis by the BCL instrument laboratory.

7.0 Procedure

Air-dry the soil. Sieve the air-dried soil to pass a 2.00 mm mesh screen. Obtain tare weights of triplicate labelled aluminum weighing pans that were placed overnight in a desiccator containing $CaSO_4$ desiccant. Weights should be obtained to the nearest 0.001 g. To each of the tared triplicate weighing pans add 1.1 g of air-dry sieved soil, using the top-loading electronic balance (accurate to 0.01g). Place the pans of soil into a desiccator containing $CaSO_4$, evacuate the desiccator for 45 minutes with a vacuum pump and shut off the vacuum lines to maintain the vacuum in the desiccator. After at least 1 hour, weigh the pans of soil, evacuate the desiccator again for 45 minutes, and weigh again at least 1 hour later. Continue this procedure until a constant weight is obtained (to nearest 0.001 g).

After constant weight has been obtained, add 3.0 ml of reagent grade EGME to each pan of soil to form a slurry. After 1 hour of equilibration of the slurry, place the pans of soil in the desiccator and evacuate for 45 minutes. After maintaining the vacuum for an additional hour, obtain weights (to the nearest 0.001 g) for each pan, place the pans back into the desiccator, evacuate for 45 minutes, and allow the pans to sit overnight under vacuum desiccation. Weigh the pans again, evacuate the desiccator for 45 minutes, and reweigh one hour later. Continue this procedure until a constant weight is obtained. Record all weights in ink in the laboratory record book.

8.0 Calculations

For each pan, calculate the total surface area (TSA) by dividing the grams of EGME adsorbed per gram of soil by $0.000286 g/m^2$, the Dyal-Hendricks value for the weight of EGME required to form a monolayer on a surface area of $1 m^2$. That is,

$$TSA = \frac{a - b}{(0.000286\ g/m^2)(b - c)}$$

where: a = weight of desiccated soil, EGME, and pan;
b = weight of desiccated soil and pan;
c = tare weight of pan.

9.0 References

Carter, D.L., M.D. Heilman, and C.L. Gonzalez. 1965. Ethylene glycol monoethyl ether for determining surface area of silicate minerals. Soil Sci. 100:356-360.

Heilman, M.D., D.L. Carter, and C.L. Gonzalez. 1965. The ethylene glycol monoethyl ether (EGME) technique for determining soil-surface area. Soil Sci. 100:409-413.

Cihacek, L.J., and J.M. Bremner. 1979. A simplified ethylene glycol monoethyl ether procedure for assessment of soil surface area. Soil Sci. Soc. Am. J. 43:821-822.

Approved by:

Section 325 SOP Representative

A. Barker

R. Mayer

Annex 1E

SOP 0-12-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR PARTICLE SIZE ANALYSIS OF SOILS

1.0 Scope

This method describes a standard way to determine the particle size distribution in soils. It is adapted from an ASTM procedure (ASTM, 1972; ASTM, 1978) and is widely applicable to soils.

2.0 Summary of Method

Air-dry soil is analyzed for particle size distribution based on sieving (for those particles larger than 75 um) or by a sedimentation process (those particles passing the 75 um sieve). A hydrometer is used in the sedimentation procedure.

3.0 Interferences

Not applicable.

4.0 Apparatus

4.1 Balance. A balance sensitive to 0.01 g for weighing the material passing a 75 um sieve and for weighing the material retained on larger sieves.

4.2 Mortar and Pestle. A ceramic mortar and pestle for breaking up larger soil clumps.

4.3 Hydrometer. An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per liter of suspension.

4.4 Sedimentation Cylinder. A glass 1000 ml graduated cylinder, for performing sedimentation analyses.

4.5 Thermometer. A thermometer accurate to 1°F (0.5°C).

4.6 Soil Sieves. A series of sieves, of square-mesh woven-wire cloth, as follows:

No. 4	4.75 mm
No. 10	2.00 mm
No. 20	850 um
No. 40	425 um
No. 60	250 um
No. 140	106 um
No. 200	75 um

4.7 Beaker. A beaker of 250 ml capacity.

4.8 Timing Device. A watch or clock with a second hand.

4.9 Electric Stirring Apparatus. A high-speed electric stirring device, such as a "milk shake stirrer."

5.0 Reagents

5.1 Dispersing Agent. A solution of sodium hexametaphosphate, freshly prepared by dissolving 40 g of sodium hexametaphosphate per 1.0 liter of Barnstead water.

6.0 Calibration

The hydrometer should be calibrated in distilled water and in a solution containing sodium hexametaphosphate. Bring a 1000 ml glass graduated cylinder to volume with Barnstead water. To another 1000 ml glass graduated cylinder add 125 ml of a solution of sodium hexametaphosphate (40 g/l) and bring to volume with Barnstead water, with constant mixing. After assuring that the cylinders of liquid are at room temperature (e.g. 22°C), add the hydrometer to the cylinder containing water and record the value as read at the top of the meniscus. Ideally this should be 0. Next, put the hydrometer in the cylinder containing the sodium hexametaphosphate solution and record the reading at the top of the meniscus. The difference between the readings is called the composite correction factor and is to be used in the calculations (Section 8.0).

7.0 Procedure

Spread the soil sample in an exhaust hood so that it will air-dry. When the soil is air-dry and can be ground in a mortar and pestle, mix the sample thoroughly to assure uniformity, gently grind the soil in the mortar and pestle until soil particles are broken up (do not grind soil and gravel particles to a fine powder), and mix the sample again. Weigh triplicate portions (about 5.0 g), oven-dry them (105°C for 24 hours) and re-weigh them to determine moisture content. Using sieves No. 4 and 10, separate the soil sample into three fractions: greater than 4.75 mm diameter, less than 4.75 mm but greater than 2.00 mm diameter, and less than 2.00 mm diameter. Record the weights of all size fractions. The results constitute the portion greater than 2.00 mm diameter.

The portion passing the 2.00 mm sieve is to be fractionated further. Add 50 g of the less than 2.00 mm air-dry soil (or more accurately, soil of known moisture content), to a 250 ml beaker. Cover the soil with 125 ml of sodium hexametaphosphate solution (40 g/l) and stir until the soil is thoroughly wet. Allow the mixture to sit at room temperature for at least 16 hours.

At the end of the soaking period, disperse the soil further by stirring for one minute with a high speed electric "milk shake stirrer." Immediately transfer the soil-water slurry to a glass 1000 ml graduate cylinder and bring to volume with Barnstead water. Cover the cylinder with parafilm (2 layers) and repeatedly invert and upright the cylinder for 1 minute. During this mixing, make sure all soil is being dispersed and that none settles in the cylinder. After the 1 minute period, place the cylinder on a level, sturdy surface at room temperature. Record the time of day and after 2, 5, 15, 30, 60, 250, and 1440 minutes, use an ASTM-approved hydrometer to obtain a sedimentation reading. Approximately 20 to 25 seconds before a reading is done, carefully insert the hydrometer into the cylinder. Read the hydrometer at the top of the meniscus formed by the suspension around the stem of the hydrometer and immediately remove the hydrometer from the cylinder.

After the final hydrometer reading is taken, i.e. at 1440 minutes, pour the suspension onto a 75 µm sieve (No. 200), wash the sieve with water until the wash water is clear, transfer the material to a suitable container such as a beaker, and dry in an oven at 105°C overnight. After the oven-drying is complete, fractionate the dried material on the following sieves: No. 20, 40, 60, 140, and the bottom pan, weighing the portion retained by each.

8.0 Calculations

The moisture content of the original soil sample is calculated as follows:

$$\frac{(\text{air-dry weight of soil + pan}) - (\text{oven-dry weight of soil + pan})}{(\text{oven-dry weight of soil + pan}) - (\text{weight of empty pan})}$$

Results can be expressed as percentage moisture by multiplying by 100.

Calculate the portion greater than 4.75 mm (A), between 4.75 mm and 2.00 mm (B), and less than 2.00 mm (C), as follows:

$$\frac{\text{Fraction in question (in grams)}}{\text{Total grams of soil } (A + B + C)} \times 100$$

The percentage (P) of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension is calculated as follows:

$$P = \left[1 - \left(\frac{Ra}{W}\right)\right] \times 100$$

where:

R = hydrometer reading minus the composite correction,

a = a correction factor for the model of hydrometer used, and in most cases equals 1.00 for a specific gravity of 2.65 for soil,

W = oven-dry mass of soil dispersed in the hydrometer test

The diameter (D) of the particles corresponding to the percentage indicated by a given hydrometer reading is calculated as follows:

$$D = K \ (L/T)^{1/2}$$

where:

K = a constant that is dependent on temperature and specific gravity. At 22°C and 2.65 specific gravity, the value of K is 0.01332. Other values can be obtained from Table 3 of ASTM, 1972.

L = distance from the surface of the suspension to the level at which the density of the suspension is measured, in cm; this value is known as the effective depth and requires the use of Table 2 in ASTM, 1972.

T = interval of time from beginning of sedimentation to the taking of the reading; in min.

9.0 References

ASTM, 1972. Standard method for particle-size analysis of soils. ASTM D422-63, Washington, D.C.

ASTM, 1978. Standard method for dry preparation of soil samples for particle-size analysis and determination of soil constants. ASTM D421-58, Washington, D.C.

Approved by:

7/25/84
Section 326 SOP Representative

A. Barker

7/25/84
R. Mayer

Annex 1F

SOP 0-13-1
July 17, 1984

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SOIL CATION EXCHANGE CAPACITY

1.0 Scope

This method is designed to determine the cation exchange capacity in soils, which indicates the capacity of soils to bind important plant nutrients. It is based on the displacement from exchange sites of nutrient cations such as Ca, K, and Mg, with an extracting cation. The extracting cation is then displaced and analyzed in the leachate. Although several different cations can be employed to displace the soil cations, the cation of choice should be low in the soil to prevent low CEC determinations. Therefore, ammonium is used most widely and is employed in this procedure.

2.0 Summary of Method

Ammonium acetate is added to a known quantity of soil to both displace the bound cations and to saturate the exchange sites. An ethanol solution is used to wash the soil, followed by the displacement of NH_4^+ ions by KCl. The displaced NH_4^+ ions are analyzed using an ammonium ion-specific electrode, and the concentration of ammonium ions in the leachate is proportional to the K^+ ions bound to the soil and thus to the CEC.

3.0 Interferences

To provide accurate CEC determination the displacing ion should not be present in high concentrations in the soil. In addition, monovalent ions have proven to give less variable results than divalent displacing cations, such as calcium or magnesium.

4.0 Apparatus

4.1 Balance. A top-loading digital display electronic balance, or similar balance or equal accuracy, for weighing soils to the nearest 0.01 g.

4.2 Shaker. A shaker capable of holding a 125 ml Erlenmeyer flask, to provide constant shaking for 1 hour.

4.3 Filter Flask and Funnel. A 250 ml filter flask with a Buchner funnel, for washing the soil sample.

4.4 Filter Paper. Whatman number 44 filter paper (or similar paper) that fits the Buchner funnel, for washing the soil.

4.5 Volumetric Flasks. A 100 ml volumetric flask, for adjusting to volume the displaced NH_4^+ solution, and 1.0 l and 500 ml volumetric flasks for preparing reagents.

4.6 Ammonium ion-specific electrode. An Orion ammonium ion-specific electrode and digital display pH/millivolt meter such as an Orion 601A.

5.0 Reagents

5.1 Ammonium acetate. One molar solution, made by dissolving 77.1 g ammonium acetate in a total of 1.0 liter of Barnstead water in a volumetric flask. Adjust to pH 7.0, using glacial acetic acid and a standardized pH meter.

5.2 Industrial Alcohol. Mix 60 ml denatured ethanol with 40 ml Barnstead water.

5.3 Potassium Chloride. A 5% solution, prepared by dissolving 50.0 g KCl in a total of 1.0 l Barnstead water.

6.0 Calibration

Calibration for this procedure includes the preparation of a standard curve for the ammonium ion-specific electrode. To prepare the standard curve, first prepare standard solutions containing 1, 10, 100, and 1000 ppm NH_4Cl from the 0.1 M NH_4Cl Orion standard. Add 93.5 ml of the Orion standard to a 500 ml volumetric flask and bring to volume with Barnstead water, to give a 1000 ppm NH_4^+ standard. Add 50.0 ml of the 1000 ppm standard to a 500 ml volumetric flask and bring to volume with Barnstead water, to give a 100 ppm standard solution. Add 50.0 ml of the 100 ppm standard to a 500 ml volumetric flask and bring to volume, giving a 10 ppm standard. Prepare the 1.0 ppm standard by adding 50.0 ml of the 10 ppm standard to a 500 ml volumetric flask and bringing to volume.

Before determining the standard curve, check the electrode slope as follows. Mix 100 ml Barnstead water and 1.0 ml of 10 M NaOH solution (Orion standard) in a 150 ml beaker. Turn the function switch on the model 501A pH/mV meter to relative millivolt, place the electrode in the beaker, and stir the beaker constantly with a magnetic stir bar. Now pipet 1.0 ml of 1000 ppm standard into the beaker and adjust the reading to 000.0 with the calibration knob. Next, add 10.0 ml of 1000 ppm standard into the beaker. Correct electrode operation is indicated by a reading of -57 (±3) mV.

To prepare the standard curve, add approximately 7 ml of 1 ppm standard to a 10 ml beaker, add a small magnetic stirring bar, and stir the solution constantly. Place the electrode in the solution, being careful not to trap bubbles under the electrode. Add 0.1 ml (100 1) of 10 M NaOH to the beaker, set the function switch to rel mv, and record the display. Return the function switch to stand-by, remove the electrode, rinse thoroughly with Barnstead water, and repeat the above procedures for the remaining standard solutions. Plot the millivolt reading on the ordinate (linear axis) against concentration on the abcissa (log axis) on semilogarithmic paper.

The standard curve should be recalibrated every two hours during the analysis.

7.0 Procedure

Add 5.0 g air-dried, sieved (<2.00 mm) soil to a 125 ml Erlenmayer flask. Add 125 ml of 1M ammonium acetate and shake the mixture for ½ hour at room temperature. Using Whatman 44 filter paper, repeatedly wash the soil under vacuum filtration in a Buchner funnel with portions of industrial ethanol solution. Use at least 100 ml of the ethanol solution and reject all washings. Leach the soil with small (20-30 ml) portions of 5% KCl until a total of 100 ml (in a volumetric flask) has been collected. Determine the NH_4^+-N concentration in the leachate using the ammonium ion-specific electrode as described earlier for the preparation of the standard curve.

8.0 Calculations

CEC should be expressed as milliequivalents per 100 g of dry soil. The conversion from ppm (g/ml) NH_4^+-N as determined with the ammonium electrode to CEC is as follows:

$$\text{CEC (meq/100 g)} = \frac{(0.111)A}{(1 - B)}$$

where:

A = ppm NH_4^+-N from the standard curve, and

B = moisture content of the soil, expressed as a decimal fraction rather than percent.

The derivation for this formula is as follows:

CEC (expressed in meq/g dry soil) =

$$\frac{(\text{ug/ml } NH_4^+\text{-N})(\text{100 ml leachate})(\text{1 mg/1000 ug})}{(\text{5 g air-dry soil})(\text{1-moisture content})(\text{18 mg/meq})}$$

9.0 References

Allen, S. E., H. M. Grimshaw, J. A. Parkinsen, and C. Quarmby. 1974. Chemical Analysis of Ecological Materials. Wiley and Son, New York. 565 pp.

Approved by:

7/25/84
Section 326 SUP Representative

A. Barker

7/25/84
R. Mayer

METHOD FOR DETERMINATION OF TOTAL SOLVENT-EXTRACTABLE CONTENT OF SOILS AND SEDIMENTS

1. Scope

1.1 This method covers the extraction and quantification of the total solvent-extractable content of soils and sediments at concentrations above 10 ug/g.

1.2 The method is suitable for the extraction of most phenols, anilines, and neutral semivolatile organic compounds.

1.3 Low boiling organic components are lost by evaporation and are not included in the total solvent-extractable content.

2. Summary of Method

2.1 The wet or dry soil or sediment sample is mixed with aqueous sodium chloride/potassium dihydrogen phosphate and equilibrated with methyl tert-butyl ether (MTBE). The MTBE extract is dried and concentrated. The concentrated extract is filtered to remove any particulate material. The solvent is evaporated from a portion of the concentrated extract and the residue weight is determined.

3. Interferences

3.1 Relatively large amounts of extractable components boiling below 200°C will make it difficult to obtain reproducible residue weights. Relatively large amounts of extractable high molecular weight polymeric material may interfer with the filtration of the concentrated extract.

4. Apparatus

4.1 Separatory Funnel — 100-mL with Teflon stopcock.

4.2 Kuderna-Danish Apparatus — 25-mL concentrator tube fitted with a micro-Snyder column.

4.3 Weighing Dish — approximately 2-cm diameter aluminum foil.

4.4 Syringe — 2-mL gas-tight with Teflon Luer Lock.

4.5 Filter Assembly -- 25-mm disposable, with Millipore Millex-SR, 0.5 um PTFE membrane filter.

4.6 Boiling Chips -- approximately 10-40 mesh carborundum.

4.7 Water Bath -- heated, capable of temperature control of ± 2°C. The bath should be used in a hood.

4.8 Microbalance -- capable of accurately weighing to ± 0.001 mg.

4.9 Rotary Mixer -- capable of rotating a 6-oz sample bottle end-over-end at approximately 30 rpm.

5. Reagents

5.1 Salt Solution -- Dissolve 300 g of NaCl and 20 g of KH_2PO_4 in 800 mL of water and dilute to one liter with water.

5.2 Methyl tert-Butyl Ether (MTBE) -- Distilled-in-glass grade.

5.3 Magnesium Sulfate -- Anhydrous powder.

5.4 Corn Oil Standard, high level -- Dissolve 500 mg of corn oil in 80 mL of MTBE and dilute to 100 mL with MTBE.

5.5 Corn Oil Standard, low level -- Dilute 10 mL of the high level corn oil standard to 100 mL with MTBE.

6. Quality Assurance

6.1 Blank -- Determine the residue weights (7.6) of triplicate 100 uL aliquots of MTBE. An average value of 0.0 ± 10 ug should be obtained.

6.2 Corn Oil Standards -- Determine the residue weights (7.6) of triplicate 100-uL aliquots of the low level corn oil standard and the high level corn oil standard. Average values of 50 ± 10 ug and 500 ± 25 ug should be obtained for the low level and high level standards, respectively.

7. Procedure

7.1 Extraction -- Add 30 mL of 30% NaCl/2% KH_2PO_4 and 30 mL of MTBE to 30 g of soil in a 6-oz wide-mouth bottle having a Teflon-lined screw cap. Tumble the bottle end over end for 16 hours. Weigh the bottle before and after the tumbling period to determine any decrease in gross weight from loss of solvent. If the gross weight has decreased by more than 0.5 g, the sample must be discarded.

7.2 Phase Separation -- Let the sample stand to allow the phases to separate. Decant the MTBE and water layers into a 100-mL separatory funnel and allow the phases to separate for 10 minutes. Withdraw the aqueous layer and discard.

7.3 Drying -- Transfer the MTBE extract to a 2-oz narrow-mouth bottle having a Teflon-lined screw cap. Add 2 g of anhydrous magnesium sulfate, shake the sample vigorously for one minute, and allow the sample to stand for at least 5 minutes.

7.4 Concentration -- Transfer 25 mL of the clear extract to a Kuderna-Danish apparatus, add two boiling chips, and concentrate the extract to 0.5-0.8 mL in a water bath at 80-90°C.

7.5 Filtration -- Transfer the concentrated extract to the barrel of a 2-mL syringe fitted with a Millipore Millex-SR 0.5 um PTFE membrane filter. Insert the plunger of the syringe and express the filtrate into a 1-mL volumetric flask. Rinse the K-D tube with 0.2 mL of MTBE, transfer the rinse to the syringe and filter the rinse into the volumetric flask. Dilute to volume by the addition of MTBE and mix thoroughly.

7.6 Determination of Residue Weight -- Transfer 100 uL of the extract to an aluminum weighing dish that has been tared on a microbalance; place the dish under a heat lamp at a distance of 8 cm from the lamp for one minute to allow the solvent to evaporate; transfer the dish to the microbalance; allow the dish to equilibrate for one minute; and, weigh to determine the residue weight.

separate 125 ml Erlenmeyer flasks that have been acid washed. Handle the standards exactly as described below in Section 7.0, Procedures.

7.0 Procedures

Weigh 1.0g of air-dry, 2 mm mesh sieved soil into a 125 ml Erlenmeyer flask. Depending on the organic matter content, the amount of soil may have to be reduced. For soils expected to contain greater than 10% organic matter, but which are not truly organic soils, use a 0.5 g sample. For an organic soil, use a 0.1 g portion. To each flask (standards and samples), add 10 ml of 2N $K_2Cr_2O_7$ and swirl to mix. Then add 20 ml concentrated H_2SO_4 and swirl to mix well. Leave each flask undisturbed for 60 minutes. Next, in the same sequence as the acid was added, add to each flask 100 ml of 0.5% $BaCl_2$ solution. Leave the flasks undisturbed overnight. Set the spectrophotometer to 611 nm, the slit width to 2.0 nm, the lamp switches to visible, and the mode to absorbance. Turn on the spectrophotometer, allow sufficient time for warm-up (approximately 2 hours), and determine the absorbance of each solution at 611 nm, including all standards, blanks, and samples.

8.0 Calculations

Plot the standard curve on graph paper or via linear regression as percent organic matter versus absorbance at 611 nm. From the absorbance of the samples, the corresponding percent organic matter can be determined from the standard curve.

9.0 References

Schulte, E.E., 1980. Recommended soil organic matter test. pp 29-31. In: Recommended Chemical Soil Test Procedures for the North Central Region, North Central Regional Publication No. 221 (Revised). North Dakota Central Agricultural Experiment Station, North Dakota State University, Fargo, North Dakota.

Watson, M.E. 1978. Soil testing procedures. Research Extension Analytical Laboratory. Ohio Agricultural Research and Development Center, Wooster, Ohio 20 pp.

Approved by:

7/25/84
Section 525 SUP Representative

A. Barker

7/25/84
R. Mayer

Annex 2

BATTELLE FINAL REPORT ON
PHYSICAL-CHEMICAL CHARACTERIZATION
OF UNCONTAMINATED SOILS FROM
JI, EGLIN, AND NCBC

FINAL REPORT

on

PHYSICAL-CHEMICAL CHARACTERIZATION OF SOILS

to

IT ENVIROSCIENCE

December 12, 1984

by

M.F. Arthur and T. C. Zwick

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Final Report

on

Physical-Chemical Characterization of Soils

to

IT Enviroscience

INTRODUCTION

IT Enviroscience (IT) submitted three soil samples to Battelle's Columbus Laboratories for selected physical and chemical characterization. The analyses to be completed included pH, electrical conductivity, organic matter content, cation exchange capacity, moisture content, surface area, oil and grease (solvent extractable) content, and particle size distribution. This report presents the results of this project in tabular and brief descriptive form.

Materials and Methods

The methods employed were the same as those included in the appendix of the previous Final Report to IT on project 526-J-8687. All analyses were performed in duplicate with the exception of surface area, in which five replicates were used.

Results and Discussion

Table 1 presents a comparison of the three IT soils and is largely self-explanatory. Each characteristic is discussed briefly below.

pH

Sample IT 4543 was considerably more acidic than the other two samples. Samples IT 4540 and IT 4569 exhibited similar pH readings, even though their physical appearances were considerably different from one another. That is, while IT 4540 resembled a normal soil, sample IT 4569 resembled ground limestone, which would be expected to exhibit a high pH. The three pH values, however, are not atypical for widely dispersed soils.

Electrical Conductivity

Electrical conductivity (EC) is an indication of soluble salts content. Samples IT 4540 and IT 4543 showed very low EC readings compared to sample IT 4569. Nevertheless, all three soils are low in soluble salts compared to truly saline soils (e.g., an EC greater than 10 millimhos/cm).

Organic Matter

All three soils were very low in organic matter content. Samples IT 4543 and IT 4569 were among the lowest we have measured. A typical midwestern agricultural soils may have an organic matter content of 4 to 7 percent, while truly organic soils, such as a Histosol in southern Florida, may have organic matter contents of 50 percent or more.

Cation Exchange Capacity

Cation exchange capacity (CEC) is a measure of the ability of a soil to bind important nutrient cations, such as calcium, magnesium, and potassium. A soil with a high CEC, e.g., 10 to 15 milliequivalents/100 g or more, generally is more fertile than soils exhibiting low CECs. The IT soils all showed low CECs, with samples IT 4543 and IT 4569 showing comparably low values. These results compare well with the organic matter contents and illustrate the influence of organic matter on CEC.

Moisture Content

The values for moisture content in Table 1 reflect the air-dry nature of the samples as received. The main reason for determining the moisture content of the air-dry samples is to correct the results of various analyses to reflect oven-dry weights of soil.

Surface Area

The surface areas of the three soils samples are considerably less than the soil sample previously analyzed for IT. This is the result of the relatively low proportion of clay in the three samples most recently analyzed (see Particle Size Distribution).

Oil and Grease (Solvent-extractable) Content

Sample IT 4540, with an average solvent-extractable content of 1759 micrograms/g, was considerably higher in total solvent-extractable content than the other two samples. The origin of the solvent extractable content, whether anthropogenic or natural, is not indicated by the analysis performed. Nevertheless, it is likely that sample IT 4540 has been subjected to a level of anthropogenic input of oil and grease. Some other analysis, such as gas chromatography-mass spectroscopy, would be required to identify the materials making up the solvent extractable content.

Particle Size Distribution

The results of the particle size analysis are for the less-than-2.00-mm fraction since the samples as received were air-dry and sieved

TABLE 5. PHYSICAL-CHEMICAL ANALYSES OF THREE IT SOIL SAMPLES

Physical-Chemical Parameters(a)	[illegible] IT [illegible]	EGLIN IT 4503	JI IT 4500
1. pH (± standard deviation)	[illegible] ± 0.021	5.21 ± 0.049	8.24 ± 0.036
2. Conductivity (millimhos/cm ± standard deviation)	0.279 ± 0.004	0.0418 ± 0.001	4.99 ± 0.148
3. Organic Matter (percent ± standard deviation)	1.5 ± 0.07	< 0.50	< 0.50
4. Cation Exchange Capacity (milliequivalents/100 g ± standard deviation)	3.9 ± 0.05	0.15 ± 0.008	0.12 ± 0.02
5. Moisture Content (percent ± standard deviation)	0.40 ± 0	0.12 ± 0.01	1.06 ± 0.13
6. Surface Area (m^2/g ± standard deviation)	12.3 ± 1.96	2.46 ± 0.706(b)	6.74 ± 1.47
7. Oil and Grease Content (micrograms/g)	1759 ± 44.5	116.0 ± 4.2	664.9 ± 30.5
8. Particle Size Distribution (percent ± standard deviation)			
a. Medium Sand (between 425 microns and 2.00 mm)	25.7 ± 2.65	41.3 ± 3.60	41.3 ± 3.04
b. Fine Sand (between 75 and 425 microns)	59.4 ± 3.13	51.9 ± 3.39	36.2 ± 1.56
c. Silt (between 5 and 75 microns)	12.3 ± 1.03	4.77 ± 0.23	19.2 ± 1.91
d. Clay (smaller than 5 microns, including colloids) (c)	2.70 ± 0.42	2.03 ± 0.52	3.31 ± 0.42
Colloids (less than 1 micron)	2.25 ± 0.36	1.67 ± 0.47	2.94 ± 0.30

(a) All analyses were done in duplicate with the exception of surface area, which was done in replicates of five.

(b) This mean and standard deviation represent four replicates. A fifth replicate yielded unreasonably high results compared to the other four replicates. If the fifth value is included, the surface area for sample IT 4503 is calculated as 3.92 ± 3.32 m^2/g.

(c) Note that the clay fraction includes the colloid fraction, so that the percentages total greater than 100 percent.

to 2.00-mm or less. All three soils had fairly high contents of medium and fine sands, and fairly low clay contents. Based on these results, the less-than-2.00-mm fraction of IT 4540, IT 4543, and IT 4569 are classified as loamy sand, sand, and loamy sand to sandy loam, respectively. These results also explain the relatively low values for CEC, organic matter, and surface areas, which are those parameters influenced considerably by the extent of the clay-size fraction in a soil.

CONCLUSION

Although the three soils included in this study represent non-contaminated background soils, the results of the physical-chemical characterization should prove useful to IT for understanding the fate of 2,3,7, 8-tetrachlorodibenzodioxin in similar soils.

Annex 3

BATTELLE DATA TABLE FOR PHYSICAL-CHEMICAL CHARACTERIZATION OF CONTAMINATED SOILS FROM JI, EGLIN, AND NCBC

TABLE 8. PHYSICAL-CHEMICAL ANALYSIS OF THREE IT SOIL SAMPLES (contaminated soil)

Physical-Chemical Parameters(a)(b)	437-3-EAFB	437-4-II	437-13-MCRC-1
1. pH (± standard deviation)	3.83 ± 0.035	8.45 ± 0.071	8.55 ± 0.071
2. Conductivity (millimhos/cm ± standard deviation)	0.146 ± 0.003	5.02 ± 0.404	0.205 ± 0.71
3. Organic Matter (percent ± standard deviation)	1.22 ± 0.042	4.22 ± 0.141	2.34 ± 0.177
4. Cation Exchange Capacity (milliequivalents/100 g ± standard deviation)	0.77 ± 0.078	0.73 ± 0.16	2.45 ± 0.95
5. Moisture Content (percent ± standard deviation)	0.56 ± 0.78	0.34 ± 0.48	0.38 ± 0.63
6. Oil and Grease Content (micrograms/g)	6069.0 ± 946.1	1804.5 ± 863.4	3106.5 ± 629.6

(a) All analyses were done in duplicate.

(b) All methods used are the same as those described in detail in Battelle's Final Report to IT Enviroscience on project 526-J-0687, dated July 25, 1984.

Annex 4

ALLIS-CHALMERS REPORT ON SMALL BATCH KILN TEST SIEVE ANALYSIS AND BULK DENSITY MEASUREMENTS OF JI, EGLIN, AND NCBC SOILS

ALLIS-CHALMERS
PROCESS RESEARCH & TEST CENTER
9180 FIFTH AVENUE

SMALL BATCH KILN TEST AND COMPUTER STUDY

For

IT CORPORATION

Test Center Project No. 85-003

Charge No. 01-6712-52357

Date Performed: January 9-11, 1985

Date Reported: January 30, 1985

Reported By: K. K. Mak

TABLE OF CONTENTS

Page No.

INTRODUCTION. 1

CONCLUSION. 1

TESTWORK AND COMPUTER STUDY SUMMARY 1

APPENDICES:

Appendix A - Small Batch Kiln Test Results

B - Small Batch Kiln Test Time-Temperature Curves

C - Bulk Densities of Soil and Product

D - Screen Analysis of Soil and Product

E - Dynamic Angle of Repose

F - TGA and DSC Test Results

INTRODUCTION

In accordance with Subcontracting Agreement and Purchase Order No. 4-12-27-01 from IT Corporation, Allis-Chalmers Energy and Minerals Systems Company performed a rotary kiln study for IT Corporation.

The objective of the study is to determine the behavior of contaminated soil on pyrolysis, and the degree of exhaust dust carry over.

The study included small batch kiln tests using three uncontaminated soils supplied by IT Corporation, and kiln heat transfer computer study.

The small batch kiln tests were performed at Allis-Chalmers Process Research & Test Center during the period of January 9-11,1985. These tests were witnessed by Mr. J. L. Fleming of IT Corporation.

CONCLUSION

Based on the small batch kiln test result, the following conclusions have been obtained:

1. All three(3) soils (4761, 4760, 4768) did not show any signs of sticking, slagging or balling when kiln gas temperature of 1038 °C was used.
2. Kiln exhaust gas dust increased with higher gas velocity through kiln.
3. Material 4768 tend to slide in kiln instead of turning over bed motion.

TESTWORK AND COMPUTER STUDY SUMMARY

Material Description

Three 5 gallon pails of uncontaminated soil were received at the Allis-Chalmers Process Research & Test Center. They were opened at the presence of Mr. J. L. Fleming.

Soil 4761 looked like a clay structure with a few occasional 2 or 3 inches lumps. Soil 4760 looked like a sandy soil. Soil 4768 looked like a sandy soil with large amounts of embedded sea shells.

Moisture Content

The moisture content of the as received soils are shown below:

Soil 4761	6.5% Moisture
Soil 4760	1.2% Moisture
Soil 4768	6.4% Moisture
Soil 4768	5.0% Moisture (oven dried at 100 °C overnight)

Small Batch Kiln Test

Eleven small batch kiln tests were performed and witnessed by Mr. J. L. Fleming of IT Corporation. Total retention time for each heat was set at 60 minutes. Kiln speed was 2.8 rpm and bed loading was around 7 percent. A summary of the test conditions used in the batch kiln tests is tabulated below.

Heat No.	Soil	Feed Moisture %	Kiln Gas Temp °C	Total Gas Input SCFM	Excess O_2 %
1	4761	6.5	1038	32	10
2	4761	6.5	1038	45	10
3	4760	1.2	1038	32	10
4	4761	6.5	1038	100	10
5	4760	1.2	1038	66	10
6	4760	1.2	1038	100	10
7	4761	6.5	1038	66	10
8	4768	6.4	1038	32	10
9	4768	6.4	1038	66	10
10	4768	6.4	1038	100	10
11	4760	1.2	1038	25	10

Test results and observations are shown in Appendix A. Time-temperature curves for each heat are shown in Appendix B.

Bulk Densities and Screen Analysis

The loose bulk densities of the as received material and batch kiln products are shown in Appendix C. The screen size structures of the as received soil and product from the small batch kiln tests are summarized and shown in Appendix D.

Dynamic Angle of Repose Inside Kiln

Dynamic angle of repose tests on as received soil, 4760 and 4768 test product were performed using a glass-faced 38 inch diameter drum. The drum was rotated at 0.82 rpm equivalent to 0.6 rpm in the EERU unit. Test results are shown in Appendix E.

Loss On Ignition

A loss-on-ignition test was performed on the as received soil sample. The sample was held at 1000 °C (1832 °F) for 30 minutes. Test result showed that the loss in weight was 46.62% in soil 4761, 2.07% in soil 4760 and 12.46% in soil 4768.

TGA AND DSC TESTS

Thermogravimetric Analysis (TGA) and Differnetial Scanning Calorimeter (DSC) tests were performed on each of the three as received soils. The results are shown in Appendix F.

Heat Transfer Computer Program Study

This will be provided in a separate addendum report.

K. Mak

K. K. Mak
Senior Engineer, Process Development
Process Research & Test Center
A-C Energy & Minerals Systems Company

KKM/sjk

APPENDIX A

Small Batch Kiln Test

Project Name IT Corporation

Date January 9, 1985

Heat No. 1 (4761-1)

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 6.5 %

Total Gas Input Rate 32 SCFM

Charge Weight 5.44 Kg

Time Min	Bed Temp °C #2	Off Gas Temp °C #1	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	30		2.0	Fine dust blown out at start.
5	708	919	29.5	1	2.5	
10	865	1040	28.0	1	3.3	
15	879	1041	28.0	1	3.0	
20	884	1039	28.5	1	2.9	
25	892	1037	28.5	1	2.8	
30	954	1031	27.3	2.4	2.4	Between 25 and 32 min - the 3 large pieces broke up. Bed temp incurred 140 °C in 8 minutes.
35	1000	1044	28.1	2.2	1.7	
40	1006	1034	28.1	2.2	1.7	
45	1013	1037	28.1	2.2	1.7	
50	1014	1037	28.1	2.2	1.7	
55	1018	1040	28.1	2.2	1.7	
60	1020	1041	28.1	2.2	1.7	

Product Weight 2.43 Kg

Exhaust Dust Weight 0.23 Kg

Floor Sweepings ________ Kg

Total Product Weight 2.66 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 9, 1985

Heat No. 2 (4761-2)

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 6.5 %

Total Gas Input Rate 45 SCFM

Charge Weight 5.44 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1040	39.1	2.7	3.2	Irregular bed action.
5	693	894	37.6	3.8	3.6	
			36.8	4.4	3.8	
10	882	1038	35.3	4.5	4.2	-10 Min regular bed action - No large pieces (broken).
			36.4	4.9	4.0	
15	920	1042	36.8	4.4	3.8	
			37.6	3.8	3.6	
20	942	1039	38.4	3.2	3.4	
25	985	1045	39.1	2.7	3.2	
30	1019	1040	39.1	2.1	3.0	
35	1028	1043	39.1	2.1	3.0	
			39.1	1.8	2.9	
40	1034	1039	39.1	1.6	2.8	
45	1036	1043	39.1	1.6	2.8	
50	1034	1037	39.1	1.6	2.8	
55	1035	1038	39.1	1.6	2.8	
60	1039	1040	39.1	1.6	2.8	

Product Weight 2.13 Kg

Exhaust Dust Weight .34 Kg

Floor Sweepings .06 Kg

Total Product Weight 2.53 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 9, 1985

Heat No. 3 (4760-1)

Kiln Ret. Time 6.0 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 1.2 %

Total Gas Input Rate 32 SCFM

Charge Weight 4.75 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	25.1	3.9	3.0	Roots burned immediately.
			25.8	3.4	2.8	
5	906	1046	27.3	2.3	2.4	
10	1010	1044	27.3	2.3	2.4	
15	1025	1040	27.3	2.3	2.4	
20	1027	1040	27.3	2.3	2.4	
25	1025	1037	27.3	2.3	2.4	
30	1026	1038	27.3	2.3	2.4	
35	1028	1038	27.3	2.3	2.4	
40	1028	1040	27.3	2.3	2.4	
45	1029	1040	27.3	2.3	2.4	
50	1030	1038	27.3	2.3	2.4	
55	1029	1039	27.3	2.3	2.4	
60	1028	1038	27.3	2.3	2.4	

Product Weight 3.03 Kg

Exhaust Dust Weight 1.4 Kg

Floor Sweepings ________ Kg

Total Product Weight 4.43 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 10, 1985

Heat No. 4 (4761-3)

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 6.5 %

Total Gas Input Rate 100 SCFM

Charge Weight 5.44 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	85	7.4	7.6	Considerable dust blown out.
5	864	1043	86	6.3	7.2	
			87	5.7	7	
10	874	1040	89	4.5	6.6	At 13 Min Bed Temp approaching Off Gas.
15	931	1041	89.6	3.9	6.4	
			91	2.9	6	
20	1010	1034	91	2.9	6	
25	1022	1040	91	2.9	6	
30	1015	1040	91	2.9	6	
35	1020	1038	91	2.9	6	
40	1019	1040	91	2.9	6	
45	1021	1039	91	2.9	6	
50	1024	1039	91	2.9	6	
55	1028	1043	91	2.9	6	
60	1029	1041	91	2.9	6	

Product Weight 1.77 Kg

Exhaust Dust Weight 0.44 Kg

Floor Sweepings 0.25 Kg

Total Product Weight 2.46 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 10, 1985

Heat No. 5 (4760-2) Sand

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 ^{o}C

Kiln Loading 7 %

Feed Moisture 1.2 %

Total Gas Input Rate 66 SCFM

Charge Weight 4.75 Kg

Time Min	Bed Temp ^{o}C	Off Gas Temp ^{o}C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0			56	4.8	5	
5	932	1012	57.7	3.7	4.6	
10	992	1034	58.5	3.1	4.4	At 10 Min 0.66 Kg leakage a Kiln Cover.
15	1009	1030	58.5	3.1	4.4	
20	1013	1040	58.5	3.1	4.4	
25	1016	1038	58.5	3.1	4.4	
30	1017	1040	58.5	3.1	4.4	
35	1018	1040	58.5	3.1	4.4	
40	1020	1041	58.5	3.1	4.4	
45	1018	1036	58.5	3.1	4.4	
50	1019	1039	58.5	3.1	4.4	
55	1022	1041	58.5	3.1	4.4	
60	1021	1040	58.5	3.1	4.4	

Kiln Leakage 0.66 Kg

Product Weight 2.57 Kg

Exhaust Dust Weight 0.90 Kg

Floor Sweepings 0.18 Kg

Total Product Weight 4.31 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 10 , 1985

Heat No. 6 (4760-3) Sand

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 1.2 %

Total Gas Input Rate 100 SCFM

Charge Weight 4.75 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	91 87	2.9 5.7	6 7	Organics burned out.
5	993	1043	89	4.5	6.6	At 8 Min Bed Off Gas approaching line out conditions.
10	1009	1038	90.4	3.4	6.2	
15	1017	1040	91	2.9	6	
20	1018	1040	91	2.9	6	
25	1020	1039	91	2.9	6	
30	1019	1039	91	2.9	6	
35	1021	1037	91	2.9	6	
40	1023	1040	91	2.9	6	
45	1021	1038	91	2.9	6	
50	1018	1038	91	2.9	6	
55	1020	1038	91	2.9	6	
60	1021	1040	91	2.9	6	

Product Weight 2.84 Kg

Exhaust Dust Weight 1.01 Kg

Floor Sweepings 0.44 Kg

Total Product Weight 4.29 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 10, 1985

Heat No. 7 (4761-4)

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 6.5 %

Total Gas Input Rate 66 SCFM

Charge Weight 5.44 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	56.2	4.8	5	
5	840	1018	55.4	5.4	6	
10	921	1038	56.2	4.8	5	
15	944	1039	58.0	3.7	4.6	
20	1009	1041	59.0	2.6	4.2	
25	1028	1035	59.0	3.1	4.4	
30	1036	1039	59.0	3.1	4.4	
35	1032	1038	59.0	3.1	4.4	
40	1037	1040	59.0	3.1	4.4	
45	1035	1039	59.0	3.1	4.4	
50	1033	1036	59.0	3.1	4.4	
55	1038	1040	59.0	3.1	4.4	
60	1035	1039	59.0	3.1	4.4	

Product Weight 2.00 Kg

Exhaust Dust Weight 0.42 Kg

Floor Sweepings 0.09 Kg

Total Product Weight 2.51 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 11, 1985

Heat No. 8 (4768-1)

Kiln Ret. Time	60	mins	Excess O_2	10	%
Kiln Speed	2.8	rpm	Kiln Temp at Time of Feed	1038	°C
Kiln Loading	7	%	Feed Moisture	6.4	%
Total Gas Input Rate	32	SCFM	Charge Weight	4.68	Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM Air	Process Gas Input Comp SCFM O_2	Process Gas Input Comp SCFM Nat. Gas	Remarks
0			25.1	3.9	3.0	Organics burning out. 3 Min to complete. Bed more free flowing at 3 Min rather than sliding.
5	865	996	23.5	5.1	3.4	
10	947	1042	24.3	4.5	3.2	Air Compressor off low pressure - change to high pressure system (8 Min Bed Temp below expected).
15	996	1041	25.1	3.9	3.0	
20						
25	800	870	25.1	3.9	3.0	
30	985	1043	25.1	3.9	3.0	
35	1009	1040	26.6	2.8	2.6	
40	1021	1038	26.6	2.8	2.6	
45	1010	1033	20.0	2.0	1.8	
50	1012	1038	20.0	2.0	1.8	
55	1014	1038	20.0	2.0	1.8	
60	1014	1038	20.0	2.0	1.8	

Product Weight	3.36	Kg	Exhaust Dust Weight	0.12	Kg
Floor Sweepings	0.02	Kg	Total Product Weight	3.50	Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation Date January 11, 1985

Heat No. 9 (4768-2)

Kiln Ret. Time 60 mins Excess O_2 10 %

Kiln Speed 2.8 rpm Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 % Feed Moisture 6.4 %

Total Gas Input Rate 66 SCFM Charge Weight 4.68 Kg

Time Min	Bed Temp oC	Off Gas Temp oC	Process Gas Input Comp SCFM Air	O_2	Nat. Gas	Remarks
0		1038	60	2.0	4	
5	929	1037	56	4.8	5	
10	1013	1037				
15	1023	1040	59.2	2.6	4.2	
20	1021	1037	59.2	2.6	4.2	
25	1023	1038	59.2	2.6	4.2	
30	1023	1039	59.2	2.6	4.2	
35	1024	1038	59.2	2.6	4.2	
40	1023	1037	59.2	2.6	4.2	
45	1021	1036	59.2	2.6	4.2	
50	1026	1040	59.2	2.6	4.2	
55	1025	1039	59.2	2.6	4.2	
60	1024	1038	59.2	2.6	4.2	

Product Weight 3.00 Kg Exhaust Dust Weight 0.52 Kg

Floor Sweepings 0.05 Kg Total Product Weight 3.57 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation

Date January 11, 1985

Heat No. 10 (4768-3)

Kiln Ret. Time 60 mins

Excess O_2 10 %

Kiln Speed 2.8 rpm

Kiln Temp at Time of Feed 1038 °C

Kiln Loading 7 %

Feed Moisture 6.4 %

Total Gas Input Rate 100 SCFM

Charge Weight 4.68 Kg

Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0		1038	91	2.9	6	Organics burned out within 2 Min from start.
5	926	1020				
10	1020	1047				
15	1029	1042	92	2.3	5.8	
20	1026	1034	92	2.3	5.8	
25	1026	1034	92	2.3	5.8	
30	1028	1036	92	2.3	5.8	
35	1029	1036	92	2.3	5.8	
40	1036	1040	92	2.3	5.8	
45	1034	1039	92	2.3	5.8	
50	1033	1035	92	2.3	5.8	
55	1033	1038	92	2.3	5.8	
60	1037	1040	92	2.3	5.8	

Product Weight 2.60 Kg

Exhaust Dust Weight 0.81 Kg

Floor Sweepings 0.13 Kg

Total Product Weight 3.54 Kg

Observations:

Small Batch Kiln Test

Project Name IT Corporation Date January 11, 1985

Heat No. 11 (4760-4)

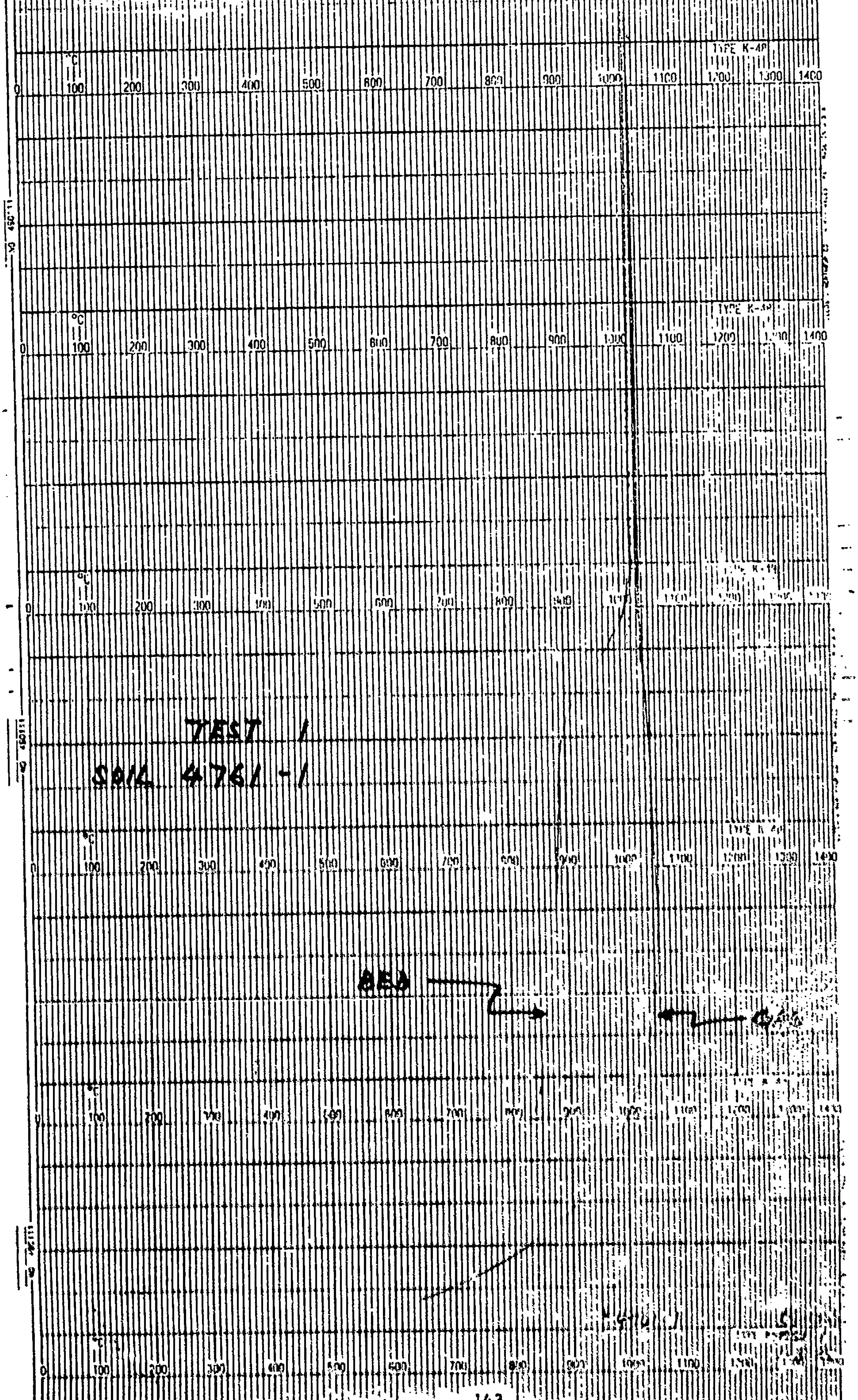
Kiln Ret. Time	60	mins	Excess O_2	10	%
Kiln Speed	2.8	rpm	Kiln Temp at Time of Feed	1038	°C
Kiln Loading	7	%	Feed Moisture	1.2	%
Total Gas Input Rate	25	SCFM	Charge Weight	4.75	Kg

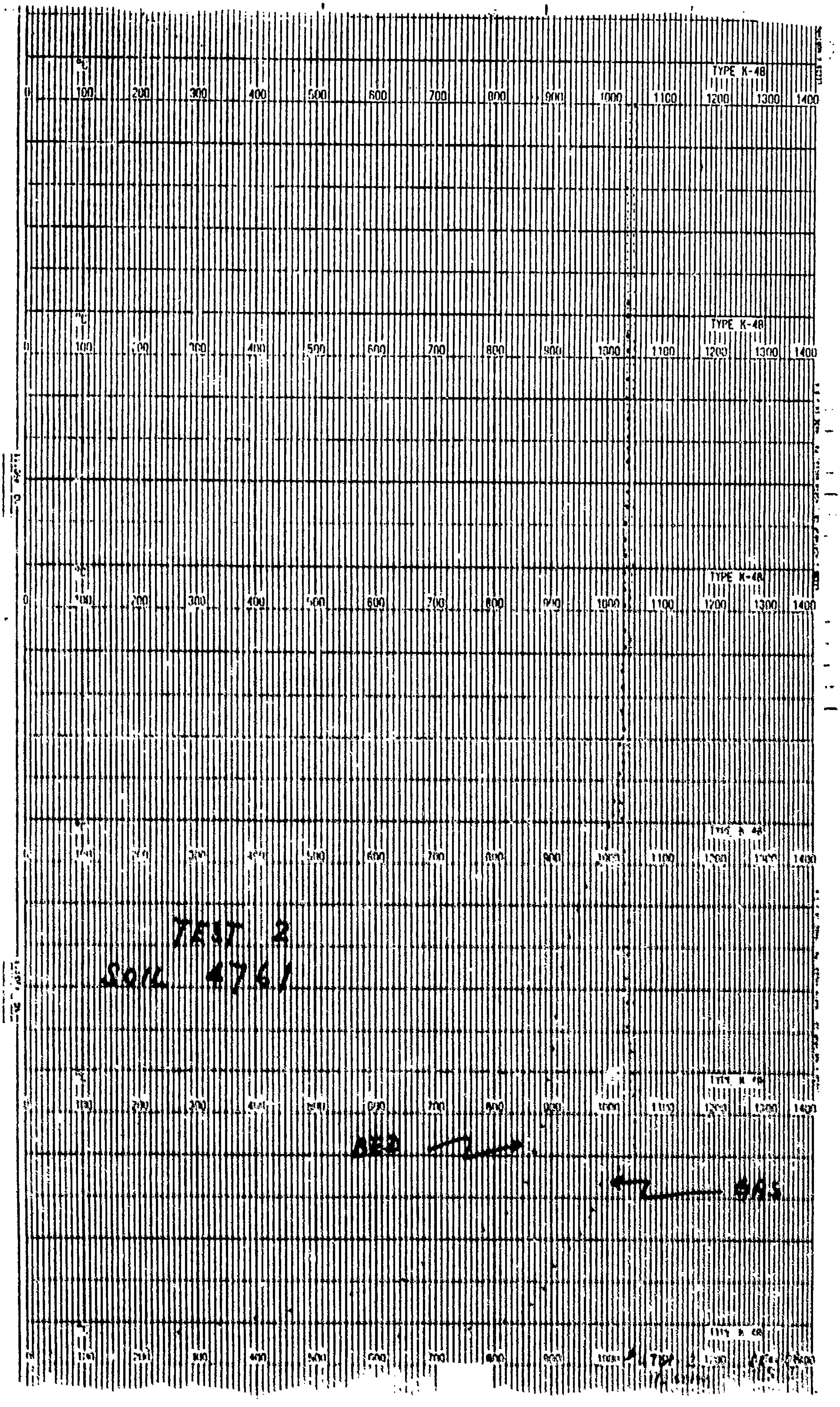
Time Min	Bed Temp °C	Off Gas Temp °C	Process Gas Input Comp SCFM			Remarks
			Air	O_2	Nat. Gas	
0						
5	996	1043	17.1	4.9	3	
10	1040	1043	19.4	3.2	2.4	
15	1034	1038	20.9	2.1	2.0	
20	1035	1038	20.9	2.1	2.0	
25	1035	1037	20.9	2.1	2.0	
30	1035	1038	20.9	2.1	2.0	
35	1035	1040	20.9	2.1	2.0	
40	1035	1038	20.9	2.1	2.0	
45	1036	1040	20.9	2.1	2.0	
50	1037	1038	20.9	2.1	2.0	
55	1032	1032	20.9	2.1	2.0	
60	1033	1038	20.9	2.1	2.0	

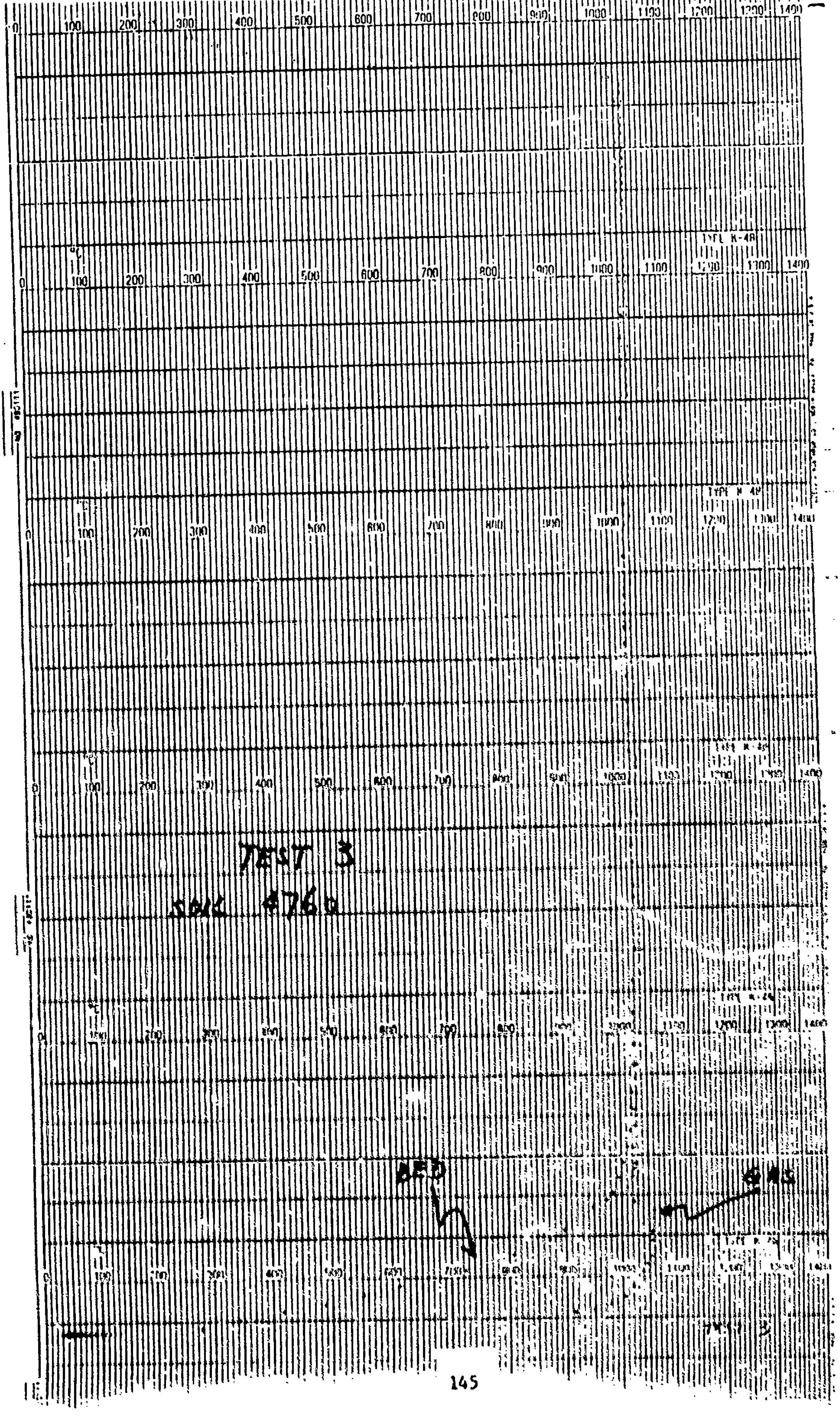
Product Weight	3.73	Kg	Exhaust Dust Weight	0.86	Kg
Floor Sweepings	0.03	Kg	Total Product Weight	4.62	Kg

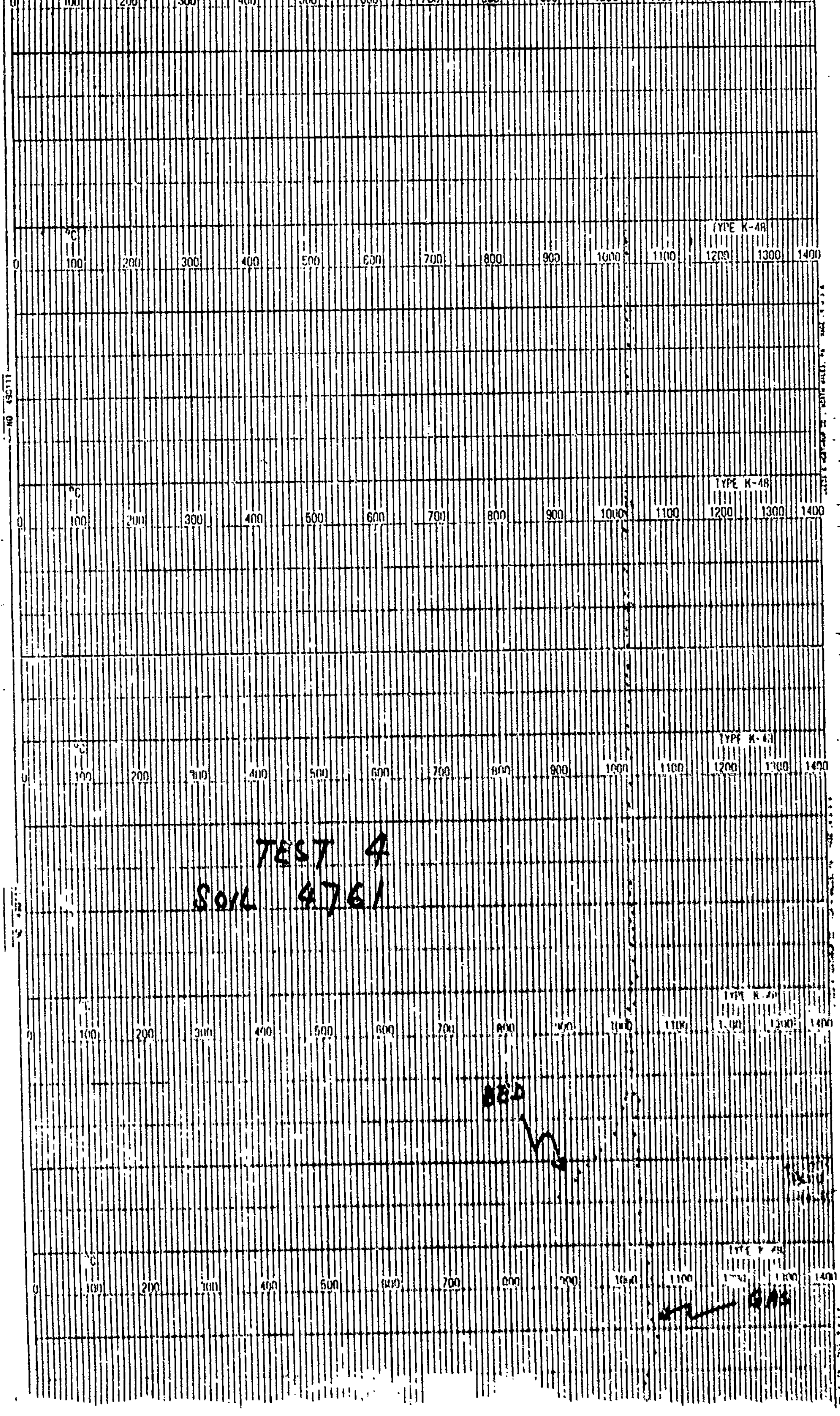
Observations:

APPENDIX B


TYPE K-40
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 1
SOIL 4761-1
BED
GAS


°C
TYPE K-48
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 2
SOIL 4741
AER
GAS

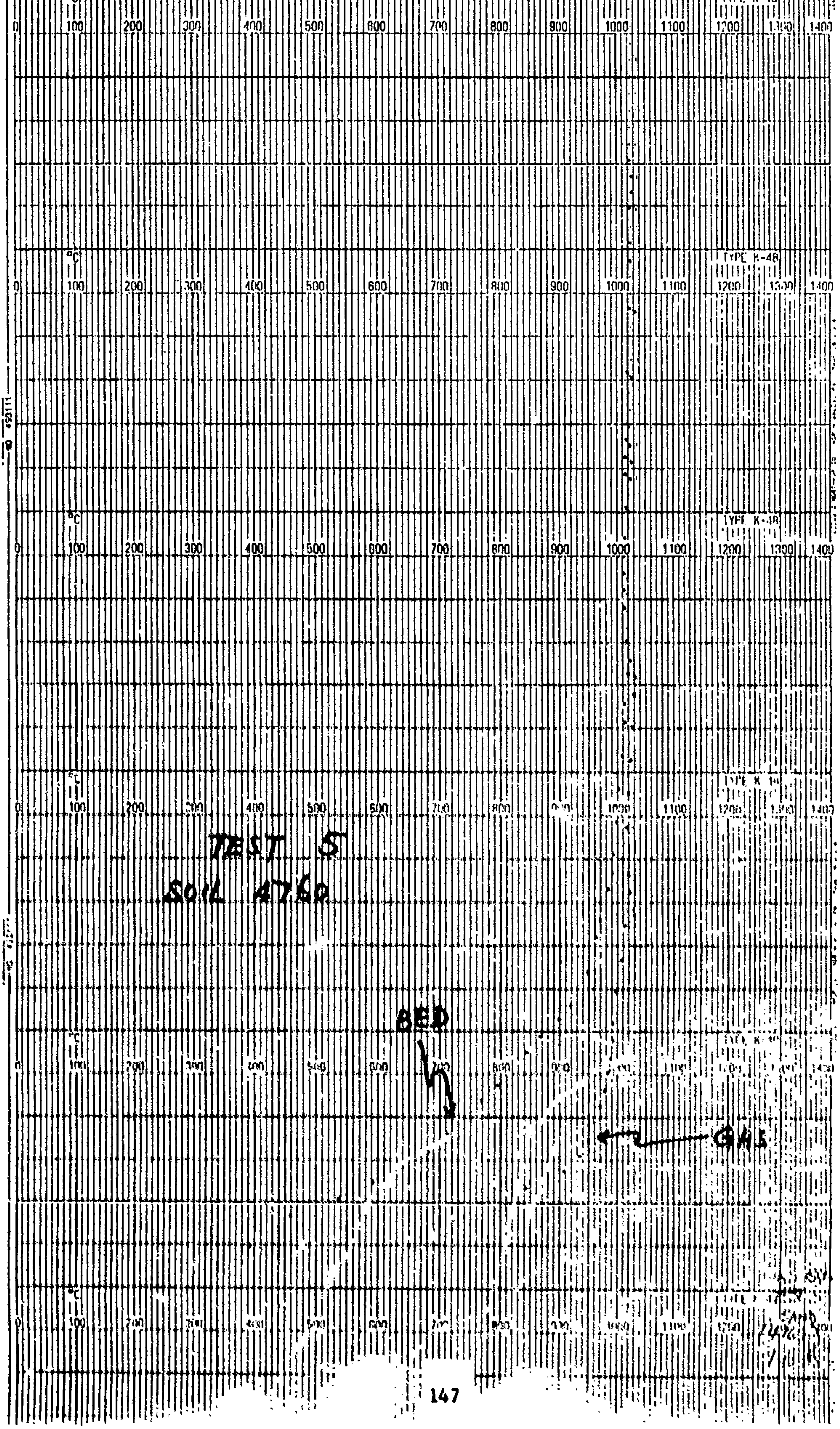

0 100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400
°C
TYPE K-48
TEST 3
SWL 4760
AFD
GAS

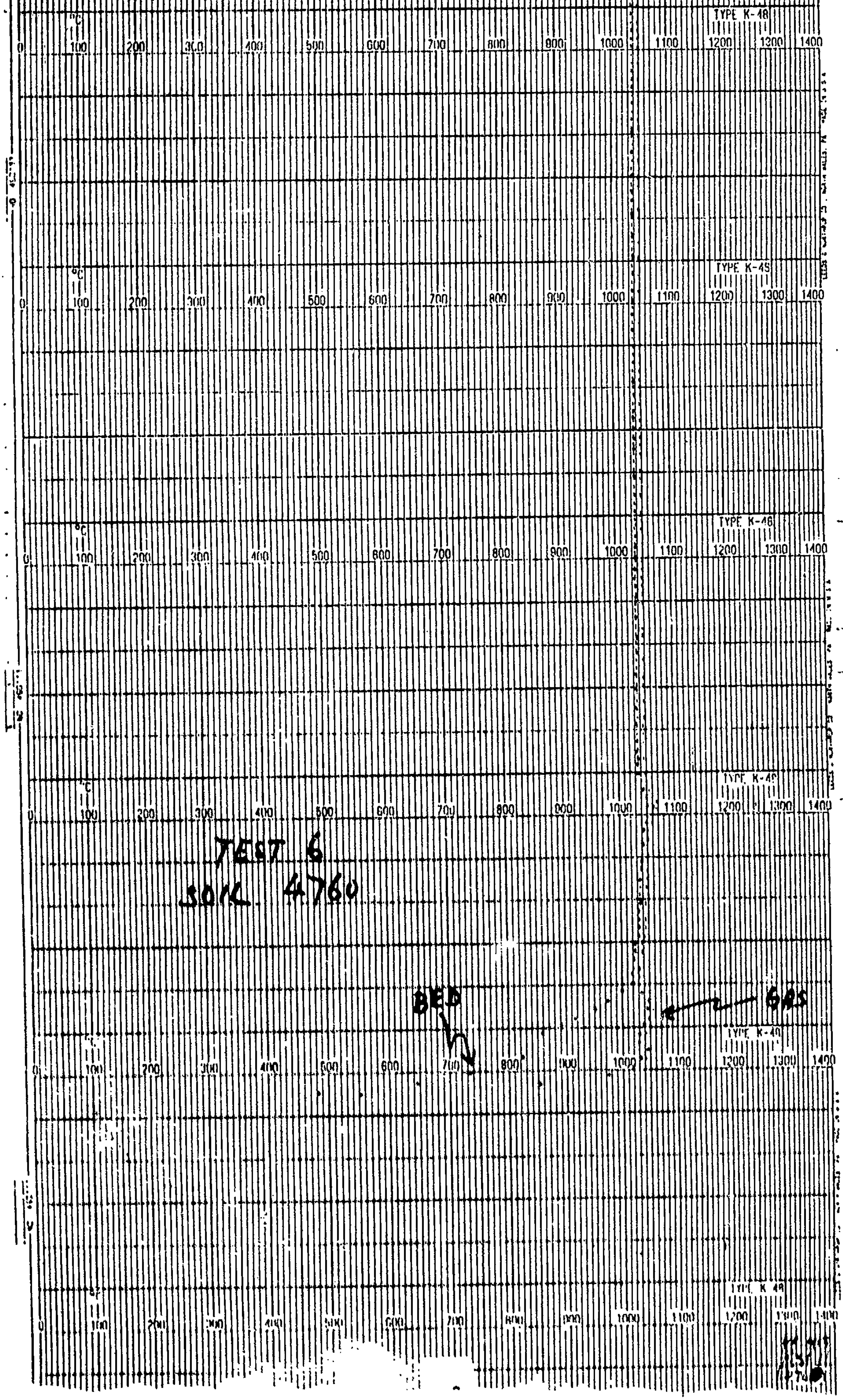


TEST 4
SOIL 4761

BED

GAS

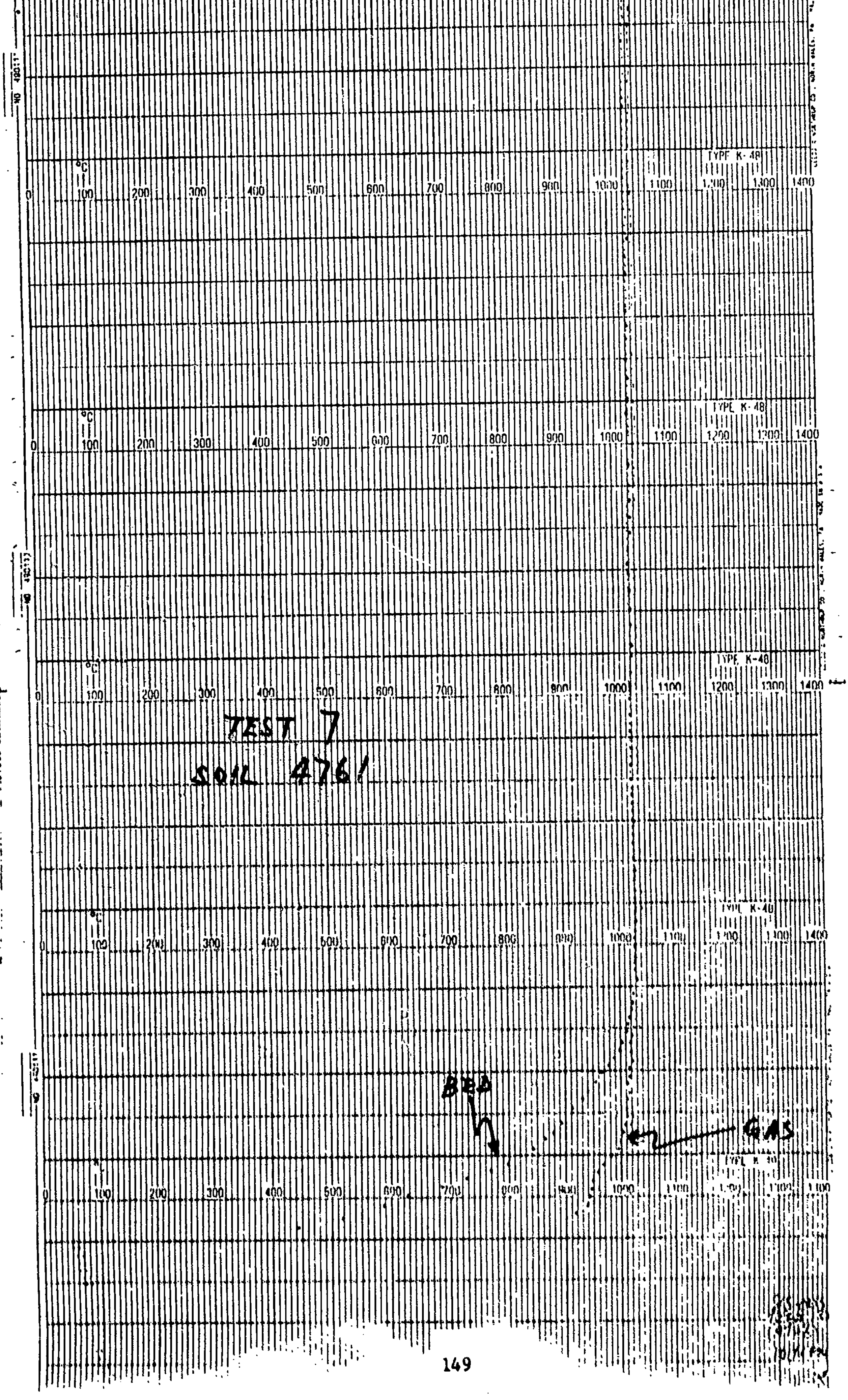

°C
TYPE K-48
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 5
SOIL AT6R
BED
GAS

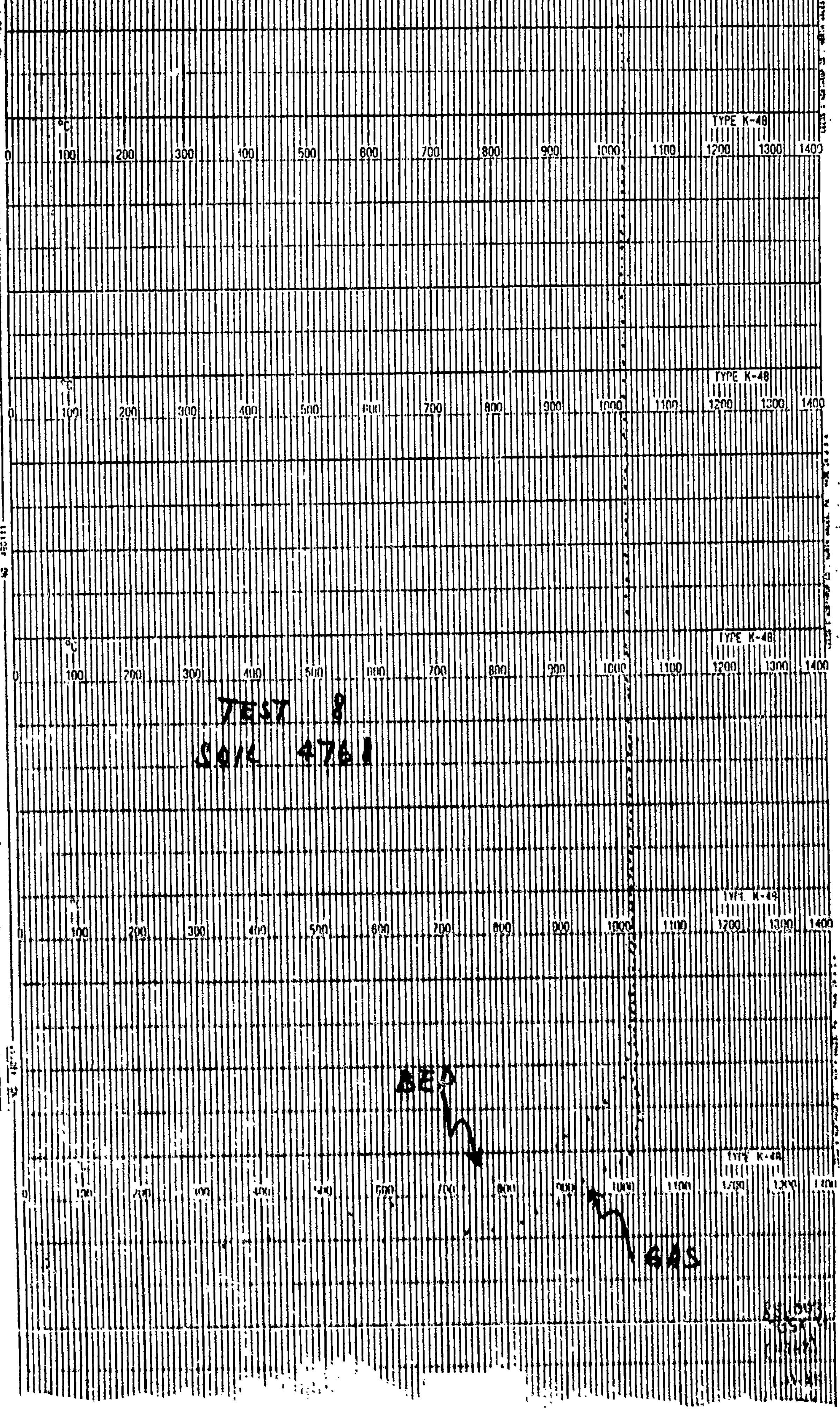


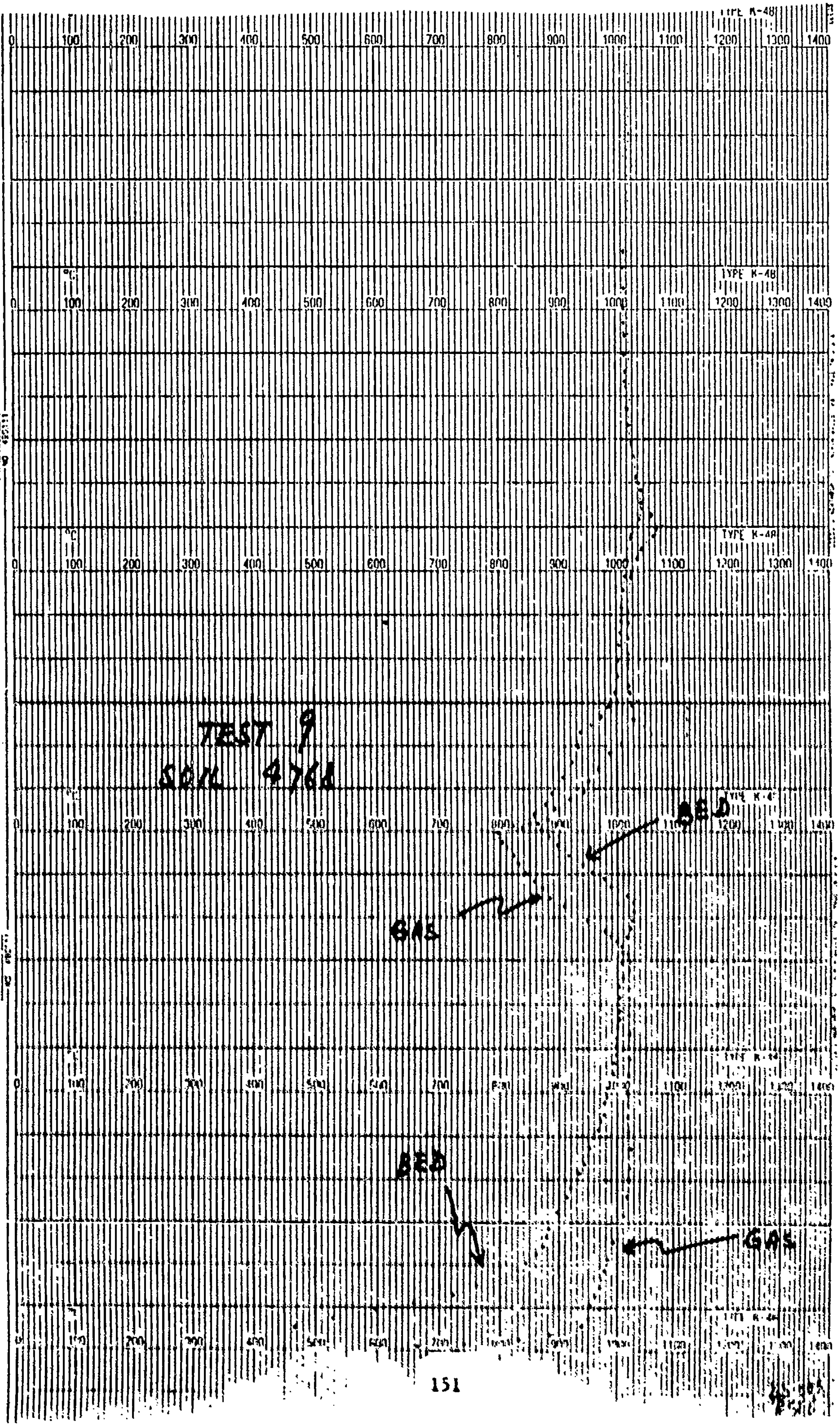
TEST 6
SOIL 4760

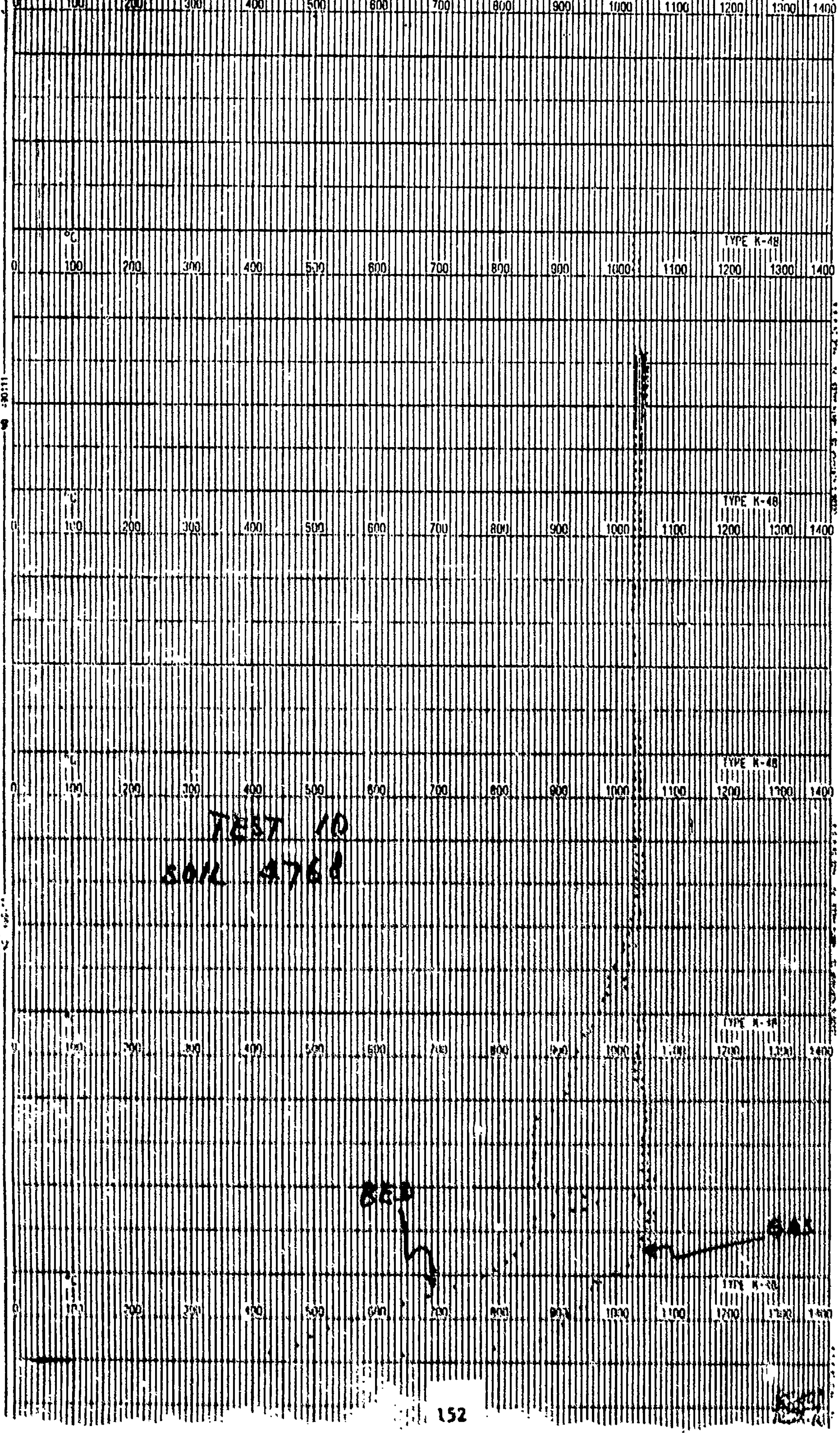
BED

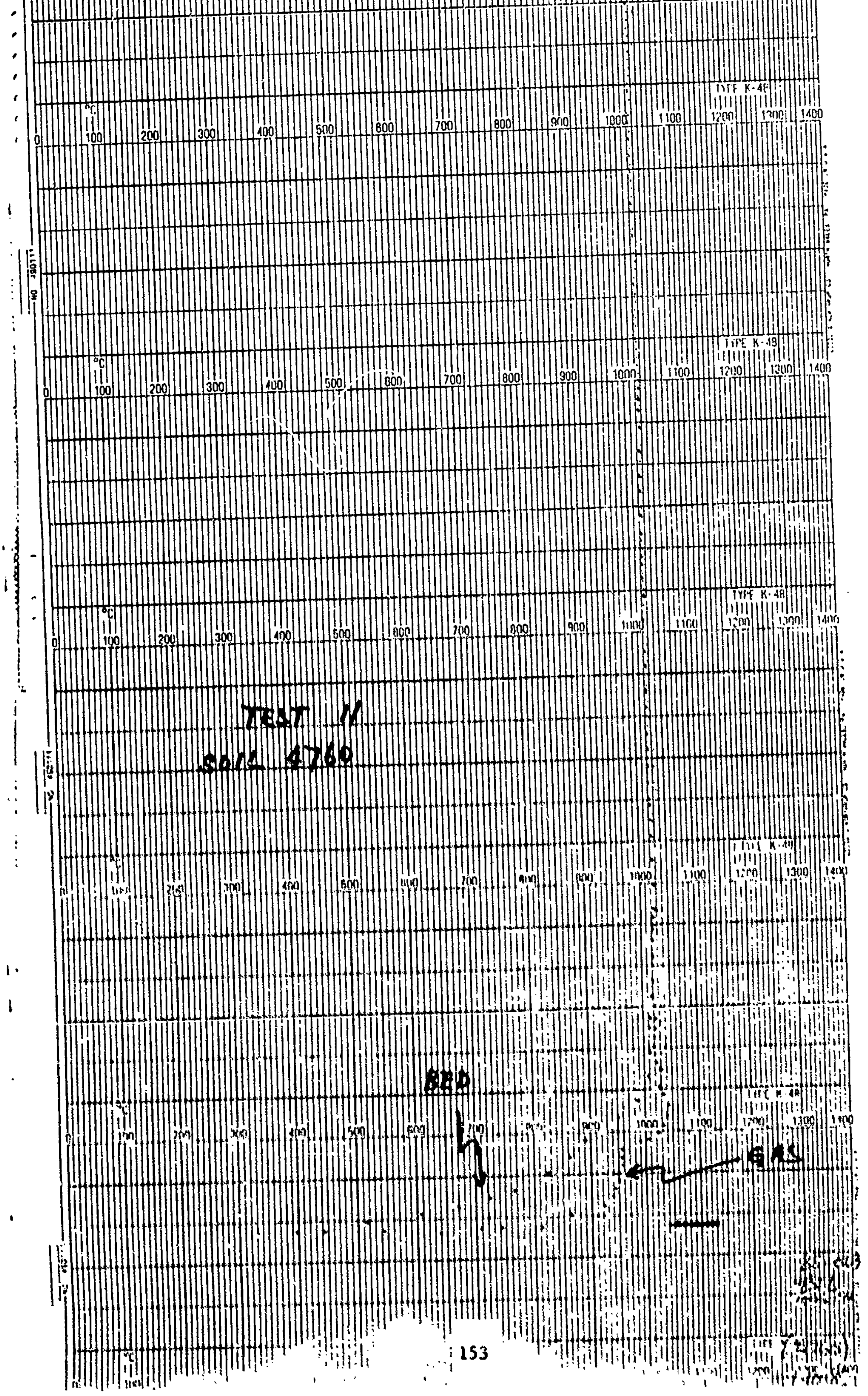
GAS


TYPE K-48
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 7
SOIL 4761
BED
GAS


TYPE K-48
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 8
SOAK 476[illegible]
BED
GAS


TYPE K-48
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 9
SOIL 4766
BED
GAS
BED
GAS


°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TYPE K-48
TEST 10
SOIL 4768
RED
BLK


TYPE K-48
°C
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
TEST 11
SOIL 4769
BED
GAS

APPENDIX C

LOOSE BULK DENSITY OF MATERIALS, lb/ft^3

As Received 4761	85.98
As Received 4760	80.04
As Received 4768	78.92
Test 1 Product	55.56
Test 2 Product	54.31
Test 3 Product	94.28
Test 4 Product	51.19
Test 5 Product	92.40
Test 6 Product	92.40
Test 7 Product	47.45
Test 8 Product	84.29
Test 9 Product	87.41
Test 10 Product	118.63
Test 11 Product	93.03

APPENDIX D

AS RECEIVED SOIL SCREEN ANALYSIS

PERCENTAGE PASSING

Screen Size	Soil 4761	Soil 4760	Soil 4768
2.1 Inch	100.00		
1.5 Inch	76.59		
1.1 Inch	-		
.75 Inch	-		100.00
.53 Inch	74.06		92.49
.375 Inch	68.31		88.08
3 Mesh	61.69		85.04
4 Mesh	54.77	100.00	72.68
6 Mesh	49.17	99.96	64.22
8 Mesh	43.52	99.90	58.66
10 Mesh	39.77	99.84	56.14
14 Mesh	35.71	99.60	54.02
20 Mesh	31.54	97.57	51.52
28 Mesh	26.36	82.92	47.01
35 Mesh	20.90	51.71	40.06
48 Mesh	15.67	22.43	30.95
65 Mesh	10.71	6.97	17.94
100 Mesh	6.94	2.77	10.58
150 Mesh	3.62	1.77	6.74
200 Mesh	1.24	1.15	3.67
Pan	0	0	0

```
SIEVE$MAIN       SIEVE$MAIN                                 62      00000250  00002450


SIEVE ANALYSIS                                                       ALLIS-CHALMERS
MATERIAL         SOIL 4761
SUBMITTED BY     IT CORP.
                 BATCH KILN TEST 1
TEST NO.85-003                                              DATE 1-10-85

A= AS REC. FEED SAMPLE
B= 4761-1 KILN PROD.      TEST 1
C= 4761-1 EXHAUST DUST, TEST 1
D=
 SIEVE SIZE             A                  B                  C                  D
EQUIV   ASTM      PERCENTAGE         PERCENTAGE         PERCENTAGE         PERCENTAGE
I.MESH  MU-M      ON   PASSING       ON   PASSING       ON   PASSING       ON   PASSING
 2.1    53000    0.00 100.00       0.00 100.00        0.00 100.00
 1.5    37500   23.41  76.59       0.00 100.00        0.00 100.00
 1.1    26500    0.00   0.00       0.00 100.00        0.00 100.00
 .75    19000    0.00   0.00       0.00 100.00        0.00 100.00
 .53    13200    2.53  74.06      12.61  87.39        0.00 100.00
 375     9500    5.75  68.31       9.04  78.35        0.00 100.00
 M=3     6700    6.62  61.69      10.98  67.37        0.00 100.00
   4     4750    6.92  54.77      10.08  57.29        0.00 100.00
   6     3350    5.60  49.17       7.56  49.73        2.35  97.65
   8     2360    5.65  43.52       7.61  42.12        0.81  96.83
  10     1700    3.74  39.77       6.26  35.86        0.77  96.06
  14     1180    4.07  35.71       7.29  28.57        1.54  94.52
  20      850    4.17  31.54       4.65  23.92        5.35  89.17
  28      600    5.18  26.36       6.80  17.12       13.14  76.04
  35      425    5.45  20.91       0.00   0.00       16.69  59.35
  48      300  . 5.23  15.68       0.00   0.00       16.56  42.79
  65      212    4.96  10.72       0.00   0.00       16.77  26.02
 100      150    3.88   6.84       0.00   0.00       14.63  11.38
 150      106    3.17   3.67       0.00   0.00        7.36   4.02
 200       75    2.33   1.34       0.00   0.00        2.57   1.45
 270       53    0.00   0.00       0.00   0.00        0.00   0.00
 325       45    0.00   0.00       0.00   0.00        0.00   0.00
 400       38    0.00   0.00       0.00   0.00        0.00   0.00
 500       25    0.00   0.00       0.00   0.00        0.00   0.00
 PAN        0    1.34   0.00      17.12   0.00        1.45   0.00
                                                 A         B         C         D
80 PCT  SIZE (LOG-LOG)                         39675.    10116.     576.
SLOPE.80% SIZE TO SMALLEST DATUM               0.664     0.944     1.829
SPECIFIC GRAVITY                              ******** ******** ********
ESTIMATED SP.GR. FOR 40% VOIDS                  0.00      0.00      0.00
VOIDS FRACTION                                ******** ******** ********
BULK WEIGHT (LBS.FT**3)                       ******** ******** ********
```

```
SIEVE ANALYSIS                                          ALLIS-CHALMERS
MATERIAL       SOIL 4761 -1
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 1
TEST NO,85-003                                     DATE 1-10-85

         100                  10   WEIGHT % PASSING  1               ,2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
4,2 107MM 7
  A= AS REC, FEED SAMPLE
3,0 75000 7
  B= 4761-1 KILN PROD,
2,1 53000 7
  C= 4761-1 EXHAUST DUST
1,5 37500 4  A

1,1 26500 4

,75 19000 4

,53 13200 C BA                                                         +

,375 9500 C  BA

M=3  6700 C   BA

  4  4750 C     1

  6  3350  C     1

  8  2360  C      AB

 10  1700  C       AB

 14  1180  C        A B

 20   850   C         A B

 28   600    C         A    B

 35   425      C          A

 48   300          C        A

 65   212              C        A

100   150                      C    A

150   106                           CA

200    75                                      CA

270    53
325    45
400    38

500    26 +                                                            +
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
1=A+B     2-A+C     3 A+D     4-B+C     5-B+D     6=C+D
7=A+B+C   8=A+B+D   9=A+C+D   0-B+C+D   + A+B+C+D
```

```
SIEVE#MAIN      SIEVE#MAIN                                     62      00000250   00002450

SIEVE ANALYSIS                                                       ALLIS-CHALMERS
MATERIAL          SOIL 4761
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST 2
TEST NO.85-003                                                DATE 1-10-85

A= AS REC. FEED SAMPLE
B= 4761-2 KILN PROD.   TEST  2
C= 4761-2 EXHAUST DUST TEST  2
D=
 SIEVE SIZE              A                 B                 C                 D
EQUIV.  ASTM      PERCENTAGE        PERCENTAGE        PERCENTAGE        PERCENTAGE
T.MESH  MU-M      ON  PASSING       ON  PASSING       ON  PASSING       ON  PASSING
 2.1    53000    0.00 100.00       0.00 100.00       0.00 100.00
 1.5    37500   23.41  76.59       0.00 100.00       0.00 100.00
 1.1    26500    0.00   0.00       0.00 100.00       0.00 100.00
 .75    19000    0.00   0.00       0.00 100.00       0.00 100.00
 .53    13200    2.53  74.06       7.25  92.75       0.00 100.00
 .375    9500    5.75  68.31       7.84  84.91       0.00 100.00
 M=3     6700    6.62  61.69      10.09  74.82       0.00 100.00
   4     4750    6.92  54.77      10.27  64.54       0.00 100.00
   6     3350    5.60  49.17       8.15  56.40       1.60  98.40
   8     2360    5.65  43.52       8.64  47.76       1.08  97.32
  10     1700    3.74  39.77       6.81  40.95       1.22  96.10
  14     1180    4.07  35.71       6.42  34.53       3.20  92.89
  20      850    4.17  31.54       7.55  26.99       7.60  85.29
  28      600    5.18  26.36       7.54  19.45      12.18  73.11
  35      425    5.45  20.90       0.00   0.00      14.51  58.61
  48      300    5.23  15.67       0.00   0.00      16.60  42.00
  65      212    4.96  10.71       0.00   0.00      16.95  25.05
 100      150    3.77   6.94       0.00   0.00      12.76  12.29
 150      106    3.32   3.62       0.00   0.00       6.93   5.36
 200       75    2.38   1.24       0.00   0.00       3.03   2.33
 270       53    0.00   0.00       0.00   0.00       0.00   0.00
 325       45    0.00   0.00       0.00   0.00       0.00   0.00
 400       38    0.00   0.00       0.00   0.00       0.00   0.00
 500       26    0.00   0.00       0.00   0.00       0.00   0.00
 PAN        0    1.24   0.00      19.45   0.00       2.33   0.00
                                                 A         B         C         D
80 PCT. SIZE (LOG-LOG)  =                    39678.     8060.      735.
SLOPE.80% SIZE TO SMALLEST DATUM             0.664     0.544     1.549
SPECIFIC GRAVITY                           ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS              0.00      0.00      0.00
VOIDS FRACTION                             ******* ******* *******
BULK WEIGHT (LBS/FT**3)                    ******* ******* *******
```

```
SIEVE ANALYSIS                                          ALLIS-CHALMERS
MATERIAL       SOIL 4761
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 2
TEST NO.85-003                                     DATE 1-10-85

         100                   10   WEIGHT % PASSING  1              .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
4.2 107MM 7
  A= AS REC. FEED SAMPLE
3.0 75000 7
  B= 4761-2 KILN PROD.
2.1 53000 7
  C= 4761-2 EXHAUST DUST
1.5 37500 4  A

1.1 26500 4

.75 19000 4

.53 13200 CB A                                                         *

.375 9500 C B A

M=3  6700 C  B A

  4  4750 C    BA

  6  3350  C    BA

  8  2360  C      1

 10  1700  C       1

 14  1180  C         AB

 20   850   C         AB

 28   600    C          A  B

 35   425       C         A

 48   300          C        A

 65   212               C       A

100   150                      C    A

150   106                             C   A

200    75                                     C     A

270    53
325    45
400    38

500    26 *                                                            *
 MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
 1=A+B    2=A+C    3=A+D    4=B+C    5=B+D    6=C+D
 7=A+B+C  8=A+B+D  9=A+C+D  0=B+C+D  +=A+B+C+D
```

```
SIEVE ANALYSIS                                                  ALLIS-CHALMERS
MATERIAL          SOIL 4760
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST 3
TEST NO.85-003                                              DATE 1-10-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= TEST 3 KILN PROD.    4760-1
C= TEST 3 EXHAUST DUST 4760-1
D=
 SIEVE SIZE             A                   B                   C                   D
EQUIV.  ASTM     PERCENTAGE          PERCENTAGE          PERCENTAGE          PERCENTAGE
T.MESH  MU-M     ON  PASSING         ON  PASSING         ON  PASSING         ON  PASSING
   4     4750    0.00 100.00         0.00 100.00         0.00 100.00
   6     3350    0.04  99.96         0.06  99.94         0.00 100.00
   8     2360    0.06  99.90         0.02  99.92         0.00 100.00
  10     1700    0.06  99.84         0.04  99.87         0.00 100.00
  14     1180    0.24  99.60         0.26  99.62         0.03  99.97
  20      850    2.03  97.57         2.32  97.30         0.49  99.48
  28      600   14.65  82.92        14.94  82.36         9.96  89.52
  35      425   31.21  51.71        30.29  52.07        38.52  51.00
  48      300   29.28  22.43        28.85  23.21        34.97  16.03
  65      212   15.47   6.97        16.15   7.07        12.73   3.30
 100      150    4.20   2.77         4.93   2.14         2.64   0.66
 150      106    1.00   1.77         1.12   1.02         0.44   0.22
 200       75    0.62   1.15         0.54   0.48         0.14   0.09
 270       53    0.00   0.00         0.00   0.00         0.00   0.00
 325       45    0.00   0.00         0.00   0.00         0.00   0.00
 400       38    0.00   0.00         0.00   0.00         0.00   0.00
 500       26    0.00   0.00         0.00   0.00         0.00   0.00
 PAN        0    1.15   0.00         0.48   0.00         0.09   0.00
                                                   A         B         C         D
80 PCT. SIZE (LOG-LOG)  =                        584.      587.      560.
SLOPE,80% SIZE TO SMALLEST DATUM                2.044     2.127     2.901
SPECIFIC GRAVITY                              ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                   0.00      0.00      0.00
VOIDS FRACTION                                ******* ******* *******
BULK WEIGHT (LBS/FT**3)                       ******* ******* *******
```

```
SIEVE ANALYSIS                                                ALLIS-CHALMERS
MATERIAL        SOIL 4760
SUBMITTED BY    IT CORP.
                BATCH KILN TEST 3
TEST NO.85-003                                           DATE 1-10-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= TEST 1 KILN PROD.   4760-1
  C= TEST 1 EXHAUST DUST 4760-1
         100                   10   WEIGHT % PASSING   1              .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2      198  6 5 4  3   2
.53 13200 7                                                            *

.375 9500 7

M=3  6700 7

  4  4750 7

  6  3350 C1

  8  2360 C1

 10  1700 C1

 14  1180  7

 20   850  7

 28   600   7

 35   425       7

 48   300              BA  C

 65   212                            1      C

100   150                                     A B          C

150   106                                         A    B              C

200    75                                             A        B

270    53
325    45
400    38

500    26 *                                                            *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2      198  6 5 4  3   2
1=A+B      2-A+C      3-A+D      4-B+C      5-B+D      6-C+D
7=A+B+C    8-A+B+D    9-A+C+D    0-B+C+D    *-A+B+C+D
```

```
SIEVE ANALYSIS                                                ALLIS-CHALMERS
MATERIAL         SOIL 4761
SUBMITTED BY     IT CORP.
                 BATCH KILN TEST 4
TEST NO.85-003                                           DATE 1-10-85

A= AS REC. FEED SAMPLE
B= 4761-3 KILN PROD.  TEST 4
C= 4761-3 EXHAUST DUST  TEST 4
D=
 SIEVE SIZE            A               B               C               D
EQUIV.  ASTM      PERCENTAGE      PERCENTAGE      PERCENTAGE      PERCENTAGE
T.MESH  MU-M      ON  PASSING     ON  PASSING     ON  PASSING     ON  PASSING
 2.1    53000    0.00 100.00     0.00 100.00     0.00 100.00
 1.5    37500   23.41  76.59     0.00 100.00     0.00 100.00
 1.1    26500    0.00   0.00     0.00 100.00     0.00 100.00
 .75    19000    0.00   0.00     0.00 100.00     0.00 100.00
 .53    13200    2.53  74.06     4.76  95.24     0.00 100.00
 .375    9500    5.75  68.31     9.24  86.00     0.00 100.00
 M=3     6700    6.62  61.69    14.39  71.60     0.00 100.00
   4     4750    6.92  54.77    14.45  57.15     0.00 100.00
   6     3350    5.60  49.17     9.07  48.08     7.81  92.19
   8     2360    5.65  43.52    10.08  38.00     4.02  88.18
  10     1700    3.74  39.77     7.37  30.63     3.93  84.25
  14     1180    4.07  35.71     6.78  23.85     5.04  79.21
  20      850    4.17  31.54     7.12  16.73     5.39  73.82
  28      600    5.18  26.36     7.42   9.31     7.37  66.45
  35      425    5.45  20.90     0.00   0.00    11.18  55.26
  48      300    5.23  15.67     0.00   0.00    15.80  39.47
  65      212    4.96  10.71     0.00   0.00    18.36  20.91
 100      150    3.77   6.94     0.00   0.00    11.69   9.22
 150      106    3.32   3.62     0.00   0.00     3.32   3.90
 200       75    2.38   1.24     0.00   0.00     2.31   1.60
 270       53    0.00   0.00     0.00   0.00     0.00   0.00
 325       45    0.00   0.00     0.00   0.00     0.00   0.00
 400       38    0.00   0.00     0.00   0.00     0.00   0.00
 500       26    0.00   0.00     0.00   0.00     0.00   0.00
 PAN        0    1.24   0.00     9.31   0.00     1.60   0.00
                                               A        B        C        D
80 PCT. SIZE (LOG-LOG)  =                  39678.    8277.    1252.
SLOPE,80% SIZE TO SMALLEST DATUM            0.664    0.820    1.390
SPECIFIC GRAVITY                          ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS              0.00     0.00     0.00
VOIDS FRACTION                            ******* ******* *******
BULK WEIGHT (LBS/FT**3)                   ******* ******* *******
```

```
SIEVE ANALYSIS                                ALLIS-CHALMERS
MATERIAL     SOIL 4761
SUBMITTED BY IT CORP.
             BATCH KILN TEST 4
TEST NO.85-003                            DATE 1-10-85

             100             10  WEIGHT % PASSING 1           .2
MESH MU-M 198 6 5 4  3   2   198 6 5 4  3   2   198 6 5 4  3   2
4.2 107MM 7
  A= AS REC. FEED SAMPLE
3.0 75000 7
  B= 4761-3 KILN PROD.
2.1 53000 7
  C= 4761-3 EXHAUST DUST
1.5 37500 4  A

1.1 26500 4

.75 19000 4

.53 13200 CB A                                                 *

.375 9500 C B A

M=3  6700 C  BA

  4  4750 C   1

  6  3350  C   1

  8  2360   C    A B

 10  1700   C     A  B

 14  1180    C     A   B

 20   850    C       A    B

 28   600     C       A       B

 35   425       C       A

 48   300         C       A

 65   212              C     A

100   150                     C  A

150   106                            CA

200    75                                   C A

270    53
325    45
400    38

500    26 *                                                    *
MESH MU-M 198 6 5 4  3   2   198 6 5 4  3   2   198 6 5 4  3   2
1=A+B      2=A+C      3=A+D      4=B+C      5=B+D      6=C+D
7=A+B+C    8=A+B+D    9=A+C+D    0=B+C+D    *=A+B+C+D
```

```
SIEVE ANALYSIS                                                  ALLIS-CHALMERS
MATERIAL         SOIL 4760
SUBMITTED BY     IT CORP.
                 BATCH KILN TEST  5
TEST NO.85-003                                             DATE 1-10-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= TEST 5 KILN PROD.  4760-2
C= TEST 5 EXHAUST DUST 4760-2 .
D=
 SIEVE SIZE           A                 B                C                 D
EQUIV.  ASTM     PERCENTAGE       PERCENTAGE       PERCENTAGE        PERCENTAGE
T.MESH  MU-M     ON  PASSING      ON  PASSING      ON  PASSING       ON  PASSING
   4    4750    0.00 100.00      0.00 100.00      0.00 100.00
   6    3350    0.04  99.96      0.00 100.00      0.00 100.00
   8    2360    0.06  99.90      0.06  99.94      0.00 100.00
  10    1700    0.06  99.84      0.06  99.89      0.04  99.96
  14    1180    0.24  99.60      0.17  99.72      0.09  99.87
  20     850    2.03  97.57      1.50  98.22      1.82  98.05
  28     600   14.65  82.92      9.80  88.42     18.33  79.72
  35     425   31.21  51.71     27.17  61.25     37.16  42.56
  48     300   29.28  22.43     34.97  26.28     24.53  18.02
  65     212   15.47   6.97     19.15   7.13     12.27   5.76
 100     150    4.20   2.77      5.35   1.78      4.34   1.42
 150     106    1.00   1.77      1.00   0.78      0.93   0.49
 200      75    0.62   1.15      0.45   0.33      0.22   0.27
 270      53    0.00   0.00      0.00   0.00      0.00   0.00
 325      45    0.00   0.00      0.00   0.00      0.00   0.00
 400      38    0.00   0.00      0.00   0.00      0.00   0.00
 500      26    0.00   0.00      0.00   0.00      0.00   0.00
 PAN       0    1.15   0.00      0.33   0.00      0.27   0.00
                                                  A       B       C        D
80 PCT. SIZE (LOG-LOG)  =                       584.    546.    604.
SLOPE,80% SIZE TO SMALLEST DATUM               2.044   2.759   2.737
SPECIFIC GRAVITY                              ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                  0.00    0.00    0.00
VOIDS FRACTION                                ******* ******* *******
BULK WEIGHT (LBS/FT**3)                       ******* ******* *******
```

```
SIEVE ANALYSIS                                           ALLIS-CHALMERS
MATERIAL       SOIL 4760
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 5
TEST NO.85-003                                      DATE 1-10-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= TEST 5 KILN PROD. 4760-2
  C= TEST 5 EXHAUST DUST. 4760-2
         100                  .10   WEIGHT % PASSING   1              .2
MESH MU-M 198  6 5 4   3   2    198   6 5 4  3   2     198  6 5 4  3   2
.53 13200 7                                                            *

.375 9500 7

M=3  6700 7

  4  4750 7

  6  3350 4A

  8  2360 C1

 10  1700  7

 14  1180  7

 20   850  7

 28   600   1C

 35   425      B A C

 48   300               B A C

 65   212                            . C

100   150                                     A   B C

150   106                                         A       B   C

200    75                                             A            B C

270    53
325    45
400    38

500    26 *                                                            *
MESH MU-M 198  6 5 4   3   2    198   6 5 4  3   2     198  6 5 4  3   2
1=A+B     2=A+C     3=A+D     4=B+C      5=B+D     6=C+D
7=A+B+C   8=A+B+D   9=A+C+D   0=B+C+D    +=A+B+C+D
```

```
SIEVE ANALYSIS                                                ALLIS-CHALMERS
MATERIAL        SOIL 4760
SUBMITTED BY    IT CORP.
                BATCH KILN TEST 6
TEST NO.85-003                                           DATE 1-10-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= TEST 6 KILN PROD.    4760-3
C= TEST 6 EXHAUST DUST  4760-3
D=
 SIEVE SIZE             A                 B                 C                 D
EQUIV.  ASTM      PERCENTAGE        PERCENTAGE        PERCENTAGE        PERCENTAGE
T.MESH  MU-M     ON  PASSING       ON  PASSING       ON  PASSING       ON  PASSING
   4    4730    0.00 100.00       0.00 100.00       0.00 100.00
   6    3350    0.04  99.96       0.03  99.97       0.00 100.00
   8    2360    0.06  99.90       0.03  99.95       0.00 100.00
  10    1700    0.06  99.84       0.03  99.92       0.08  99.92
  14    1180    0.24  99.60       0.16  99.76       0.16  99.76
  20     850    2.03  97.57       1.79  97.97       1.55  98.20
  28     600   14.65  82.92      15.07  82.90       8.61  89.60
  35     425   31.21  51.71      34.18  48.72      21.91  67.69
  48     300   29.28  22.43      30.91  17.81      31.33  36.35
  65     212   15.47   6.97      13.60   4.21      24.89  11.46
 100     150    4.20   2.77       3.26   0.95       8.89   2.57
 150     106    1.00   1.77       0.58   0.37       2.00   0.57
 200      75    0.62   1.15       0.21   0.16       0.45   0.12
 270      53    0.00   0.00       0.00   0.00       0.00   0.00
 325      45    0.00   0.00       0.00   0.00       0.00   0.00
 400      38    0.00   0.00       0.00   0.00       0.00   0.00
 500      26    0.00   0.00       0.00   0.00       0.00   0.00
 PAN       0    1.15   0.00       0.16   0.00       0.12   0.00
                                                   A       B       C        D
80 PCT. SIZE (LOG-LOG)  -                        584.    586.    522.
SLOPE,80% SIZE TO SMALLEST DATUM                2.044   3.029   3.341
SPECIFIC GRAVITY                              ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                   0.00    0.00    0.00
VOIDS FRACTION                                ******* ******* *******
BULK WEIGHT (LBS/FT**3)                       ******* ******* *******
```

```
SIEVE ANALYSIS                                          ALLIS-CHALMERS
MATERIAL       SOIL 4760
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 6
TEST NO.85-003                                     DATE 1-10-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= TEST 6 KILN PROD.     4760-3
  C= TEST 6 EXHAUST DUST, 4760-3
         100                  10   WEIGHT % PASSING  1               .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
.53 13200 7                                                           *

.375 9500 7

M=3  6700 7

  4  4750 7

  6  3350 C1

  8  2360 C1

 10  1700  7

 14  1180  7

 20   850  7

 28   600   7

 35   425    C  1

 48   300          C    A B

 65   212                    C    A    B

100   150                                  2         B

150   106                                      A          C   B

200    75                                          A

270    53
325    45
400    38

500    26 *                                                           *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
1=A+B     2-A+C     3-A+D     4-B+C     5-B+D     6-C+D
7=A+B+C   8=A+B+D   9=A+C+D   0=B+C+D   *=A+B+C+D
```

```
SIEVE ANALYSIS                                              ALLIS-CHALMERS
MATERIAL          SOIL 4761
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST 7
TEST NO.85-003                                         DATE 1-10-85

A= AS REC. FEED SAMPLE
B= 4761-4 KILN PROD. TEST 7
C= 4761-4 EXHAUST DUST TEST 7
D=
 SIEVE SIZE            A               B               C               D
EQUIV.  ASTM      PERCENTAGE      PERCENTAGE      PERCENTAGE      PERCENTAGE
T.MESH  MU-M     ON  PASSING     ON  PASSING     ON  PASSING     ON  PASSING
 2.1    53000    0.00 100.00     0.00 100.00     0.00 100.00
 1.5    37500   23.41  76.59     0.00 100.00     0.00 100.00
 1.1    26500    0.00   0.00     0.00 100.00     0.00 100.00
 .75    19000    0.00   0.00     0.00 100.00     0.00 100.00
 .53    13200    2.53  74.06     6.13  93.87     0.00 100.00
 .375    9500    5.75  68.31    10.14  83.73     0.00 100.00
 M=3     6700    6.62  61.69    13.37  70.36     0.00 100.00
   4     4750    6.92  54.77    13.46  56.90     0.00 100.00
   6     3350    5.60  49.17     8.82  48.08     1.77  98.23
   8     2360    5.65  43.52     8.48  39.61     3.43  94.81
  10     1700    3.74  39.77     6.47  33.13     5.30  89.50
  14     1180    4.07  35.71     5.54  27.60     6.63  82.87
  20      850    4.17  31.54     6.01  21.59     6.08  76.80
  28      600    5.18  26.36     6.77  14.82     8.18  68.62
  35      425    5.45  20.90     0.00   0.00    12.27  56.35
  48      300    5.23  15.67     0.00   0.00    16.46  39.89
  65      212    4.96  10.71     0.00   0.00    18.90  20.99
 100      150    3.77   6.94     0.00   0.00    13.70   7.29
 150      106    3.32   3.62     0.00   0.00     5.19   2.10
 200       75    2.38   1.24     0.00   0.00     1.44   0.66
 270       53    0.00   0.00     0.00   0.00     0.00   0.00
 325       45    0.00   0.00     0.00   0.00     0.00   0.00
 400       38    0.00   0.00     0.00   0.00     0.00   0.00
 500       26    0.00   0.00     0.00   0.00     0.00   0.00
 PAN        0    1.24   0.00    14.82   0.00     0.66   0.00
                                                A        B        C        D
80 PCT. SIZE (LOG-LOG)  =                    39678.    9670.    1014.
SLOPE-80% SIZE TO SMALLEST DATUM              0.664    0.631    1.841
SPECIFIC GRAVITY                            ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                 0.00     0.00     0.00
VOIDS FRACTION                              ******* ******* *******
BULK WEIGHT (LBS/FT**3)                     ******* ******* *******
```

```
SIEVE ANALYSIS                                                ALLIS-CHALMERS
MATERIAL          SOIL 4761
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST 7
TEST NO.85-003                                          DATE 1-10-85

        100                 10   WEIGHT % PASSING  !             .2
MESH MU-M 198  6 5 4  3   2    198  6 5 4  3   2    196  6 5 4  3   2
4.2 107MM 7
  A= AS REC. FEED SAMPLE
3.0 75000 7
  B= 4761-4 KILN PROD. TEST 7
2.1 53000 7
  C= 4761-4 EXHAUST DUST TEST 7
1.5 37500 4  A

1.1 26500 4

.75 19000 4

.53 13200 CB A

.375 9500 C B A

M=3  6700 C  BA

  4  4750 C    1

  6  3350  C    1

  8  2360  C     AB

 10  1700   C      A B

 14  1180   C       A  B

 20   850    C       A  B

 28   600     C       A     B

 35   425      C       A

 48   300         C       A

 65   212              C      A

100   150                         2

150   106                               A    C

200    75                                        A    C

270    53
325    45
400    38

500    26 +                                                        +
MESH MU-M 198  6 5 4  3   2    198  6 5 4  3   2    198  6 5 4  3   2
1=A+B     2-A+C     3-A+D         +C          3+D       A-C+D
7=A+B+C   8=A+B+D   9                                   C+D
```

```
SIEVE ANALYSIS                                                   ALLIS-CHALMERS
MATERIAL          SOIL 4768
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST 8
TEST NO.85-003                                                 DATE 1-11-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= 4768-1 TEST 8 KILN PROD.
C= 4768-1 TEST 8 EXHAUST DUST
D=
 SIEVE SIZE            A               B               C               D
EQUIV.  ASTM      PERCENTAGE      PERCENTAGE      PERCENTAGE      PERCENTAGE
T.MESH  MU-M      ON  PASSING     ON  PASSING     ON  PASSING     ON  PASSING
 .75    19000    0.00 100.00     0.00 100.00     0.00 100.00
 .53    13200    7.51  92.49     1.89  98.11     0.00 100.00
 .375    9500    4.41  88.08     2.67  95.44     0.00 100.00
 M=3     6700    3.04  85.04     3.40  92.04     0.00 100.00
   4     4750   12.36  72.68    11.83  80.21     0.00 100.00
   6     3350    8.46  64.22    10.63  69.59     2.83  97.17
   8     2360    5.56  58.66     8.19  61.40     1.70  95.48
  10     1700    2.53  56.14     3.96  57.44     1.21  94.26
  14     1180    2.12  54.02     3.26  54.18     1.62  92.65
  20      850    2.50  51.52     3.31  50.87     2.34  90.31
  28      600    4.31  47.21     5.52  45.35     5.83  84.48
  35      425    6.95  40.06     8.01  37.33    11.63  72.86
  48      300    9.11  30.95    10.43  26.90    15.27  57.59
  65      212   13.01  17.94    13.56  13.34    22.54  35.06
 100      150    7.37  10.58     7.79   5.55    17.53  17.53
 150      106    3.84   6.74     3.73   1.82    10.50   7.03
 200       75    3.06   3.67     1.44   0.37     6.54   0.48
 270       53    0.00   0.00     0.00   0.00     0.00   0.00
 325       45    0.00   0.00     0.00   0.00     0.00   0.00
 400       38    0.00   0.00     0.00   0.00     0.00   0.00
 500       26    0.00   0.00     0.00   0.00     0.00   0.00
 PAN        0    3.67   0.00     0.37   0.00     0.48   0.00
                                                  A       B       C       D
80 PCT. SIZE (LOG-LOG)                         5862.   4719.    528.
SLOPE,80% SIZE TO SMALLEST DATUM               0.707   1.296   2.616
SPECIFIC GRAVITY                             ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                 0.00    0.00    0.00
VOIDS FRACTION                               ******* ******* *******
BULK WEIGHT (LBS/FT**3)                      ******* ******* *******
```

```
SIEVE ANALYSIS                                                ALLIS-CHALMERS
MATERIAL       SOIL 4768
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 8
TEST NO.85-003                                           DATE 1-11-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= 4768-1 TEST 8 KILN PROD
  C= 4768-1 TEST 8 EXHAUST DUST
         100                   10   WEIGHT % PASSING  1              .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
.75 19000 7

.53 13200 C1                                              *            *

.375 9500 CBA

N=3  6700 CBA

  4  4750 C  BA

  6  3350  C  BA

  8  2360  C   BA

 10  1700  C    1

 14  1180  C    1

 20   850  C     1

 28   600   C     1

 35   425     C     AB

 48   300       C     AB

 65   212            C     A  B

100   150                  C    A     B

150   106                           2             B

200    75                                 A                  C  B

270    53
325    45
400    38

500    26 *                                                            *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
1=A+B    2=A+C    3=A+D    4 B+C    5=B+D    6=C+D
7=A+B+C  8=A+B+D  9=A+C+D  0=B+C+D  *=A+B+C+D
```

```
SIEVE ANALYSIS                                                  ALLIS-CHALMERS
MATERIAL         SOIL 4768
SUBMITTED BY     IT CORP.
                 BATCH KILN TEST  9
TEST NO.85-003                                             DATE 1-11-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= 4768-2 TEST 9 KILN PROD.
C= 4768-2 TEST 9 EXHAUST DUST
D=
 SIEVE SIZE             A               B               C               D
EQUIV.  ASTM       PERCENTAGE      PERCENTAGE      PERCENTAGE      PERCENTAGE
T,MESH  MU-M       ON  PASSING     ON  PASSING     ON  PASSING     ON  PASSING
  .75   19000     0.00 100.00     0.00 100.00     0.00 100.00
  .53   13200     7.51  92.49     0.94  99.06     0.00 100.00
 .375    9500     4.41  88.08     0.66  98.40     0.00 100.00
  M=3    6700     3.04  85.04     2.59  95.81     0.00 100.00
    4    4750    12.36  72.68    12.28  83.53     0.00 100.00
    6    3350     8.46  64.22    13.62  69.91     2.16  97.84
    8    2360     5.56  58.66     8.72  61.20     1.28  96.56
   10    1700     2.52  56.14     4.36  56.84     1.28  95.28
   14    1180     2.12  54.02     3.36  53.48     2.08  93.19
   20     850     2.50  51.52     3.76  49.72     2.68  90.52
   28     600     4.51  47.01     6.95  42.76     4.11  86.41
   35     425     6.95  40.06    10.11  32.65     5.37  81.04
   48     300     9.11  30.95    11.62  21.03    10.71  70.33
   65     212    13.01  17.94    12.74   8.29    29.38  40.95
  100     150     7.37  10.58     5.98   2.31    20.99  19.96
  150     106     3.84   6.74     1.68   0.63    12.69   7.26
  200      75     3.06   3.67     0.37   0.26     5.85   1.41
  270      53     0.00   0.00     0.00   0.00     0.00   0.00
  325      45     0.00   0.00     0.00   0.00     0.00   0.00
  400      38     0.00   0.00     0.00   0.00     0.00   0.00
  500      26     0.00   0.00     0.00   0.00     0.00   0.00
  PAN       0     3.67   0.00     0.26   0.00     1.41   0.00
                                                A       B       C       D
80 PCT. SIZE (LOG-LOG)  -                     5862.   4364.    412.
SLOPE,80% SIZE TO SMALLEST DATUM              0.707   1.413   2.369
SPECIFIC GRAVITY                            ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                 0.00    0.00    0.00
VOIDS FRACTION                              ******* ******* *******
BULK WEIGHT (LBS/FT**3)                     ******* ******* *******
```

```
SIEVE ANALYSIS                                              ALLIS-CHALMERS
MATERIAL       SOIL 4768
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 9
TEST NO.85-003                                         DATE 1-11-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= 4768-2 TEST 9 KILN PROD.
  C= 4768-2 TEST 9 EXHAUST DUST
         100                    10   WEIGHT % PASSING  1              .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2      198  6 5 4  3   2
.75 19000 7

.33 13200 C1                                                          *

.375 9500 CBA

M=3  6700 CBA

  4  4750 C B A

  6  3350  C  BA

  8  2360  C   BA

 10  1700  C    1

 14  1180  C    1

 20   850  C     1

 28   600   C     AB

 35   425    C     A B

 48   300     C       A  B

 65   212          C       A      B

100   150                 C     A              B

150   106                           2                      B

200    75                                 A        C               B

270    53
325    45
400    38

500    26 *                                                           *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2      198  6 5 4  3   2
1=A+B     2-A+C     3-A+D     4-B+C     5-B+D     6-C+D
7-A+B+C   8=A+B+D   9-A+C+D   0-B+C+D   +-A+B+C+D
```

```
- SIEVE$MAIN     SIEVE$MAIN                                62     00000250  00002450




SIEVE ANALYSIS                                                    ALLIS-CHALMERS
MATERIAL          SOIL 4768
SUBMITTED BY      IT CORP.
                  BATCH KILN TEST  10
TEST NO.85-003                                              DATE 1-11-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= 4768-3 TEST 10 KILN PROD.
C= 4768-3 TEST 10 EXHAUST DUST
D=
 SIEVE SIZE            A                B                C                D
EQUIV.  ASTM     PERCENTAGE       PERCENTAGE       PERCENTAGE       PERCENTAGE
T.MESH  MU-M     ON  PASSING      ON  PASSING      ON  PASSING      ON  PASSING
 .75    19000    0.00 100.00      0.00 100.00      0.00 100.00
 .53    13200    7.51  92.49      0.87  99.13      0.00 100.00
 .375    9500    4.41  88.08      3.26  95.88      0.00 100.00
 M=3     6700    3.04  85.04      2.48  93.40      0.00 100.00
   4     4750   12.36  72.68     13.92  79.48      0.00 100.00
   6     3350    8.46  64.22     13.92  65.56      2.39  97.61
   8     2360    5.56  58.66      9.17  56.39      2.39  95.22
  10     1700    2.52  56.14      4.66  51.73      2.02  93.20
  14     1190    2.12  54.02      4.15  47.58      1.17  92.03
  20      850    2.50  51.52      4.90  42.68      1.03  90.99
  28      600    4.51  47.01      8.60  34.08      2.30  88.70
  35      425    6.95  40.06     10.90  23.18      5.11  83.58
  48      300    9.11  30.95     10.51  12.66     11.73  71.86
  65      212   13.01  17.94      9.23   3.43     29.78  42.07
 100      150    7.37  10.58      2.75   0.69     24.34  17.73
 150      106    3.84   6.74      0.36   0.33     12.62   5.11
 200       75    3.07   3.67      0.18   0.15      4.27   0.84
 270       53    0.00   0.00      0.00   0.00      0.00   0.00
 325       45    0.00   0.00      0.00   0.00      0.00   0.00
 400       38    0.00   0.00      0.00   0.00      0.00   0.00
 500       26    0.00   0.00      0.00   0.00      0.00   0.00
 PAN        0    3.67   0.00      0.15   0.00      0.84   0.00
                                             A         B         C         D
80 PCT. SIZE (LOG-LOG)  =                 5862.     4816.      384.
SLOPE,80% SIZE TO SMALLEST DATUM          0.707     1.510     2.786
SPECIFIC GRAVITY                        ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS             0.00      0.00      0.00
VOIDS FRACTION                          ******* ******* *******
BULK WEIGHT (LBS/FT**3)                 ******* ******* *******
```

```
MATERIAL       SOIL 4768
SUBMITTED BY   IT CORP.
               BATCH KILN TEST 10
TEST NO.85-003                                      DATE 1-11-85

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= 4768-3 TEST 10 KILN PROD.
  C= 4768-3 TEST 10 EXHAUST DUST
         100                   10   WEIGHT % PASSING  1              .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
.75 19000 7

.53 13200 C1                                                          *

.375 9500 CBA

M=3  6700 CBA

  4  4750 C  BA

  6  3350  C   1

  8  2360  C    1

 10  1700  C    AB

 14  1180  C    A B

 20   850  C     A B

 28   600   C     A  B

 35   425   C      A    B

 48   300     C       A       B

 65   212          C       A               B

100   150                  C    A                         B

150   106                           A  C                         B

200    75                                 A             C

270    53
325    45
400    38

500    26 *                                                           *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
1=A+B     2=A+C     3=A+D     4=B+C     5=B+D     6=C+D
7=A+B+C   8=A+B+D   9=A+C+D   0=B+C+D   +=A+B+C+D
```

```
SIEVE ANALYSIS                                                    ALLIS-CHALMERS
MATERIAL         SOIL 4760
SUBMITTED BY     IT CORP.
                 BATCH KILN TEST //
TEST NO.85-003                                               DATE 1-11-85

A= AS REC. FEED SAMPLE DRIED AT 100 C
B= TEST 11 KILN PROD. 4760-4
C= TEST 11 EXHAUST DUST 4760-4
D=
 SIEVE SIZE             A               B                C                D
EQUIV.   ASTM      PERCENTAGE      PERCENTAGE       PERCENTAGE       PERCENTAGE
T.MESH   MU-M     ON   PASSING    ON   PASSING     ON   PASSING     ON   PASSING
   4     4750    0.00 100.00     0.00 100.00     0.00 100.00
   6     3350    0.04  99.96     0.08  99.92     0.09  99.91
   8     2360    0.06  99.90     0.08  99.83     0.14  99.77
  10     1700    0.06  99.84     0.04  99.79     0.09  99.68
  14     1180    0.24  99.60     0.26  99.53     0.09  99.59
  20      850    2.03  97.57     2.29  97.25     0.54  99.05
  28      600   14.65  82.92    14.94  82.31    10.76  88.29
  35      425   31.21  51.71    27.08  55.23    38.08  50.20
  48      300   29.28  22.43    28.99  26.24    33.41  16.80
  65      212   15.47   6.97    17.90   8.34    12.26   4.54
 100      150    4.20   2.77     5.59   2.75     2.95   1.59
 150      106    1.00   1.77     1.27   1.48     0.91   0.68
 200       75    0.62   1.15     0.80   0.68     0.41   0.27
 270       53    0.00   0.00     0.00   0.00     0.00   0.00
 325       45    0.00   0.00     0.00   0.00     0.00   0.00
 400       38    0.00   0.00     0.00   0.00     0.00   0.00
 500       26    0.00   0.00     0.00   0.00     0.00   0.00
 PAN        0    1.15   0.00     0.68   0.00     0.27   0.00
                                                  A        B        C        D
90 PCT. SIZE (LOG-LOG)  -                       584.     585.     565.
SLOPE,90% SIZE TO SMALLEST DATUM               2.064    2.322    2.814
SPECIFIC GRAVITY                             ******* ******* *******
ESTIMATED SP.GR. FOR 40% VOIDS                  0.00     0.00     0.00
VOIDS FRACTION                               ******* ******* *******
BULK WEIGHT (LBS/FT**3)                      ******* ******* *******
```

```
                                                         ALLIS-CHALMERS
SIEVE ANALYSIS
MATERIAL       SOIL 4760
SUBMITTED BY   IT CORP.
               BATCH KILN TEST  11
                                                    DATE 1-11-85
TEST NO.85-003

  A= AS REC. FEED SAMPLE DRIED AT 100 C
  B= TEST 11 KILN PROD.  4760-4
  C= TEST 11 EXHAUST DUST.4760-4
         100                      10   WEIGHT % PASSING  1               .2
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
                                                                        *
.53 13200 7

.375 9500 7

M=3  6700 7

  4  4750 7

  6  3350  7

  8  2360  7

 10  1700  7

 14  1180  7

 20   850  7

 28   600   7

 35   425        B2

 48   300               B A  C

 65   212                          B A   C

100   150                                     1    C

150   106                                         A B      C

200    75                                             A    B        C

270     53
325     45
400     38
                                                                        T
500     26 *
MESH MU-M 198  6 5 4  3   2     198  6 5 4  3   2     198  6 5 4  3   2
1=A+B     2-A+C     3-A+D     4-B+C     5-B+D     6-C+D
7=A+B+C   8=A+B+D   9-A+C+D   0-B+C+D   +-A+B+C+D
```

APPENDIX E

Dynamic Angle of Repose Test

Company Name IT Corporation

Test No. 1

Test Date January 9, 1985

Test Apparatus Size 38" ID x 10" Wide

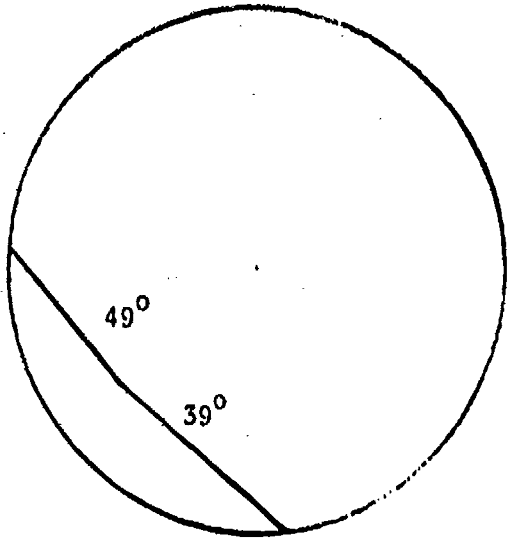
Feed Material 4761 As Received

Feed Moisture (%) 6.5

Test Apparatus Speed (rpm) 0.82

52" Mobile Kiln Equivalent Speed (rpm) 0.6

Bed Loading (%) 7



Dynamic Angle of Repose Test

Company Name IT Corporation

Test No. 2

Test Date January 9, 1985

Test Apparatus Size 38" ID x 10" Wide

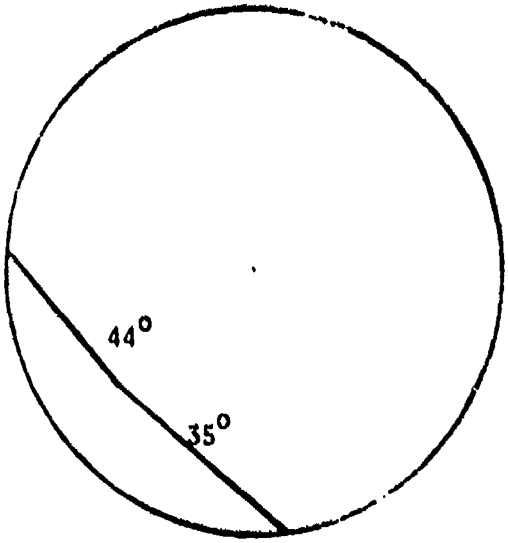
Feed Material 4760 As Received

Feed Moisture (%) 1.2

Test Apparatus Speed (rpm) 0.82

52" Mobile Kiln Equivalent Speed (rpm) 0.60

Bed Loading (%) 7



Dynamic Angle of Repose Test

Company Name IT Corporation

Test No. 3

Test Date January 10, 1985

Test Apparatus Size 10" Wide x 38" ID

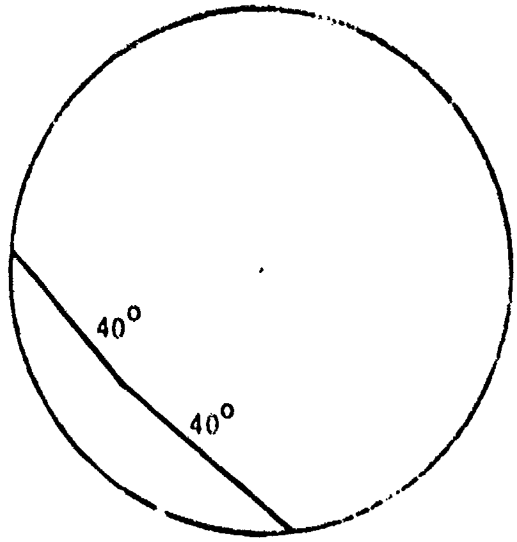
Feed Material 4768 As Received

Feed Moisture (%) 6.4

Test Apparatus Speed (rpm) 0.82

52" Mobile Kiln Equivalent Speed (rpm) 0.6

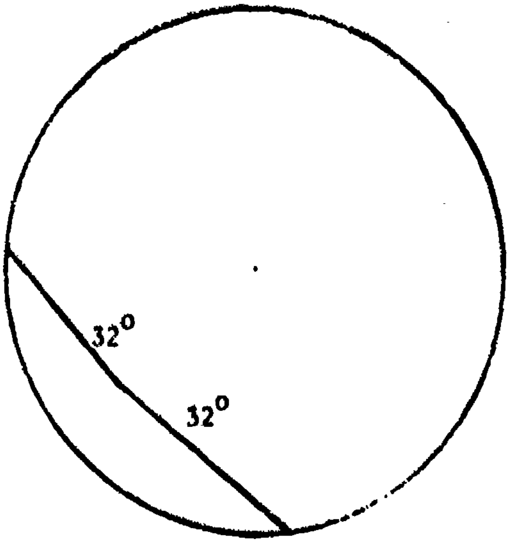
Bed Loading (%) 7



NOTE: Material did not turn over in kiln. Bed slides on kiln wall.

Dynamic Angle of Repose Test

Company Name	IT Corporation
Test No.	4
Test Date	January 14, 1985
Test Apparatus Size	38" ID x 10" Wide
Feed Material	4760 (Tests 3, 5, 6 and 11 Batch Kiln)
Feed Moisture (%)	0
Test Apparatus Speed (rpm)	0.82
52" Mobile Kiln Equivalent Speed (rpm)	0.6
Bed Loading (%)	4.42%



NOTE: Only 12.25 Kg available for test (16.66 desired).

Dynamic Angle of Repose Test

Company Name IT Corporation

Test No. 5

Test Date January 14, 1985

Test Apparatus Size 10" Wide x 38" ID

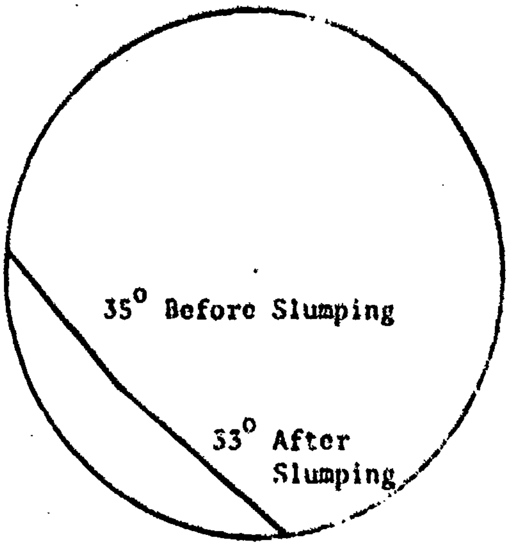
Feed Material 4768 Test Product

Feed Moisture (%) 0

Test Apparatus Speed (rpm) 0.82

52" Mobile Kiln Equivalent Speed (rpm) 0.6

Bed Loading (%) 2.54%

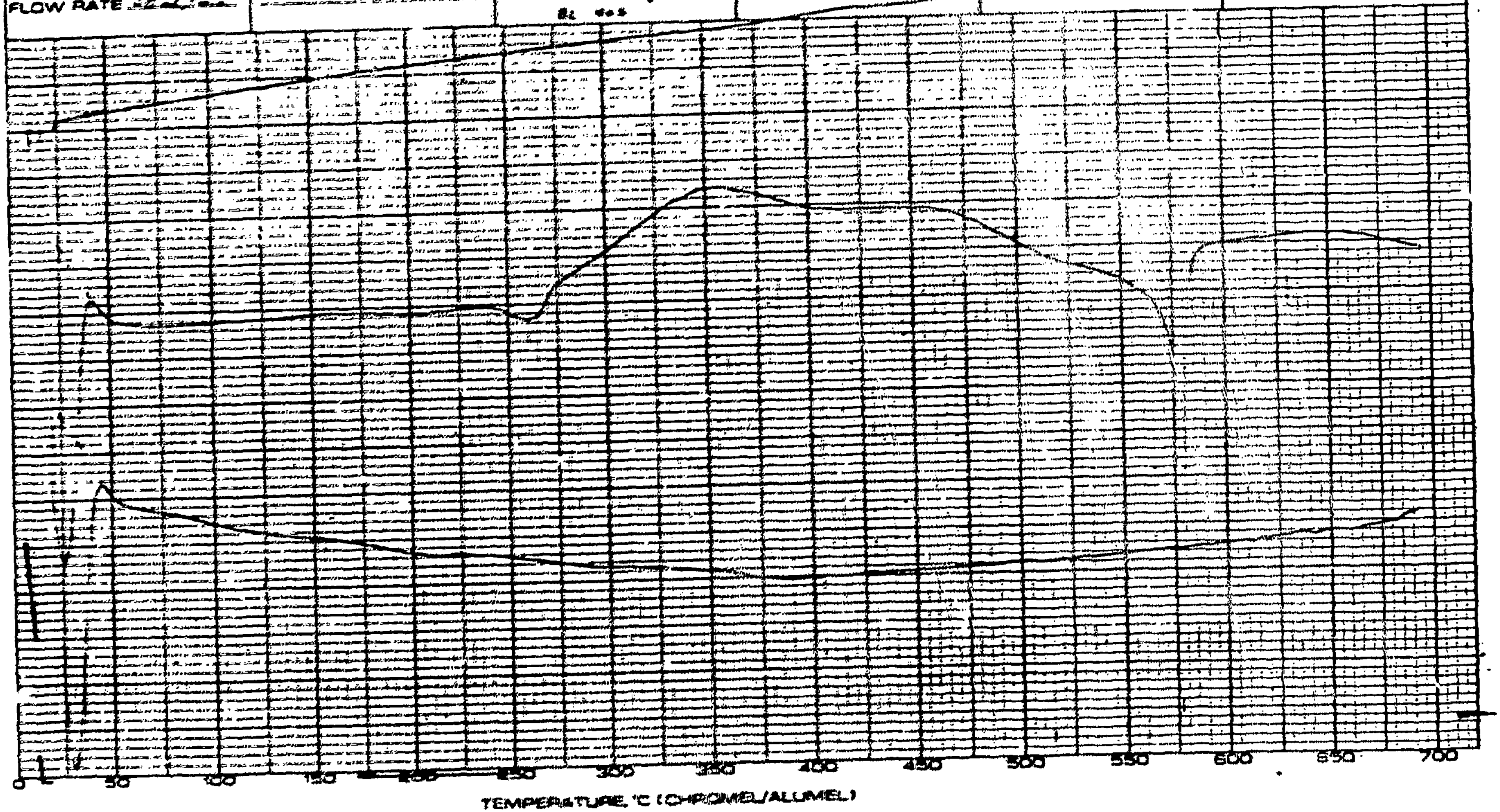

35° Before Slumping
33° After Slumping

NOTE: 9 Kg available for test (16.43 Kg desired).

APPENDIX F

PART NO. 990088

[illegible] RUN NO. __ DATE [illegible] OPERATOR E.T. SAMPLE: #763 [illegible] ATM [illegible] FLOW RATE [illegible]	**T-AXIS** SCALE, °C/in 50 PROG. RATE, °C/min 20 HEAT ✓ COOL __ ISO __ SHIFT, in __ 0	**DTA-DSC** SCALE, °C/in 1.0 (mcal/sec)/in __ WEIGHT mg 31.94 REFERENCE -0- [illegible] 53.33 mg [illegible]	**TGA** SCALE mg/in __ SUPPRESSION, mg __ WEIGHT, mg __ TIME CONST., sec __ dY, (mg/min)/in __	**TMA** SCALE mils/in __ MODE __ SAMPLE SIZE __ LOAD, g __ dY, (DOW) (mils/min)/in __



```
IT CORP PROJECT [illegible] OCT. 82

INPUT DATA

WTAL203  59.77 MG./WT.SAMPLE  31.94 MG./HR 20.DEG.C/MIN./ DELTAW 1.00

N=NUMBER OF DATA POINTS =   27

CALCULATED DATA

  TEMP      DELTA Y    DELTA Y        E            CP           H          TOT. HEAT
 DEG.C      AO IN.   SAMP IN.   INST.CONST   KCAL/KG-C     KCAL/KG       KCAL/KG
  25.0       3.18       1.35       1.1652       0.1478       0.0000          0.00
  50.0       4.13       2.25       0.9522       0.2012       4.3624          4.36
  75.0       4.42       2.37       0.9352       0.2101       5.1417          9.50
 100.0       4.62       2.45       0.9349       0.2151       5.3154         14.82
 125.0       4.80       2.50       0.9338       0.2193       5.4701         20.25
 150.0       4.93       2.53       0.9357       0.2231       5.5291         25.78
 175.0       5.07       2.59       0.9382       0.2282       5.6412         31.42
 200.0       5.24       2.67       0.9297       0.2332       5.7675         37.19
 225.0       5.32       2.67       0.9349       0.2345       5.8453         43.03
 250.0       5.42       2.80       0.9344       0.2457       6.0024         49.04
 275.0       5.56       2.55       0.9253       0.2216       5.8420         54.88
 300.0       5.64       2.24       0.9249       0.1946       5.2027         60.08
 325.0       5.72       1.91       0.9231       0.1656       4.5024         64.58
 350.0       5.82       1.71       0.9171       0.1473       3.9113         68.49
 375.0       5.96       1.83       0.9042       0.1554       3.7840         72.28
 400.0       6.05       2.07       0.8985       0.1747       4.1264         76.40
 425.0       6.07       2.14       0.9026       0.1814       4.4515         80.86
 450.0       6.08       2.20       0.9076       0.1876       4.6122         85.47
 475.0       6.13       2.37       0.9063       0.2017       4.8662         90.33
 500.0       6.16       2.73       0.9075       0.2327       5.4306         95.76
 525.0       6.23       3.05       0.9027       0.2586       6.1413        101.91
 550.0       6.27       3.32       0.9070       0.2813       6.7484        108.65
 575.0       6.30       4.37       0.9026       0.3705       8.1471        116.80
 600.0       6.32       3.10       0.9045       0.2634       7.9233        124.72
 625.0       6.35       3.13       0.9049       0.2660       6.6174        131.34
 650.0       6.38       3.17       0.9051       0.2712       6.7149        138.06
 675.0       6.40       3.33       0.9065       0.2835       6.9339        144.99

 PROGRAM EQNS

 E=WT.AL203*HR*CPAL/(60*DELTYA),MCAL/SEC-DEG.C
 CPSAMP=60*E*DELTYS/(WTSAMP*HR),KCAL/KG.DEG.C

 H/KG=CPSAMP*DELTAT,KCAL/KG.DEG.C

 CPAL203=1.72398141E-1 + 5.34395190E-4T - 1.0735742E-6(T**2)
 +1.17693296E-9(T**3) - 4.62227060E-13(T**4)
```

PART NO. 99X066

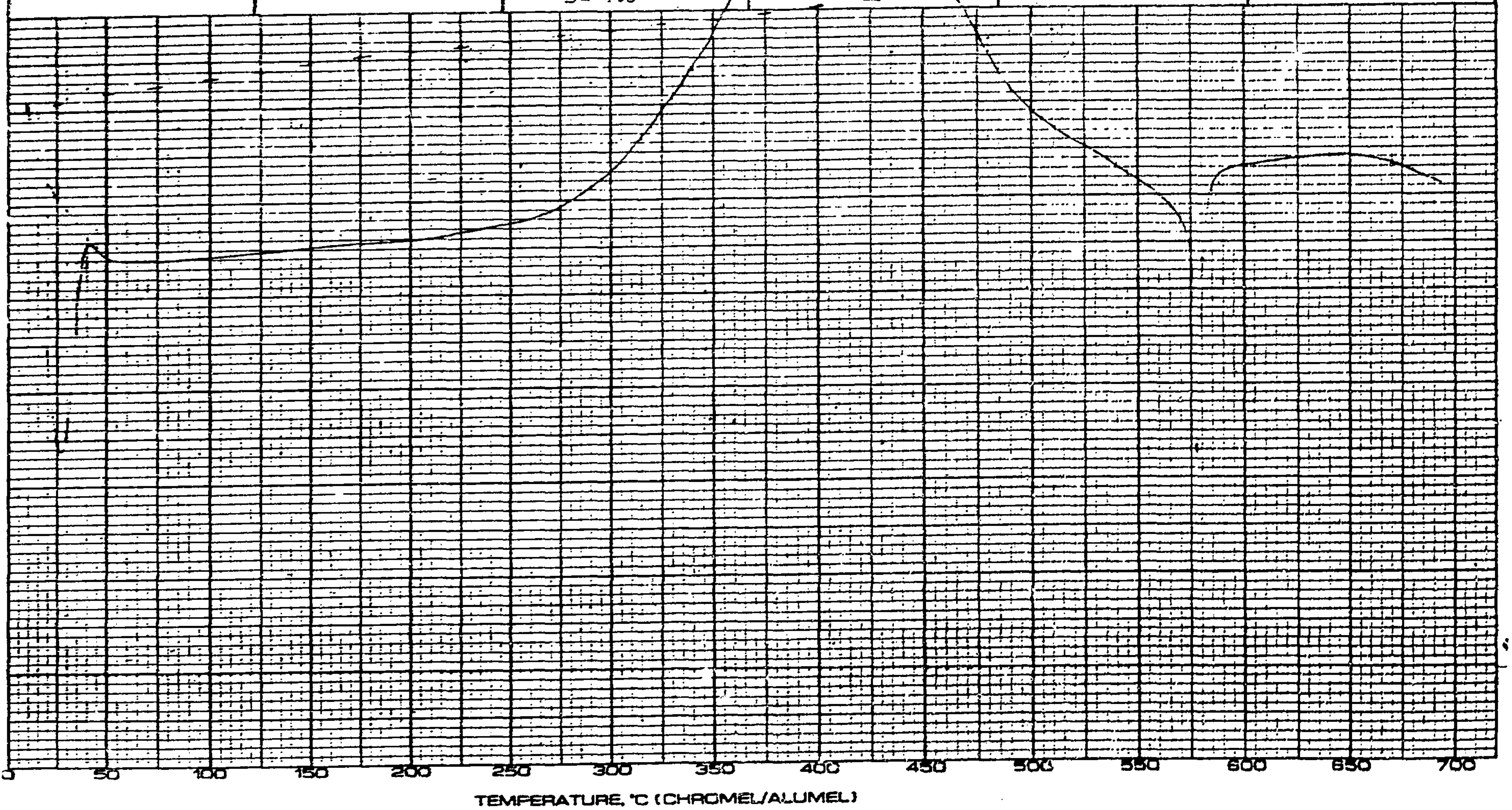
RUN NO. [illegible] DATE [illegible]
OPERATOR [illegible]
SAMPLE #768
ATM [illegible]
FLOW RATE [illegible]

T-AXIS
SCALE, °C/in 50
PROG. RATE, °C/min 20
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in 1.0
(mcal/sec)/in
WEIGHT, mg 29.53
REFERENCE -0-
A.
BL 400

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec
dY, (mg/min)/in

TMA
SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10 X); (mils/min)/in



```
IT CORP. PROJECT [illegible] DSC RUN [illegible] 10 PCT 02

INPUT DATA

WTAL203  59.77 MG./WT.SAMPLE  29.53 MG./HR 20.DEG.C/MIN./ DELTAQ 1.00

N=NUMBER OF DATA POINTS =    27

CALCULATED DATA

 TEMP     DELTA Y   DELTA Y        E           CP          H        TOT. HEAT
DEG.C     AD IN.   SAMP IN.  INST.CONST  KCAL/KG-C    KCAL/KG     KCAL/KG
  25.0     3.18      1.00      1.1652      0.1184      0.0000        0.00
  50.0     4.13      1.74      0.9522      0.1683      3.5838        3.58
  75.0     4.42      1.85      0.9359      0.1759      4.3028        7.89
 100.0     4.62      1.89      0.9349      0.1795      4.4426       12.33
 125.0     4.80      1.93      0.9338      0.1831      4.5325       16.86
 150.0     4.93      1.93      0.9387      0.1840      4.5892       21.45
 175.0     5.07      1.96      0.9382      0.1868      4.6358       26.09
 200.0     5.24      2.00      0.9297      0.1889      4.6966       30.78
 225.0     5.32      1.99      0.9349      0.1890      4.7239       35.51
 250.0     5.42      1.93      0.9344      0.1832      4.6527       40.16
 275.0     5.56      1.81      0.9253      0.1701      4.4169       44.58
 300.0     5.64      1.49      0.9249      0.1400      3.8769       48.45
 325.0     5.72      0.92      0.9231      0.0863      2.8285       51.28
 350.0     5.82      0.13      0.9171      0.0121      1.2299       52.51
 375.0     5.96     -0.94      0.9042     -0.0863     -0.9280       51.58
 400.0     6.05     -1.71      0.8985     -0.1561     -3.0305       48.55
 425.0     6.07     -1.47      0.9026     -0.1348     -3.6360       44.92
 450.0     6.06     -0.56      0.9076     -0.0516     -2.3304       42.59
 475.0     6.13      0.49      0.9063      0.0451     -0.0815       42.51
 500.0     6.16      1.37      0.9075      0.1263      2.1428       44.65
 525.0     6.23      1.81      0.9027      0.1660      3.6537       48.30
 550.0     6.27      2.25      0.9020      0.2062      4.6521       52.95
 575.0     6.30      3.20      0.9026      0.2934      6.2453       59.20
 600.0     6.32      2.27      0.9045      0.2086      6.2754       65.47
 625.0     6.35      2.29      0.9049      0.2105      5.2388       70.71
 650.0     6.38      2.35      0.9051      0.2161      5.3323       76.05
 675.0     6.40      2.55      0.9065      0.2348      5.6365       81.68

PROGRAM EQNS

E=WT.AL203*HR*CPAL/(60*DELTYA),MCAL/SEC-DEG.C
CPSAMP=60*E*DELTYS/(WTSAMP*HR),KCAL/KG.DEG.C

H/KG=CPSAMP*DELTAT,KCAL/KG.DEG.C

CPAL203=1.72398141E-1 + 5.34395190E-4T - 1.0735742E-6(T**2)
+1.17693296E-9(T**3) - 4.8282706E-13(T**4)
```

PART NO. 990088

KM-5
RUN NO.__ DATE 1/23/85
OPERATOR ETL
SAMPLE: 4761

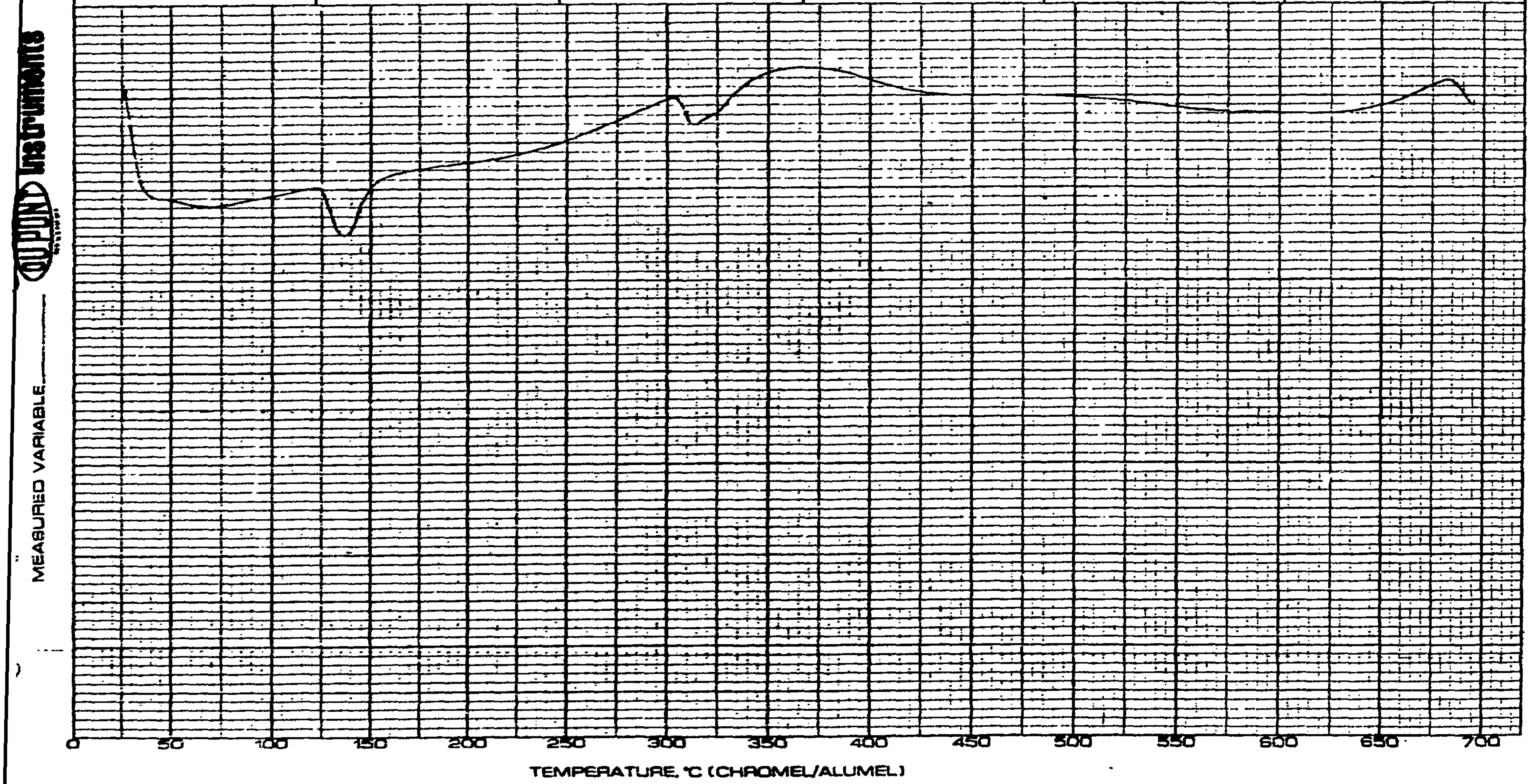
Ar-H2
ATM ~10% H2 @ ~2 cm H2O
FLOW RATE 60 ml/min

T-AXIS
SCALE, °C/in 50
PROG. RATE, °C/min 20
HEAT ✓ COOL __ ISO __
SHIFT, in 0

DTA-[DSC]
SCALE, °C/in 1.0
(mcal/sec)/in __
WEIGHT, mg 21.73
REFERENCE -0-
Al2O3 57.77 mg
BL 400

TGA
SCALE, mg/in __
SUPPRESSION, mg __
WEIGHT, mg __
TIME CONST., sec __
dY, (mg/min)/in __

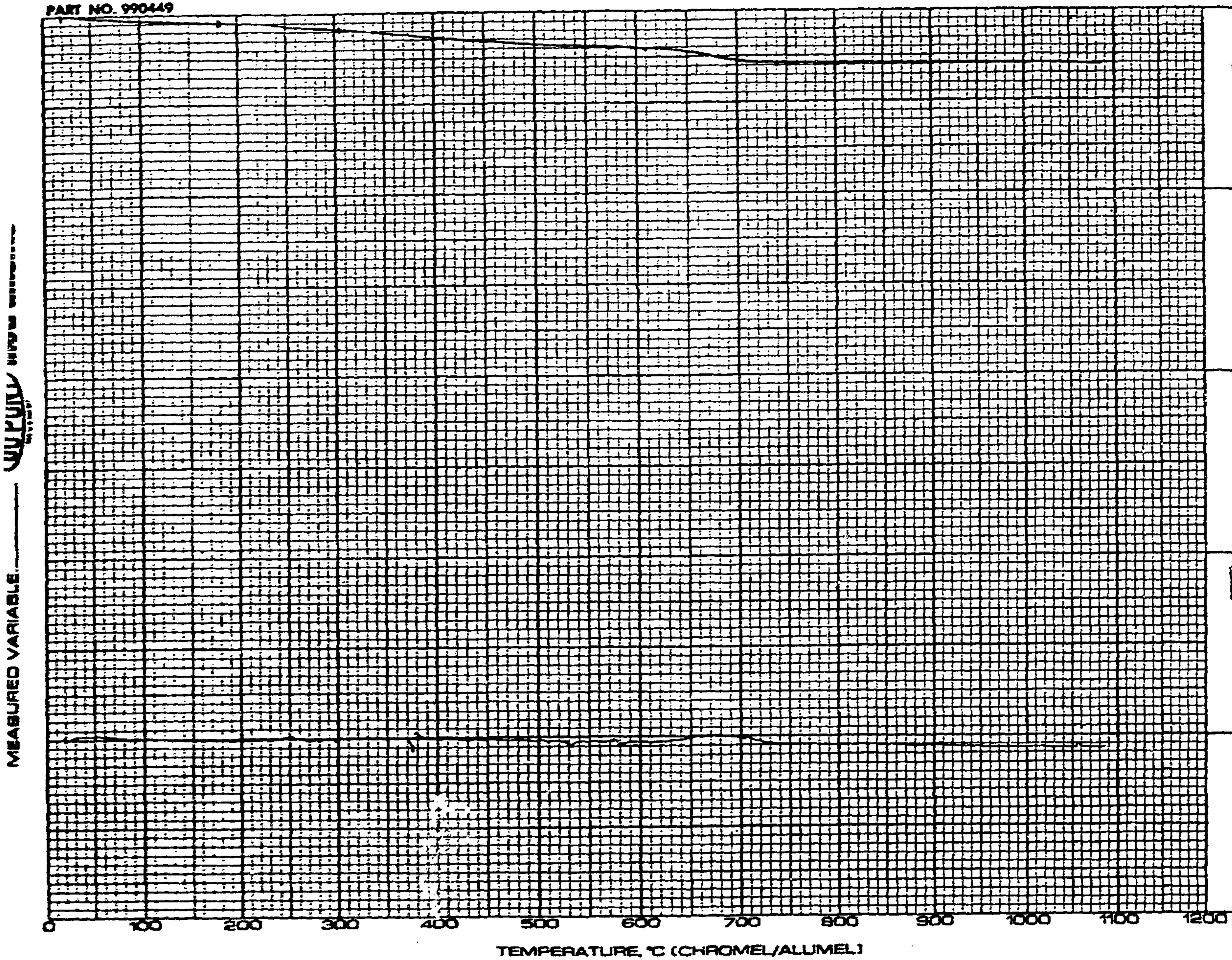
TMA
SCALE, mils/in __
MODE __
SAMPLE SIZE __
LOAD, g __
dY, (10X), (mils/min)/in __



DU PONT instruments

```
IT CORP. PROJECT 65-003 [illegible] 10 PCT O2

INPUT DATA

WTAL203  59.77 MG./WT.SAMPLE  21.73 MG./HR 20.DEG.C/MIN./ DELTAQ 1.00

N=NUMBER OF DATA POINTS =   27

CALCULATED DATA

 TEMP      DELTA Y    DELTA Y        E            CP           H          TOT. HEAT
DEG.C      AO IN.    SAMP IN.   INST.CONST   KCAL/KG-C     KCAL/KG       KCAL/KG
 25.0       3.18       0.00       1.1652       0.0000       0.0000          0.00
 50.0       4.13       1.30       0.9522       0.1709       2.1363          2.14
 75.0       4.42       1.42       0.9359       0.1835       4.4297          6.57
100.0       4.62       1.33       0.9349       0.1781       4.5199         11.09
125.0       4.80       1.40       0.9338       0.1805       4.4825         15.57
150.0       4.93       1.45       0.9367       0.1879       4.6049         20.17
175.0       5.07       1.31       0.9382       0.1697       4.4599         24.64
200.0       5.24       1.31       0.9297       0.1681       4.2229         28.87
225.0       5.32       1.25       0.9349       0.1613       4.1186         32.98
250.0       5.42       1.19       0.9344       0.1535       3.9356         36.92
275.0       5.55       1.00       0.9253       0.1277       3.5157         40.44
300.0       5.64       0.83       0.9249       0.1060       2.9216         43.36
325.0       5.72       1.04       0.9231       0.1325       2.9815         46.34
350.0       5.82       0.63       0.9171       0.0798       2.6538         48.99
375.0       5.96       0.63       0.9042       0.0786       1.9801         50.97
400.0       6.05       0.84       0.8995       0.1042       2.2855         53.26
425.0       6.07       1.03       0.9026       0.1284       2.9069         56.17
450.0       6.08       1.12       0.9076       0.1403       3.3587         59.52
475.0       6.13       1.20       0.9063       0.1501       3.6311         63.16
500.0       6.16       1.31       0.9075       0.1641       3.9234         67.08
525.0       6.23       1.42       0.9027       0.1770       4.2637         71.35
550.0       6.27       1.56       0.9020       0.1943       4.6404         75.99
575.0       6.30       1.69       0.9026       0.2106       5.0608         81.05
600.0       6.32       1.78       0.9045       0.2223       5.4110         86.46
625.0       6.35       1.83       0.9049       0.2349       5.7142         92.17
650.0       6.38       1.88       0.9051       0.2349       5.8720         98.05
675.0       6.40       1.74       0.9065       0.2178       5.6584        103.70

PROGRAM EQNS

E=WT.AL203*HR*CPAL/(60*DELTYA),MCAL/SEC-DEG.C
CPSAMP=60*E*DELTYS/(WTSAMP*HR),KCAL/KG.DEG.C

H/KG=CPSAMP*DELTAT,KCAL/KG.DEG.C

CPAL203=1.72398141E-1 + 5.34395190E-4T - 1.0735742E-6(T**2)
+1.17693296E-9(T**3) - 4.32827060E-13(T**4)
```

PART NO. 990449



KM-1
RUN NO.___ DATE 1/21/74
OPERATOR ETL
SAMPLE: 4760
Air - N2
ATM ~10% O2 @ ~2cm, H2O
FLOW RATE 60ml/min

T-AXIS
SCALE, °C/in 100
PROG. RATE, °C/min 20
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in ___
WEIGHT, mg ___
REFERENCE ___

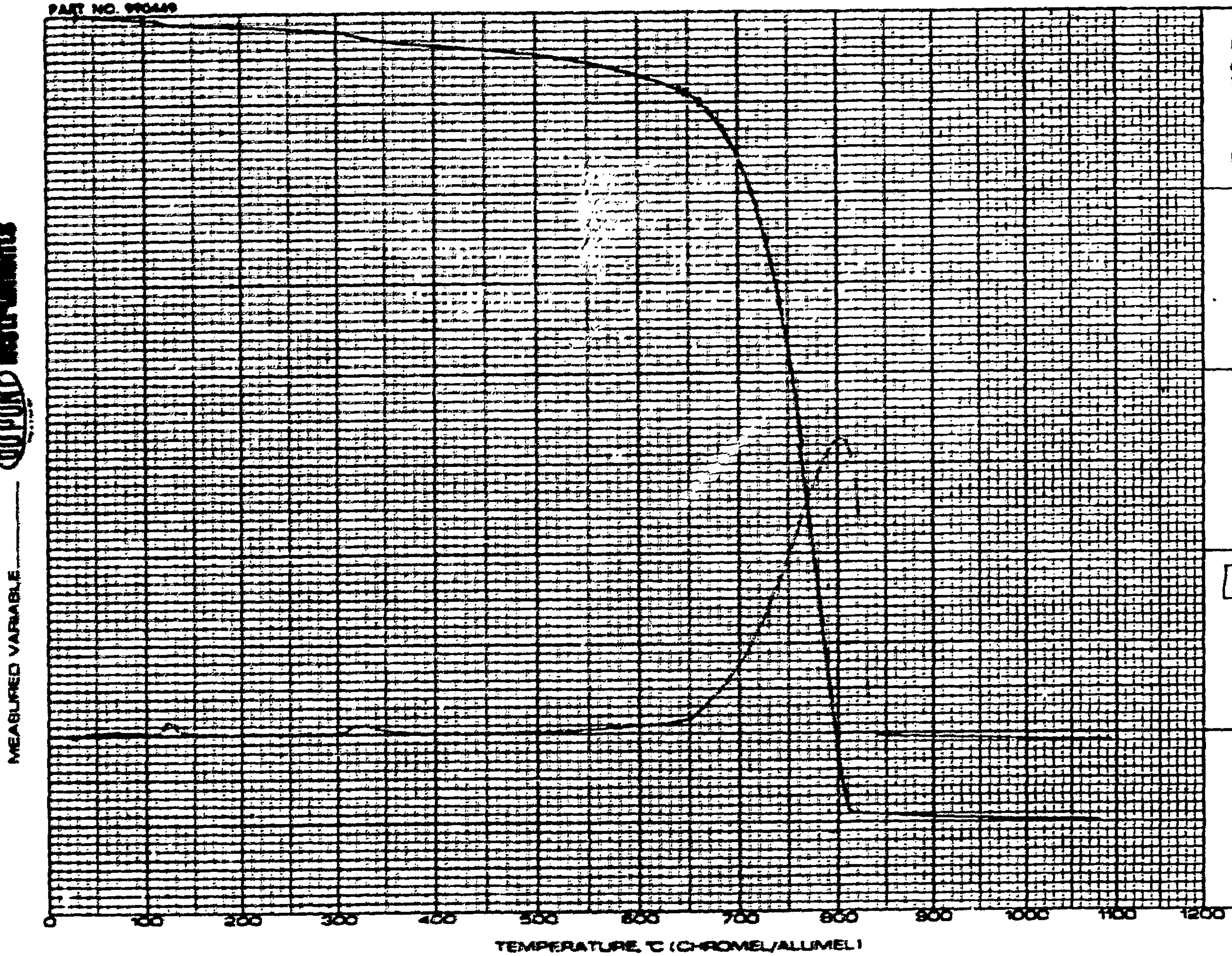
TGA
SCALE, mg/in 1.0
SUPPRESSION, mg 20.54
WEIGHT, mg 30.53 mg
TIME CONST., sec 5
dY, (mg/min)/in 0.5

TMA
SCALE, mils/in C.P. ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___

SOIL 4760 As Received TGA

0.72 mg. total Wt loss @ 1050°C

or $\frac{0.72}{30.53} \times 100 = 2.36$ %



RUN NO. KM-7 DATE 2-85
OPERATOR E-L
SAMPLE: -7½1

ATM Air + N2 ~15% O2 @ ~2cm H2O
FLOW RATE 60ml/min

T-AXIS
SCALE, °C/in 100
PROG. RATE, °C/min 2.2
HEAT ✓ COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in ___
WEIGHT, mg ___
REFERENCE ___

TGA
SCALE, mg/in 1.0
SUPPRESSION, mg 10.26
WEIGHT, mg 20.29
TIME CONST., sec 5
dY, (mg/min)/in 0.5

TMA
SCALE, mils/in C.R. ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___

Soil 4761 As Received TGA

8.90 mg. total loss @ 1050°C

2.80mg loss @ 350 °C

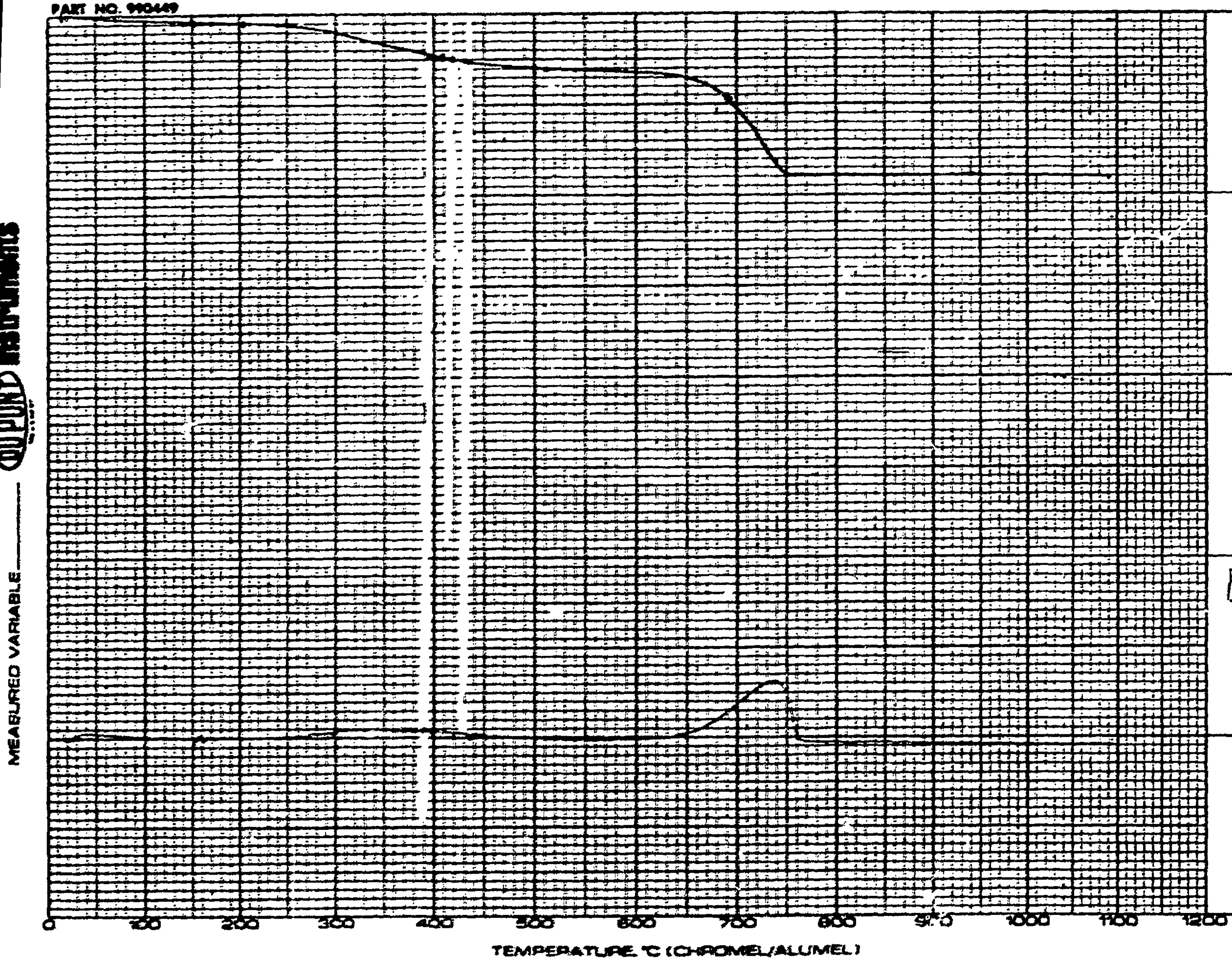
total Wt loss = $\frac{8.90}{20.26} \times 100 = 43.93\%$

Calcination Wt. loss

$$\frac{8.90 - 2.80}{20.26} \times 100 = 30.11 \%$$

DU PONT Instruments

PART NO. 990449



RUN NO. 1.4-3 DATE [illegible]
OPERATOR STL
SAMPLE: 4763

ATM [illegible]
FLOW RATE [illegible]

Y-AXIS
SCALE, °C/in 100
PROG RATE, °C/min 20
HEAT / COOL ___ ISO ___
SHIFT, in 0

DTA-DSC
SCALE, °C/in ___
(mcal/sec)/in ___
WEIGHT, mg ___
REFERENCE ___

TGA
SCALE, mg/in 1.0
SUPPRESSION, mg 21.34
WEIGHT, mg 31.34
TIME CONST., sec 5
dY, (mg/min)/in 0.5

TMA
SCALE, mils/in C.P. ___
MODE ___
SAMPLE SIZE ___
LOAD, g ___
dY, (10X), (mils/min)/in ___

Soil 4768 As Received TGA

1.73 mg. loss @ 1090°C

$\frac{1.73}{31.34} \times 100 = 5.52\ \%$ Wt loss

0.55 mg. loss @ 520°C

$\frac{0.55}{31.34} \times 100 = 1.75\ \%$ Wt. loss

0.10 mg. loss @ 200°C

$\frac{0.10}{31.34} \times 100 = 0.32\ \%$ Wt loss

ANALYTICAL METHODS FOR 2,3,7,8-TCDD

5A SAMPLE PREPARATION PROCEDURES - JAR METHOD

5B SAMPLE PREPARATION PROCEDURES - SOXHLET METHOD

5C ANALYSES PROCEDURES

5D STANDARD VERIFICATION

NOTE: THESE ARE REPRINTED AS USED

APPENDIX A

SAMPLE PREPARATION PROCEDURES FOR DIOXIN IN SOILS - JAR METHOD

1.0 SUMMARY OF METHOD

1.1 Soil and sediment samples are extracted with a methanol/hexane mixture using a jar extraction technique after spiking the sample with $^{37}Cl_4$-2,3,7,8-TCDD and $^{13}C_{12}$-2,3,7,8-TCDD. After filtering, sample cleanup procedures are followed before analysis by GC/MS is performed.

2.0 REAGENTS

2.1 Spiking standard solution (contains both internal and surrogate standards). $^{13}C_{12}$-2,3,7,8-TCDD internal standard at a concentration of 500 ng/ml and $^{37}Cl_4$-2,3,7,8-TCDD surrogate standard at a concentration of 100 ng/ml, both in the same hexane solution. The standard ID number is 358:32-IFB SOIL. GC/MS IS mixture is prepared at a concentration of 200 ng/ml in isooctane.

2.2 Sulfuric acid (concentrated); ACS grade; specific gravity 1.84.

2.3 Potassium hydroxide; 20% aqueous. Prepare by cautiously adding, with stirring, 200 g of potassium hydroxide pellets to 800 ml of distilled/deionized water contained in a beaker in a cold water bath. After the potassium hydroxide has dissolved and the solution is at room temperature, transfer to a plastic bottle.

2.4 Methylene chloride; pesticide quality or equivalent.

2.5 Hexane; pesticide quality or equivalent.

2.6 Methanol; pesticide quality or equivalent.

2.7 Toluene; pesticide quality or equivalent.

2.8 Sodium sulfate; ACS, granular, anhydrous. Prepare by a 24 hour methylene chloride extraction. After preparation, store in an oven maintained at 110°C.

2.9 Silica gel; type 60, EM reagent, 70-230 mesh, or equivalent. Prepare by soxhlet extraction with methylene chloride overnight, drying, and then activating in an aluminum foil covered glass container for 24 hours at 130°C.

2.10 Alumina-neutral; Fisher brand, 80-200 mesh. Prepare by soxhlet extraction with methylene chloride, followed by drying and then activating, in an aluminum foil-covered glass container for 24 hours at 190°C.

2.11 Sulfuric acid; impregnated silica gel, 40% w/w. To prepare, add two parts (200 g) concentrated sulfuric acid to three parts silica gel (300 g) contained in a one-glass liter bottle equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label the bottle with "Sulfuric Acid - impregnated silica gel" and "Caution - contains concentrated sulfuric acid."

2.12 Sodium hydroxide modified silica gel, 33% w/w. To prepare, add 1 part 1M NaOH to two parts activated silica gel contained in a glass jar equipped with a teflon lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label bottle with "NaOH - impregnated silica gel".

2.13 Carbopak C (activated carbon) on Celite; prepare by thoroughly mixing 3.6 grams of Carbopak C (80/100 mesh) and 16.4 grams of Celite 545 in a 40-ml vial. Activate at 130°C for six hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopak/Celite to ensure TCDD recovery of > 50%. Subject the low level concentration calibration solution to the procedure in section 6.3.0-6.3.12 and measure the quantity of labeled and unlabeled TCDD.

3.0 CAUTIONS

3.1 Samples received for this preparation procedure are of unknown composition but may be potentially carcinogenic, mutagenic, toxic or in other ways hazardous.

3.2 It is mandatory that the initial weighing of the samples, addition of isotopically labelled 2,3,7,8-TCDD, and mixing of the sample, be performed inside of the designated dioxin hood, in the high hazard laboratory.

3.3 Personnel involved in this sample preparation procedure should be thoroughly familiar with laboratory SOP's on the processing of high hazard samples.

4.0 EQUIPMENT AND MATERIALS

4.1 Electrical platform shakers.

4.2 500 ml glass jars (amber) with teflon-lined screw caps to be used on the platform shakers.

4.3 Kuderna-Danish concentration apparatuses, consisting of a three-ball macro Snyder column, a 500 ml evaporative flask and a 10 ml graduated concentrator tube.

4.4 Mini vials (reactivials); 1.0 ml. capacity with conical interiors and graduated at 0.1 ml; equipped with teflon-faced rubber septa and screw caps.

4.5 Concentration vials; 20 ml screw top, septum sealed, scintillation vials.

4.6 Concentration device; nitrogen blowdown apparatus, Organomation and Pierce concentration devices, or equivalent.

4.7 Glass filtering funnels, short stem.

4.8 Filter paper, Whatman #4 or equivalent.

5.0 SAMPLE EXTRACTION: Jar extraction. NOTE: Extremely wet samples may require centrifuging to remove water before addition of sodium sulfate. The liquid will be run as a water sample (see water method).

5.1 Prepare designated dioxin hood as instructed in laboratory SOP for processing high hazard samples. This includes obtaining, preparing and labelling the requisite number of 500 ml jars before placing in the hood.

5.2 Transfer 10 g of the soil or sediment (wet weight) to a tared 500 ml jar (± 0.5 g weighed to 3 significant figures).

5.3 Spike the sample with 100 µl of spiking solution, containing both internal and surrogate standards, adding the solution at several sites over the surface of the sample.

5.4 Add 20 g anhydrous sodium sulfate. Stir the mixture thoroughly with a stainless steel spatula.

5.5 Allow the mixture to stand for two hours, mix thoroughly with the spatula and allow the mixture to stand for an additional six hours. Mix the sample again, insuring that no lumps are present. During the period that the samples set, turn off the light in the hood and close the hood sash.

5.5 Add 20 ml of methanol, stir, and add 150 ml of hexane. Remove the spatula while rinsing it with hexane.

5.7 Place the extraction jar containing the soil, sodium sulfate, and solvents in the shaker and shake for at least 3 hours.

5.8 After the three hour shaking period, turn off the shaker and allow the solids to settle before proceeding.

5.9 Into the top of a 500 ml KD flask, insert a glass filter funnel containing Whatman #4 filter paper (or equivalent) rinsed with hexane.

5.10 Carefully decant the extract through the filter funnel, using a stainless steel spatula to facilitate the transfer process.

5.11 Rinse the inside of the jar and contents with hexane, using the spatula to mix the hexane with the solid material remaining in the beaker.

5.12 Decant the washing into the filter funnel, using the spatula to facilitate the transfer.

5.13 Concentrate the extract volume to approximately 3 ml with a Kuderna-Danish apparatus.

5.14 Transfer the concentrated extract to a 20 ml scintillation vial. Rinse the evaporator flask with three 5 ml portions of hexane; transfer each rinse into the scintillation vial. During these transfers, evaporate the solvent using a gentle stream of dry nitrogen.

5.15 After the final rinse has been added, reduce the extract volume to approximately 1 ml.

6.0 DUAL COLUMN CLEAN-UP

6.1 Either the necessary columns have been prepared and are in a drying oven, or the silica gel and alumina columns need preparation. If columns need to be prepared, obtain enough 1 x 20 cm columns (for the silica gel columns) and 1 x 30 cm columns, for the alumina columns.

6.2 Place a small wad of glass wool in the bottom of the 1 x 20 cm columns and add 1.0 g of silica gel, 2.0 g of the NaOH impregnated silica gel, 1 g silica gel, 4 g of the 40% w/w sulfuric acid treated silica gel and 2.0 g of silica gel. Gently tap the columns to allow the contents to settle, after each addition.

6.3 Place a small wad of glass wool in the bottom of the 1 x 30 cm columns and add 6 g of alumina and a 1 cm layer of sodium sulfate. Gently tap the columns to allow the contents to settle.

6.4 Attach the silica gel and alumina columns to lab supports so that the silica gel column is above the alumina column and the lower tip of the silica gel column is inserted into the top of the alumina column.

6.5 Rinse both columns with hexane to remove any air bubbles and discard the hexane.

6.6 When the silica gel and alumina columns have stopped dripping hexane, place a clean Erlenmeyer flask under the alumina column and transfer the hexane concentrate obtained from step 5.15 to the top of the silica gel column. Rinse the scintillation vial with 2 x 0.5 ml portions of hexane and add the washings to the top of the silica gel column.

6.7 Wash the silica gel column with 120 ml of hexane, added in aliquots by means of a transfer pipet. Remove the silica gel column.

6.8 Wash the alumina column by placing 20 ml of hexane on the column and elute until the hexane has dropped below the sodium sulfate layer. Remove the Erlenmeyer flask and retain (with aluminum foil cap) until directions are given to discard.

6.9 Place a clean, labelled 125 ml Erlenmeyer flask under the alumina column and place 20 ml of 20% v/v methylene chloride in hexane on the alumina column, catching the eluent in the Erlenmeyer flask.

6.10 Place the 125 Erlenmeyer flask on the Organomation or Pierce concentration apparatus and reduce the volume of the solvent until less than 10 ml of solvent is remaining, but do not evaporate to dryness.

6.11 Quantitatively transfer the hexane extract to labelled 10 ml K-D concentrator tubes. Rinse the Erlenmeyer flask with several small (1-2 ml) portions of hexane, adding the washes to the K-D concentrator tube.

6.12 Transfer the K-D concentrator tubes to the concentration device and concentrate the hexane extract to approximately 1.0 ml, using a gentle stream of nitrogen and heat if necessary.

6.13 After the 1.0 ml volume has been obtained, remove the K-D concentrator tube and quantitatively transfer the contents of the K-D concentrator tube to designated conical mini-vials. Rinse the concentrator tube with 2 X 0.5 ml portions of hexane and transfer the washes to the concentrate vial. Complete necessary paperwork (See QA section.)

6.14 Store the hexane concentrate in a freezer until just prior to GC/MS analysis.

6.15 Concentrate the hexane to near dryness and add 50 µl of 200 ppb standard of ^{13}C-TCDF. Return sample to the reach-in refrigerator.

7.0 ACTIVATED CARBON CLEAN-UP

7.1 After GC/MS analysis of samples processed through the dual column methodology in Section 6.0, the possibility exists that certain matrices may produce indeterminable results. In such cases an activated carbon column clean-up technique will be employed.

7.2 Obtain from the desiccator the activated Celite/Carbopak (from Section 2.13).

7.3 Insert a small wad of glass wool into a small (7 mm O.D. x 15 cm) disposable pipet and, using vacuum aspiration at the pointed tip of the pipet, add the Celite/Carbopak mixture until a 2 cm column is obtained.

7.4 Prepare the column by adding the following solvents/solvent mixtures in the designated aliquot sizes: (place a clean 125 ml Erlenmeyer flask under the column)

- 2 ml toluene,
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene,
- 1 ml 50/50 (v/v) cyclohexane/methylene chloride,
- 2 ml hexane.

7.5 When the bottom of the hexane meniscus just touches the top of the Celite/Carbopak material, quantitatively transfer the 50 µl of sample onto the column.

7.6 Rinse the sample container with 2 1 ml portions of hexane, adding the rinses onto the column.

7.7 Sequentially wash the column with the following solvents/solvent mixtures in the designated aliquot sizes:

- 1 ml 50/50 (v/v) cyclohexane/methylene chloride
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene.

7.8 Remove the 125 ml Erlenmeyer flask and replace with a concentrator tube or Reacti-vial.

7.9 Elute the TCDD from the column with 2 ml of toluene into the concentrator tube or Reacti-vial.

7.10 Store the toluene eluent in a freezer until the GC/MS analysis is to be performed.

7.11 Shortly before the analysis, concentrate the extract to near dryness and add 50 ul of isooctane for GC/MS analysis.

7.12 Complete any paperwork requirements and store the concentrate in the reach-in refrigerator (See QA section for examples of paperwork.)

8.0 CAUSTIC AND ACID CLEANUP (OPTIONAL)

8.1 Certain samples may require additional cleanup before column chromatography in order to achieve acceptable detection limits. The following is a procedure which involves acid and caustic wash of the sample.

8.2 After step 5.15 is completed, quantitatively transfer the extract to a 125-ml separatory funnel.

8.3 Wash the extract with 30 ml 20% aqueous potassium hydroxide by shaking for 10 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.4 Wash the extract with 25 ml of distilled deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.5 Slowly add 50 ml concentrated sulfuric acid to the extract and shake for 10 minutes. Let stand for 10 minutes and discard the acid layer. Repeat until acid layer remains colorless after extraction.

8.6 Wash extract with 20 ml distilled/deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.7 Quantitatively transfer the organic layer to a 20-ml scintillation vial and dry over 10 g anhydrous sodium sulfate.

8.8 Reduce the extract volume to approximately 1 ml.

8.9 Proceed to dual column cleanup if required.

9.0 GLASSWARE PREPARATION PROCEDURES

9.1 Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water; drain dry. Rinse with high purity acetone and hexane and allow to air dry. When dry, heat in a muffle furnace to 400°C for 1 hour. (Volumetric glassware should not be heated in a muffle furnace.) Remove from the oven when cool; store inverted in a clean environment.

APPENDIX B

SAMPLE PREPARATION PROCEDURES FOR DIOXIN IN SOILS - SOXHLET METHOD

1.0 SUMMARY OF METHOD

1.1 Soil samples are spiked with isotopically labeled TCDD, pretreated with 1 N-HCl for 1 hour and air dried. The dry soil is transferred to a glass soxhlet thimble and subsequently soxhlet extracted with benzene for 16 hours. The extract is concentrated and cleaned up using liquid column chromatography steps. The extract is analyzed by HRGC/LRMS for 2,3,7,8-TCDD.

2.0 REAGENTS

2.1 Spiking standard solution (contains both internal and surrogate standards). $^{13}C_{12}$-2,3,7,8-TCDD internal standard at a concentration of 500 ng/ml and $^{37}Cl_4$-2,3,7,8-TCDD surrogate standard at a concentration of 100 ng/ml, both in the same hexane solution. The standard ID number is 358:32-IFB. GC/MS IS mixture is prepared at a concentration of 200 ng/ml in isooctane.

2.2 Sulfuric acid (concentrated); ACS grade; specific gravity 1.84.

2.3 Methylene chloride; pesticide quality or equivalent.

2.4 Hexane; pesticide quality or equivalent.

2.5 1-N HCl

2.6 Benzene; pesticide quality or equivalent.

2.7 Sodium sulfate: ACS, granular, an hydrous. Prepare by a 24-hour methylene chloride extraction. Store in an oven maintained at 110°C.

2.8 Silica gel; type 60, EM reagent, 70-230 mesh, or equivalent. Prepare by soxhlet extraction with methylene chloride overnight, drying, and then activating in an aluminum foil covered glass container for 24 hours at 130°C.

2.9 Alumina-neutral; Fisher brand, 80-200 mesh. Prepare by soxhlet extraction with methylene chloride, followed by drying and then activating in an aluminum foil-covered glass container for 24 hours at 190°C.

2.10 Sulfuric acid; impregnated silica gel, 40% v/v. To prepare, add two parts (200 g) concentrated sulfuric acid to three parts silica gel (300 g) contained in a glass liter bottle equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label the bottle with "Sulfuric Acid - impregnated silica gel" and "Caution - contains concentrated sulfuric acid," date prepared and person preparing the reagent. All information will be recorded in the laboratory standard notebook.

2.11 Sodium hydroxide modified silica gel, 33% v/v. To prepare, add 1 part 1M NaOH to two parts activated silica gel contained in a glass jar equipped with a Teflon-lined screw cap. Mix thoroughly with a glass rod until no lumps are visible. Label bottle with "NaOH - impregnated silica gel," date prepared and person preparing the reagent. All information will be recorded in the laboratory standard notebook.

2.12 Carbopak C (activated carbon) on Celite; prepare by thoroughly mixing 3.6 g of Carbopak C (80/100 mesh) and 16.4 g of Celite 545 in a 40-ml vial. Activate at 130°C for six hours. Store in a desiccator. CAUTION: Check each new batch of mixed Carbopak/Celite to ensure TCDD recovery of > 50%. Subject the low level concentration calibration solution to the procedure in section 6.3.0-6.3.12 and measure the quantity of labeled and unlabeled TCDD. Label bottle with "Activated Carbopak/Celite," date prepared and person preparing the the reagent. All information will be recorded in the laboratory standard notebook.

2.13 Potassium hydroxide; 20% aqueous. Prepare by cautiously adding, with stirring, 200 g of potassium hydroxide pellets to 800 ml of distilled/deionized water contained in a beaker in a cold water bath. After the potassium hydroxide has dissolved and the solution is at room temperature, transfer to a plastic bottle.

3.0 CAUTIONS

3.1 Samples received for this preparation procedure are of unknown composition but may be potentially carcinogenic, mutagenic, toxic or in other ways hazardous.

3.2 It is mandatory that all handling of the samples be performed inside of the designated dioxin hood in the high hazard laboratory.

3.3 Personnel involved in this sample preparation procedure should be thoroughly familiar with laboratory SOPs on the processing of high hazard samples.

4.0 EQUIPMENT AND MATERIALS

4.1 Glass soxhlet system with glass thimbles.

4.2 Heating mantles with temperature control.

4.3 Kuderna-Danish concentration apparatuses, consisting of a three-ball macro Snyder column, a 500-ml evaporative flask, and a 10-ml graduated concentrator tube.

4.4 Mini vials (reactivials); 1.0 ml. capacity with conical interiors and graduated at 0.1 ml; equipped with Teflon-faced rubber septa and screw caps.

4.5 Concentration vials; 20-ml screw top, septum sealed, scintillation vials.

4.6 Concentration device; nitrogen blowdown apparatus, Organomation and Pierce concentration devices, or equivalent.

4.7 Glass filtering funnels, short stem.

4.8 Filter paper, Whatman No. 4 or equivalent.

4.9 Filter paper, Whatman No. 2 or equivalent.

4.10 Bucnner funnel set up.

5.0 SAMPLE PREPARATION AND EXTRACTION

5.1 Prepare designated dioxin hood as instructed in laboratory SOP for processing high hazard samples. This includes obtaining, preparing and labeling the requisite number of soxhlet systems.

5.2 Weight out 10g of soil into a 250 ml amber jar and spike with 100 μl of the internal/surrogate mixture.

5.3 Add 100 ml of 1-N HCl to the jar and shake on a platform shaker for 1 hour.

5.4 Pour the soil/acid mixture through a Bucnner Funnel and neutralize the soil by pouring 300 ml Distilled water through the Funnel. Allow the soil to air dry for 16 hours.

5.5 Place a small amount of silica gel in a glass extraction thimble.

5.6 Carefully transfer the dry sample into the extraction thimble. Charge the soxhlet extractor with fresh benzene and soxhlet extract for 16 hours.

5.7 Concentrate the extract volume to approximately 3 ml with a K-D apparatus.

5.8 Transfer the concentrated extract to a 20-ml scintillation vial. Rinse the evaporator flask with three 5-ml portions of hexane; transfer each rinse into the scintillation vial. During these transfers, evaporate the solvent using a gentle stream of dry nitrogen.

5.9 After the final rinse has been added, reduce the extract volume to approximately 1 ml.

6.0 DUAL COLUMN CLEAN-UP

6.1 Either the necessary columns have been prepared and are in a drying oven, or the silica gel and alumina columns need preparation. If columns need to be prepared, obtain enough 1 X 20 cm columns (for the silica gel columns) and 1 X 30 cm columns, for the alumina columns.

6.2 Place a small wad of glass wool in the bottom of the 1 X 20 cm columns and add 1.0 g of silica gel, 2.0 g of the NaOH impregnated silica gel, 1 g silica gel, 4 g of the 40% w/w sulfuric acid treated silica gel and 2.0 g of silica gel. Gently tap the columns to allow the contents to settle after each addition.

6.3 Place a small wad of glass wool in the bottom of the 1 X 30 cm columns and add 6 g of alumina and a 1-cm layer of sodium sulfate. Gently tap the columns to allow the contents to settle.

6.4 Attach the silica gel and alumina columns to lab supports so that the silica gel column is above the alumina column and the lower tip of the silica gel column is inserted into the top of the alumina column.

6.5 Rinse both columns with hexane to remove any air bubbles and discard the hexane.

6.6 When the silica gel and alumina columns have stopped dripping hexane, place a clean Erlenmeyer flask under the alumina column and transfer the hexane concentrate obtained from step 5.8 to the top of the silica gel column. Rinse the scintillation vial with two 0.5-ml portions of hexane and add the washings to the top of the silica gel column.

6.7 Wash the silica gel column with 90 ml of hexane, added in aliquots by means of a transfer pipet. Remove the silica gel column.

6.8 Wash the alumina column by placing 20 ml of hexane on the column and eluting until the hexane has dropped below the sodium sulfate layer. Remove the Erlenmeyer flask and retain (with aluminum foil cap) until directions are given to discard.

6.9 Place a clean, labeled 125-ml Erlenmeyer flask under the alumina column and place 20 ml of 20% v/v methylene chloride in hexane on the alumina column, catching the eluent in the Erlenmeyer flask.

6.10 Place the 125-Erlenmeyer flask on the Organomation or Pierce concentration apparatus and reduce the volume of the solvent until less than 10 ml of solvent remains, but do not evaporate to dryness.

6.11 Quantitatively transfer the hexane extract to labeled 10-ml K-D concentrator tubes. Rinse the Erlenmeyer flask with several small (1 to 2 ml) portions of hexane, adding the washes to the K-D concentrator tube.

6.12 Transfer the K-D concentrator tubes to the concentration device and concentrate the hexane extract to approximately 1.0 ml, using a gentle stream of nitrogen and heat if necessary.

6.13 After the 1.0 ml volume has been obtained, remove the K-D concentrator tube and quantitatively transfer the contents of the K-D concentrator tube to designated conical mini-vials. Rinse the concentrator tube with two 0.5-ml portions of hexane and transfer the washes to the concentrate vial. Complete necessary paperwork (see QA section).

6.14 Store the hexane concentrate in a freezer until just prior to GC/MS analysis.

6.15 Concentrate the hexane to near dryness and add 50 µl of 200 ppb standard of an appropriate Is. Return sample to the reach-in refrigerator.

7.0 ACTIVATED CARBON CLEAN-UP

7.1 After GC/MS analysis of samples processed through the dual column method in Section 6.0, the possibility exists that certain matrices may produce indeterminable results. In such cases an activated carbon column clean-up technique will be employed.

7.2 Obtain from the desiccator the activated Celite/Carbopak (from Section 2.13).

7.3 Insert a small wad of glass wool into a small (7 mm O.D. X 15 cm) disposable pipet and, using vacuum aspiration at the pointed tip of the pipet, add the Celite/Carbopak mixture until a 2-cm column is obtained.

7.4 Prepare the column by adding the following solvents/solvent mixtures in the designated aliquot sizes: (place a clean 125-ml Erlenmeyer flask under the column)

- 2 ml toluene,
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene,
- 1 ml 50/50 (v/v) cyclohexane/methylene chloride,
- 2 ml hexane.

7.5 When the bottom of the hexane meniscus just touches the top of the Celite/Carbopak material, quantitatively transfer the 50 µl of sample onto the column.

7.6 Rinse the sample container with two 1-ml portions of hexane, adding the rinses onto the column.

7.7 Sequentially wash the column with the following solvents/solvent mixtures in the designated aliquot sizes:

- 1 ml 50/50 (v/v) cyclohexane/methylene chloride
- 1 ml 75/20/5 (v/v/v) methylene chloride/methanol/benzene.

7.8 Remove the 125-ml Erlenmeyer flask and replace with a concentrator tube or Reacti-vial.

7.9 Elute the TCDD from the column with 2 ml of toluene into the concentrator tube or Reacti-vial.

7.10 Store the toluene eluent in a freezer until the GC/MS analysis is to be performed.

7.11 Shortly before the analysis, concentrate the extract to near dryness and add 50 µl of isooctane for GC/MS analysis.

7.12 Complete any paperwork requirements and store the concentrate in the reach-in refrigerator (see QA section for examples of paperwork).

8.0 CAUSTIC AND ACID CLEANUP (OPTIONAL)

8.1 Certain samples may require additional cleanup before column chromatography in order to achieve acceptable detection limits. The following is a procedure which involves acid and caustic wash of the sample.

8.2 After step 5.12 is completed, quantitatively transfer the extract to a 125-ml separatory funnel.

8.3 Wash the extract with 30 ml 20% aqueous potassium hydroxide by shaking for 10 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.4 Wash the extract with 25 ml of distilled deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.5 Slowly add 50 ml of concentrated sulfuric acid to the extract and shake for 10 minutes. Let stand for 10 minutes and discard the acid layer. Repeat until acid layer remains colorless after extraction.

8.6 Wash extract with 20 ml of distilled/deionized water by shaking for 2 minutes. Let stand for 10 minutes and discard the aqueous layer.

8.7 Quantitatively transfer the organic layer to a 20-ml scintillation vial and dry over 10 g anhydrous sodium sulfate.

8.8 Reduce the extract volume to approximately 1 ml.

8.9 Proceed to dual column cleanup if required.

9.0 GLASSWARE PREPARATION PROCEDURES

9.1 Rinse glassware with the last solvent used in it. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water; drain dry. Rinse with high purity acetone and hexane and allow to air dry. When dry, heat in a muffle furnace to 400°C for 1 hour. (Volumetric glassware should not be heated in a muffle furnace.) Remove from the oven when cool; store inverted in a clean environment.

APPENDIX — C

DIOXIN ANALYSES - ANALYSIS PROCEDURES

1.0 INTRODUCTION

1.1 This is a qualitative and quantitative (high resolution) GC/(low resolution) MS analysis specific for the 2,3,7,8 isomer of tetrachlorodibenzo-p-dioxin using selected ion monitoring. A sample is spiked with isotopically labeled $^{13}C_{12}$-2,3,7,8-TCDD as internal standard and with $^{37}Cl_4$-2,3,7,8-TCDD as surrogate. Quantitation is based on the response of native TCDD relative to the internal standard. Performance is based on surrogate standard results. Percent recovery of the IS/SURR mixture is based on the GC/MS internal standard ^{13}C-2,3,7,8-TCDF.

2.0 SAFETY

2.1 Samples are sent to IT Corporation from suspected or known hazardous waste sites. Samples are to be handled from receipt to storage by qualified personnel only. Analysts must have a working knowledge of safety protocols and be adept at safety procedures. GC/MS instruments must be equipped with vapor contamination traps on the capillary split and sweep vents and on the rough pump effluent lines prior to use (see Safety SOP).

3.0 SET-UP AND INSTALLATION

3.1 Install a 60 meter, 0.25 mm i.d., fused silica SP2330, 0.20 micron film thickness capillary column. Set the head pressure to approximately 20-25 psi and the split and sweep flows to 30 ml/min and 3 ml/min respectively.

3.2 Create a reasonable unit resolution tune for PFTBA. Adjust the zero according to the instrument manufacturers suggested settings. Set the preamp sensitivity to 10^{-8} amps/volts. The electron multiplier must be set to achieve 800,000 area units for 2 ng of $^{13}C_{12}$-TCDD as injected for M/E 334. Calibrate the instrument.

3.3 Establish the following MID descriptors:

3.3.1 NAME "TC" for running column performance mixture

MID DESC: TC
INST: FINN CALI: CAL19

MASS DEFECT AT 100 AMU	30 MMU
MASTER RATE	1024
TOTAL ACQU TIME	0.318 SECS
TOTAL SCAN TIME	0.450 SECS
CENT SAMP INT	0.200 MS
CALI MASS RANGE	44 TO 614 AMU

INT NO.	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
1	319.649	320.150	0.050	0.054	1	100	1	1	0	POS
2	321.649	322.150	0.050	0.052	1	100	1	1	0	POS
3	322.649	323.105	0.050	0.052	1	100	1	1	0	POS
4	327.649	328.150	0.050	0.054	1	100	1	1	0	POS
5	331.649	332.150	0.050	0.052	1	100	1	1	0	POS
6	333.649	334.150	0.050	0.052	1	100	1	1	0	POS

3.3.2 NAME "TD" for running standards and samples

MID DESC: TD
INST: FINN CALI CAL39

MASS DEFECT AT 100 AMU	30 MMU
MASTER RATE	1024
TOATL ACQU TIME	0.430 SECS
TOTAL SCAN TIME	0.450 SECS
CENT SAMP INT	0.200 MS
CALI MASS RANGE	44 TO 614 AMU

INT NO.	BEGIN MASS	END MASS	TIME REQUEST	(SECS) ACTUAL	MPW	MFW	MA	TH	BL	ION
1	256.649	257.150	0.050	0.052	1	100	1	1	0	POS
2	319.649	320.150	0.050	0.054	1	100	1	1	0	POS
3	321.649	322.150	0.050	0.052	1	100	1	1	0	POS
4	327.649	328.150	0.050	0.054	1	100	1	1	0	POS
5	331.649	332.150	0.050	0.052	1	100	1	1	0	POS
6	333.649	334.150	0.050	0.052	1	100	1	1	0	POS
7	315.649	316.150	0.050	0.052	1	100	1	1	0	POS
8	317.649	318.150	0.050	0.054	1	100	1	1	0	POS

3.4 Set the GC conditions as follows:

Injection Port Temp	250°C
Separator Temp	250°C
Initial Temp	70°C
Initial Time	4 min
Ramp Rate 1	20°C/min
Temp 2	200°C
Hold Time 2	0 min
Ramp Rate 2	4°C/min
Temp 3	250°C
Hold Time 3	5 min*
Split/Sweep	85 sec
Filament/Mult turn on time	10 min

* Hold for at least 2 min beyond the retention time of the last isomer of TCDD in the performance mixture.

3.5 Analyze the 7 isomer EPA test mixture. If no isomers are co-eluting with 2,3,7,8-TCDD, conditions stated above are acceptable to proceed. If co-elution does occur with 2,3,7,8-TCDD, the column must be changed or conditions modified in order to stop co-elution.

3.6 Create a library with the following entries:

Entry 1 - ^{13}C - 2,3,7,8-TCDD (Internal standard)
- delete all masses except 322/334
- amount = 1000
- units = pg/ul
- quant mass = 332

Entry 2 - ^{13}C - 2,3,7,8-TCDD (Secondary ion)
- delete all masses except 322/334
- amount = 1000
- units = pg/ul
- quant mass = 334

Entry 3 - 2,3,7,8-TCDD
- delete all masses except 320, 322, 332, 257
- amount = 1000
- units = pg/ul
- quant mass = 320

Entry 4 - 2,3,7,8-TCDD (Secondary ion)
- delete all masses except 320, 322, 332, 257
- amount = 1000
- units = pg/ul
- quant mass = 322

Entry 5 - 2,3,7,8-TCDD (Tertiary ion)
- delete all masses except 320, 322, 332, 328
- amount = 1000
- units = pg/ul
- quant mass = 257

Entry 6 - ^{37}Cl -2,3,7,8-TCDD (Recovery int. std.)
- delete all masses except 328
- amount = 200
- units = pg/ul
- quant mass = 328

4.0 ANALYSIS

4.1 General Description

4.1.1 A five point calibration consisting of a 200 pg/ul (1 ppb equivalent), a 1000 pg/ul (5 ppb equivalent), a 5000 pg/ul (25 ppb equivalent), a 20,000 pg/ul (100 ppb equivalent), and a 20,000 pg/ul (200 ppb equivalent) standard must be run in triplicate and a linear response curve generated before samples are analyzed. The 200 pg/ul standard is analyzed at the beginning of each eight hour shift to verify system performance and conformity to the multipoint calibration (see QA section on standard paperwork). Samples are received in 50 ul volumes and require no further preparation by the GC/MS laboratory.

4.1.2 COMPOSITION OF CONCENTRATION CALIBRATION SOLUTIONS

Solution #	Concentration of 2,3,7,8-TCDD			
	Isotopically Labeled			Unlabeled
	$^{13}C_{12}$	$^{37}Cl_4$	^{13}C-TCDF	
1	1 ng/uL	0.06 ng/uL	0.2 ng/ul	0.2 ng/uL
2	1 ng/uL	0.12 ng/uL	0.2 ng/ul	1 ng/uL
3	1 ng/uL	0.2 ng/uL	0.2 ng/ul	5 ng/uL
4	1 ng/uL	0	0.2 ng/ul	20 ng/uL
5	1 ng/uL	0	0.2 ng/ul	40 ng/uL

4.2 Procedures for GC/MS Analysis Initial Calibration

4.2.1 The GC conditions for all standards, samples, and the column performance mixture are as stated in Section 3.4.

4.2.2 Tune and calibrate the instrument as in step 3.2 or verify that the instrument has been tuned and calibrated within the past week and has performed satisfactorily when last used. If the method has not been performed successfully within the last seven days, check the tune and recalibrate.

4.2.3 Acquire the seven isomer EPA test mix. If no isomers are co-eluting with 2,3,7,8-TCDD, proceed with 4.2.4. If co-elution does occur, the conditions must be modified or the column must be changed. The MID descriptor TC must be used for this analysis (section 3.3).

4.2.3.1 Determine and document acceptable system performance with the following criteria:

A. Five data points for each GC peak are acquired.

B. GC column performance -- The valley between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers must be resolved with a valley <25%. Valley % = x/y x 100 when y is peak height of 2,3,7,8-TCDD and x is baseline to valley height (Fig. 1).

C. Ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be ≥ 0.67 and ≤ 0.87.

mean RFs for each compound do not differ by more than $\pm$ 10%, the RF can be considered to be independent of analyte quantity for the calibration concentration range, and the mean of the five mean RFs shall be used for concentration calculations.

4.2.7 Fill out all necessary paperwork for the standard calibration QA/QC (see QA/QC section for paperwork).

4.2.8 Plot the response factor vs concentration for the five point calibration curve for QA/QC reporting.

4.3 Calibration before the start of each eight hour shift

4.3.1 Inject 2 ul of the performance check solution as in 4.2.3 - 4.2.3.1F.

4.3.2 Inject 2 ul of the concentration calibration solution #1 (200 pg/ul) determine and document acceptable performance for

4.3.2.1 MS sensitivity - signal-to-noise (S/N) ratio of $\geq$ 2.5 for m/z 257 and $\geq$ 10 for m/z 322 for unlabeled 2,3,7,8-TCDD. The ratio of integrated ion current for m/z 257 to m/z 322 must be $\geq$ 0.20 and $\leq$ 0.45.

4.3.2.2 Measured response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD is within $\pm$ 10% of the mean values established (Section 4.2) by triplicate analyses of the concentration calibration solutions.

4.3.2.3 If both these criteria are met, fill out the bottom portion of form 248A (QA/QC section). If the RF ratios are within 10% of the calibration average samples may then be analyzed. DO NOT UPDATE the shift standard to the response list. Use R;S;T only. If the 10% criteria are not met, reanalyze the shift standard. If still out of bounds, a new multipoint must be run. Multipoints may continue to be used for as long as the shift standards conform to this criteria. Xerox a copy of Form 248A for inclusion with the shift standard packages and an extra copy to be placed in the instrument log book so that subsequent shift standard entries may be made on the same form.

4.3.2.4 Acquire sample analyses. Samples may be analyzed following a successful shift standard analysis. Performance standards must be rerun within every eight hours. The injection procedure must be carefully adhered to to avoid cross contamination. If the background of a sample analysis remains high towards the end of an acquisition, the column should be baked out for an extra period of time to avoid possible chromatographic carryover into the next sample injection.

Septa should be changed after approximately 40 injections. Capillary injection port liners should be cleaned or exchanged with every other septum change. Good sense and experience prevail.

4.3.3 After eight hours from the injection of the column performance check solution (4.3.1), the tune is over and the performance check solution must be analyzed again. If all criteria (4.2.3.1 a-f) are met, the samples analyzed during that eight hour period are acceptable. If the criteria are not met, the samples must be reanalyzed.

4.4 For all injections, a hot needle injection technique is used.

4.4.1 Injection Technique (Hot Needle) - The syringe must be thoroughly cleaned between injections to avoid cross contamination. Remove the plunger between injections and wipe it thoroughly with a kimwipe. Rinse the syringe with ten to fifteen full syringe volumes of hexane solvent wash. Replace the solvent wash with pesticide quality hexane daily. If a hamilton syringe cleaner is available that is equipped with a vacuum source, use this also. Do not use the hamilton syringe cleaner if there is no vacuum pump attached. Insert the needle into the septum port, wait approximately ten seconds for the needle to heat, then pump the plunger back and forth a few times. Rinse with the solvent wash hexane again. Work the plunger up and down in the syringe barrel to reduce excess hexane wash. There should be approximately 0.5 μl of solvent left in the syringe barrel following this final rinse.

Draw back the plunger so that there are about 2 μl of air in the barrel. Draw 2.1 ml of sample into the needle. Usually to get a total of 2 μl of sample, it is necessary to pull the plunger back approximately 1.2 μl. The sample should be drawn up into the barrel and the amount confirmed to be 2 μl. If it is not, the sample should be expelled and process repeated.

After getting 2.0 μl of sample into the barrel, insert the needle into the injector port and wait 6 seconds. Rapidly make the injection.

After making the injection, remove the needle as quickly as possible. As soon as the injection is made, start the GC.

4.5 Identification criteria for native 2,3,7,8-TCDD

4.5.1 Retention time (at maximum peak height) of the sample component must be within 3 seconds of the retention time of the $^{13}C_{12}$-2,3,7,8-TCDD. Retention times are required for all chromatograms, but scan numbers are optional. These parameters should be printed next to the appropriate peak.

4.5.2 The integrated ion currents detected for m/z 257, 320, and 322 must maximize simultaneously. If there are peaks that will affect the maximization or quantitation of peaks of interest, attempts should be made to narrow the scan window to eliminate the interfering peaks. This should be reported on a separate chromatogram.

4.5.3 The integrated ion current for each analyte and surrogate compound ion (m/z 257, 320, 322 and 328) must be at least 2.5 times background noise and must not have saturated the detector; internal standard ions (m/z 332 and 334) must be at least 10 times background and must not have saturated the detector.

4.5.4 Relative abundance of m/z 257 to m/z 322 should be ≥ 20% and ≤ 45%.

4.5.5 Abundance of integrated ion counts detected for m/z 320 must be ≥ 67% and ≤ 87% of integrated ion counts detected for m/z 322.

5.0 DELIVERABLES

5.1 Each sample "package" must include the following:

a) RIC (1000 - end of run)
b) Complete quantitation report. (Input area and scan # manually if missed)
c) (EICP of m/e 332; m/e 334; m/e 316, and m/e 318)
d) (EICP of 320; 322; 332; and 257)
e) (EICP of 320; 322; 332; and 328)
f) Quan (320; 322; 257; 5 scans) The center of the 5 scan window is the retention time of $^{13}C_{12}$-2,3,7,8-TCDD
g) A standard package including all of (a) through (e) plus an attached copy of the TCDD calibration summary (Form 243A)

5.1.1 See QA/QC section for batch report deliverables

6.0 TOTAL ION CONFORMATION

6.1 Inject 2 .l of a DFTPP solution into the GC/MS system using the same GC conditions as stated in section 3.4 with the MS scanning from 35-450 at 1 sec/scan. Required calibration criteria for DFTPP shall be:

m/z	Relative Intensity
51	30 - 60 percent of base peak
68	< 2 percent of m/z = 69
70	< 2 percent of m/z = 69
127	40 - 60 percent of base peak
197	< 1 percent of base peak
198	100 percent (base peak)
199	5 - 9 percent of base peak
275	10 - 30 percent of base peak
365	> 1 percent of base peak
441	less than m/z = 443
442	> 40 percent of base peak
443	17 - 23 percent of m/z = 442

6.2 Inject 4 µl of the positive TCDD sample using the same GC conditions in section 3.4. MS data acquisition requirements shall be:

6.2.1 Cycle time ≤ 1.5 seconds.

6.2.2 Acquisition of ≥ 5 spectra during elution of 2,3,7,8-TCDD from the GC.

6.2.3 MS scanning from 150-350 at 1 sec/scan.

6.3 Subtract an appropriate background spectrum, and plot a spectrum of 2,3,7,8-TCDD after background subtraction. (The person responsible for MS data interpretation is responsible for demonstrating that the background spectrum selected for subtraction was an appropriate spectrum.) Provide a hard copy of the background spectrum, the TCDD spectrum before subtraction, and the TCDD spectrum after subtraction. The quality of the plotted spectrum will be affected by other sample components that have approximately the same GC retention time and will be highly variable. Desired spectral features are:

Base peak = m/z 322
Ratio of m/z 320 to 322 = 0.77
Ratio of m/z 320 to 324 = 1.58
Ratio of m/z 257 to 322 = 0.32
Ratio of m/z 257 to 259 = 1.03
Ratio of m/z 194 to 196 = 1.54
m/z 160 and 161 = ≥ 10% of m/z 322

Because $^{13}C_{12}$-2,3,7,8-TCDD, the internal standard, is present in every sample and has essentially the same retention time as unlabeled 2,3,7,8-TCDD, the spectrum after background subtraction will represent a mixture. When $^{13}C_{12}$-2,3,7,8-TCDD is present at a higher concentration than unlabeled 2,3,7,8-TCDD, the resultant spectrum must be normalized to m/z 322 to demonstrate desired spectral features.

7.0 SEE QA/QC SECTION FOR EXAMPLES OF ALL PAPERWORK NECESSARY FOR COMPLETE REPORTING OF GC/MS DATA

0150-AP

APPENDIX D

DIOXIN ANALYSIS - STANDARD VERIFICATION

1.0 Summary

1.1 All standards bought commercially must be checked for both purity and accuracy of concentration. The benchmark standard for verification of accuracy is a 7.37 ug/ml standard of native 2,3,7,8-TCDD supplied by the USEPA.

2.0 Receipt of Standards and Standard Checks of Stock Solutions

2.1 Standards received from commercial suppliers of dioxin are checked into the building and logged into a standard notebook. The standards are transferred to volumetric flasks and brought up to volume in toluene. A known dilution of the stock is analyzed on GC/MS to check for both purity and accuracy of concentration vs. an already approved standard of the USEPA benchmark standard. If the response factor agrees ± 10% from the check standard, the new lot is approved for use. The hardcopies of the standard check will contain EICP's for all compounds of interest from both the new standard and the benchmark standard and will be filed for future reference.

3.0 Storage of Approved Standards

3.1 The approved stock will be transferred to tared numbered and labeled vials (2 ml size). The weights of the standards will be recorded (Form 1). When a new standard bottle is used, it is weighed first to insure no volume loss during storage. The standards are kept at -4°C for no longer than six months.

4.0 Working Standard Solutions

4.1 Dilutions of the stock solutions are made for all working standards to be used in the extraction laboratory and the GC/MS laboratory.

4.2 Before use of the working level standards on sample analysis, the solutions must be approved for concentration and purity as in the stock solutions. The extraction lab standard is analyzed by GC/MS and compared to the shift calibration solution. If the calculated amounts are ± 10% of expected amount, the standard is approved for use in the extraction laboratory. When new GC/MS shift standards are made and approved as above, a new five point calibration in triplicate must be run.

4.3 Working standard solutions are stored as per Section 3.0

5.0 Standard Notebook

5.1 All standards made are logged into a standard notebook that includes volumes used; solvents; any problems encountered; date made; date expired; weights of each standard container; concentration; approval date.

Standard or Stock Solution # ______________ Ref. # __________ Lot # __________________

Compound(s): ____

Original Conc. _______________ Solvent ______________ Total Volume __________________

Sol'n Prepared By ______________________________ at Date/Time __________________

Container Tare (grams) _______________ Container - Solvent (grams) __________________

Container Gross (grams) _______________

Special Notes : ____

____

____

____

Date Used	User Initials	Weight Before Use (grams)	Weight After Use (grams)	Volume Removed (mls/µl)	Weight After Septum (grams) Replacement

SUMMARY OF QA/QC RESULTS FOR 2,3,7,8-TCDD ANALYSES

6A COMPARISON OF SOIL PREPARATION PROCEDURES AND TRIPLICATE ANALYSES OF CONTAMINATED SOILS

6B ACCURACY AND PRECISION RESULTS FOR 2,3,7,8-TCDD ANALYSES

6C RESULTS OF DUPLICATE LABORATORY THERMAL DESORPTION EXPERIMENTS

COMPARISON OF SAMPLE PREPARATION TECHNIQUES FOR ANALYSIS OF 2,3,7,8-TCDD IN UNTREATED SOILS

Soil identification	Concentration of 2,3,7,8-TCDD (ppt) Acid pretreatment and soxhlet w/benzene	 Jar method w/hexane-methanol
JI	110	99.1
JI	111	98
JI	97.2	104
	$\bar{x}$ = 106 RSD = 7.2%	$\bar{x}$ = 100.4 RSD = 3.2%
Eglin	93.9	71
Eglin	98	79
Eglin	112	68
	$\bar{x}$ = 101.3 RSD = 9.3%	$\bar{x}$ = 72.7 RSD = 7.2%
NCBC	470	497
NCBC	501	420
NCBC	510	450
	$\bar{x}$ = 493.7 RSD = 4.3%	$\bar{x}$ = 455.7 RSD = 8.5%
NCBC >2 mm	148	
	149	
	$\bar{x}$ = 148.5	

[illegible] TA-T-9

SUMMARY OF 2,3,7,8-TCDD QA/QC RESULTS

Mean percent accuracy = 97.4
Standard deviation = 24.6
Percent RSD = 25.2
Number of data points = 55

Precision

Triplicate analyses of starting soil

	Soil/Method	Mean (ppb)	SD(ppb)	% RSD
1)	JI/soxhlet	101	9.4	9.4
2)	Eglin/soxhlet	106	7.7	7.3
3)	NCBC/soxhlet	493	20.9	4.2
4)	JI/jar	72.6	5.6	7.8
5)	Eglin/jar	100	3.2	3.2
6)	NCBC/jar	455	38.8	8.5

Duplicate Analyses

1) Samples ID = J2155/J2156
Variation[a] = 0.67%
Mean Concentration = 148.5 ppb

2) Samples ID = J1942/J1945
Variation[a] = 89%
Mean Concentration = 0.36 ppb

3) Samples ID = J2204/J2265
Variation[a] = 6.4%
Mean Concentration = 25.5 ppb

10 blanks out of 45 samples = 22%

---

[a] $\frac{S_1 - S_2}{\bar{S}}$ X 100 = RPD (relative percent difference).

0301 TA-SUMMARY

RESULTS OF DUPLICATE TREATMENT TESTS

Test number	Soil type	Test temp.[a] (°C)	Time at test temp. (min)	Total time in oven (min)	Initial soil weight (gm)	Purge gas (atmosphere)	Purge[b] gas flow (cc/min)	Weight loss (%)	Final 2,3,7,8-TCDD conc. (ppb)
2	EGLIN	481	15	23.0	31.89	Air	100 10	1.79	1.3
4	EGLIN	481	15	23.0	32.83	Air	100 10	1.79	0.8
8	NCBC	556	15	23.0	30.63	Air	100 10	3.20	0.99
13	NCBC	568	6	23.0	30.19	Air	100 10	3.08	0.53

[a] See time-temperature logs, Appendix F.

[b] Precision based on calibration data for rotameter.

D277/EPA-T-11

ANALYTICAL DATA SHEETS

7A 2,3,7,8-TCDD RESULTS FOR UNCONTAMINATED AND CONTAMINATED SOIL SAMPLES USED FOR LABORATORY THERMAL DESORPTION TESTS

7B 2,3,7,8-TCDD RESULTS FOR SOIL SAMPLES AFTER LABORATORY THERMAL DESORPTION TESTS

7C ANALYTICAL REPORTS FOR HERBICIDE COMPOUNDS IN SAMPLES BEFORE AND AFTER LABORATORY THERMAL DESORPTION TESTS

ANALYTICAL DATA FOR 2,3,7,8-TCDD CONTAMINATED AND UNCONTAMINATED SOIL SAMPLES

Lab: ITAS Knoxville

Report Date: 4/3/85
Column: 60m SP2330

Sample Number	Extra Cleanup	Aliquot Wet Wt. (g)	PPB TCDD Meas.	DL	GC/MS Analysis Instr. ID	Date	Time	Rel. Ion Abund. 320/322	332/334	PPB Surrogate Meas.	% Acc'y	Relative Ion Abundance 320	322	257	332	334	[illegible]	(1% ... Rec.) Comments
J 1077	Yes	10.47	ND	0.20	4000	10/31/84	14:17		0.79	196	99				115920	970181	1127940	Soxhlet
J 1078	Yes	10.38	ND	0.44	4000	10/31/84	14:50		0.77	213	106				249128	595416	757902	Soxhlet
J 1079	Yes	9.91	ND	0.58	4000	10/31/84	15:23		0.84	213	106				91100	220693	290073	Soxhlet
J 1080	Yes	1.3	93.9		4000	10/31/84	15:57	0.79	0.80	200	100	500640	645460	230778	177512	410908	571512	Soxhlet
J 1081	Yes	1.06	90.0		4500	12/04/84	11:22	0.84	0.84	259	124	178962	211531	77002	79696	171778	284030	Soxhlet
J 1082	Yes	1.13	112		4500	12/04/84	12:41	0.84	0.81	254	127	200552	333125	122736	98484	210512	259574	Soxhlet
J 1083	Yes	1.27	110		4500	12/04/84	13:34	0.84	0.83	252	126	410444	515017	200136	151151	314720	340451	Soxhlet
J 1084	Yes	1.20	111		4500	12/04/84	14:12	0.81	0.83	243	122	452170	610712	202540	171509	378764	342100	Soxhlet
J 1085	Yes	1.43	97.2		4000	11/01/84	0:29	0.74	0.85	190	95	564052	730524	236064	170711	411520	625600	Soxhlet
J 2014	Yes	0.56	470		4000	11/19/84	16:19	0.78	0.78	140	70	2511500	3165100	1079620	144570	537100	677047	Soxhlet
J 2015	Yes	1.02	501		4500	01/20/85	22:55	0.84	0.86	180	90	1581790	2156090	792224	113075	377419	393168	Soxhlet
J 2016	Yes	1.29	510		4500	01/20/85	23:33	0.84	0.75	204	102	477712	605739	225416	31157	71216	85197	Soxhlet
J 2155	Yes	3.58	109		4000	01/19/85	14:13	0.80	0.81	200	100	3990560	5475610	2302230	287106	747570	912010	Soxhlet
J 2156	Yes	3.71	100		4000	01/19/85	12:42	0.79	0.80	202	101	4276830	5424520	2439990	290187	753072	912544	Soxhlet
J 1066	Yes	1.12	71.0		4000	11/01/84	10:36	0.80	0.81	253	126	518144	656444	212340	275119	700706	870121	Jar
J 1067	Yes	1.27	78.9		4000	11/01/84	11:11	0.78	0.80	203	102	670240	917592	285930	219711	711114	969400	Jar
J 1068	Yes	1.27	68.0		4000	11/01/84	11:56	0.80	0.80	200	100	213624	410000	158469	167656	476607	544300	Jar
J 1069	Yes	1.44	95.1		4000	10/31/84	11:21	0.82	0.79	229	114	601960	580592	265591	117569	477312	379797	Jar
J 1070	Yes	1.29	98.6		4000	11/01/84	13:23	0.80	0.82	201	101	591952	753907	204710	106427	585260	677204	Jar
J 1071	Yes	1.07	104		4000	10/31/84	11:56	0.76	0.79	205	103	810201	1046190	311756	313150	787270	971100	Jar

APPENDIX E-1. (Continued)

Lab: IIAS Knoxville

Report Date: 9/3/85
Column: 60m SP2330 FSCC

Sample Number	Extra Cleanup	Aliquot Wet Wt. (g)	PPB TCDD Meas.	PPB TCDD DL	GC/MS Analysis Instr. ID	GC/MS Analysis Date	GC/MS Analysis Time	Rel. Ion Abund. 320/322	Rel. Ion Abund. 332/334	PPB Surrogate Meas.	PPB Surrogate % Acc'y	Relative Ion Abundance 320	Relative Ion Abundance 322	Relative Ion Abundance 257	328*	332	334	(IS % Rec.) Comments
J 1930	Yes	1.90	497		4000	12/03/84	10:52	0.79	0.81	143	72	19466600	23891000	11050230	634919	[illegible]	[illegible]	Jar
J 2012	Yes	1.57	420		4000	01/19/85	15:01	0.79	0.78	133	67	12195700	15377600	6950000	453790	[illegible]	2222970	Jar
J 2013	Yes	1.17	450		4000	01/19/85	16:45	0.80	0.82	137	69	5712690	7590870	3295470	271611	1062200	1291450	Jar
J 1939	Yes	1.91	873		4000	12/03/84	11:41	0.79	0.82	146	73	16227000	20730700	9436130	261696	956610	[illegible]	Jar
J 1900	Yes	2.11	464		4000	12/03/84	13:09	0.79	0.79	146	73	13516400	17754300	8032530	379555	1175710	1691760	Jar
TBlank135(RB)	Yes	10.0	ND	0.62	4500	11/15/84	8:51		0.80	253	126				106720	216900	255760	Jar
TBlank137A(RB)	Yes	10.0	ND	0.40	4000	11/09/84	13:00		0.80	226	113				163510	419375	530613	Soxhlet
TBlank137B(RB)	Yes	10.0	ND	0.70	4000	11/09/84	14:14		0.79	226	113				57760	150790	106572	Soxhlet
TBlank149(RB)	Yes	10.0	ND	0.40	4530	11/29/84	4:58		0.44	151	76				[illegible]	731664	[illegible]	Jar
TBlank167(RB)	Yes	10.0	ND	0.21	4000	01/19/85	8:59		0.80	133	67				455696	1011750	2276010	Jar
TBlank184(RB)	Yes	10.0	ND	0.55	4500	01/19/85	9:44		0.79	274	137				132105	190120	268030	Soxhlet
TBlank190(RB)	Yes	10.0	ND	0.69	4500	01/19/85	9:06		0.80	215	107				157349	[illegible]	[illegible]	Soxhlet

DJ01:IA-IAM E11A

ANALYTICAL DATA FOR 2,3,7,8 TCDD IN TREATED SOIL SAMPLES

Lab: ITAS Knoxville

Report Date: 4/1/85
Column: 60M SP2330 FSC

Sample Number	Extra Cleanup	Aliquot Wet Wt. (g)	PPB TCDD Meas.	PPB TCDD DL	GC/MS Analysis Instr. ID	GC/MS Analysis Date	GC/MS Analysis Time	Rel. Ion Abund. 320/322	Rel. Ion Abund. 332/334	PPB Surrogate Meas.	PPB Surrogate % Acc'y	Relative Ion Abundance 320	Relative Ion Abundance 322	Relative Ion Abundance 257	Relative Ion Abundance 328*	332	334	(IS % Rec.) Comments
J 1940	Yes^a	9.97	4.5		4500	11/12/84	12:15	0.83	0.84	233	116	109021	135120	408270	117127	271388	269811	Suitable
J 1941	Yes	9.25	1.6		4000	12/04/84	11:13	0.78	0.83	144	72	251400	300432	155997	472560	1739010	2137300	Suitable
J 1942	Yes	9.93	0.81		4000	12/04/84	11:46	0.80	0.85	144	72	86784	112416	92140^b	287910	1067670	1298740	Suitable
J 1945	Yes	9.44	0.31		4000	12/04/84	14:14	0.77	0.83	139	70	21464	28240	10912	196210	740000	970004	Suitable
J 2150	Yes	10.39	ND	0.64	4000	01/19/85	9:31		0.80	194	97				192440	510712	607152	Suitable
J 1981	Yes	10.2	1.26		4500	11/12/84	12:51	0.86	0.86	240	120	11049	12866	3270	39760	75303	93006	Suitable
J 1983	Yes	10.0	0.83		4500	01/09/85	22:20	0.84	0.68	127	64	81344	131149	46112	322540	1101250	1300000	Suitable
J 1944	Yes	10.87	0.45		4000	12/04/84	13:35	0.82	0.80	148	74	35696	43304	18290	206669	717760	931616	Suitable
J 1946	Yes	9.91	0.71		4000	12/04/84	15:06	0.80	0.81	136	68	39640	49212	28512	126282	493669	640736	Suitable
J 2153	Yes	9.46	10.3		4000	01/19/85	12:09	0.79	0.81	200	100	1213450	1573050	703641	472050	1245030	1560000	Suitable
J 2154	Yes	10.60	6.6		4000	01/19/85	11:31	0.70	0.79	202	101	300144	300939	167840	252475	654808	812736	Suitable
J 2049	Yes	10.13	0.99		4000	01/19/85	10:12	0.79	0.82	132	66	46240	64992	20512	110583	475160	587512	Suitable
J 2157	Yes	10.29	0.74		4000	01/19/85	10:57	0.77	0.81	202	101	47296	60112	20012	248475	652752	793824	Suitable
RBlank 100(RB)	Yes	10.0	ND	0.54	4500	11/21/84	14:29		0.80	206	103				593170	1300175	1550000	Suitable
RBlank 150(RB)	Yes	10.0	ND	0.40	4500	12/04/84	15:34		0.83	175	87				870830	2013744	3009370	Suitable
RBlank 190(RB)	Yes	10.0	ND	0.69	4500	01/19/85	9:06		0.80	215	107				357144	2940601	4069720	Suitable
RBlank 160(RB)	Yes	10.0	ND	0.56	4500	01/19/85	9:44		0.79	214	107				1123019	1991120	2606100	Suitable
J 2203	Yes	10.48	0.53		4500	02/21/85	14:21	0.79	0.82	215	108	16309	29712	12504	112279	397445	4506620	Suitable
J 2204	Yes	10.36	27.4		4500	02/21/85	16:46	0.83	0.83	216	108	796600	993005	424515	1090141	2011420	3172044	Suitable
J 2205	Yes	10.71	25.7		4500	02/21/85	22.16	0.82	0.81	244	122	1049670	1317310	805065	1607515	3005115	9654030	Suitable
J 2206	Yes	9.94	30.5		4500	02/21/85	21.20	0.80	0.81	197	99	2357890	2970060	1761900	2172851	6038629	7640076	Suitable
J 2207	Yes	9.97	4.4		4500	02/21/85	21.59	0.78	0.83	200	140	77008	1010000	1000003^b	800050	1760460	2770064	Suitable
RBlank 219(RB)	Yes	10.0	ND	0.25	4500	02/21/85	13:40		0.80	213	107				1064920	5131777	6154064	Suitable

^a Corrected for contribution by native TCDD; 0.9% of m/z 322 subtracted.

RB - Reagent Blank
FB - Field Blank
P - Partial Scan/Confirmatory Analysis
ND - Not Detected
N - Native TCDD Spike
DL - Detection Limited
D - Duplicate/Fortified Field Blank

^b Interference on 257 ion.

[illegible]

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 29, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 9736.04

Sample Description: Six (6) solid samples received November 20, 1984

Concentration units are ug/gram (ppm)

	2,4-D	2,4,5-T
437-14-3	0.47	0.15
437-14-4	0.16	0.21
437-14-5	0.16	0.24
437-3-EAFB	1200.	1700.
437-4-JI	900.	890.
437-13-1	370.	710.
Blank	0.039	0.032

Sworn to and subscribed before me this 29th
day of November, 1984
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

AALA Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO: IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 30, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 9736.04

Sample Description: 437-14-3

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND	2,4,5-Cl_3 phenol	~~1.9~~ Reanalyzed

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988
Notary Public

Approved by
Laboratory Manager
Title

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615 588-[illegible]

CERTIFICATE OF ANALYSIS

TO IT Corporation
ATTN: A. Groen
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED June 25, 1985
PROJECT CODE ITDK 20085
ORDER NUMBER Project #9736.04

Sample Description: #4540, Clean soil, 5-2-85

Concentration units are ug/gram (ppm)

	437-14-3
2,4,5-Trichlorophenol	ND

Remarks: ND = Not detected.

Sworn to and subscribed before me this 25th
day of June, 1985
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED: November 30, 1984
PROJECT CODE: ITDK 19028
ORDER NUMBER: ITTD 9736.04

Sample Description: 437-14-4 ✓2

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND		

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988
Notary Public

Approved by
Laboratory Manager
Title

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 30, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 3736.04

Sample Description: 437-14-5
[illegible]

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND		

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

AALA Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • [illegible]

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED January 16, 1985
PROJECT CODE ITDK 19206
ORDER NUMBER 9736.03

Sample Description: Three (3) soil samples received December 18, 1984

Concentration units are ug/gram (ppm)

	2,4-D	2,4,5-T
437-25-1	<0.047	0.016
437-25-2	<0.012	0.0008
437-25-3	<0.031	0.003

Sworn to and subscribed before me this 16th
day of January, 1985
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

AALA Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615 588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 30, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 9736.04

Sample Description: 437-13-1
NcBc cont.

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	1.1	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND	2,4,5-Cl_3 phenol	53.

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

[illegible] Tennessee [illegible]

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 30, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 9736.04

Sample Description: 437-4-JI

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	1.9	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	1.1
2-nitrophenol	ND	2,4,5-Cl_3 phenol	38.

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988
Notary Public

Approved by
Laboratory Manager
Title

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current AALA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

3315 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED November 30, 1984
PROJECT CODE ITDK 19028
ORDER NUMBER ITTD 9736.04

Sample Description: 437-3-EAFB

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	2.4	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND	2,4,5-Cl_3 phenol	20.

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million).

Sworn to and subscribed before me this 30th
day of November, 1984
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

A2LA Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current A2LA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED January 16, 1985
PROJECT CODE ITDK 19206
ORDER NUMBER 9736.03

Sample Description: 437-25-3

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND		

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million)

Sworn to and subscribed before me this 16th
day of January, 1985
My commission expires January 16, 1988

Notary Public

Respectfully,

Laboratory Manager
Title

AALA [illegible]

IT CORPORATION

IT ANALYTICAL SERVICES
[illegible] Pike • Knoxville Tennessee [illegible]

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED	January 16, 1985
PROJECT CODE	ITDK 19206
ORDER NUMBER	9736.03

Sample Description: 437-25-2 -

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND		

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million)

Sworn to and subscribed before me this 16th
day of January, 1985
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

AALA [illegible]

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-[illegible]

CERTIFICATE OF ANALYSIS

TO IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED January 16, 1985
PROJECT CODE ITDK 19206
ORDER NUMBER 9736.03

Sample Description: 437-25-1

ACID EXTRACTABLE ORGANICS - PRIORITY POLLUTANT ANALYSIS

Compound	Concentration ppm	Compound	Concentration ppm
2-chlorophenol	ND	4-nitrophenol	ND
2,4-dichlorophenol	ND	p-chloro-m-cresol	ND
2,4-dimethylphenol (m-xylenol)	ND	pentachlorophenol	ND
4,6-dinitro-O-cresol	ND	phenol	ND
2,4-dinitrophenol	ND	2,4,6-trichlorophenol	ND
2-nitrophenol	ND		

Remarks: ND = Not detected
<1.0 = Detected but at a level less than the lower quantitation limit of 1.0 ppm (parts per million)

Sworn to and subscribed before me this 16th
day of January, 1985
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager
Title

A2LA Accredited by the American Association for Laboratory Accreditation in the chemical [illegible] testing as listed in the current A2LA Directory of Accredited Laboratories

IT CORPORATION

IT ANALYTICAL SERVICES

5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

CERTIFICATE OF ANALYSIS

TO: IT Technology Development
ATTN: Dick Helsel
312 Directors Drive
Knoxville, TN 37923

DATE REPORTED: November 29, 1984
PROJECT CODE: ITDK 19028
ORDER NUMBER: ITTD 9736.04

Sample Description: Eight (8) solid samples received November 20, 1984

Concentration units are ug/gram (ppm)

	Arsenic
437-14-1	<10.
437-14-2	<10.
437-14-3	<10.
437-14-4	<10.
437-14-5	<10.
437-3-EAFB	<10.
437-4-J1	<10.
437-13-1	<10.

Sworn to and subscribed before me this 20th
day of November, 1984
My commission expires January 16, 1988

Notary Public

Approved by

Laboratory Manager

Accredited by the American Association for Laboratory Accreditation in the chemical field of testing as listed in the current A2LA Directory of Accredited Laboratories

EXPERIMENTAL DATA - LABORATORY TREATABILITY TESTS

8A SUMMARY OF TEST DATA

8B TEST DATA LOGS

SUMMARY OF INCINERABILITY DATA

Chronological test number	Data book sample number	Analytical sample numbers	Nominal test temperature[a] (°C)	Time at test temperature (min.)	Total time in oven (min.)	Initial soil weight (gm)	Soil identification	Initial 2,3,7,8-TCDD concentration (ppb)[b,c]	Final 2,3,7,8-TCDD concentration (ppb)[c]
1	437-9-1	1900	481	15	21.6	30.08	J1	106	4.5
2	437-10-1	1901	481	15	23.0	31.09	Eglin	101	1.3
3	437-11-1	1914	483	30	36.7	36.62	Eglin	102	0.45
4	437-12-1[d]	1903	481	15	23.0	32.03	Eglin	101	0.8
5	437-15-1	1941	480	10	37.2	34.21	J1	106	1.6
6	437-16-1	1942	558	8	13.4	36.13	J1	106	0.81
	437-17-4[e]	1945							0.33
7	437-18-1	1946	558	6	14.5	31.04	Eglin	101	0.71
8	437-21-8	2019	556	15	23.0	30.63	NCBC	495	0.99
9	437-26-1	2153	483	15	22.5	31.38	NCBC	494	10.1
10	437-27-1	2154	482	30	38.0	31.56	NCBC	494	4.6
11	437-29-1	2157	481	30	39.0	41.19	NCBC (>2mm)	140[f]	0.74
12	437-30-1	2158	482	30	38.0	36.90	J1 (>2mm)	106[g]	0.44
13[h]	437-32-1	2203	560	15	23.0	30.19	NCBC	494	0.51
14	437-31-1	2204	433	20	25.0	29.49	NCBC	494	27.4
	437-33-2[e]	2205							25.7
15	437-34-1	2206	411	15	25.6	29.93	J1	106	16.5
16	437-35-1	2207	431	15	25.6	30.71	Eglin	102	4.4

[a] See time-temperature data or test logs; temperature given above is approximate average after "time zero."
[b] Average of analyses of triplicate aliquots of soil except as noted.
[c] All 2,3,7,8-TCDD analyses were obtained using acid pretreatment and soxhlet extraction w/benzene.
[d] Experimental duplicate of test 2.
[e] Analytical duplicate.
[f] Average of analyses for duplicate aliquots of coarse fraction.
[g] Starting concentration of coarse (>2 mm) assumed to be equal to <2 mm material.
[h] Experimental duplicate of test 8.

[illegible] 1A-1-11

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Expt # AF1
Treatment Test Data Log

Untreated soil identification #437-4-SI Date of Test 11/5/84
Treated soil identification 437-9-AFIT By A. Green

A. Treatment Test Conditions

Target temperature 476 °C (test) Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 15 minutes Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 10/29/84
2. Soil quantity

Check soil depth/uniformity	-	✓
Weight of tray A	-	433.67 grams
Weight of tray plus soil-start	-	464.47 grams
Weight of untreated soil-	-	30.80 grams
Weight of tray plus soil- end	-	462.67 grams
Weight loss during treatment	-	1.80 g grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	
0	54	21	21:30	454	481	**
1	139	52				
2	283	169				
3	396	309				
4	452	418				
5	451	451				
6	452	469				
6:30	452	477 *				
7:00	454	481				
8:00	454	479				
10:00	455	481				
15:00	453	482				
20 00	453	481				

Purge gas flow - initial 0.1 ℓ/min
- final 0.1 ℓ/min.

D. Comments/Observations

* start t = 0
** Stop t = 15 min

- reduce set point 2°C @ 13:00 min 435 @ 464 E
- some black carbon like particles mainly in treated sample when removed from oven.

Figure 10-1A

Expt *AF2

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Treatment Test Data Log

Untreated soil identification 437-3-EAF8
Date of Test 11/5/84

Treated soil identification 437-10-AF2T
By H

A. Treatment Test Conditions

Target temperature 478 °C (test)
Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 15 minutes
Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 11/5/84
2. Soil quantity

Check soil depth/uniformity - ✓
Weight of tray A - 433.77 grams
Weight of tray plus soil-start - 465.66 grams
Weight of untreated soil- - 31.89 grams
Weight of tray plus soil- end - 465.09 grams
Weight loss during treatment - 0.57 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	24	18	454	481
1	139	55	20	454	481
2	288	173	23	454	480 **
3	401	318			
4	449	421			
5	451	449			
6	453	462			
7	454	470			
8	459	474 *			
10	455	479			
12	455	482			
14	454	481			
16	454	481			

Purge gas flow - initial 0.1 ℓ/min
- final 0.1 ℓ/min.

D. Comments/Observations

* start t=0.
** stop t=15 min

[illegible] 2°C @ 12.30

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment # AF3

Treatment Test Data Log

Untreated soil identification 437-3-EAFB

Date of Test 11/15/84

Treated soil identification 437-11-AF3T

By A. Kh

A. Treatment Test Conditions

Target temperature 478 °C (test) Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 30 minutes Soil depth 2.3 mm

Special conditions none

B. Actual Test Data

1. Test tray, utensils cleaned 11/5/84
2. Soil quantity

Check soil depth/uniformity -	✓	
Weight of tray -	431.56	grams
Weight of tray plus soil-start -	468.20	grams
Weight of untreated soil- -	36.62	grams
Weight of tray plus soil- end -	467.46	grams
Weight loss during treatment -	0.74	grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	51	22	20	452	482
1	146	57	25	453	482
2.30	355	253	30	452	481
3	407	320	35	452	480
4	449	426	36:30	453	480
5	452	456			
6	455	470			
6:50	454 *	475			
8:00	455	480 •			
10:00	453	484			
12	453	485			
14	454	485			
15	454	485 +			

Purge gas flow - initial 0.1 ℓ/min
- final 0.1 ℓ/min.

D. Comments/Observations

* reduce set point 2°C · final set pt = 441°C
B reduce set point 2°C
upper layer of soil appear more whitish than subsurface soil

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment *AF4

Treatment Test Data Log

Untreated soil identification 437-3-EAFA

Treated soil identification 437-12-AF4T

Date of Test 11/15/84

By

A. Treatment Test Conditions

Target temperature 478 °C (test)

Purge Gas & Flow Rate 0.1 l/min

Target residence time 15 minutes

Soil depth 2-3 mm

Special conditions none

B. Actual Test Data

1. Test tray, utensils cleaned 11/15/84

2. Soil quantity

Check soil depth/uniformity	-	✓
Weight of tray A	-	433.76 grams
Weight of tray plus soil-start	-	466.59 grams
Weight of untreated soil-	-	32.83 grams
Weight of tray plus soil- end	-	466.01 grams
Weight loss during treatment	-	0.58 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	23	18	452	482
1	141	54	20	451	481 *
2	291	179	22		
3	400	322			
4	445	426			
5.30	449	456			
6	450	463			
7	450	470			
8	451	474 *			
10	451	479			
12	451	482			
14	451	482			
16	452	482			

Purge gas flow - initial 0.1 l/min
- final 0.1 l/min.

D. Comments/Observations

# start t=0.
* Stop t=15 min

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

[illegible] AFS

Treatment Test Data Log

Untreated soil identification 437-4-[illegible]
Date of Test 11/21/84

Treated soil identification 437-15-AFST
By A [illegible]

A. Treatment Test Conditions

Target temperature 478 °C (test)
Purge Gas & Flow Rate 0.1 l/min [illegible]

Target residence time 30 minutes
Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 11/20/84

2. Soil quantity

Check soil depth/uniformity	- ✓	
Weight of tray A	- 433.77	grams
Weight of tray plus soil-start	- 467.98	grams
Weight of untreated soil-	- 34.21	grams
Weight of tray plus soil- end	- 465.99	grams
Weight loss during treatment	- 1.99	grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	23	20	453	481
1	149	55	25	453	480
2	302	172	30	453	480
3	416	317	35	453	479
4	445	412	37:10	453	479 **
5	448	441			
6	448	460			
7	450	474			
7:10		475 *			
8	450	473 +			
10	453	477			
12:15	453	480			
15	453	481			

Purge gas flow - initial 0.1 l/min
- final 0.1 l/min.

C. Comments/Observations [illegible] - black carbon like specks visible in treated soil
[illegible] AA7
[illegible]

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment* AF6

Treatment Test Data Log

Untreated soil identification 437-4-JI

Date of Test 11/21/[illegible]

Treated soil identification 437-16-AF6T

By A. [illegible]

A. Treatment Test Conditions

Target temperature 550 °C (test)

Purge Gas & Flow Rate 0.1 air l/min

Target residence time ~~15~~ 8 minutes

Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned

2. Soil quantity

set point = 574
reduce to 530°C

Check soil depth/uniformity -	✓
Weight of tray B -	431.58 grams
Weight of tray plus soil-start -	467.71 grams
Weight of untreated soil- -	36.13 grams
Weight of tray plus soil- end -	465.30 grams
Weight loss during treatment -	2.41 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	53	20			
1	161	59			
2	318	185			
3	435	338			
4	520	465			
5	535	535			
5:25	536	550 *			
6	538	560 A			
7	536	556			
8	537	557			
10	535	559			
12	535	559			
13:25	535	559, [illegible] t=0			

Purge gas flow - initial 0.1 l/min
- final 0.1 l/min.

D. Comments/Observations

* [illegible] t=0
A - reduced set point to 530°C

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment * AF7

Treatment Test Data Log

Untreated soil identification 437-3-EAFB Date of Test 11/21/84

Treated soil identification 437-18-AF7T By

A. Treatment Test Conditions

Target temperature 550 °C (test) Purge Gas & Flow Rate 0.1 air l/min

Target residence time 6 minutes Soil depth 2-3 mm

Special conditions —

B. Actual Test Data

1. Test tray, utensils cleaned 11/21/84

2. Soil quantity

Check soil depth/uniformity	-	✓
Weight of tray A	-	433.77 grams
Weight of tray plus soil-start	-	464.81 grams
Weight of untreated soil-	-	31.04 grams
Weight of tray plus soil- end	-	464.14 grams
Weight loss during treatment	-	0.67 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	20			
1	189	71			
2	332	202			
3	442	353			
4	525	475			
5	534	526			
6	539	542			
6:30		547 *			
7:00	535	550			
8:00	536	555			
10	537	559			
12	537	559			
14:30	537	559 [illegible] +8 min			

Purge gas flow - initial 0.1 l/min
- final 0.1 l/min.

D. Comments/Observations

* [illegible]

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment # AF8

Treatment Test Data Log

Untreated soil identification 437-13-NCBC-1　　Date of Test 12-13-84

Treated soil identification 437-20-AF8T　　By F. Groen

A. Treatment Test Conditions

Target temperature 550 °C (test)　　Purge Gas & Flow Rate 0· air l/min

Target residence time 15 minutes　　Soil depth 2-3 mm

Special conditions -

B. Actual Test Data

1. Test tray, utensils cleaned 12/13/84
2. Soil quantity

Check soil depth/uniformity	-	
Weight of tray	-	431.62 grams
Weight of tray plus soil-start	-	462.25 grams
Weight of untreated soil-	-	30.63 grams
Weight of tray plus soil- end	-	461.27 grams
Weight loss during treatment	-	0.98 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	21			
1	144	43			
2	295	140			
3	410	276			
4	503	409			
5	530	490			
6	535	522			
8	537	543			
9:50	537	550			
10	537	552			
15	539	557			
20	540	557			
23:50	539	556			

Purge gas flow - initial 0.1 l/min

- final 0.1 l/min.

D. Comments/Observations

+ start time

- sample appeared unchanged when hot in the oven - more reddish colour after cooling - lower layer appears slightly darker

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

SpitAF9

Treatment Test Data Log

Untreated soil identification 437-13-NCBL-1

Treated soil identification 437-26-AFTT

Date of Test 1/12/85

By H [illegible]

A. Treatment Test Conditions

Target temperature 478 °C (test)   Purge Gas & Flow Rate [illegible] ℓ/min

Target residence time 15 minutes   Soil depth 2-3 mm

Special conditions ______

B. Actual Test Data

1. Test tray, utensils cleaned 12/10/84
2. Soil quantity

Check soil depth/uniformity	-	
Weight of tray	-	431.58 grams
Weight of tray plus soil-start	-	462.96 grams
Weight of untreated soil-	-	31.38 grams
Weight of tray plus soil- end	-	462.12 grams
Weight loss during treatment	-	0.84 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0 [illegible]	≤ 2	20	20	452	403
1	210	97	22'1.	452	482 [illegible]
2	360	210			
3	452	362			
4	450	424			
5	451	448			
6	452	463			
7	452	472			
7'20		475 [illegible]			
8	453	477			
9	454	481			
10	452	483			
15:00	452	484			

Purge gas flow - initial ______ ℓ/min
- final ______ ℓ/min.

D. Comments/Observations

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Soil #AF 10

Treatment Test Data Log

Untreated soil identification 437-13-NC8C-1 Date of Test 1/12/85

Treated soil identification 437-27-AF10T By

A. Treatment Test Conditions

Target temperature 475 °C (test) Purge Gas & Flow Rate [illegible] L/min

Target residence time 30 minutes Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 1/12/85
2. Soil quantity

Check soil depth/uniformity - ✓

Weight of tray - 433.73 grams

Weight of tray plus soil-start - 465.29 grams

Weight of untreated soil- - 31.56 grams

Weight of tray plus soil- end - 464.49 grams

Weight loss during treatment - 0.80 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	23	30	453	481
1	178	68	35	453	480
2	337	187	38	453	480
3	450	347			
4	452	416			
5	452	441			
6	452	456			
7	453	466			
8	453	473 steady			
10	453	480			
15	453	483			
20	453	482			
25	453	482			

Purge gas flow - initial 0.1 L/min

- final 0.1 L/min.

C. Comments/Observations

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Spl #4 #11

Treatment Test Data Log

Untreated soil identification ~~[illegible]~~ #4684 [illegible] Date of Test 1/12/85

Treated soil identification 437-29-4#11T By A. [illegible]

A. Treatment Test Conditions

Target temperature 478 °C (test) Purge Gas & Flow Rate 0 / ℓ/min

Target residence time 30 minutes Soil depth — mm

Special conditions - [illegible]

B. Actual Test Data

1. Test tray, utensils cleaned 1/12/85

2. Soil quantity

Check soil depth/uniformity -
Weight of tray A - 433.71 grams
Weight of tray plus soil-start - 474.90 grams
Weight of untreated soil- - 41.19 grams
Weight of tray plus soil- end - 474.00 grams
Weight loss during treatment - 0.90 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	55	25	39	454	481 [illegible]
1	194	65			
2	315	165			
3	425	312			
4	440	405			
5	449	430			
7	452	460			
8	453	468			
9	453	473 start t0			
10	454	476			
15	454	483			
20	454	482			
30	454	481			

Purge gas flow - initial ____ ℓ/min
- final ____ ℓ/min.

D. Comments/Observations

@ ~ 450°C popping & cracking [illegible] inside oven
- still occasional pop @ 470°C

- some weight loss may be due to loss of sample cracking

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Experiment #AF12

Treatment Test Data Log

Untreated soil identification #4565 · +10 mesh Johnston Island Date of Test 1/12/85

Treated soil identification 437-36-AF12T By

A. Treatment Test Conditions

Target temperature 475° °C (test) Purge Gas & Flow Rate 0.1 L/min

Target residence time 30 minutes Soil depth — mm

Special conditions +10 mesh soil (oversize from screening)

B. Actual Test Data

1. Test tray, utensils cleaned 1/12/85
2. Soil quantity

Check soil depth/uniformity	-	
Weight of tray	-	431.55 grams
Weight of tray plus soil-start	-	468.45 grams
Weight of untreated soil-	-	36.90 grams
Weight of tray plus soil- end	-	466.38 grams
Weight loss during treatment	-	grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	54	22			
1	164	52			
2	305	160			
3	425	310			
4	447	400			
5	441	438			
7	453	471			
8	453	474 start tr.			
10	453	479			
15	453	483			
25	453	482			
35	454	481			
38	454	461 stop tr.			

Purge gas flow - initial 0.1 L/min
- final 0.1 L/min.

C. Comments/Observations

Figure 10-1A

[illegible] 14

Treatment Test Data Log

Untreated soil identification #437-13-NABS-1 Date of Test 2/7/85

Treated soil identification 437-33-AFKT By A [illegible]

A. Treatment Test Conditions

Target temperature 430 °C (test) Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 30 minutes Soil depth 2-3 mm

Special conditions ______

B. Actual Test Data

1. Test tray, utensils cleaned 2/7/85
2. Soil quantity

Check soil depth/uniformity	-	✓
Weight of tray B	-	431.56 grams
Weight of tray plus soil-start	-	461.05 grams
Weight of untreated soil-	-	29.49 grams
Weight of tray plus soil- end	-	460.39 grams
Weight loss during treatment	-	0.74 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	50	21	30	404	431
1	153	49	35	404	431 stop t-30 min
2	301	170			
3	405	300			
4	406	376			
5	408	400 start t=0			
9:30	408	428			
10	408	430			
17.30	407	434			
18	406	435 [illegible] 436			
19	405	433 [illegible] 436			
20	404	432			
25	403	431			

Purge gas flow - initial 0.1 ℓ/min

- final 0.1 ℓ/min.

D. Comments/Observations

analytical duplicate sample prepared & submitted

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

[illegible] AF 15

Treatment Test Data Log

Untreated soil identification 437-4-ST

Date of Test 2/7/85

Treated soil identification 437-34-AF15T

By

A. Treatment Test Conditions

Target temperature 430 °C (test)

Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 20 minutes

Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 2/7/85

2. Soil quantity

Check soil depth/uniformity	-	
Weight of tray	-	433.76 grams
Weight of tray plus soil-start	-	463.69 grams
Weight of untreated soil-	-	29.93 grams
Weight of tray plus soil- end	-	462.18 grams
Weight loss during treatment	-	1.51 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Temp. (°C) Test (NBS)
0	52	21	25:40	402	432 stop t=20 min
1	147	45			
2	300	153			
3	407	293			
4	402	365			
5	402	390			
5:40	404	400 * start t=0			
6:00	404	405			
8:00	406	426			
10:00	406	430			
13	402	432 [illegible]			
15	463	432			
20	463	432			

Purge gas flow - initial 0.1 ℓ/min
- final 0.1 ℓ/min.

D. Comments/Observations

Figure 10-1A

Section No.: 10A
Revision No.: 2
Date: Oct. 15, 1984
Page: 2 of 2

Expt # AF16

Treatment Test Data Log

Untreated soil identification 437-3-EAFB Date of Test 2/7/85

Treated soil identification 437-35-AF16T By A

A. Treatment Test Conditions

Target temperature 430 °C (test) Purge Gas & Flow Rate 0.1 ℓ/min

Target residence time 20 minutes Soil depth 2-3 mm

Special conditions

B. Actual Test Data

1. Test tray, utensils cleaned 2/7/85
2. Soil quantity

Check soil depth/uniformity - ✓
Weight of tray D - 431.56 grams
Weight of tray plus soil-start - 462.29 grams
Weight of untreated soil- - 30.78 grams
Weight of tray plus soil- end - 461.74 grams
Weight loss during treatment - 0.55 grams

3. Data Record

Time (min.)	Temp. (°C) Oven Indicator	Test (NBS)	Time (min.)	Temp. (°C) Oven Indicator	Test (NBS)
0	52	23	21	403	432
1	156	52	24	403	432
2	306	163	25:40		
3	407	303			
4	403	366			
5	403	391			
5:40	403	400 [illegible]			
6:00	403	403			
7:00	[illegible]	420 [illegible]			
10:00	406	428			
12	405	432			
15	403	432			
18	403	432			

Purge gas flow - initial 0.1 ℓ/min
- final 0.1 ℓ/min.

D. Comments/Observations

Figure 10-1A

Annex 9

PARTICLE ENTRAINMENT THEORY

PARTICLE ENTRAINMENT THEORY

There are three equations of interest to estimate entrainment velocities of particles in air. These correlations do not determine the rate of entrainment.

1. The horizontal fluid velocity required to cause a spherical particle at rest on a bed of solids (soil) to lift off the bed is depicted in Figure G-1, assuming a particle density of 2.6 g/cc and air temperature of 2000°F.

$$U = \sqrt{\frac{4g\ D_p\ (\rho_p - \rho_f)}{3\ \rho_f}} \qquad \text{(Reference A)}$$

where:
U = fluid velocity (ft/sec),
g = gravitational constant (32.3 ft/sec^2),
D_p = particle diameter (ft),
ρ_p = particle density (lb/ft^3),
ρ_f = fluid density (lb/ft^3)

This equation does not account for viscosity of the air or the static friction of the particle against the bed surface. Similar correlations are reported for non-spherical particles, multiple particles, and inclined ducts.

2. The terminal settling velocity of a particle falling through a fluid is expressed by:

$$U_T = \sqrt{\frac{4g\ D_p\ (\rho_p - \rho_f)}{3\ \rho_f C_d}} \qquad \text{(Reference B)}$$

This equation is not for horizontal air velocities. The C_d term or coefficient of drag accounts for the characteristics of the fluid and particle.

3. The last correlation developed by Zenz (Reference C) shown in Figure G-2 estimates the horizontal velocity in pipes required to pick up a particle at rest and entrain it in the air and also the saltation velocity required to keep the particle suspended based on the particle Reynolds number and drag coefficient. This last correlation accounts for both the density and viscosity of the fluid.

The difficulty in using the Zanz correlation is determining the velocity profile within the kiln and accounting for the kiln rotation and particle size distribution. The turbulance profile due to the burner nozzles should be considered as well. The effect of large particles in soil samples with the same mean particle distribution has been observed to reduce the entrainment rate significantly.

In summary, the following factors are thought to effect any theoretical based estimate of entrainment.

- Velocity profile in kiln
  - Entrance and end effects
  - Burner nozzles
- Kiln diameter and length
- Kiln rotation effects
- Particle size distribution effects

REFERENCES

A. Zenz, F. A. and Othmer, D. F., "Fluidization and Fluid-Particle Systems," Reinhold Chemical Engineering Series, C. W. Wilke, ed., Reinhold Publishing Corp., NY.

B. Boucher, D. F., "Fluid and Particle Dynamics, "Chemical Engineers' Handbook, 5th ed., H. B. Crawford and R. J. Kepler, eds., McGraw-Hill Book Company, NY, 1973, 68 pp.

C. Zenz, F. A., "Conveyability of Materials of Mixed Particle Size, "I & EC Fundamentals, 3:1, February 1964.

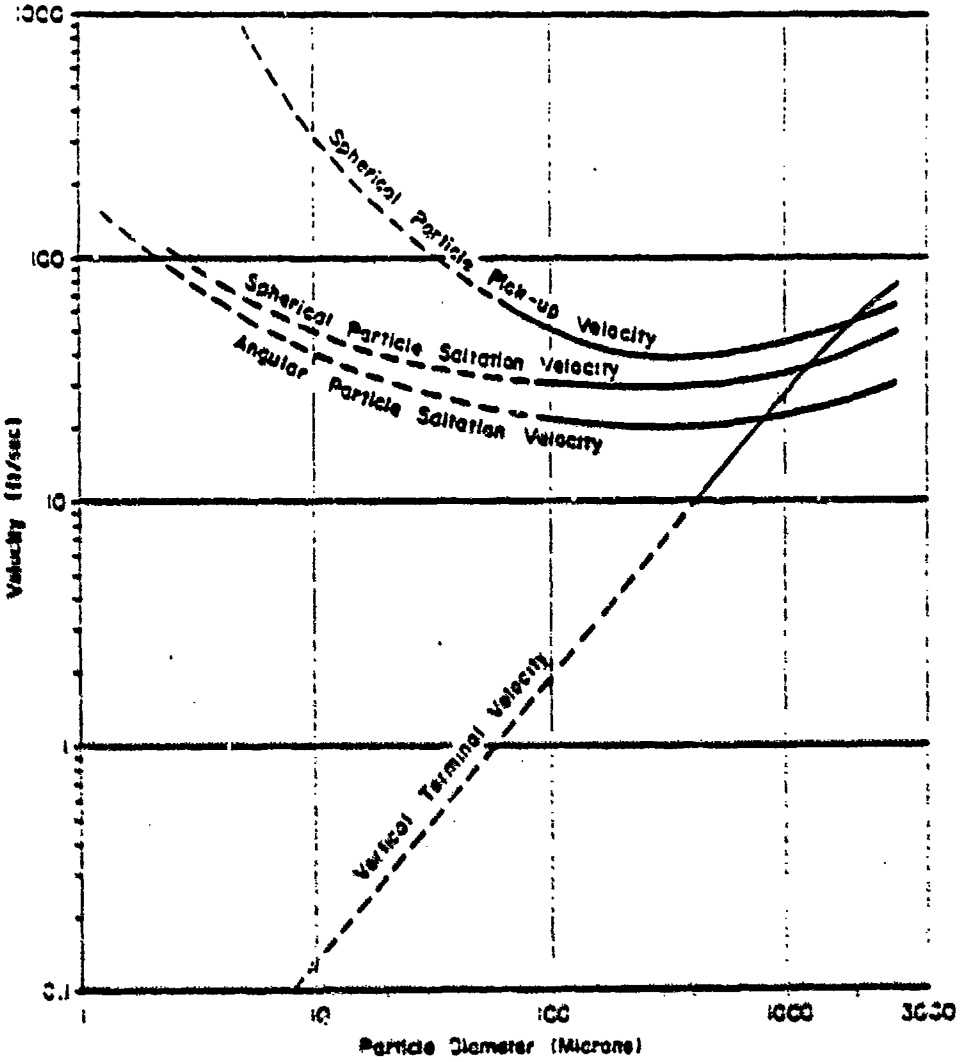


Figure G-2 Correlation of Single Particle Entrainment Saltation Velocities

9728.C8/JWM/5-3-83

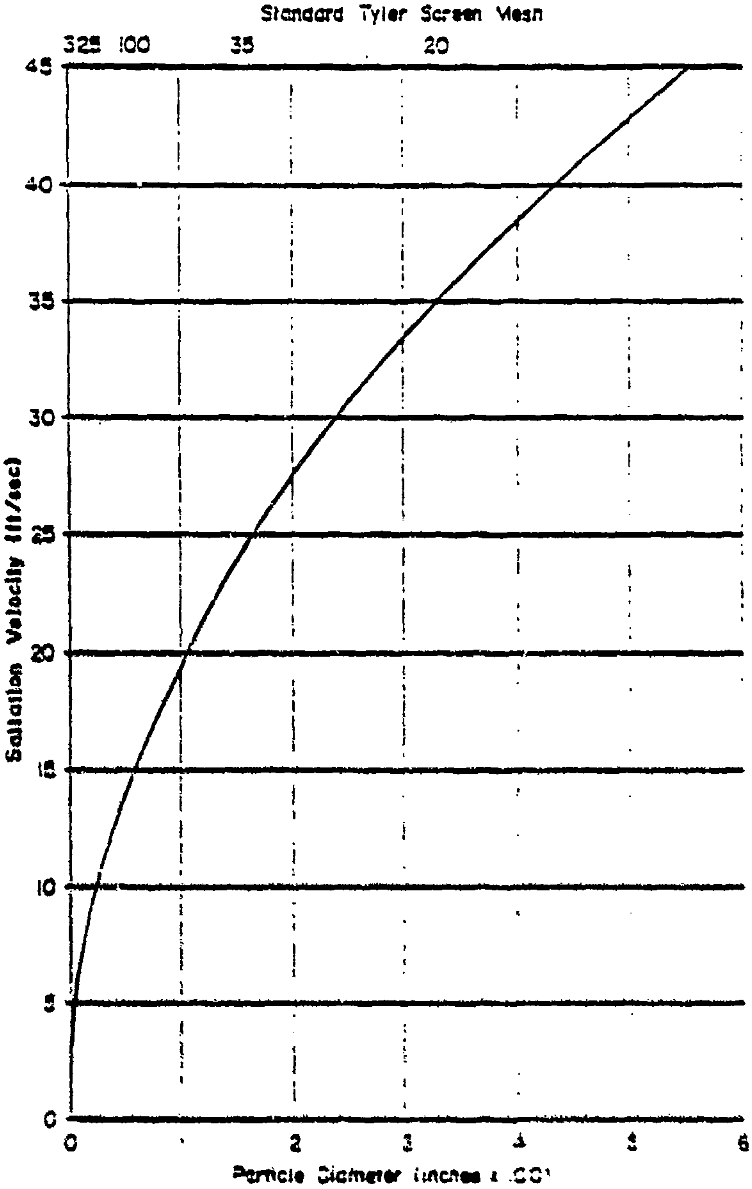


Figure G-1
Theoretical Saltation Velocity versus Particle Diameter

9738.06/JWH/8-3-85

SAMPLE COMPUTER HEAT TRANSFER AND HEAT MATERIAL BALANCE CALCULATIONS

10A ALLIS-CHALMERS HEAT TRANSFER COMPUTER PRINTOUT - RUN 2

10B ITC HEAT AND MATERIAL BALANCE COMPUTER PRINTOUT - RUN 2

2/13/85

```
$NAME
E= 0.899999976    ,ES= 0.899999976    ,EGAS=  520.000000   ,D1=  4.42000007    ,D2=  5.42000007    ,FA= 0.399999991E-01,FG=
  8354.00000    ,FS=  2400.00000    ,XK= 0.410000026    ,TS1=  60.0000000    ,TT2=  1450.00000    ,TA=  60.0000000    ,TJ=
 0.000000000E+00,MFC= 0.000000000E+00,TG1=  100.000000   ,CS= 0.230000019    ,QCOMB=  91667.0000    ,XLENGH=  16.0000000    ,EF=
 0.800000011    ,FLENGH=  6.00000000    ,AMOIST= 0.119999922E-01
$END
INPUT DATA:                                  SOIL 4760    RUN 2

 INTERNAL KILN DIAMETER (FT)                                =     4.42
 EXTERNAL KILN DIAMETER (FT)                                =     5.42
 AREA LOAD FRACTION                                         =     0.040
 GAS FLOW RATE (LB/HR)                                      =  8354.
 CLINKER FLOW RATE(LB/HR)                                   =  2430.
 AVERAGE GAS CONDUCTIVITY (BTU/FT-HR-F)                     =     0.0445
 KILN WALL CONDUCTIVITY (BTU/FT-HR-F)                       =     0.410
 AVERAGE GAS VISCOSITY (LB/HR-FT)                           =     0.116
 AVERAGE GAS DENSITY (LB/CUFT)                              =     0.0169
 AVERAGE PRANDTL NUMBER OF GAS                              =     0.71
 AVERAGE GAS EMISSIVITY                                     =   520.00
 AVERAGE SOLID EMISSIVITY                                   =     0.90
 AVERAGE KILN WALL EMISSIVITY                               =     0.90
 INCREMENT OF KILN LENGTH (FT)                              =     0.10
 INITIAL HEAT TRANSFER COEF FROM WALL TO FLUID (BTU/HR-F-FT2)=    2.38
 CONVERGENCE CRITERION FOR INNER WALL TEMPERATURE           =     0.0050
 CONVERGENCE CRITERION FOR OUTER WALL "                     =     0.0100
 CONVERGENCE CRITERION FOR SOLIDS TEMPERATURE               =     5.
 AMBIENT FLUID CONDUCTIVITY (BTU/HR-FT-F)                   =     0.0174
 AMBIENT FLUID DENSITY (LB/CUFT)                            =     0.0700
 PRANDTL NUMBER OF THE AMBIENT FLUID                        =     0.71
 AMBIENT FLUID VISCOSITY (LB/HR-FT)                         =     0.045
 AMBIENT AIR TEMPERATURE (F)                                =    60.

 HEAT INPUT BY FUEL COMBUSTION (BTU/MIN)                    = 91667.0
 FLAME LENGTH ESTIMATED (FT)                                =     6.00
 EMISSIVITY OF FLAME                                        =     0.80
 FLAME HEAT RELEASE DISTRIBUTION, GAMMA                     =     6
 FLAME HEAT RELEASE DISTRIBUTION, DELTA                     =     6.0
 FLAME DEAD LENGTH FROM SOLID EXIT (FT)                     =     0.0
 HEAT OF CLINKER FORMATION (BTU/MIN)                        =     0.0
 CLINKERING HEAT RELEASE LENGTH  (FT)                       =     0.0
 CLINKERING HEAT RELEASE, GAMMA                             =     0
 CLINKERING HEAT RELEASE, DELTA                             =     0.0
 CLINKERING DEAD LENGTH  (FT)                               =     0.0
 CALCINATION HEAT REQUIRED  (BTU/MIN)                       =     0.0
 MOISTURE FRACTION IN FEED SOLIDS                           =     0.012
 MAXIMUM SOLID TEMPERATURE (F)                              =  1900.0

 THE CALCULATED TOTAL BED ANGLE   (DEGREE)=     67.22
 THE CALCULATED BED DEPTH FT. =     0.3654

              DATA SUMMARY                                                    ACTIVE HEAT TRANSFER AREA
 INLET GAS TEMPERATURE (F)              =      100.0          GAS TO SOLIDS        (CONVECTION)   3.54 SQ.FT/FT
 INLET SOLID TEMPERATURE (F)            =     1405.4          GAS TO SOLIDS         (RADIATION)   2.45 SQ.FT/FT
 GAS EXIT TEMPERATURE (F)               =     1465.7          GAS TO SOLIDS (FREE CONVECTION)    55.20 SQ.FT/FT
 SOLID EXIT TEMPERATURE (F)          =          60.0          GAS TO WALL          (CONVECTION)  11.29 SQ.FT
 MAXIMUM SOLID TEMPERATURE (F)          =     1305.6          GAS TO WALL           (RADIATION)  11.29 SQ.FT/FT
 KILN LENGTH (FT)                       =       16.1          SOLIDS TO WALL       (CONVECTION)   2.59 SQ.FT/FT
 FUEL CONSUMPTION (BTU/MIN)             =    91657.0          SOLIDS TO WALL        (RADIATION)   2.59 SQ.FT/FT
                                                              THROUGH THE WALL     (CONDUCTION)  30.81 FT/FT
                                                              WALL TO AMBIENT      (CONVECTION)  17.93 SQ.FT/FT
```

```
                                        SUMMARY OF TEMPERATURES AND FLOWS
                                    DEGREES F. AND LBS/HR

        AXIAL
       DISTANCE                        SOLIDS                              GASES                                    KILN WALL
        (FEET)                TS                FS              TG                  FG            TW          TWO          HWO          HWR

          0.0                40.0            2400.00           100.0             8354.00          79.1        65.5        0.950        0.956
          1.0                62.9            2400.00           165.5             8354.00         102.8        71.4        1.139        0.902
          2.0                88.5            2400.00           822.6             8354.00         596.0       161.1        1.926        1.174
          3.0               218.1            2400.00          1499.2             8354.00        1452.7       251.9        2.245        1.622
          4.0               575.8            2400.00          2066.2             8354.00        1783.7       322.0        2.364        1.770
          5.0               844.5            2400.00          2117.1             8354.00        1789.1       322.2        2.364        1.786
          6.0              1133.2            2400.00          2055.1             8354.00        1710.4       313.1        2.347        1.746
          7.0              1320.9            2400.00          2047.1             8354.00        1734.1       316.1        2.353        1.754
          8.0              1466.7            2400.00          2006.3             8354.00        1753.1       318.3        2.357        1.765
          9.0              1546.3            2400.00          1972.6             8354.00        1760.3       319.1        2.359        1.768
         10.0              1627.4            2400.00          1946.6             8354.00        1773.6       320.6        2.361        1.775
         11.0              1693.0            2400.00          1925.8             8354.00        1786.2       322.0        2.364        1.782
         12.0              1738.3            2400.00          1909.5             8354.00        1794.5       322.9        2.366        1.786
         13.0              1771.4            2400.00          1895.8             8354.00        1799.0       323.4        2.366        1.788
         14.0              1787.7            2400.00          1886.7             8354.00        1800.5       323.6        2.367        1.789
         15.0              1799.3            2400.00          1875.1             8354.00        1799.9       323.5        2.367        1.789
         16.0              1805.6            2400.00          1866.7             8354.00        1797.7       323.3        2.366        1.786
         16.1              1805.8            2400.00          1865.9             8354.00        1797.4       323.2        2.366        1.786
```

```
                                        SUMMARY OF HEAT TRANSFER PATHS
                                           BTU'S/HOUR/AXIAL FOOT

  AXIAL       GAS -TO- SOLIDS                             GAS -TO- WALL        WALL -TO- SOLIDS                               WALL -TO- AMBIENT
 DISTANCE  CONVECTION RADIATION                      CONVECTION  RADIATION  CONVECTION  RADIATION                            CONV. AND RAD.
  (FEET)      QGSC       QGSR       QCLEM     QALEM     QGWC       QGWR       QSWC        QSWR       QCOMB        QCALX       QWD

   0.0        523.        85.        0.        0.        405.       236.       861.          9.          0.         0.        171.
   1.0       1081.       204.        0.        0.       1642.       558.      1763.         19.     606093.         0.        397.
   2.0       9605.      7386.        0.        0.       9097.     18579.     21958.       1652.    2418927.         0.       5366.
   3.0      19379.     47353.        0.        0.       9477.     84421.     54491.      25019.    1703165.         0.      14789.
   4.0      20285.     67997.        0.        0.      10881.    121252.     55960.      57691.     596747.         0.      19462.
   5.0      16388.     52309.        0.        0.      12609.    109353.     40907.      62629.     141541.         0.      18529.
   6.0      12429.     30602.        0.        0.      14330.     78815.     25677.      50019.      26279.         0.      17649.
   7.0       9347.     25455.        0.        0.      11813.     64732.     17072.      40663.       4120.         0.      17910.
   8.0       7029.     20601.        0.        0.       9068.     53002.     12639.      35886.        571.         0.      18123.
   9.0       5578.     17012.        0.        0.       8184.     44701.      9445.      25217.         72.         0.      19203.
  10.0       4176.     13303.        0.        0.       6859.     36669.      8451.      18517.          5.         0.      18352.
  11.0       3046.     10092.        0.        0.       5357.     29527.      6112.      12289.          1.         0.      14494.
  12.0       2237.      7574.        0.        0.       4414.     24321.      2480.       7569.          0.         0.      18586.
  13.0       1687.      5735.        0.        0.       3775.     20521.      1549.       4261.          0.         0.      18037.
  14.0       1269.      4408.        0.        0.       3235.     17709.       566.       1613.          0.         0.      18655.
  15.0        992.      3486.        0.        0.       2992.     15972.        28.         90.          0.         0.      18648.
  16.0        801.      2806.        0.        0.       2654.     14517.      -343.      -1108.          0.         0.      18623.
  16.1        786.      2750.        0.        0.       2635.     14405.      -375.      -1205.          0.         0.      18619.

 SUM OF HEAT                                                                                                       0.     278214.
 AVERAGE HEAT LOSS, BTU/(FT**2,HR)                                                                                             906.
```

```
**********  INPUT DATA SUMMARY  **********

 1,EERU KILN CAPACITY ESTIMATION
 2,AF SOIU-#1
 2,2/18/85
 3,9736.05
 4,JLF
 5,60,406.8,3,6,0,0/
 6,6/
06,C     H2    O2    N2    WAT   ASH
07,SOIL        1.0/
07,0,0,0,0,0,0,0,0,0,0,0,0,100,0/
08,2400,0,0/
09,.001,60,0/
07,WATER       1.0/
07,0,0,0,0,100,0,0,0,0,0,0,0,0,0/
08,29,0,0/
09,.001,60,0/
07,AUX1        3.0/
07,91,9,0,0,0,0,0,0,0,0,0,0,0,0/
08,0,0,0/
09,18000,60,0/
07,AUX2        3.0/
07,91,9,0,0,0,0,0,0,0,0,0,0,0,0/
08,0,0,0/
09,18000,60,0/
07,FUEL1       1.0/
07,91,9,0,0,0,0,0,0,0,0,0,0,0,0/
08,306,0,0/
09,18000,60,0/
07,FUEL2       2.0/
07,91,9,0,0,0,0,0,0,0,0,0,0,0,0/
08,0,0,0/
09,18000,60,0/
10,0,406.3,3,.29689,1,100,60,.01,0,45,100/
10,
15,2200,405.8,3,.5,1,50,60,.01,0,50,50/
15,AUX2
```

```
JOB NO 9736.05 :AF SOIU-#1          ENGINEER:JLF          ( 60.0 DEG F & 14.696 PSI)  2/18/85    PAGE  1

 I. FUEL TO PRIMARY BURNER

 FUEL NAME  ************************************** COMPONENT FLOW TO FURNACE  ***********************************
             CARBON    HYDROGEN    OXYGEN    NITROGEN     WATER        ASH
 SOIL
  PERCENT     0.000      0.000      0.000      0.000      0.000     100.000
  POUNDS      0.000      0.000      0.000      0.000      0.000    2400.000
  LB-MOLE     0.000      0.000      0.000      0.000      0.000       0.000

 WATER
  PERCENT     0.000      0.000      0.000      0.000    100.000       0.000
  POUNDS      0.000      0.000      0.000      0.000     29.000       0.000
  LB-MOLE     0.000      0.000      0.000      0.000      1.610       0.000

 FUEL1
  PERCENT    91.000      9.000      0.000      0.000      0.000       0.000
  POUNDS    278.460     27.540      0.000      0.000      0.000       0.000
  LB-MOLE    23.184     13.661      0.000      0.000      0.000       0.000

 TOT FUEL
  POUNDS    278.460     27.540      0.000      0.000     29.000    2400.000
  LB-MOLE    23.184     13.661      0.000      0.000      1.610       0.000
```

```
JOB NO 9736.05 :AF SOIU-#1         ENGINEER:JLF           ( 60.0 DEG F & 14.696 PSI)  2/18/85   PAGE 2

  1. PRIMARY BURNER HEAT AND MATERIAL BALANCE

  *****  HEAT IN  *****             TEMP DEG F        LBS / HR          BTU / LB          MM BTU/HR
  FUEL  SOIL                          60.000          2400.000             0.001              0.000
  FUEL  WATER                         60.000            29.000             0.001              0.000
  FUEL  FUEL1                         60.000           306.000         18000.000              5.508

        DRY AIR                       60.000          8284.038             0.000              0.000
        AIR HUM.                      60.000            82.840          1059.900              0.088

  OVERALL TOTAL                        0.000         11101.879                                5.596



  *****  HEAT OUT *****             LB-MOLES          LBS / HR            BTU/LB          MM BTU/HR

  FLU GAS OUT: 1777.83 DEG F
        CO2                           23.184          1020.340           461.733              0.471
        H2O                           19.869           357.952          1935.832              0.693
        N2                           113.563          3181.568           457.803              1.457
        AIR (XS)                     143.125          4142.019           447.551              1.854
  TOTAL FLU GAS                      299.740          8701.879                                4.474

  SOLIDS OUT BOTTOM:
        ASH                            0.000          2400.000           343.567              0.825
        INERT                          0.000             0.000           343.567              0.000

      RAD LOSS                                                                                0.297

  OVERALL TOTAL                      299.740         11101.879                                5.596
```

```
JOB NO 9736.05 :AF SOIU-#1        ENGINEER:JLF          ( 60.0 DEG F & 14.696 PSI)  2/18/85    PAGE  3

  III. FUEL TO SECONDARY BURNER

  FUEL NAME  **************************************  COMPONENT FLOW TO FURNACE  ******************************
               CARBON    HYDROGEN    OXYGEN   NITROGEN     WATER       ASH
  FUEL2
   PERCENT     91.000      9.000      0.000      0.000      0.000      0.000
   POUNDS       0.000      0.000      0.000      0.000      0.000      0.000
   LB-MOLE      0.000      0.000      0.000      0.000      0.000      0.000

  AUX2
   PERCENT     91.000      9.000      0.000      0.000      0.000      0.000
   POUNDS     356.929     35.301      0.000      0.000      0.000      0.000
   LB-MOLE     29.717     17.510      0.000      0.000      0.000      0.000

  TOT FUEL
   POUNDS     356.929     35.301      0.000      0.000      0.000      0.000
   LB-MOLE     29.717     17.510      0.000      0.000      0.000      0.000
```

```
JOB NO 9736.05 :AF SOIU-#1        ENGINEER:JLF          ( 60.0 DEG F & 14.696 PSI)   2/18/85    PAGE  4

 IV. SECONDARY BURNER HEAT AND MATERIAL BALANCE

 *****  HEAT IN  *****            TEMP DEG F      LBS / HR        BTU / LB        MM BTU/HR
 FUEL  FUEL2                         60.000          0.000       18000.000           0.000
 FUEL  AUX2                          60.000        392.230       18000.000           7.060

 PRIM. FLU GAS                     1777.833       8701.879                           4.474

       DRY AIR                       60.000       7963.846           0.000           0.000
       AIR HUM.                      60.000         79.638        1059.900           0.084

 OVERALL TOTAL                        0.000      17137.593                          11.619



 *****  HEAT OUT *****             LB-MOLES       LBS / HR         BTU/LB         MM BTU/HR

 FLU GAS OUT: 2200.00 DEG F
       CO2                           52.901       2328.209         593.726           1.382
       H2O                           41.799        753.055        2189.468           1.649
       N2                           259.127       7259.694         581.862           4.224
       AIR (XS)                     234.854       6796.634         568.466           3.864
 TOTAL FLU GAS                      588.681      17137.593                          11.119

 SOLIDS OUT BOTTOM:
       ASH                            0.000          0.000         428.000           0.000
       INERT                          0.000          0.000         428.000           0.000

    RAD LOSS                                                                         0.500

 OVERALL TOTAL                      588.681      17137.593                          11.619
```

```
V. PRIMARY BURNER AIR SUMMARY

                                 ******* OXYGEN  *******      ****** NITROGEN ******
                   LBS  AIR          POUNDS     LB-MOLE           POUNDS    LB-MOLE
TOTAL              8284.038        1920.903      60.028         6363.136    227.125
THEORETICAL        4142.019         960.451      30.014         3181.568    113.563

TOT - THEO.        4142.019         960.451      30.014         3181.568    113.563

PERCENT XS AIR FOR PRIMARY BURNER =  100.00


VI. SECONDARY BURNER AIR SUMMARY

                                 ******* OXYGEN  *******      ****** NITROGEN ******
                   LBS  AIR          POUNDS     LB-MOLE           POUNDS    LB-MOLE
TOTAL              7963.846        1846.657      57.708         6117.189    218.346
THEORETICAL        5309.231        1231.104      38.472         4078.126    145.564

TOT - THEO.        2654.615         615.552      19.236         2039.063     72.782

PERCENT XS AIR FOR SECONDARY BURNER =   50.00


VII. TOTAL AIR TO PRIM. AND SEC. BURNERS

                                 ******* OXYGEN  *******      ****** NITROGEN ******
                   LBS  AIR          POUNDS     LB-MOLE           POUNDS    LB-MOLE
TOTAL             16247.884        3767.559     117.736        12480.325    445.471
THEORETICAL        9451.250        2191.556      68.486         7259.694    259.127

TOT - THEO.        6796.634        1576.004      49.250         5220.631    186.345

TOTAL PERCENT XS AIR =   71.91


VIII. GAS FLOW RATES

                            TEMPERATURE     PRESSURE
 STREAM                       (DEG F)        (PSI)          ACFM
1 PRIM OUT                   1777.833        14.678       8172.188

2 SEC OUT                    2200.000        14.660      19101.663

  PRIM AIR                     60.000        14.696       1810.575

  SEC AIR                      60.000        14.696       1682.457
```

STATISTICAL DATA

11A PARAMETERS USED IN REGRESSION ANALYSIS LISTED BY SOIL TYPE

11B PARAMETERS USED IN REGRESSION ANALYSIS LISTED IN ORDER OF INCREASING IVP

PARAMETERS USED IN REGRESSION ANALYSES LISTED IN ORDER OF SOIL TYPE

Soil Identification	INCONC	Nominal temperature (°C)	Time at nominal temperature (min)	ITEMP	D	IVP	LINC	FD	LFD	LNFD	LD	LOGVP	VP	IVPSQ	TEMPSQ	TITP
JI	106	482	15	8375	4.50	42047	2.02531	0.04245	-1.3721	-3.1594	0.65321	3.4461	2793.2	1767950209	232324	7230
JI	106	481	30	16573	1.60	86165	2.02531	0.01509	-1.8212	-4.1934	0.20412	3.4389	2747.3	7424407225	231361	14430
JI	106	559	8	5516	0.56	69608	2.02531	0.00528	-2.2773	-5.2433	-0.25181	3.9403	8717.8	4845273664	312481	4472
JI	106	432	20	9957	38.50	23722	2.02531	0.36321	-0.4398	-1.0128	1.58546	3.0613	1151.6	562733284	186624	8640
JI	106	23	0	0	106.00	0	2.02531	1.00000	0.0000	0.0000	2.02531	-4.9651	0.0	0	529	0
Eglin	101	482	15	9942	1.30	48839	2.00432	0.01287	-1.8904	-4.3528	0.11394	3.4461	2793.2	2385247921	232324	7230
Eglin	101	485	30	16554	0.45	90021	2.00432	0.00446	-2.3511	-5.4136	-0.34679	3.4676	2934.0	8103780441	235225	14550
Eglin	101	482	15	9616	0.80	49284	2.00432	0.00792	-2.1012	-4.8383	-0.09691	3.4461	2793.2	2428912656	232324	7230
Eglin	101	559	8	6525	0.71	82805	2.00432	0.00703	-2.1531	-4.9576	-0.14874	3.9403	8717.1	6856668025	312481	4472
Eglin	101	432	20	9926	4.40	22681	2.00432	0.04356	-1.3609	-3.1335	0.64345	3.0613	1151.6	514427761	186624	8640
Eglin	101	23	0	0	101.00	0	2.00432	1.00000	0.0000	0.0000	2.00432	-4.9651	0.0	0	529	0
NCBC	494	557	15	5516	0.99	152489	2.69373	0.00200	-2.6981	-6.2126	-0.00436	3.9364	8637.0	23252895121	310249	8355
NCBC	494	484	15	9609	10.10	49654	2.69373	0.02045	-1.6894	-3.8900	1.00432	3.4604	2887.0	2465519716	234256	7260
NCBC	494	483	30	16774	4.60	79309	2.69373	0.00931	-2.0310	-4.6765	0.66276	3.4533	2839.0	6289917481	233289	14490
NCBC	494	558	15	10923	0.53	153070	2.69373	0.00107	-2.9695	-6.8374	-0.27572	3.9423	8756.0	23430424900	311364	8370
NCBC	494	431	30	13974	26.60	33631	2.69373	0.05385	-1.2688	-2.9216	1.42488	3.0530	1129.9	1131044161	185761	12930
NCBC	494	23	0	0	494.00	0	2.69373	1.00000	0.0000	0.0000	2.69373	-4.9651	0.0	0	529	0

INCONC - Initial 2,3,7,8-TCDD concentration (ppb)
ITEMP - Integral of temperature over treatment period (°C · min)
D - Final 2,3,7,8-TCDD concentration (ppb)
IVP - Integral of vapor pressure over treatment period (mmHg · min)
LINC - $\log_{10}$ of initial 2,3,7,8-TCDD concentration
FD - Weight fraction of 2,3,7,8-TCDD remaining after treatment
LFD - $\log_{10}$ of FD
LNFD - $\log_e$ of FD
LD - $\log_{10}$ of final D
LOGVP - $\log_{10}$ of 2,3,7,-TCDD vapor pressure in mmHg
IVPSQ - IVP squared
TITP - (Time) · (Temp)

0391 1-AP-2

PARAMETERS USED IN REGRESSION ANALYSES LISTED IN ORDER OF INCREASING TVP

Soil Identification	INCONC	Nominal Temperature (°C)	Time at nominal temperature (min)	ITEMP	D	TVP	LINC	FD	LFD	LNFD	LD	LOGVP	VP	TVPSQ	ITMPSQ	TTTP
JI	106	23	0	0	106.00	0	2.02531	1.00000	0.0000	0.0000	2.02531	-4.9651	0.0	0	529	0
Eglin	101	23	0	0	101.00	0	2.00432	1.00000	0.0000	0.0000	2.00432	-4.9651	0.0	0	529	0
NCBC	494	23	0	0	494.00	0	2.69373	1.00000	0.0000	0.0000	2.69373	-4.9651	0.0	0	529	0
Eglin	101	432	20	9926	4.40	22808	2.00432	0.04356	-1.3609	-3.1335	0.64345	3.0613	1151.6	[illegible]	186624	8640
JI	106	432	20	9957	38.50	23722	2.02531	0.36321	-0.4398	-1.0128	1.58546	3.0613	1151.6	562733284	186624	8640
NCBC	494	431	30	13974	26.60	33631	2.69373	0.05385	-1.2688	-2.9216	1.42488	3.0530	1129.9	1131044161	185761	12930
JI	106	482	15	8375	4.50	42047	2.02531	0.04245	-1.3721	-3.1594	0.65321	3.4461	2793.2	1767950209	232324	7230
Eglin	101	482	15	8887	1.30	[illegible]	2.00432	0.01287	-1.8904	-4.3528	0.11394	3.4461	2793.2	[illegible]	232324	7230
Eglin	101	482	15	9816	0.80	[illegible]	2.00432	0.00792	-2.1012	-4.8383	-0.09691	3.4461	2793.2	[illegible]	232324	7230
NCBC	494	484	15	9640	10.30	49654	2.69373	0.02085	-1.6809	-3.8704	1.01284	3.4600	2887.0	2465519716	234256	7260
JI	106	509	8	5516	0.56	[illegible]	2.02531	0.00528	-2.2771	-5.2433	-0.25181	3.9001	[illegible]	[illegible]	259081	4072
NCBC	494	483	30	16774	4.60	[illegible]	2.69373	0.00931	-2.0310	-4.6765	0.66276	3.4533	2839.0	[illegible]	233289	14490
Eglin	101	559	8	8625	0.71	[illegible]	2.00432	0.00703	-2.1531	-4.9576	-0.14870	3.9073	8077.1	[illegible]	312481	4472
JI	106	481	30	16571	1.60	86165	2.02531	0.01509	-1.8212	-4.1934	0.20412	3.4389	2741.3	7424407225	231361	14430
Eglin	101	485	30	16554	0.45	99921	2.00432	0.00446	-2.3511	-5.4136	-0.34679	3.4679	2936.0	[illegible]	235225	14550
NCBC	494	557	15	5616	0.99	[illegible]	2.69373	0.00200	-2.6981	-6.2126	-0.00436	3.9364	8637.0	[illegible]	310249	8355
NCBC	494	558	15	[illegible]	0.53	[illegible]	2.69373	0.00107	-2.9695	-6.8374	-0.27572	3.9423	8756.0	[illegible]	311364	8370

INCONC - Initial 2,3,7,8-TCDD concentration (ppb)
ITEMP - Integral of temperature over treatment period (°C · min)
D - Final 2,3,7,8-TCDD concentration (ppb)
TVP - Integral of vapor pressure over treatment period (mmHg · min)
LINC - Log10 of initial 2,3,7,8-TCDD concentration
FD - Weight fraction of 2,3,7,8-TCDD remaining after treatment compared with initial concentration
LFD - Log10 of FD
LNFD - Loge of FD
LD - Log10 of D
LOGVP - Log10 of 2,3,7,8-TCDD vapor pressure in mmHg
TVPSQ - TVP squared
TTTP - (Time) x (Temp)

[illegible]