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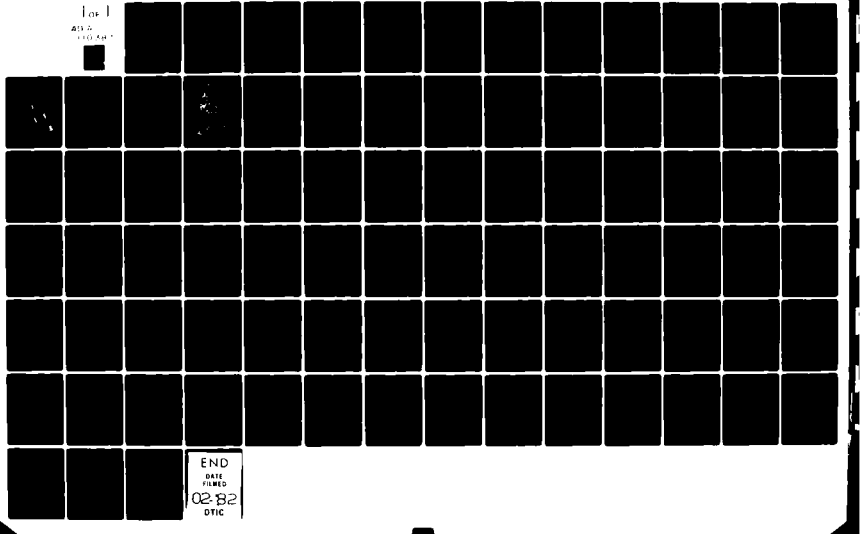

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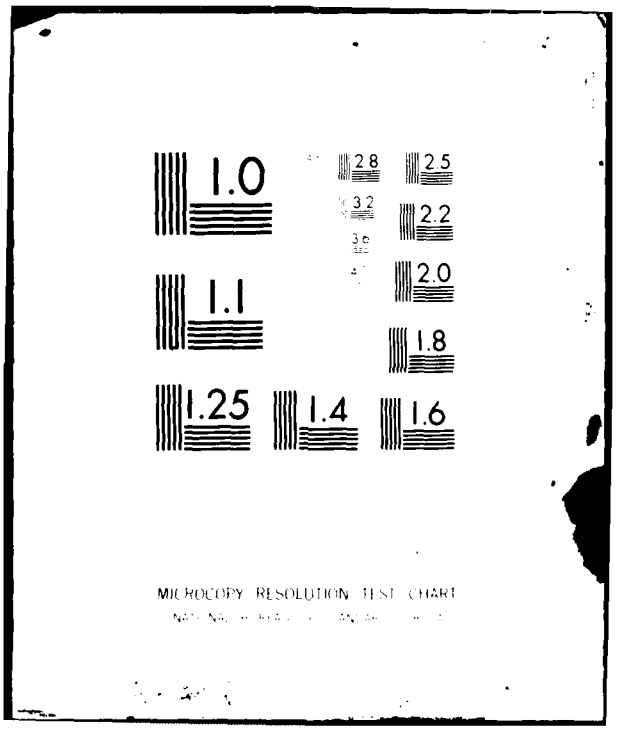
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EXCESS AREA CONTAMINATION SURVEY OF INDIANA ARMY AMMUNITION PLANT

Prepared by:

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25 September 1981

FINAL REPORT

Under Contract to:
Battelle Scientific Services Program
Delivery Order 0015 for:

U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
Aberdeen Proving Ground, Maryland 21010

USATHAMAMISC.1/INAAP/DIST.1
9/24/81

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A limited environmental survey of a proposed excess area in the northern portion of Indiana Army Ammunition Plant was performed to determine the extent of residual hazardous contamination. Two areas, the Burning Ground and the Static Test Area, were surveyed. Soils of the Burning Ground were sampled and analyzed for nitrocellulose. Buildings surfaces in the Static Test Area were sampled and analyzed for nitrocellulose and nitroglycerin. In addition, paints on building surfaces were tested for heavy metals and asbestos content.

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Nitrocellulose was not found in soil, sediment or sewer samples. Building surfaces were free of nitrocellulose and nitroglycerin contamination. Paint samples contained high levels of copper, chromium and zinc and low levels of lead and cadmium. Mercury was not found in any paint samples.

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1.0 INTRODUCTION

Indiana Army Ammunition Plant (INAAP) is located in Charlestown, Indiana, north of Louisville, Kentucky, and bordering the Ohio River. ICI Americas (ICI) currently operates the ammunition plant. A limited Environmental Survey of a 1,333-acre area of INAAP identified for excessing action was conducted by ESE from July 27 to July 30, 1981.

1.1 PURPOSE OF SURVEY

The proposed excess area is located in the northern portion of INAAP (Figure 1-1). A determination of the extent of residual hazardous contamination was required before release of the property to civilian access. Potential residual contamination by nitrocellulose and nitroglycerin existed in two sites within the area. The Burning Ground in the eastern part of the excess area was used until 1958 for open burning of explosives containing nitrocellulose. The Static Test Area, located centrally, was used briefly in 1945 for nitrocellulose/nitroglycerin rocket motor testing, and again from 1970 to 1973 for nitrocellulose/nitroglycerin propellant safety testing. Reportedly, this site was decontaminated after these operations. An incomplete industrial sewer system with the remains of two manholes still in place is in the Static Test Area. Sediments in these manholes had the potential for contamination from past operations in the Static Test Area.

Further evaluation of the extent of nitrocellulose contamination in the excess area was required for sediments of streams traversing the tract. Fourteen Mile Creek receives runoff from the Burning Ground and the Static Test area. Lick Creek, which feeds into Fourteen Mile Creek, drains the western part of the proposed excess area and receives runoff from the Single Base Propellant Area (Number 7, Figure 1-1), the former landfill (Number 4, Figure 1-1), and effluent from the City of Charlestown sewage disposal plant. These creeks are mapped in Figure 1-1.

Some buildings in the Static Test Area had painted surfaces which had deteriorated. The red, non-heat conductive paint was porous, resembling concrete, and contained fibrous materials. The potential for metals

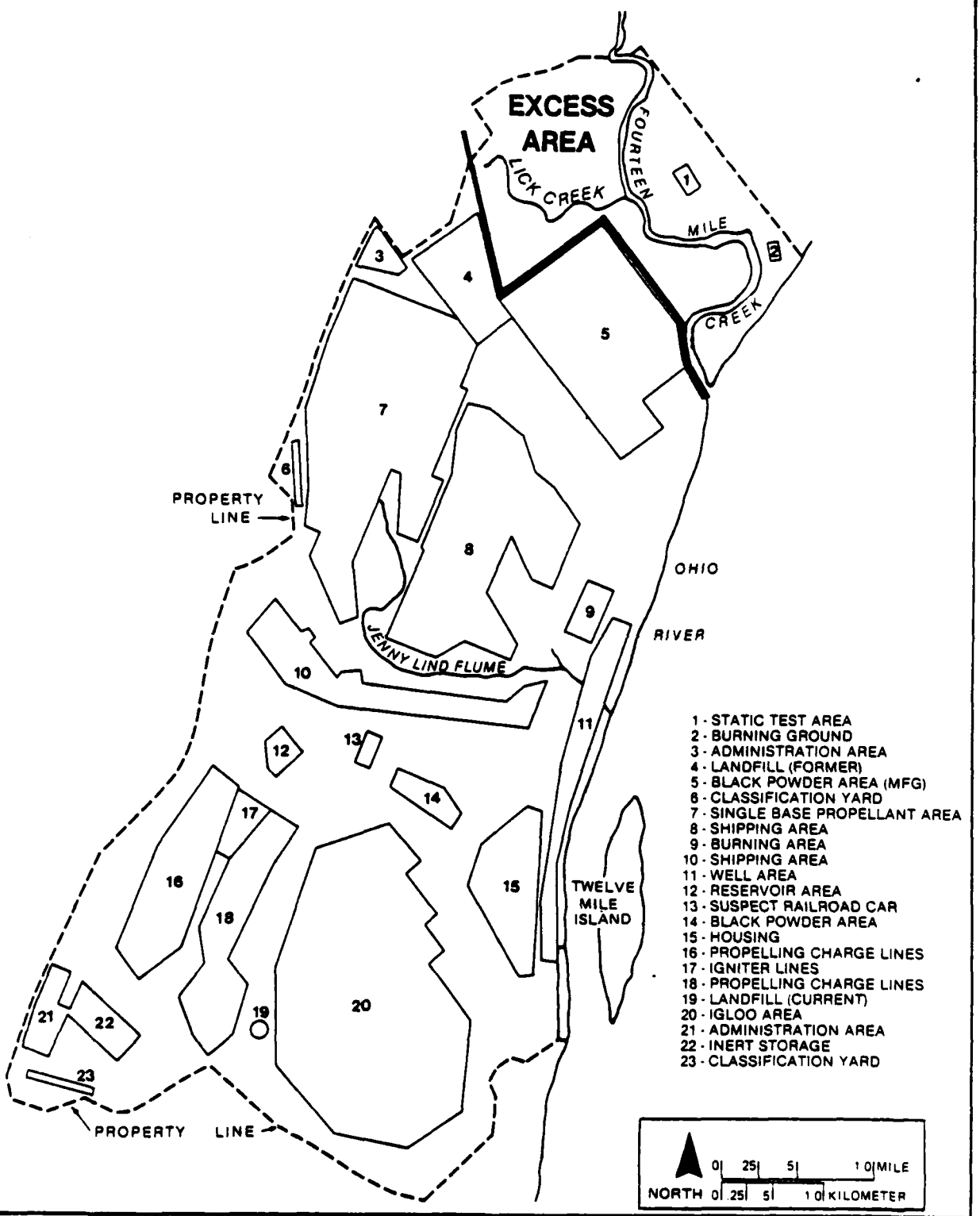

Figure 1-1
INDIANA ARMY AMMUNITION PLANT INCLUDING PROPOSED EXCESS AREA

SOURCES: USATHAMA, 1981. ESE, 1981.

Limited Environmental Survey

INDIANA ARMY AMMUNITION PLANT
CHARLESTOWN, INDIANA

contamination arising from these paints was determined in the Environmental Survey. Fibers contained in the paint were analyzed for asbestos.

The limited Environmental Survey conducted at INAAP included the analysis of soils, sediments, and sewers for nitrocellulose, building surfaces for nitrocellulose and nitroglycerin, and paint samples for metals and asbestos. Only the potentially contaminated sites in the proposed excess area were sampled. A comprehensive evaluation of the extent of residual contamination was carried out to satisfy the requirements for preparation of certification documents for excessing actions.

1.2 TECHNICAL APPROACH

The ESE field team and the USATHAMA Project Officer arrived in Charlestown, Indiana, on July 27, 1981 and met government and ICI personnel who provided access to the proposed Excess Area. The sampling program was begun under the supervision of the USATHAMA Project Officer. Sample sites in the Burning Ground were marked and half of the soil samples were collected.

The following day, manholes in the Static Test Area were sampled. Paint samples were collected from buildings in this area. One sediment sample was obtained from Lick Creek and the remaining soil samples from the Burning Ground were collected. On July 29, the sediments from Fourteen Mile Creek were sampled.

Building swabs for nitrocellulose and nitroglycerin were taken in the Static Test Area on July 30, the final day of the survey. Samples were shipped to ESE, Gainesville, by guaranteed air freight and were analyzed during the period from August 10 to September 10. Results of these analyses are presented in this report.

2.0 DISCUSSION

2.1 FIELD SAMPLING

ESE's chain-of-custody procedures were followed for the INAAP sampling program. These procedures allow precise accounting for the location and status of samples throughout the sampling and analysis process by a computer-controlled management program.

Prior to field sampling, kits of sample collection vessels were prepared and labeled. Vessels were thoroughly washed, rinsed with acetone and hexane, and air dried.

Acquisition of labels was part of the Pre-Field Setup procedure, in which sample stations, fractions, sample trip itinerary, personnel, and analyses to be performed were entered in the data management system. Labels were printed with the information necessary for their efficient use in sample collection and documentation. The containers were then labeled, packed, and shipped to INAAP.

At the time of sampling, the sample point, time, date, and the sampler's initials were marked on the label with waterproof ink. Sampling information was also entered on log sheets, which served as shipping forms. Complete records were maintained in the field team's notebook.

Samples were kept at room temperature (25°C), packed, and shipped by guaranteed air freight to ESE, Gainesville. Upon arrival, samples were checked against the log sheets shipped with them and were logged in to ESE's data management system. Prior to analysis, samples were kept at 4°C.

2.1.1 Burning Ground

Soil samples were collected from thirty points in the Burning Ground Area (Figure 1-2). The exact sample points were inspected jointly by the ESE field team and the USATHAMA Project Officer. Each site was marked with a wooden stake painted fluorescent orange, and each was

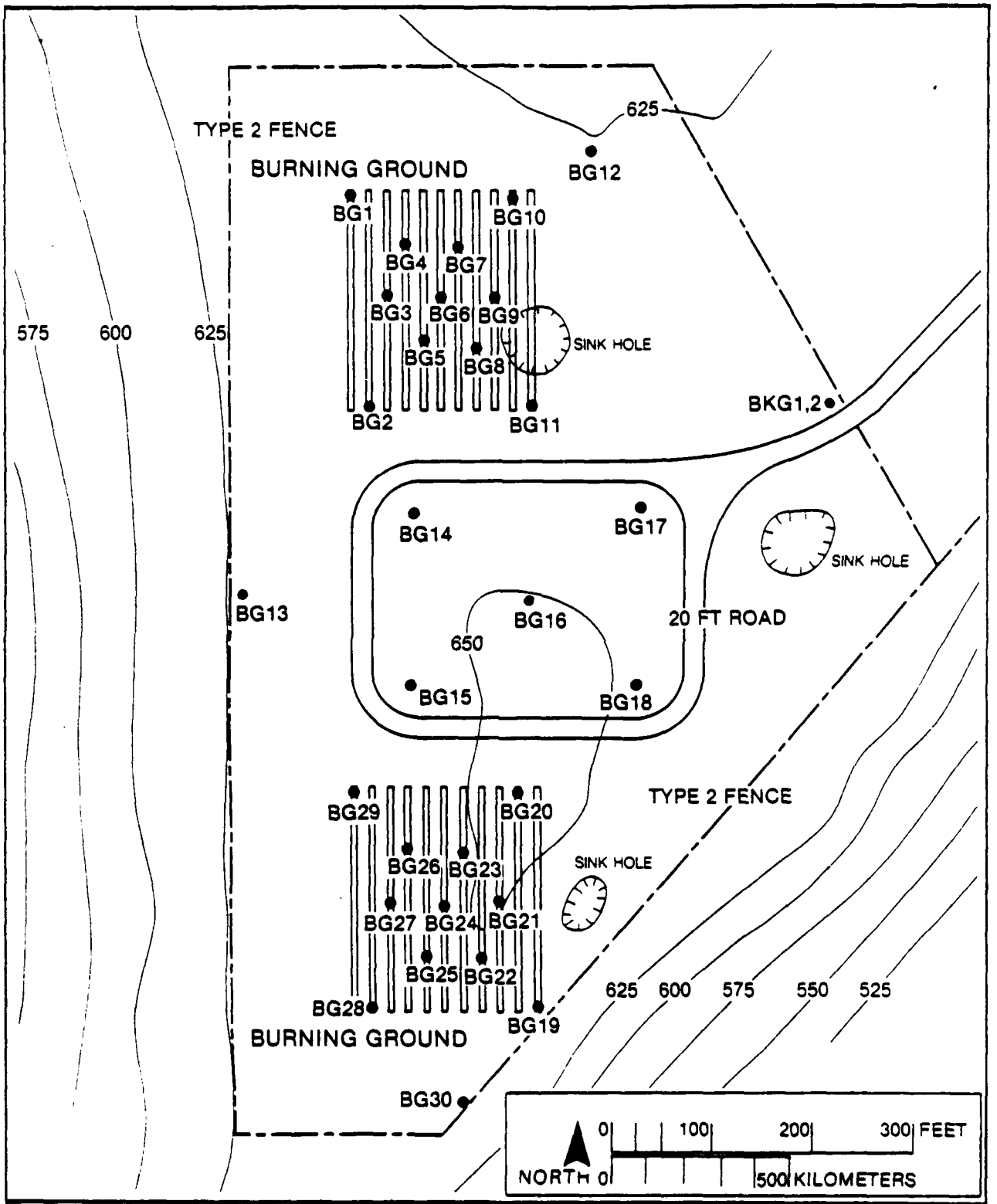

Figure 1-2
BURNING GROUND SOIL SAMPLING SITES

SOURCE: ESE, 1981.

Limited Environmental Survey

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given a sample point code number. Sites were labeled BG01 through BG30, consecutively. These points included sites in burning ground trenches (last used for nitrocellulose burning in 1958), drainage sites, and sample points in the turnaround bordered by the service road.

The area was densely forested with deciduous and coniferous trees. Ground cover was dense, with leaf litter, honeysuckle, ferns, grasses, and forbs. Sinkholes were scattered about the Burning Ground Area. The trenches used for burning were still evident, though overgrown. Drainage Sites BG12, BG13, and BG30 were gullies which carried runoff from the site. Site BG12, particularly, had metal debris and trash lying in it.

Two additional soil samples, BKG1 and BKG2, were collected east of the Burning Ground at a site which was uncontaminated. These samples were used as quality control blanks and for spiking according to the USATHAMA August 1980 Quality Assurance Program.

Prior to coring, surface vegetation, rocks, leaves, and debris were removed from the sample point to allow collection of a clean soil sample. One-foot-deep soil samples were taken with a soil auger, 1 inch in diameter. Distilled water, approved by the USATHAMA Project Officer, was used for cleaning the coring device after each sample was taken. Soil samples were placed in prelabeled, 1-quart Mason® jars with Teflon®-lined lids.

2.1.2 Static Test Area

There were nine buildings or building foundations in the Static Test Area which were sampled for specified parameters during the Environmental Survey. Four were sampled for nitrocellulose and nitroglycerin and five were sampled for metals in paint. These buildings are shown in Figure 1-3.

2.1.2.1 Building Sampling for Nitrocellulose and Nitroglycerin

Four buildings in the Static Test Area were sampled for residual nitrocellulose and nitroglycerin: 2750-2, 2750-2A, 2750-3, and

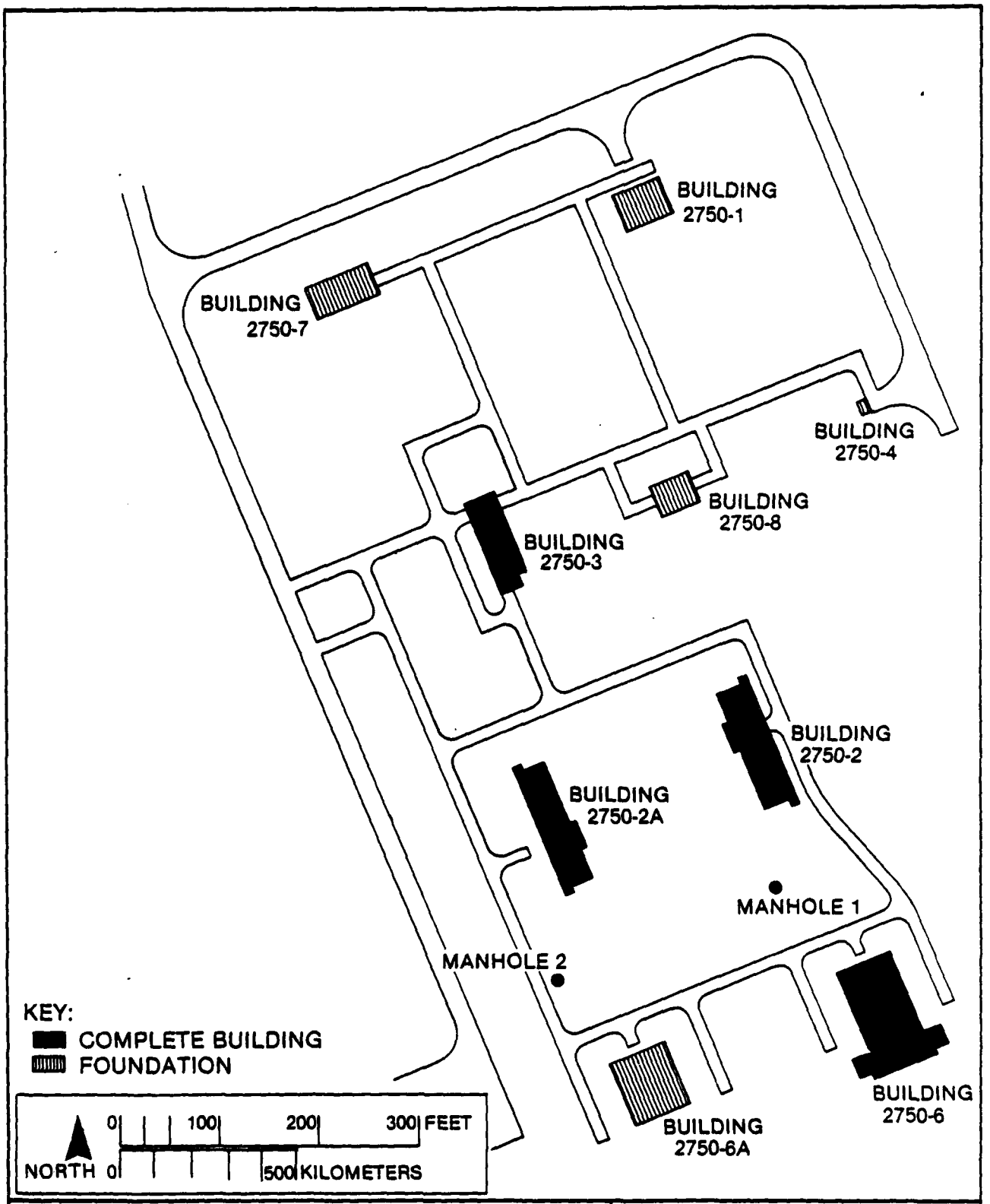

Figure 1-3
STATIC TEST AREA BUILDING AND SEWER SAMPLING SITES

SOURCES: USATHAMA, 1981. ESE, 1981.

Limited Environmental Survey

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2750-6, shown in Figure 1-3. These buildings were constructed of reinforced concrete. Building 2750-6 was surrounded by a concrete pad and showed evidence of propellant testing, with blackened wall and floor surfaces. Buildings 2750-2 and 2750-2A were surrounded by vegetation 4 feet high, making access to the buildings difficult. The buildings were used for shelter by cattle and birds. Building 2750-3, which had no roof, was surrounded by vegetation approximately 2 feet high. A sheet metal shed with evidence of testing operations stood in the center of the structure.

Spot tests for nitrocellulose and nitroglycerin were performed on these building surfaces. Building surfaces that yielded positive spot test results were sampled for confirmatory thin-layer chromatography (TLC) tests for nitrocellulose and nitroglycerin. A total of 16 samples was taken.

Samples were collected by wiping the surface to be tested with a cotton swab soaked with acetone. A 20-square centimeter (cm^2) area was sampled, the swab rinsed with acetone, and the acetone collected in a 4-milliliter (ml) glass vial with a Teflon®-lined cap. The surface was re-swabbed three times, with a final volume of 3 ml of acetone collected.

2.1.2.2 Building Sampling for Metals in Paint

The floors and baseboards of several buildings or building foundations in the Static Test Area were coated with red, conductive paint about 1/4 to 1/2 inch thick. It had been weathered and was cracked and crumbled in some places. The USATHAMA Project Officer supervised the collection of paint samples and specified the sample sites in Buildings 2750-1, 2750-3, 2750-4, 2750-7, and 2750-8.

Paint samples were collected by filling 50-cubic centimeter (cm^3) Nalgene® bottles with pieces of paint pried from the surface. Also, paint which had been ground by weathering to a coarse sand consistency

was collected by scooping by hand into the bottles. While sampling, the field team wore rubber gloves which were changed for every sample site.

2.1.2.3 Sewer Sampling for Nitrocellulose

The two manholes in the Static Test Area were sampled for nitrocellulose. Both were located under high brush about 4 to 5 feet high. The manhole walls were brick, with steel ladder steps to the concrete bottom. The tops of the manholes rose 1-1/2 feet above the ground and were about 3 feet in diameter. The walls of the manholes sloped 8 feet downward to a diameter of about 5 feet at the base. Both manholes were in disrepair, with the walls collapsing. The condition of the manholes dictated sampling from above rather than climbing down into the sewer.

Extenders were put on the soil auger and cores of sediment were lifted out of the manholes. Because of the concrete floor, 2-inch cores were taken in areas where sediment had collected. Samples were placed in 1-quart Mason® jars sealed with Teflon®-lined lids. One sample, composed of several cores, was taken from each of the two manholes in the Static Test Area. The sites were marked MH01 and MH02.

2.1.3 Fourteen Mile Creek; Lick Creek

Sediments in these streams were sampled to assess the contamination of tributaries in the proposed excess area.

The Lick Creek sample site (SE01), shown in Figure 1-4, was selected to measure levels of nitrocellulose in sediment potentially contaminated by areas adjacent to the proposed excess area. Four samples were taken with a scoop sampler at equally spaced points traversing the creek and were composited in a bucket. The samples were taken to a depth of 3 inches under 4 inches to 1 foot of water. The sediment sample was then transferred to a 1-quart Mason® jar with a Teflon®-lined lid.

Fourteen Mile Creek was sampled to measure contamination in runoff from the Burning Ground and the Static Test Area. Site SE02 (Figure 1-4) was

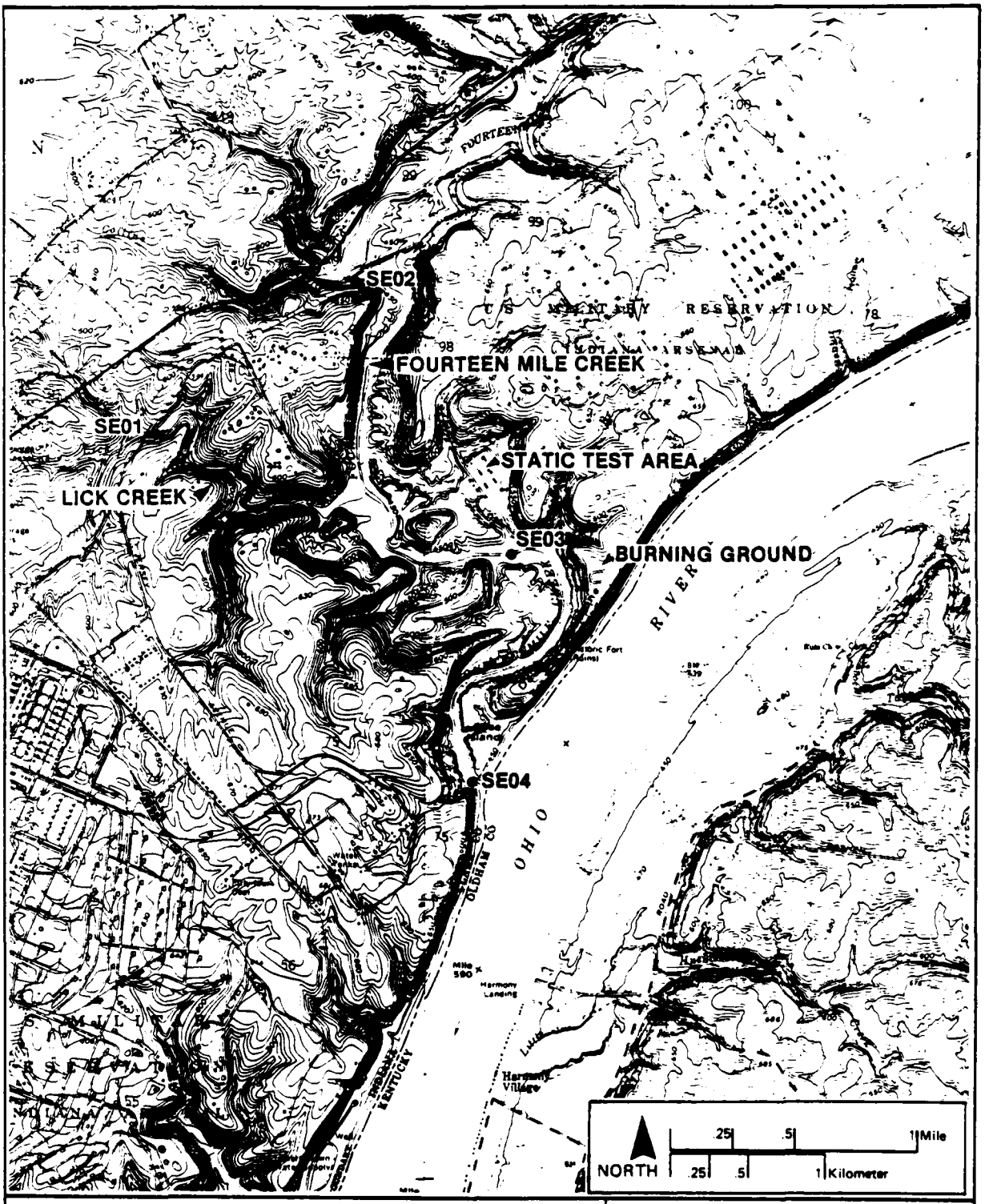

Figure 1-4
PROPOSED EXCESS AREA SEDIMENT SAMPLING SITES

SOURCES: USGS QUADRANGLE MAPS, 1963. ESE, 1981.

Limited Environmental Survey

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collected with a Ponar sampler from a bridge crossing the creek. Four sediment samples were taken under water depths of 1 to 8 feet, composited in a bucket, and transferred to the sample container.

Sites SE03 and SE04 were accessed by boat. The first site, SE03, received drainage from the Static Test Area. Four samples were taken with a Ponar sampler under water 2 to 12 feet deep. Site SE04 was at the point where Fourteen Mile Creek meets the Ohio River. Four samples were collected in the same manner as for Site SE03, under water from 1 to 10 feet deep. These sediments were each composited in a bucket and transferred to the sample container.

At each sediment site, a fluorescent orange marker was placed on the east side of the creek above the high water mark. The sample sites were selected and sampled under the direction of the USATHAMA Project Officer.

2.2 ANALYTICAL METHODOLOGY

2.2.1 Organonitrates in Soil and Sediments

Sample analysis was performed with quality control as dictated by the USATHAMA 1980 Quality Assurance Plan. For quantitative determinations of organonitrates in soil and metals in paints, background soil was analyzed unspiked and spiked at three levels with standard solutions of the analytical parameters. Semi-quantitative analyses included one blank and one spike at the detection limit of the method. Reagent blanks were run with every set of samples. Field duplicates, collected at a rate of 10 percent of the samples, served as checks on method reproducibility. Before analysis, soils were observed for Munsell color chart classifications. Soil and sediment samples were air-dried and sieved on a 30-mesh screen before analysis.

An extraction in acetone under basic conditions was used to isolate nitrocellulose from soil. Nitrite ion, released from nitrocellulose by the strong base, was measured as an indication of the presence of

nitrocellulose. This measure was made by diazotizing nitrite with sulfanilamine and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which was measured colorimetrically. This method (USATHAMA Number 1Q), with detection limit calculations required in the Statement of Work, is given in Appendix A.

2.2.2 Spot Test for Nitrocellulose and Nitroglycerin

The spot test was used as a field screening technique for determining nitrocellulose and nitroglycerin on building surfaces. A 400-cm^2 area of each building surface site was wiped thoroughly with a 2.4-cm glass-fiber filter held with metal tongs and soaked with acetone. Acetone was also applied to the test surface to keep the surface wet so that the filter absorbed any nitrocellulose and nitroglycerin (soluble in acetone) present.

The filter was then sprayed with methanolic potassium hydroxide, followed by the procaine, N,N-dimethyl-1-naphthylamine color reagent. The intensity of the color, which is only roughly proportional to the amount of explosives present, was noted. Details of the nitrocellulose/nitroglycerin field sampling procedure are given in Appendix A.

2.2.3 Thin-Layer Chromatography for Nitrocellulose and Nitroglycerin

Building surfaces that yielded positive spot test results were swabbed for confirmatory thin-layer chromatography (TLC) analysis. This technique provides rapid screening of up to 13 samples on a single TLC plate. A volume of sample was spotted on each available portion of the plate, which was developed with a 2:1:1 ratio of toluene, methanol, and ethylacetate. After drying, the plates were sprayed with methanolic-potassium hydroxide, then with Greiss reagent, followed by acetic acid. A pink and yellow spot with the R_f (distance traveled up the TLC plate in a specific developing solution) corresponding to nitrocellulose or nitroglycerin was recorded as a positive result. This method (USATHAMA Number 4E) is described in detail in Appendix A. Documentation data

required for certification of this method for nitroglycerin is also included in Appendix A.

2.2.4 Metals in Paint

Metals in paint samples were analyzed by atomic absorption using the methods for metals in soils (USATHAMA Numbers 1N and 2D) described in Appendix A. Digestion of the samples with nitric acid was vigorous. All samples required dilution for analysis within the range of the method.

2.2.5 Asbestos in Paint

A water and soil modification of the NIOSH procedure (NIOSH Publication 77-1699) for determining asbestos, formulated by Earl F. McFarren of the Water Supply Research Laboratory, National Environmental Research Center, Environmental Protection Agency (1976), was used for asbestos analysis. Paint samples were crushed and the fibrous material mounted on microscope slides with a dimethyl phthalate, diethyl oxalate, and cellulose ester filter mixture. Slides were observed by phase contrast microscopy.

Ashing tests were performed on the paint samples to further characterize the fibrous material found in the paint. Crushed paint samples were placed on stainless steel planchets and placed in a muffle furnace at 250°C for 12 hours. Another set of samples was treated in a similar manner in the muffle furnace at 550°C for 24 hours. After ashing, the samples were observed microscopically.

3.0 RESULTS

Field and chemical data were entered in USATHAMA format into the ESE data system. After computer quality control checks and careful review by an ESE Quality Assurance Supervisor, data were validated. Data have been submitted to USATHAMA on 9-track tape, in Data Level 1. The map file is included in Appendix B. A full printout of the chemical data, in ESE report format, is given in Appendix C.

3.1 NITROCELLUOSE AND NITROGYLCERIN

The soils of the Burning Ground area were classified according to the Munsell color chart. These data are shown in Table 3-1. All soils were varieties of clayey, sandy silt and ranged from light yellowish-brown to reddish-yellow in color.

No contamination was found in Burning Ground soils as a result of nitrocellulose analysis. Likewise, all four sediment and two sewer samples yielded negative results. The detection limit of the method was found to be 2.0 micrograms per gram (ug/g). A full report of the soil, sediment, and sewer chemical data is given in Appendix C.

Field testing of building surfaces in the Static Test Area for nitrocellulose and nitroglycerin yielded approximately 30 percent positive spot tests (Table 3-2). Confirmatory TLC analyses of swabs of these sites showed no nitroglycerin or nitrocellulose present.

3.2 METALS IN PAINT

The paints collected in the Static Test Area were analyzed for zinc, copper, lead, cadmium, chromium, and mercury. These analyses showed high levels of copper and zinc (see Table 3-3). Samples from Buildings 2750-3 and 2750-7 also had elevated chromium levels. The significance of these high levels will depend on the proposed use of this site. The stability of the complex state of the metals is unknown. Stable complexes would not release heavy metals into the environment, but would keep them bound in the paint matrix.

Table 3-1. Munsell Classification of Burning Ground Soils

Site	Description	Munsell Color Code
BG01	Red sandy silt with black burnt vegetable matter	2.5 YR 4/8
BG02	Reddish-yellow clayey, sandy silt; trace of roots	7.5 YR 6/8
BG03	Reddish-yellow clayey, sandy silt; trace of roots	7.5 YR 6/8
BG04	Yellow clayey silt	10 YR 7/6
BG05	Yellow clayey silt; trace of sand	10 YR 7/6
BG06	Brownish-yellow clayey silt	10 YR 6/6
BG07	Reddish-yellow clayey silt; few roots	7.5 YR 6/6
BG08	Reddish-yellow clayey, sandy silt	7.5 YR 6/6
BG09	Strong brown clayey, sandy silt	7.5 YR 5/8
BG10	Reddish-yellow clayey silt; trace of gravel	7.5 YR 7/6
BG11	Reddish-yellow clayey, sandy silt	7.5 YR 6/8
BG12	Reddish-yellow clayey, sandy silt	7.5 YR 6/8
BG13	Reddish-yellow clayey, sandy silt	7.5 YR 6/6
BG14	Very pale brown clayey silt; trace of roots	10 YR 7/4
BG15	Yellowish-brown clayey, sandy silt	10 YR 5/6
BG16	Brownish-yellow clayey, sandy silt	10 YR 6/6
BG17	Yellow clayey silt with some angular, flinty gravel	10 YR 7/6
BG18	Light yellowish-brown clayey silt; few roots	10 YR 6/4
BG19	Very pale brown clayey silt	10 YR 7/4
BG20	Yellow clayey silt; trace of roots	10 YR 7/6
BG21	Yellow clayey silt	10 YR 7/8
BG22	Yellow clayey silt; trace of roots	10 YR 7/6
BG23	Yellow clayey silt	10 YR 7/6
BG24	Very pale brown clayey silt; trace of organic debris	10 YR 7/4
BG25	Light yellowish-brown clayey silt; some flinty gravel	10 YR 6/4
BG26	Yellow clayey silt; trace of fine gravel	10 YR 7/6
BG27	Yellow clayey silt	10 YR 7/6
BG28	Reddish-yellow clayey silt; trace of gravel	7.5 YR 6/6
BG29	Very pale brown clayey silt	10 YR 7/4
BG30	Very pale brown clayey silt; trace of roots	10 YR 7/3
BKG1	Very pale brown clayey silt	10 YR 7/4
BKG2	Very pale brown clayey, sandy silt; few stone chips	10 YR 7/4

Source: ESE, 1981.

Table 3-2. Results of Building Surface Spot Tests

Building	Positive	Negative
2750-2	8	2
2750-2A	0	5
2750-3	6	8
2750-6	2	18
TOTAL SPOT TESTS	16	33

Source: ESE, 1981.

Table 3-3. Metals in Paint Samples, Static Test Area

Site	Parameter--milligrams per kilogram (mg/kg)					
	Zinc	Copper	Lead	Cadmium	Chromium	Mercury
2750-1	378	28,800	68	3	33	<0.2
2750-1	397	49,800	<10	7	37	<0.2
2750-3	326	49,100	<10	3	26	<0.2
2750-3	567	58,300	41	4	45	<0.2
2750-3	414	55,200	26	5	50	<0.2
2750-3	478	60,900	<10	11	133	<0.2
2750-4	468	57,000	<10	5	52	<0.2
2750-7	549	54,600	23	5	723	<0.2
2750-7	419	54,100	43	5	43	<0.2
2750-8	834	48,000	97	6	78	<0.2
2750-8	644	40,400	160	7	38	<0.2

Source: ESE, 1981.

3.3 ASBESTOS IN PAINT

The fibers in paint were first observed in the field, where their white appearance in the red paint was striking. Their length ranged from less than 2 up to 5 millimeters.

Microscopic observation of paint samples showed large crystalline fibers with the 3:1 length-to-width ratio characteristic of asbestos. The fibers were considerably longer than 5 micrometers in length, a criterion for discrimination used for airborne asbestos.

After ashing the paint samples at 250°C, the paint and fibers remained intact. However, after 24 hours at 550°C, the paint was reduced to ash, but the fibers remained unaffected by the treatment. This extremely high heat resistance suggests that the fibers are asbestos. The composition of fibers believed to be asbestos can be confirmed and quantified by X-ray diffraction or electron microscopy techniques.

The fibers are firmly bound in the paint matrix and will only be released when the paint is disturbed, as evidenced by field observation of paint in various stages of cracking and crumbling due to weathering. The buildings which are painted are 2750-1, 2750-3, 2750-4, 2750-7, and 2750-8.

4.0 CONCLUSIONS

Results of analyses of soil, sediment, and sewer samples for nitrocellulose revealed that the Burning Ground Area, Lick Creek, Fourteen Mile Creek, and the sewers of the Static Test Area are uncontaminated. The method used to analyze for nitrocellulose, with a detection limit of 2.0 ug/g, yielded results that were more than adequate for classifying these sites as uncontaminated with nitrocellulose.

There were no positive results from confirmatory TLC nitrocellulose and nitroglycerin analyses. Based on these findings, the buildings of the Static Test Area are free of these contaminants.

The paint samples analyzed for heavy metals yielded high values for copper, zinc, and chromium. Considering that the paint is deteriorating by weathering, there is a high possibility for ground contamination. The paints are cracking and crumbling and can be washed from the buildings by storm runoff. Whether the heavy metals are released in the ground or remain bound in the paint matrix is unknown.

Qualitative screening of the paint samples showed evidence that the fibrous material in the paints was asbestos. Microscopic observation and high temperature resistance strongly suggest the presence of asbestos.

The limited environmental survey of INAAP conducted by ESE in July 1981 showed no nitrocellulose or nitroglycerin concentrations in any of the samples collected. The paints sampled in the Static Test Area showed significant levels of heavy metals and possibly asbestos.

The data reported herein satisfy the requirements of Battelle Delivery Order 0015 for the Scientific Services Program Excess Area Contamination Survey of INAAP.

APPENDIX A

ANALYTICAL METHODS

1. Organonitrates/Soil/Technicon (IQ)

15 MAY 1981

ORGANONITRATES/SOIL/TECHNICON (1Q)

APPLICATION;

METHOD USED FOR THE DETERMINATION OF NITROCELLULOSE (NC) IN SOIL SAMPLES.

A.TESTED CONCENTRATION RANGE; (UG/GRAM SOIL)
50 TO 1000 UG/G

B.SENSITIVITY; NORMALIZED RESPONSE (CHART UNITS TIMES DILUTION FACTOR) AT DETECTION LIMIT.
500UG NC - 82 CHART UNITS

C.DETECTION LIMIT; (UG/GRAM SOIL)
NC - 50 UG/G

D.INTERFERENCES; COLOR IN EXTRACT INTERFERED WITH ANALYSIS. COLOR BLANKS RUN BY REMOVING CHROMAGIN FROM COLOR DEVELOPING REAGENT. PHOSPHORIC ACID LEFT IN COLOR REAGENT WHILE SULFANILAMIDE AND N-1-NAPHTHYLETHYLENEDIAMINE DIHYDROCHLORIDE REAGENTS OMITTED FOR COLOR BLANK DETERMINATION. HIGH CONCENTRATIONS OF IRON, COPPER, AND OTHER METALS INTERFERE (EDTA IS ADDED TO ELIMINATE INTERFERENCE).

E.ANALYSIS RATE; AFTER INSTRUMENT CALIBRATION, ONE ANALYST CAN EXTRACT AND ANALYZE 8 SAMPLES IN AN 8 HOUR DAY. 24 HOUR EXTRACT HOLDING TIME.

CHEMISTRY;

(C12H14N6022)X CELLULOSE HEXANITRATE
CAS RN 9004-70-0
MELTING PT; 160-170C (IGNITES)

NITROCELLULOSE USED AS A COMPONENT OF ALL SINGLE-, DOUBLE-, AND TRIPLE-BASED PROPELLANTS. COMPOUND FORMULATED BY NITRATING CELLULOSE DERIVED FROM COTTON OR WOOD PULP. CHEMISTRY OF NITROCELLULOSE AND ITS TOXICITY TO AQUATIC ORGANISMS RECENTLY REVIEWED BY SULLIVAN ET.AL.,1978. MAMMALIAN TOXICITY REVIEWED BY GLENNON ET.AL.,1976. CHIEF CONSTITUENT OF GUN COTTON IS CELLULOSE HEXANITRATE AND PENTANITRATE. THESE NITROCELLULOSE FORMS ARE INSOLUBLE IN MOST COMMON SOLVENTS, EXCEPT ACETONE. NITROCELLULOSE RESISTANT TO BIOTRANSFORMATION AND THEREFORE PERSISTANT IN ENVIRONMENT. IN AQUATIC ENVIRONMENT, COMPOUND APPARENTLY NONTOXIC TO FISH AND INVERTEBRATES.

IN ACETONE SOLUTION, STRONG BASE (NAOH) RELEASES NITRITE ION FROM NITROCELLULOSE MOLECULES. NITRITE FORMED MEASURED AS INDICATION OF PRESENCE OF NITRO CELLULOSE. NITROCELLULOSE MEASURED AS NITRITE BY DIAZOTIZING WITH SULFANILAMIDE AND COUPLING WITH N-(1-NAPHTHYL)-ETHYLENEDIAMINE DIHYDROCHLORIDE TO FORM A HIGHLY COLORED AZO DYE WHICH IS MEASURED COLORIMETRICALLY.

APPARATUS;

A.INSTRUMENTATION;

TECHNICON AUTOANALYZER II USING A NITRITE CARTRIDGE

B.PARAMETERS;

TECHNICON AUTOANALYZER SET UP IN ACCORDANCE WITH SPECIFICATIONS IN METHOD 353.2 (AAII) DESCRIBED IN METHODS OF CHEMICAL ANALYSIS OF WATER AND WASTES (EPA, 1979). SPECIFIC PROCEDURE FOR NITRITE (NO_2^-) ION USED.

STANDARD CALIBRATION KNOB OF AUTO-ANALYZER COLORIMETER SET AT 1. (A 0.5 MG/L AS N NITRITE STANDARD SHOULD READ APPROX 80 +-5 CHART UNITS)

USE A 40/HR SAMPLE RATE, 4 TO 1 CAM, AND A COMMON WASH

C.HARDWARE/GLASSWARE;

CENTRIFUGE CAPABLE OF HANDLING 50ML SCREW CAP TUBES AND 3000RPM SPEEDS

50ML GLASS CENTRIFUGE TUBES W TEFLON-LINED SCREW CAPS

520NM COLORIMETER FILTER

AUTOANALYZER SAMPLE CUPS (DISPOSABLE)

ASSORTED CLASS A VOLUMETRIC FLASKS AND PIPETS

D.CHEMICALS;

METHANOL, NANOGRADE DISTILLED IN GLASS

ACETONE, NANOGRADE

1.0 N NAOH REAGENT

NITROGEN OR HELIUM GAS, HIGH PURITY

COPPER-CADMIUM REAGENT - CADMIUM GRANULES (40 TO 60 MESH) ARE CLEANED WITH DILUTE HCL, RINSED WITH DISTILLED WATER AND COPPERIZED WITH 2% COPPER SULFATE SOLUTION BY ADDING 10G OF CD TO 100ML PORTIONS OF 2% COPPER SULFATE SOLUTION FOR 5 MIN, DECANT AND REPEAT WITH FRESH COPPER SULFATE UNTIL A BROWN COLLOIDAL PRECIPITATE FORMS. WASH WITH DISTILLED WATER FOR AT LEAST 10 TIMES TO REMOVE ALL PRECIPITATED COPPPER.

COLOR REAGENT - TO APPROX 800ML OF DISTILLED WATER, ADD, WHILE STIRRING, 100ML CONC PHOSPHORIC ACID, 40G SULFANILAMIDE, AND 2G OF N-1-NAPHTHYLETHYLENEDIAMINE DIHYDROCHLORIDE. STIR UNTIL DISSOLVED AND DILUTED TO 1L. SOLUTION STABLE FOR SEVERAL MONTHS.

HYDROCHLORIC ACID, 6N

COPPER SULFATE, 2% - DISSOLVE 20G OF $CUSO_4$-5 H_2O IN 500ML OF DISTILLED WATER AND DILUTE TO 1L.

WASH SOLUTION - (UNPRESERVED SAMPLES) DISTILLED WATER. (PRESERVED SAMPLES) 2ML CONC H_2SO_4 PER LITER WASH WATER.

AMMONIUM CHLORIDE-EDTA SOLUTION - DISSOLVE 85G OF REAGENT GRADE AMMONIUM CHLORIDE AND 0.1G OF DISODIUM ETHYLENEDIAMINE TETRACETATE IN 900ML OF DISTILLED WATER. ADJUST PH TO 8.5 WITH CONC AMMONIUM HYDROXIDE AND DILUTE TO 1L. ADD 0.5ML OF BRIJ-35 (TECHNICON CORP).

POTASSIUM NITRITE

NITRITE-FREE DEIONIZED WATER

NITROCELLULOSE STANDARD, PROVISIONAL SARM OBTAINED FROM PICATINY ARSENAL, LOT 3129

STANDARDS;

A.CALIBRATION STANDARDS;

PREPARE CALIBRATION STANDARD STOCK BY DISSOLVING 6.072G OF POTASSIUM NITRITE (KNO2) IN A FEW MILLILITERS OF DEIONIZED WATER IN A 1L VOLUMETRIC FLASK AND DILUTING TO VOLUME WITH DEIONIZED WATER.

PRESERVE CALIBRATION STANDARD STOCK WITH 2ML OF CHLOROFORM. SOLUTION STABLE FOR 6 MONTHS.

PREPARE DILUTE STOCK CALIBRATION STANDARD BY PIPETTING 1.0ML OF CALIBRATION STANDARD STOCK INTO A 100ML VOLUMETRIC FLASK AND DILUTING TO VOLUME WITH DEIONIZED WATER (1.0ML = 0.01MG NO2).

PREPARE WORKING CALIBRATION STANDARD BY PIPETTING 5.0ML OF DILUTE STOCK CALIBRATION STANDARD INTO A 100ML VOLUMETRIC FLASK AND DILUTING TO VOLUME WITH DEIONIZED WATER (1ML = 0.5UG NO2). PREPARE FRESH FOR EACH RUN.

B.CONTROL SPIKES;

PREPARE WORKING CONTROL SPIKE BY DISSOLVING 0.500G OF NITROCELLULOSE SARM IN A FEW MILLILITERS OF ACETONE IN A 100ML VOLUMETRIC FLASK AND DILUTING VOLUME. (IT MAY BE NECESSARY TO PLACE FLASK IN AN ULTRASONIC BATH FOR SEVERAL MINUTES TO DISSOLVE NITROCELLULOSE).

PIPET KNOWN AMOUNT OF WORKING CONTROL SPIKE INTO 10G STANDARD SOIL SAMPLES TO PROVIDE CONCENTRATION OF 0.5 TO 20 TIMES DETECTION LIMIT.

LET EACH SPIKE AIR-DRY FOR AT LEAST 1 HOUR.

PERFORM PROCEDURE.

DETERMINE PRECISION, ACCURACY, AND DETECTION LIMIT FOR NITROCELLULOSE IN STANDARD SOIL AS FOLLOWS;

PIPET OUL (BLANK),100UL,200UL,1.0ML,AND 2.0ML ALIQUOTS OF WORKING CONTROL SPIKE IN TRIPLICATE INTO 10G STANDARD SOIL SAMPLES.

PROCEDURE;

WEIGH 10G OF AIR-DRIED, SIEVED SOIL INTO A 50ML TEFLON-LINED SCREW CAPPED CENTRIFUGE TUBE.

ADD 30ML OF METHANOL TO TUBE, APPLY CAP, AND SHAKE VIGOROUSLY FOR 3 MINUTES.

CENTRIFUGE TUBE AT MEDIUM HIGH SPEED FOR 5 MINUTES OR UNTIL SOLIDS SETTLE COMPLETELY.

DRAW OFF AND DISCARD THE 30ML OF METHANOL.

REPEAT PRIOR 3 STEPS WITH ANOTHER 30ML PORTION OF METHANOL.

ADD 15ML OF ACETONE TO REMAINING SOLIDS IN TUBE, THEN CAP AND SHAKE TUBE FOR 3 MINUTES.

CENTRIFUGE TO SETTLE SOLIDS, THEN DRAW OFF ACETONE WITH A 20ML VOLUMETRIC PIPET AND TRANSFER TO A 50-ML CULTURE TUBE.

REPEAT ACETONE EXTRACTION 2 MORE TIMES, EACH TIME COMBINING ACETONE EXTRACT.

ADD 3ML OF 1.0 N NAOH SOLUTION TO EACH CULTURE TUBE, CAP, AND SHAKE THE TUBE TO MIX. EVAPORATE ACETONE UNDER A STREAM OF NITROGEN AND GENTLE HEATING ON A 30C WATER BATH.

UPON REMOVAL OF ACETONE, DILUTE AQUEOUS BASE SOLUTION TO 10ML WITH DEIONIZED WATER.

ANALYZE EXTRACT FOR NITRITE WITHIN 24 HOURS. KEEP SOLUTIONS COLD (4C) UNTIL ANALYZED.

PREPARATION OF REDUCTION COLUMN AAII; COLUMN IS A U-SHAPED, 35 CM LONG, 2 MM I.D. GLASS TUBE (OR 0.081 I.D. PUMP TUBE). FILL REDUCTION COLUMN WITH DISTILLED WATER TO PREVENT ENTRAPMENT OF AIR BUBBLES DURING FILLING. TRANSFER CU-CD GRANULES TO THE REDUCTION COLUMN AND PLACE A GLASS WOOL PLUG IN EACH END. TO PREVENT ENTRAPMENT OF AIR BUBBLES IN COLUMN, INSURE THAT ALL PUMP TUBES ARE FILLED WITH REAGENTS BEFORE PUTTING COLUMN INTO SYSTEM.

ADJUST PH TO BETWEEN 5 AND 9 WITH CONC HCL OR CONC NH4OH. ALLOW BOTH COLORIMETER AND RECORDER TO WARM UP FOR 30 MIN. OBTAIN STABLE BASELINE WITH ALL REAGENTS, FEEDING DISTILLED WATER THROUGH SAMPLE LINE.

PLACE STANDARDS IN SAMPLER IN ORDER OF DECREASING CONCENTRATION. COMPLETE LOADING OF TRAY WITH UNKNOWN SAMPLES. SWITCH SAMPLE LINE TO SAMPLER AND START ANALYSIS.

FOR INSTRUMENT CALIBRATION, SET STANDARD CALIBRA-

TION KNOB TO 1.0 AND COMPARE RESULTING CHART READING OF A 0.5MG/L NITRITE STANDARD TO HISTORICAL CHART READINGS (80+-5 UNITS).

FOR STANDARD WORKING CURVE, PIPET OUL (BLANK),100-UL,200UL,1.0ML,AND 2.0ML ALIQUOTS OF WORKING CONTROL SPIKE INTO SEPARATE 50ML CULTURE TUBES CONTAINING 45ML OF ACETONE (PREPARE FRESH FOR EACH RUN). PERFORM PROCEDURE, STARTING AT THE 1.0 N NAOH STEP.

CALCULATIONS;

PLOT RESPONSE (IN TERMS OF PEAK HEIGHT OF STANDARD) VERSUS STANDARD NITROCELLULOSE CONCENTRATION (CONCENTRATIONS OF STANDARDS EXPRESSED IN TERMS OF UG NITROCELLULOSE PER 10G OF SOIL). DETERMINE EQUATION FOR LEAST-SQUARES LINE FROM UNEXTRACTED STANDARDS (EQUIVALENT TO UG/G FOR 10G OF SOIL) VERSUS RESPONSE (CORRECTED FOR DILUTION OF EXTRACT). DETERMINE CONCENTRATION (CS) CORRESPONDING TO CORRECTED SAMPLE RESPONSE FROM STANDARD CURVE. CORRECT SAMPLE DATA FOR PERCENT MOISTURE.

REFERENCES;

GLENNON,DACRE,PEARSON,WARNER,BARKLEY,AND ROSENBLATT, 1977. MUNITIONS ENVIRONMENTAL QUALITY STANDARDS RESEARCH REPORT. PREPARED FOR U S ARMY MEDICAL RESEARCH AND DEVELOPMENT COMMAND.

SULLIVAN,PUTNAM,KEIRN,PRUITT,AND NICHOLS, 1978. A SUMMARY AND EVALUATION OF AQUATIC ENVIRONMENTAL DATA IN RELATION TO ESTABLISHING WATER QUALITY CRITERIA FOR MUNITIONS UNIQUE COMPOUNDS - NITROCELLULOSE; FINAL REPORT. PREPARED FOR U S ARMY MEDICAL RESEARCH AND DEVELOPMENT COMMAND, CONTRACT DAMD 17-77-C-7027. WATER AND AIR RESEARCH, INC, GAINESVILLE, FLORIDA.

U S ENVIRONMENTAL PROTECTION AGENCY, 1979. METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES, METHOD 353.2, EPA-600/4-79-020.

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STORET= 99574 METHOD=  1  QUAL DET. LMT=        2.5000
         CORR. COEFF.= 0.9984 SLOPE(FOUND/TARGET)=        1.6356 INTCPT.=      0.6209 USATHAMA ABBR.=NC
         UNITS=PPM  INSTR # =11 TVALUE=      4.7145 CALIBR. SLOPE STD. DEV=      0.7462
         NITROCELLULOSE,SOIL(UG/G)
          SXY =         0.18174  STD DEV OF ACC. SLOPES        0.06882
          # OF SLOPES=  6 AVG SLOPE=       12.23721 STD DEV SLOPE=        0.74623
          # OF DET. LIMITS=  6 AVG. DET. LIMIT=        0.35270 STD DEV. OF DET. LIMIT=        0.33089
          # OF 2X RESP.=  6 AVG 2X RESP.=        0.42717 STD DEV. OF 2X RESP.=        0.02239

         **** TARGET VERSUS FOUND RESULTS ****
                0.0000 =TARGET       -0.2900 =FOUND        0.0000 =TARGET       -0.3000 =FOUND
                2.5000 =TARGET        1.0500 =FOUND        2.5000 =TARGET        1.0500 =FOUND
                5.0000 =TARGET        2.6100 =FOUND        5.0000 =TARGET        2.5100 =FOUND
                7.5000 =TARGET        4.0500 =FOUND        7.5000 =TARGET        4.5700 =FOUND
               10.0000 =TARGET        5.9300 =FOUND       10.0000 =TARGET        5.6300 =FOUND
               15.0000 =TARGET        8.5500 =FOUND       15.0000 =TARGET        8.9700 =FOUND
                5.0000 =TARGET        2.7032 =FOUND

         **** SLOPES OF CALIBRATION CURVES ****
           12.9260 = SLOPE       12.4750 = SLOPE       11.3110 = SLOPE       12.4679 = SLOPE
      11.3107 = SLOPE       12.9327 = SLOPE
OK,
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1

NITROCELLULOSE,SOIL(UG/G)

SPIKED CONCENTRATION	FOUND CONCENTRATION	PERCENT INACCURACY
0.00000	-0.29000	0.00000
0.00000	-0.30000	-116.00002
2.50	1.05	-97.60002
2.50	1.05	-85.06670
5.00	2.61	-84.40002
5.00	2.51	-83.20001

2. Nitrocellulose in Building Swabs (4E)

NITROCELLULOSE IN BUILDING SWABS

1. APPLICATION

This method is applicable to the qualitative determination of nitrocellulose (NC) in acetone swab samples of building surfaces using thin-layer chromatography (TLC).

A. TESTED CONCENTRATION RANGE

The tested concentration range is 0.63 to 10.1 microgram (ug) NC per square centimeter (cm^2) of surface area swabbed.

B. DETECTION LIMIT

The detection limit for NC on various surfaces is as follows:

Surface	Detection Limit (ug/cm^2)
Concrete	5.04
Metal	1.26
Wood	9.45
Transite	2.52

These detection limits were determined by the rank-sum tests and correspond to the smallest quantities of NC that give a colored spot that can be reliably visually detected on the TLC plate after development. This visual detection limit has been determined to be 2.5 ug of NC spotted on the TLC plate.

C. INTERFERENCES

The method is not subject to interferences from nitrate or nitrite ions since these substances do not elute with the solvents used and give a very pale pink color on development with the indicating reagent.

D. ANALYSIS RATE

One analyst can analyze aproximately 100 extracts in an 8-hour day.

2. APPARATUS

A. HARDWARE/GLASSWARE

	Manufacturer
Cotton-tipped Applicators--6 inch	Peerless, Diamond International
TLC Glass Developing Tanks for 20x20 cm plates	Scientific Products
Dessicator	Scientific Products
Analytical Balance	Metler
Pipets, 25 ul and 200 ul	Dade
Pipet Syringe, 1 ml	Scientific Products
TLC Plates--Precoated 19 Channels-LK5DF	Whatman
Sample Vials (4 ml) calibrated at 3 ml level	Scientific Products
Drying Oven, 100°C Setting	Scientific Products
Silicone Vacuum Grease	Dow Corning
Filter Paper Sheets (#1)	Whatman
Chromist TLC Sprayer	Scientific Products
Graduated Cylinder, 100 ml	Corning
Volumetric Flask, 10 ml	Corning

B. CHEMICALS

Ethyl Acetate
Toluene
Methanol
Acetone
Potassium Hydroxide
Sulfanilic Acid
alpha-Naphthylamine
Acetic Acid

3. STANDARDS

A. CALIBRATION STANDARDS

1. A standard analytical reference material (SARM) for nitrocellulose was supplied by USATHAMA. This material is 13.45-percent nitrated cellulose Lot No. C3129, ID No. PA276.

2. The stock calibration standard was prepared by weighing 50.4 milligrams (mg) of NC into a 10-milliliter (ml) volumetric flask, dissolving the NC in a few milliliters of acetone, and diluting to volume with acetone. The NC standard required sonication to facilitate dissolution.

3. A working calibration standard was prepared by pipetting 0.2 ml of the stock calibration standard into a 10-ml volumetric flask and diluting to volume with acetone. The concentration of the working calibration standard is 0.101 ug/ul.

4. Twenty-five ul of the working calibration standard solution was spotted on one of the channels of the TLC plate and developed along with 25-ul sample spots in adjacent channels.

5. This technique allows for side-by-side comparison of the sample spots with the standard spotted at the equivalent of the visual detection limit with 2.5 ug of NC.

B. CONTROL SPIKES

1. Natural Sample Spiking--Rank-Sum Test

To determine the detection limit of the method on various natural surfaces, the rank-sum procedure was performed. The

rank-sum procedure consists of spiking various surfaces in triplicate with various levels of NC. The surfaces are wiped and the swabs analyzed for NC according to the procedure outlined in Section 4.0. The same surfaces are also wiped in triplicate without prior spiking with NC to provide three blank values. If the three blanks yield negative results for NC on a particular surface, then that spiked concentration which produces three positive values of NC (greater than the visual TLC detection limit) is determined to be the detection limit of the method on that surface.

The surfaces spiked and the spiking levels are as follows:
Spiking solution--Stock Calibration Standard (5.04 ug/ul)

Surface	Volume Spiked (ul)	Concentration Spiked (ug/cm^2)
Unpainted Wood	100	1.26
	200	3.78
	400	6.30
	750	9.45
	800	10.08
Concrete	50	0.63
	100	1.26
	200	2.52
	400	5.04
	500	6.30
	700	8.82
Steel (ungalvanized)	50	0.63
	100	1.26
Transite	100	1.26
	200	2.52
	300	3.78
	400	5.04

The spiking aliquot was distributed uniformly in small increments over the entire surface area to be wiped.

2. Laboratory Spiking

In addition to the natural sample spiking according to the rank-sum procedure, various other surfaces were spiked and analyzed in the laboratory. Various aliquots of the stock calibration standard were spiked on glass, metal, varnished wood, painted wood, and concrete.

4. PROCEDURE

A. SURFACE SWABBING TECHNIQUE

1. Mark off an area of 20 cm by 20 cm on the surface to be wiped. Alternatively, on irregular surfaces, mark off an area of approximately 400 cm^2.

2. Soak the cotton tip of a cotton-tipped applicator thoroughly with acetone from a squeeze bottle.

3. Holding the applicator by the wooden handle, wipe the surface to be tested with the acetone-saturated cotton tip using a uniform circular motion to cover the entire marked-off surface.

4. Roll the applicator stick between the thumb and fingers while performing the circular wiping motion to prevent distortion of the cotton tip on rough surfaces.

5. After the surface has been wiped once, hold the applicator with the cotton-tipped end in the mouth of a 4-ml glass vial and rinse thoroughly with approximately 1 ml of acetone from a squeeze bottle.

6. Remove the applicator from the vial, and repeat the wiping of the surface as described in Steps 3 and 4.

7. Rinse the cotton-tipped end of the applicator again with approximately 1 ml of acetone into the same 4-ml glass vial.

8. Repeat the wiping of the surface and rinsing of the cotton tips once more.

9. After the third wiping operation, break off the cotton tip of the applicator, and place it in the 4-ml vial containing the acetone rinses.

10. Adjust the volume of acetone in the glass vial to the precalibrated 3-ml mark, and seal the vial.

B. CHROMATOGRAPHY

1. Prepare the developing solvent by mixing together 500 ml of toluene, 250 ml of methanol, and 250 ml of ethylacetate in a 1-liter flask.

2. Soak several sheets of Whatman #1 filter paper with the developing solvent, and completely line the sides of the developing tank with the saturated paper.

3. Add the developing solvent to the tank. The amount of solvent added should wet no more than the bottom 3 millimeters (mm) of the pre-adsorbent area of a TLC plate when the plate is placed in the tank.

4. Shake the glass vial containing the sample acetone rinses and cotton swab tip vigorously for 1 minute.

5. Spot 25 ul of the swab solution onto the pre-adsorbent area of a single channel on the TLC plate. The sample should not be spotted in the region 5 mm from the bottom of the plate or 5 mm from the silica gel/pre-adsorbent area interface.

6. Thoroughly air-dry the TLC plate.

7. Onto 4 other channels of the TLC plate, spot 25 ul of the working calibration standard solution, which is equivalent to the visual detection limit of 2.5 ug of NC. Air-dry the plate.

8. Place the TLC plate into the developing tank, place the cover on the tank, and begin development.

9. Continue development until the solvent front has travelled about 12 cm above the pre-adsorbent area.

10. Remove the TLC plate and thoroughly air-dry or dry with a gentle stream of hot air from a heat-gun.

C. VISUALIZATION TECHNIQUE

1. Prepare the spray reagents as follows:
   Methanolic-KOH--Dissolve 8.5 grams of potassium hydroxide (KOH) in 100 ml of methanol in a volumetric flask.
   Greiss-reagent-Prepare a mixture of equal volumes of 0.5-percent [weight/volume (W/V)] sulfanilic acid in water and 0.5-percent (W/V) alpha-naphthylamine in methanol. Refrigerate and prepare fresh weekly.

2. Spray the dried plate with the methanolic-KOH solution. Place the plate in the oven at 100°C for 30 minutes.

3. Remove the plate from the oven and let cool to room temperature.

4. Spray the plate with Greiss reagent followed by a solution of 30-percent acetic acid in water.

5. Several other munitions compounds containing nitrate esters react with the reagents used, and characteristic colors of the reaction products are described below:

Procedure	Compound	Color of Spot
Spray with Methanolic KOH	TNT	Orange-Brown
	TETRYL	Orange
Heat at 100°C for 30 min	TNT	Dark Brown
	TETRYL	Yellow
Spray with Greiss reagent followed by 30-percent acetic acid	Nitroglycerine	Pink & Yellow
	PETN	Pink & Yellow
	Nitrocellulose	Pink & yellow
	RDX	Pink

6. The presence of nitrocellulose is indicated by the Greiss reagent color test and the R_f which is 0.98 for the elution solvent used.

7. Qualitative identification of nitrocellulose in a sample is based on comparison of the color and R_f of NC standard spots with those of the sample.

5. CALCULATIONS

The amount of NC present in the sample is determined by comparison of the intensity of the sample spot with that of the standard. The amount of NC is reported as either less than or greater than the visual detection limit.

The amount of NC on the surface area tested which yields a concentration equivalent to the visual detection limit on the TLC plate assuming 100-percent recovery from the surface wiped can be calculated to be:

$$\text{surface conc. of NC } (\mu g/cm^2) = \frac{(2.5\ \mu g)(3\ ml)\ (1{,}000\ \mu l/ml}{(25\ \mu l)\ (400 cm^2)}$$

$$= 0.75\ \mu g/cm^2$$

6. REFERENCES

USATHAMA-supplied TLC procedures.

7. DATA

The photograph below shows the results of the spiking experiments and gives an indication of the intensity of the NC spots at the detection limit.

The results of the rank-sum test are given in Table 7-1.

Table A-1. Results of Rank-Sum Test for NC on Various Natural Surfaces

Surface	Blank Samples*			Spiked Samples*			Detection Limit (ug/cm^2)
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	
Concrete	--	--	--	+	+	+	5.04
Steel (ungalvanized)	--	--	--	+	+	+	1.26
Wood (unpainted)	--	--	--	+	+	+	9.45
Transite	--	--	--	+	+	+	2.52

* Results are reported as follows:
-- indicates negative response to NC.
+ indicates positive response to NC greater than visual TLC detection limit.

Source: ESE, 1981.

3. Nitroglycerin in Building Swabs

NITROGLYCERIN IN BUILDING SWABS

1. APPLICATION

This method is applicable to the qualitative determination of nitroglycerin (NG) in acetone swab samples of building surfaces using thin-layer chromatography (TLC).

A. TESTED CONCENTRATION RANGE

The tested concentration range for nitroglycerin is 0.025 to 20 micrograms per square centimeter (ug/cm^2) of surface area swabbed.

B. DETECTION LIMIT

The detection limit for NG on various surfaces is as follows:

Surface	NG Detection Limit (ug/cm^2)
Concrete	15.0
Metal	0.75
Wood	4.25
Transite	0.75

These detection limits were determined by the rank-sum tests and correspond to the smallest quantities of NG that give a colored spot that can be reliably visually detected on the TLC plate after development. This visual detection limit has been determined to be 1.0 ug of NG spotted on the TLC plate.

C. INTERFERENCES

The method is not subject to interferences from nitrate or nitrite ions since these substances do not elute with the solvents used and give a very pale pink color on development with the indicating reagent.

D. ANALYSIS RATE

One analyst can analyze aproximately 100 extracts in an 8-hour day.

2. APPARATUS

A. HARDWARE/GLASSWARE

	Manufacturer
Cotton-tipped Applicators--6 inch	Peerless, Diamond International
TLC Glass Developing Tanks for 20x20 cm plates	Scientific Products
Dessicator	Scientific Products
Analytical Balance	Metler
Pipets, 25 ul and 200 ul	Dade
Pipet Syringe, 1 ml	Scientific Products
TLC Plates--Precoated 19 Channels-LK5DF	Whatman
Sample Vials (4 ml) calibrated at 3 ml level	Scientific Products
Drying Oven, 100°C Setting	Scientific Products
Silicone Vacuum Grease	Dow Corning
Filter Paper Sheets (#1)	Whatman
Chromist TLC Sprayer	Scientific Products
Graduated Cylinder, 100 ml	Corning
Volumetric Flask, 10 ml	Corning

B. CHEMICALS

Ethyl Acetate

Toluene

Methanol

Acetone

Potassium Hydroxide

Sulfanilic Acid

alpha-Naphthylamine

Acetic Acid

Nitroglycerin--Standard Analytical Reference Material

3. STANDARDS

A. CALIBRATION STANDARDS

1. A standard analytical reference material (SARM) for nitroglycerin was supplied by USATHAMA. The SARM was obtained as 200 mg of nitroglycerin dissolved in acetone.

2. The stock calibration standard was prepared by quantitatively transferring the entire contents of the SARM vial (200 mg NG) into a 100-milliliter (ml) volumetric flask and diluting to volume with acetone. The NG concentration of this solution is 2,000 ug/ml NG.

3. An intermediate calibration standard was prepared by pipetting 10 ml of the stock calibration standard into a 100-ml volumetric flask and diluting to volume with acetone. The concentration of the intermediate calibration standard is 200 ug/ml.

4. A working calibration standard was prepared by pipetting 20 ml of the intermediate calibration standard into a 100-ml volumetric flask and diluting to volume with acetone. The concentration of the working calibration standard is 40 ug/ml.

5. Twenty-five microliters (ul) of the working calibration standard solution was spotted on one of the channels of the TLC plate and developed along with 25-ul sample spots in adjacent channels.

6. This technique allows for side-by-side comparison of the sample spots with the standard spotted at the equivalent of the visual detection limit with 1.0 ug of NG.

B. CONTROL SPIKES

Natural Sample Spiking--Rank-Sum Test

To determine the detection limit of the method on various natural surfaces, the rank-sum procedure was performed. The rank-sum procedure consists of spiking various surfaces in triplicate with various levels of NG. The surfaces are wiped and the swabs analyzed for NG according to the procedure outlined in Section 4.0. The same surfaces are also wiped in triplicate without prior spiking with NG to provide three blank values. If the three blanks yield negative results for NG on a particular surface, then that spiked concentration which produces three positive values of NG (greater than the visual TLC detection limit) is determined to be the detection limit of the method on that surface.

The surfaces spiked and the spiking levels are as follows:
Spiking solution A--Stock Calibration Standard (2.0 ug/ul)
B--Intermediate Calibration Standard (0.2 ug/ul)

Surface	Spiking Solution	Volume Spiked (ul)	Concentration Spiked (ug/cm^2)
Unpainted Wood	B	250	0.13
	B	500	0.25
	A	100	0.50
	A	300	1.5
	A	500	2.5
	A	750	3.75
	A	850	4.25
Concrete	B	100	0.05
	B	500	0.25
	A	500	2.5
	A	1,000	5.0
	A	2,000	10.0
	A	2,500	12.5
	A	3,000	15.0

Surface	Spiking Solution	Volume Spiked (ul)	Concentration Spiked (ug/cm^2)
Steel	B	50	0.04
	B	100	0.08
	B	250	0.20
	B	500	0.40
	A	100	0.50
	A	150	0.75
Transite	B	50	0.04
	B	100	0.08
	B	250	0.20
	B	500	0.40
	A	100	0.50
	A	150	0.75
	A	300	1.5

The spiking aliquot was distributed uniformly in small increments over the entire surface area to be wiped.

4. PROCEDURE

A. SURFACE SWABBING TECHNIQUE

1. Mark off an area of 20 cm by 20 cm on the surface to be wiped. Alternatively, on irregular surfaces, mark off an area of approximately 400 cm^2.

2. Soak the cotton tip of a cotton-tipped applicator thoroughly with acetone from a squeeze bottle.

3. Holding the applicator by the wooden handle, wipe the surface to be tested with the acetone-saturated cotton tip using a uniform circular motion to cover the entire marked-off surface.

4. Roll the applicator stick between the thumb and fingers while performing the circular wiping motion to prevent distortion of the cotton tip on rough surfaces.

5. After the surface has been wiped once, hold the applicator with the cotton-tipped end in the mouth of a 4-ml glass vial and rinse thoroughly with approximately 1 ml of acetone from a squeeze bottle.

6. Remove the applicator from the vial, and repeat the wiping of the surface as described in Steps 3 and 4.

7. Rinse the cotton-tipped end of the applicator again with approximately 1 ml of acetone into the same 4-ml glass vial.

8. Repeat the wiping of the surface and rinsing of the cotton tips once more.

9. After the third wiping operation, break off the cotton tip of the applicator, and place it in the 4-ml vial containing the acetone rinses.

10. Adjust the volume of acetone in the glass vial to the precalibrated 3-ml mark, and seal the vial.

B. CHROMATOGRAPHY

1. Prepare the developing solvent by mixing together 500 ml of toluene, 250 ml of methanol, and 250 ml of ethylacetate in a 1-liter flask.

2. Soak several sheets of Whatman #1 filter paper with the developing solvent, and completely line the sides of the developing tank with the saturated paper.

3. Add the developing solvent to the tank. The amount of solvent added should wet no more than the bottom 3 millimeters (mm) of the pre-adsorbent area of a TLC plate when the plate is placed in the tank.

4. Shake the glass vial containing the sample acetone rinses and cotton swab tip vigorously for 1 minute.

5. Spot 25 ul of the swab solution onto the pre-adsorbent area of a single channel on the TLC plate. The sample should not be spotted in the region 5 mm from the bottom of the plate or 5 mm from the silica gel/pre-adsorbent area interface.

6. Thoroughly air-dry the TLC plate.

7. Onto 4 other channels of the TLC plate, spot 25 ul of the working calibration standard solution, which is equivalent to the visual detection limit of 1.0 ug of NG. Air-dry the plate.

8. Place the TLC plate into the developing tank, place the cover on the tank, and begin development.

9. Continue development until the solvent front has travelled about 12 cm above the pre-adsorbent area.

10. Remove the TLC plate and thoroughly air-dry or dry with a gentle stream of hot air from a heat-gun.

C. VISUALIZATION TECHNIQUE

1. Prepare the spray reagents as follows:
   Methanolic-KOH--Dissolve 8.5 grams of potassium hydroxide (KOH) in 100 ml of methanol in a volumetric flask.
   Greiss-reagent-Prepare a mixture of equal volumes of 0.5-percent [weight/volume (W/V)] sulfanilic acid in water and 0.5-percent (W/V) alpha-naphthylamine in methanol. Refrigerate and prepare fresh weekly.

2. Spray the dried plate with the methanolic-KOH solution. Place the plate in the oven at 100°C for 30 minutes.

3. Remove the plate from the oven and let cool to room temperature.

4. Spray the plate with Greiss reagent followed by a solution of 30-percent acetic acid in water.

5. Several other munitions compounds containing nitrate esters react with the reagents used, and characteristic colors of the reaction products are described below:

Procedure	Compound	Color of Spot
Spray with Methanolic KOH	TNT	Orange-Brown
	TETRYL	Orange
Heat at 100°C for 30 min	TNT	Dark Brown
	TETRYL	Yellow
Spray with Greiss reagent followed by 30-percent acetic acid	Nitroglycerine	Pink & Yellow
	PETN	Pink & Yellow
	Nitrocellulose	Pink & yellow
	RDX	Pink

6. The presence of NG is indicated by the Greiss reagent color test and the R_f which is 0.85 for the elution solvent used.

7. Qualitative identification of NG in a sample is based on comparison of the color and R_f of NG standard spots with those of the sample.

5. CALCULATIONS

The amount of NG present in the sample is determined by comparison of the intensity of the sample spot with that of the standard. The amount of NG is reported as either less than or greater than the visual detection limit.

The amount of NG on the surface area tested which yields a concentration equivalent to the visual detection limit on the TLC plate assuming 100-percent recovery from the surface wiped can be calculated to be:

$$\text{surface conc. of NG (ug/cm2)} = \frac{(1.0 \text{ ug})(3 \text{ ml})\ (1{,}000 \text{ ul/ml}}{(25 \text{ ul})\ (400\text{cm}^2)}$$

$$= 0.3 \text{ ug/cm}^2$$

6. REFERENCES

USATHAMA-supplied TLC procedures.

The results of the rank-sum test are given in Table A-2.

Table A-2. Results of Rank-Sum Test for NG on Various Natural Surfaces

Surface	Blank Samples*			Spiked Samples*			Detection Limit (ug/cm^2)
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	
Concrete	--	--	--	+	+	+	15.0
Steel (ungalvanized)	--	--	--	+	+	+	0.75
Wood (unpainted)	--	--	--	+	+	+	4.25
Transite	--	--	--	+	+	+	0.75

* Results are reported as follows:
-- indicates negative response to NG.
+ indicates positive response to NG greater than visual TLC detection limit.

Source: ESE, 1981.

4. Nitroglycerin and Nitrocellulose on Surfaces

NITROGYLCERIN (NG) AND NITROCELLULOSE (NC) ON SURFACES

I. Application: This method is applicable for the qualitative determination of nitroglycerin and nitrocellulose on surfaces.

A. Tested Concentration Range - 0.4 to 40 micrograms per square centimeter.

B. Sensitivity - N/A.

C. Detection Limits - 0.4 micrograms per square centimeter determined by testing the above concentration range for visible color response.

D. Interferences - Other nitrate esters will give a positive response. The color of the surface can affect ability to distinguish the color.

E. Analysis Rate - Approximately 2 minutes are required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

II. Chemistry

A. Alternate Nomenclautre and Chemical Abstracts Registry Number - Nitroglycerin: NB; Trinitroglycerin; Glyceryl trinitrate CAS RN-55-63-0 Nitrocellulose: NC; Cellulose nitrate Pyroxylin; colloidon cotton; soluble gun cotton. CAS RN-9004-70-0

B. Physical and Chemical Properties - NG: mp = 13°C, decomposes at 145°C NC: decomposes on heating. Hazards: Explosive hazard, avoid heat, shock, or open flame. Toxic inhalation and skin absorption hazards exist.

C. Chemical Reactions - Nitrite ion is cleaved in basic solution which diazotizes procaine, in acidic solution, which in turn couples with N,N-dimethyl-1-naphthylamine to produce an azo-dye. This presence or absence of color is decided by visual inspection.

III. Apparatus

A. Instrumentation - N/A

B. Parameters - N/A

C. Hardware/Glassware

1. cotton-tipped swabs
2. 1 dram screw cap vials
3. volumetric flasks - 100 ml, 10 ml
4. pipettes - 50 ml, 1 ml, 10 µl, and 5 ml graduated
5. 100-ml graduated cyclinder
6. 1-cm^2 confined area spottest paper
7. Sprayers and reservoir bottles

D. Chemicals

1. KOH, Analytical Reagent grade
2. ethanol, ACS grade
3. glacial acetic acid, ACS grade
4. procaine
5. N,N-dimethyl-1-naphthylamine

E. Reagents

1. Reagent A (5% ethanolic KOH) - Weigh 5 g of KOH into the sprayer reservoir. Add 100 ml of ethanol, measured using the graduated cylinder. Swirl to dissolve KOH.

2. 50% acetic acid - water (nitrate free). Dilute to volume with deionized water.

3. Reagent B - Weigh 0.35 grams each of procaine and N,N-dimethyl-1-naphthylamine into a second sprayer reservoir. Add the 100 ml of 50% acetic acid. Cap and shake until the reagents are dissolved.

IV. Standards

A. Calibration Standards

1. A 4 g/l solution of NC is prepared by weighing 40 mg of NC in a 10 ml volumetric flask and diluting to the mark with acetone. When 10 µl are spotted on confined area spottest paper, this concentration corresponds to 40 µg/cm^2.

2. A 0.4 g/l solution of NC is prepared by pipetting 1 ml of the 4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10 µl are spotted on confined area spottest paper, this concentration corresponds to 4 µg/cm^2.

3. A 40 mg/l solution of NC is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10 µl are spotted on confined area spottest paper, this concentration corresponds to 4 µg/cm^2.

4. The NG standards having 4 g/l, 0.4 g/l, and 40 mg/l are prepared in a way analogous to Steps 1, 2, and 3. The 4 g/l solution of NG is prepared by pipetting 4 ml of a 1% NG in acetone SARM into a 10 ml volumetric flask and diluting to the mark with acetone.

5. A 100 mg/l solution of NC is prepared by pipetting 2.5 ml of the 0.4 g/l solution into a 10 ml volumetric flask, using the 5 ml graduated pipette, and diluting to the mark with acetone. This solution is used routinely for a procedure check.

B. Control Spikes - N/A.

V. Procedure

A. Testing the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of NC.

2. Withdraw the swab and allow it to air dry.

3. Spray the swab with Reagent A and allow it to dry.

4. Spray the swab with Reagent B.

5. A positive test is indicated by a violet or red violet color and indicates that the spray solution is adequate for testing.

B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately 6 inches from the surface, apply Reagent A for approximately one second. Allow the alcohol to evaporate, then spray with Reagent B in the same manner and in the same location as Reagent A. A postive test is indicated by a violet or red violet color against a pale pink background.

VI. Calculations - N/A.

VII. References - NAVEODFAC Technical Report TR-185 Development of a Simple Portable Detection Kit for Selected Explosives, Robert E. Wyant, Battelle Columbus Laboratories, September 1977.

Supporting Data - Using confined spot test paper.

Spike Level ($\mu g/cm^2$)	Response
0	-
0.4	+
4	+
40	+

5. Metals/Soils/AA (IN)

METALS/SOIL/AA (1N)

APPLICATION;

METHOD APPLICABLE FOR ANALYSIS OF THE FOLLOWING COMPOUNDS IN SOIL SAMPLES;

CADMIUM	CD
CHROMIUM	CR
COPPER	CU
IRON	FE
NICKEL	NI
LEAD	PB
ZINC	ZN

A.TESTED CONCENTRATION RANGE; (UG/GRAM SOIL)

CD	-	2.50	TO 15.0	UG/G
CR	-	9.38	TO 37.5	UG/G
CU	-	6.25	TO 25.0	UG/G
FE	-	21250	TO 85000	UG/G
NI	-	9.38	TO 37.5	UG/G
PB	-	10.0	TO 40.0	UG/G
ZN	-	178	TO 710	UG/G

B.SENSITIVITY;

	ABSORBANCE X 1000	QUANTITY (UG/25ML EXTRACT)
CD	38	5
CR	121	19
CU	150	13
FE	234	43000
NI	97	19
PB	27	20
ZN	62	360

C.DETECTION LIMIT; (UG/GRAM SOIL)

CD	-	2.5	UG/G
CR	-	9.4	UG/G
CU	-	6.3	UG/G
FE	-	21250	UG/G
NI	-	9.4	UG/G
PB	-	10.0	UG/G
ZN	-	178	UG/G

D.INTERFERENCES; NITROGEN GAS AND HIGH CONCENTRATIONS OF DISSOLVED SOLIDS.

E.ANALYSIS RATE; (CD,CR,CU,FE,NI,PB,AND ZN) AFTER INSTRUMENT CALIBRATION, ONE ANALYST CAN ANALYZE 50 AND DIGEST 15 INDIVIDUAL METAL SAMPLES IN AN 8-HOUR DAY.

CHEMISTRY;

CD CADMIUM
CAS RN 7440-43-9
MELTING PT; 321C BOILING PT; 765C

CR CHROMIUM
CAS RN 7440-47-3
MELTING PT; 1857C BOILING PT; 2672C

CU COPPER
CAS RN 7440-50-8
MELTING PT; 1083C BOILING PT; 2567C

FE IRON
CAS RN 7439-89-6
MELTING PT; 1535C BOILING PT; 2750C

NI NICKEL
CAS RN 7440-02-0
MELTING PT; 1453C BOILING PT; 2732C

PB LEAD
CAS RN 7439-92-1
MELTING PT; 328C BOILING PT; 1740C

ZN ZINC
CAS RN 7440-66-6
MELTING PT; 420C BOILING PT; 907C

APPARATUS;

A.INSTRUMENTATION;

PERKIN ELMER MODEL 703 FLAME ATOMIC ABSORPTION SPECTROPHOTOMETER AND MODEL 503 FLAME ATOMIC ABSORPTION SPECTROPHOTOMETER EQUIPPED WITH A MODEL HGA-2100 GRAPHITE FURNACE ACCESSORY.

B.PARAMETERS;

DIRECT ASPIRATION;

EPA METH	HOLLOW CATHODE LAMP	WAVE-LENGTH (NM)	FUEL	OXIDANT	TYPE OF FLAME
CD 213.1	CD	228.8	ACETYLENE	AIR	OXIDIZE
CR 218.1	CR	357.9	ACETYLENE	NITROUS OXIDE	FUELRICH
CU 220.1	CU	324.7	ACETYLENE	AIR	OXIDIZE
FE 236.1	FE	248.3	ACETYLENE	AIR	OXIDIZE
NI 249.1	NI	232.0	ACETYLENE	AIR	OXIDIZE
PB 239.1	PB	283.3	ACETYLENE	AIR	OXIDIZE
ZN 289.1	ZN	213.9	ACETYLENE	AIR	OXIDIZE

OTHER OPERATING PARAMETERS SHOULD BE SET AS SPECIFIED BY THE INSTRUMENT MANUFACTURER.

C.HARDWARE/GLASSWARE;

HEAVY-DUTY PYREX BEAKERS, 500 AND 100 ML
HOT PLATE
WATER BATH
OXFORD MACRO-PIPETTES W DISPOSABLE TIPS
DISPOSABLE BEAKERS, 10ML CAPACITY
GELMAN 42MM GLASS FIBER FILTER PADS

D.CHEMICALS;

NITRIC ACID, CONC, SPEC GRADE
HYDROCHLORIC ACID 1 TO 1 - PREPARE A 1 TO 1

SOLUTION OF SPEC GRADE HCL WITH DE-IONIZED, DISTILLED WATER.

1000PPM FISHER SCIENTIFIC ATOMIC ABSORPTION REFERENCE STANDARD SOLUTIONS; CD,CR,CU, FE,NI,PB, AND ZN

STANDARDS;

A.CALIBRATION STANDARDS;

OBTAIN CALIBRATION STANDARD STOCK FOR EACH METAL (FISHER SCIENTIFIC ATOMIC ABSORPTION REFERENCE STANDARD SOLUTIONS, 1000 MG/L).

PREPARE DILUTE STOCK CALIBRATION STANDARDS BY PIPETTING 1ML OF EACH CALIBRATION STANDARD STOCK INTO SEPARATE 100ML VOLUMETRIC FLASKS AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID. DILUTE STOCK CALIBRATION STANDARDS STABLE FOR ONE MONTH.

PREPARE WORKING CALIBRATION STANDARDS FRESH FOR EACH RUN BY PIPETTING THE FOLLOWING VOLUMES OF DILUTE STOCK CALIBRATION STANDARDS INTO SEPARATE 100 ML VOLUMETRIC FLASKS AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID.

	WORKING CALIBRATION STANDARD CONCENTRATION (UG/L)	DILUTE STOCK CALIBRATION STANDARD VOLUME (ML)
CD	200	2
CR	1000	10
CU	2000	20
FE	2000	20
NI	20	0.2
PB	200	2
ZN	300	3

MAKE SERIAL DILUTIONS OF INDIVIDUAL WORKING CALIBRATION STANDARDS TO OBTAIN WORKING STANDARD CURVES.

B.CONTROL SPIKES;

PREPARE WORKING CONTROL SPIKE A (DIRECT ASPIRATION TECHNIQUE) BY PIPETTING THE FOLLOWING VOLUMES OF CALIBRATION STANDARD STOCKS INTO SEPARATE 100ML VOLUMETRIC FLASKS AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID. WORKING CONTROL SPIKE A STABLE FOR 6 MONTHS.

	WORKING CONTROL SPIKE A CONCENTRATION (MG/L)	CALIBRATION STANDARD STOCK VOLUME (ML)
CD	10	1.0
CR	10	1.0

CU	10	1.0	
FE	10000	7.026	
		FERROUS AMMONIUM SULFATE	
NI	10	1.0	
PB	10	1.0	
ZN	100	10.0	

PIPET KNOWN AMOUNTS OF WORKING CONTROL SPIKE A INTO 2.0G STANDARD SOIL SAMPLES. QUANTITY SPIKED SHOULD BE SELECTED TO PROVIDE THE FOLLOWING CONCENTRATIONS;

UNSPIKED,R/4,R/2,R/1.33, AND UL

WHERE; UL = UPPER LIMIT OF THE METHOD
L = DETECTION LIMIT OF STANDARD SAMPLE
R = RANGE (DIFFERENCE BETWEEN UL AND L)

DETERMINE PRECISION, ACCURACY, AND DETECTION LIMIT FOR EACH METAL IN STANDARD SOIL AS FOLLOWS;

PIPET FOLLOWING VOLUMES OF WORKING CONTROL SPIKES INTO TRIPLICATE 2.0 GRAM SOIL SAMPLES.

	WORKING CONTROL SPIKE VOLUME (ML)				
	UNSPIKED	R/4	R/2	R/1.33	UL
CD	0.0	0.5	1.0	2.0	3.0
CR	0.0	1.88	3.75	5.63	7.5
CU	0.0	1.25	2.5	3.75	5.0
FE	0.0	4.25	8.5	12.75	17.0
NI	0.0	1.88	3.75	5.63	7.5
PB	0.0	2.0	4.0	6.0	8.0
ZN	0.0	3.55	7.1	10.65	14.2

PERFORM PROCEDURE.

PROCEDURE;

DIRECT ASPIRATION AND FURNACE TECHNIQUES - CD,CR, CU,FE,NI,PB,AND ZN;

WEIGH 2.0G SOIL SAMPLES AND QUANTITATIVELY TRANSFER TO 100ML BEAKERS.

ADD 3.0ML OF CONCENTRATED NITRIC ACID, COVER BEAKERS WITH WATCH GLASSES, PLACE ON A HOT PLATE, EVAPORATE TO DRYNESS, AND COOL.

REPEAT PRIOR STEP UNTIL DIGESTION IS COMPLETE THEN ADD 12.0ML OF 1:1 HYDROCHLORIC ACID TO RESIDUE, AND HEAT UNTIL REIDUE DISSOLVES.

WASH DOWN SIDES OF BEAKERS AND WATCH GLASS COVERS WITH DEIONIZED WATER.

FILTER SAMPLES THROUGH NITRIC ACID-WASHED GELMAN 42MM GLASS-FIBER FILTER PADS.

DILUTE EACH SAMPLE TO A FINAL VOLUME OF 25.0

ML WITH DEIONIZED WATER.

FOLLOW THE INSTRUMENT OPERATING INSTRUCTIONS PROVIDED BY INSTRUMENT MANUFACTURER.

ALLOW CATHODE LAMP TO WARM UP FOR AT LEAST 15 MIN UNLESS OPERATED IN A DOUBLE BEAM MODE. ALIGN INSTRUMENT, POSITION MONOCHROMATOR AT CORRECT WAVELENGTH, SELECT PROPER SLIT WIDTH, AND ADJUST CATHODE CURRENT ACCORDING TO THE MANUFACTURER'S INSTRUCTIONS. LIGHT FLAME, REGULATE FLOW OF FUEL AND OXIDANT, ADJUST BURNER AND NEBULIZER FLOW RATE FOR MAXIMUM PERCENT ABSORPTION AND STABILITY, AND BALANCE PHOTOMETER.

RUN A SERIES OF STANDARDS AND CONSTRUCT A CALIBRATION CURVE BY PLOTTING CONCENTRATION VS ABSORBANCE

	EPA METHOD	TECHNIQUE
CD	213.1	DIRECT ASPIRATION
CR	218.1	DIRECT ASPIRATION
CU	220.1	DIRECT ASPIRATION
FE	236.1	DIRECT ASPIRATION
NI	249.1	DIRECT ASPIRATION
PB	239.1	DIRECT ASPIRATION
ZN	289.1	DIRECT ASPIRATION

CALCULATIONS;

CONSTRUCT A STANDARD CURVE OF EACH PARTICULAR METAL BY PLOTTING ABSORBANCE X 1000 VERSUS MICROGRAMS OF METAL. DETERMINE ABSORBANCE X 1000 OF EACH PARTICULAR METAL, AND READ VALUE FROM STANDARD CURVE. DETERMINE CONCENTRATION OF METAL FROM CALIBRATION CURVE ACCORDING TO FOLLOWING FORMULA;

$$\text{CONCENTRATION (UG/L)} = \frac{C \times VF}{VI}$$

WHERE; C = CONCENTRATION OF METAL FROM CALIBRATION CURVE (UG/L)
VF = VOLUME OF FINAL SAMPLE (L)
VI = VOLUME OF INITIAL SAMPLE (L)

DETERMINE CONCENTRATION OF METAL IN SOIL MATRIX (ON A DRY-WEIGHT BASIS) ACCORDING TO THE FOLLOWING FORMULA;

$$\text{CONCENTRATION (UG/G)} = \frac{\text{UG/L METAL} \times VE}{WD}$$

WHERE; WD = DRY WEIGHT OF SAMPLE IN EXTRACT (G)
VE = VOLUME OF EXTRACT (L)

REFERENCES;

U.S. ENVIRONMENTAL PROTECTION AGENCY, 1979. METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES, EPA

-600/4-79-020, CINCINNATI, OHIO.

6. Metals/Soil/Coldvapor AA (2D)

METALS/SOIL/COLDVAPOR AA (2D)

APPLICATION;

METHOD APPLICABLE FOR ANALYSIS OF THE FOLLOWING METALS IN SOIL SAMPLES;

MERCURY HG

A.TESTED CONCENTRATION RANGE; (UG/GRAM SOIL)

HG - 0.20 TO 0.50 UG/G

B.SENSITIVITY;

	ABSORBANCE X 1000	QUANTITY (UG/25ML EXTRACT)
HG	93	0.4

C.DETECTION LIMIT; (UG/GRAM SOIL)

HG - 0.2 UG/G

D.INTERFERENCES; SULFIDE, COPPER, AND CERTAIN VOLATILE ORGANICS WHICH ABSORB AT 254NM.

E.ANALYSIS RATE; AFTER INSTRUMENT CALIBRATION, ONE ANALYST CAN ANALYZE AND DIGEST 30 SAMPLES IN AN 8-HOUR DAY.

CHEMISTRY;

HG MERCURY
CAS RN 7439-97-6
MELTING PT; -39C BOILING PT; 357C

APPARATUS;

A.INSTRUMENTATION;

PERKIN ELMER MODEL 703 FLAME ATOMIC ABSORPTION SPECTROPHOTOMETER AND MODEL 503 FLAME ATOMIC ABSORPTION SPECTROPHOTOMETER EQUIPPED WITH A MODEL HGA-2100 GRAPHITE FURNACE ACCESSORY.

B.PARAMETERS;

INSTRUMENT SETTINGS RECOMMENDED BY THE PARTICULAR MANUFACTURER SHOULD BE FOLLOWED.

WAVELENGTH - 253.7 NM
CATHODE LAMP - WESTINGHOUSE WL-22847,ARGON FILLED, OR EQUIVALENT
RECORDER - MULTI-RANGE VARIABLE SPEED COMPATABLE WITH THE UV DETECTION SYSTEM
ABSORPTION CELL - 10 CM LONG, HAVING QUARTZ END WINDOWS
AIR PUMP - PERISTALTIC PUMP CAPABLE OF DELIVERING 1L/MIN OF AIR

C.HARDWARE/GLASSWARE;

HEAVY-DUTY PYREX BEAKERS, 500 AND 100 ML
300ML BOD BOTTLES
HOT PLATE

WATER BATH
OXFORD MACRO-PIPETTER W DISPOSABLE TIPS
DISPOSABLE BEAKERS, 10ML CAPACITY
GELMAN 42MM GLASS FIBER FILTER PADS
AERATION TUBING, STRAIGHT GLASS FRIT HAVING A COARSE POROSITY
TYGON TUBING
DRYING TUBE, 6" X 3/4" ID TUBE

D.CHEMICALS;

SULFURIC ACID, CONC, REAGENT GRADE
NITRIC ACID, CONC, REAGENT GRADE LOW HG
STANNOUS SULFATE REAGENT - ADD 25G OF STANNOUS SULFATE OR STANNOUS CHLORIDE TO 250ML OF 0.5N SULFURIC ACID
SODIUM CHOLIDE-HYDROXYLAMINE SULFATE REAGENT DISSOLVE 12G OF NACL AND 12G OF HYDROXYL AMINE SULFATE OR HYDROXYLAMINE HYDROCHLORIDE IN DISTILLED WATER AND DILUTE TO 100ML
POTASSIUM PERMANGANATE, 5% SOLUTION, W/V
POTASSIUM PERSULFATE, 5% SOLUTION, W/V
MAGNESIUM PERCHLORATE
1000PPM FISHER SCIENTIFIC ATOMIC ABSORPTION REFERENCE STANDARD SOLUTIONS; HG

STANDARDS;

A.CALIBRATION STANDARDS;

OBTAIN CALIBRATION STANDARD STOCK FOR EACH METAL (FISHER SCIENTIFIC ATOMIC ABSORPTION REFERENCE STANDARD SOLUTIONS, 1000 MG/L).

PREPARE DILUTE STOCK CALIBRATION STANDARDS BY PIPETTING 1ML OF EACH CALIBRATION STANDARD STOCK INTO SEPARATE 100ML VOLUMETRIC FLASKS AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID. DILUTE STOCK CALIBRATION STANDARDS STABLE FOR ONE MONTH.

PREPARE WORKING CALIBRATION STANDARDS FRESH FOR EACH RUN BY PIPETTING THE FOLLOWING VOLUMES OF DILUTE STOCK CALIBRATION STANDARDS INTO SEPARATE 100 ML VOLUMETRIC FLASKS AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID.

	WORKING CALIBRATION STANDARD CONCENTRATION (UG/L)	DILUTE STOCK CALIBRATION STANDARD VOLUME (ML)
HG	10	0.1

MAKE SERIAL DILUTIONS OF INDIVIDUAL WORKING CALIBRATION STANDARDS TO OBTAIN WORKING STANDARD CURVES.

B.CONTROL SPIKES;

PREPARE DILUTE STOCK CONTROL SPIKE B (COLD VAPOR TECHNIQUE) BY PIPETTING 1ML OF HG CALIBRATION STANDARD STOCK INTO A 100ML VOLUMETRIC FLASK AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID. DILUTE STOCK CONTROL SPIKE B STABLE FOR 6 MONTHS.

PREPARE WORKING CONTROL SPIKE B BY PIPETTING 1ML OF DILUTE STOCK CONTROL B INTO A VOLUMETRIC FLASK AND DILUTING TO VOLUME WITH 0.5% (VOL/VOL) NITRIC ACID (1ML = 0.100 UG OF HG). PREPARE FRESH WORKING CONTROL SPIKE B FOR EACH RUN.

PIPET KNOWN AMOUNTS OF WORKING CONTROL SPIKE B INTO 2.0G STANDARD SOIL SAMPLES. QUANTITY SPIKED SHOULD BE SELECTED TO PROVIDE THE FOLLOWING CONCENTRATIONS;

UNSPIKED,R/4,R/2,R/1.33, AND UL

WHERE; UL = UPPER LIMIT OF THE METHOD
L = DETECTION LIMIT OF STANDARD SAMPLE
R = RANGE (DIFFERENCE BETWEEN UL AND L)

DETERMINE PRECISION, ACCURACY, AND DETECTION LIMIT FOR EACH METAL IN STANDARD SOIL AS FOLLOWS;

PIPET FOLLOWING VOLUMES OF WORKING CONTROL SPIKES INTO TRIPLICATE 2.0 GRAM SOIL SAMPLES.

	WORKING CONTROL SPIKE VOLUME (ML)				
	UNSPIKED	R/4	R/2	R/1.33	UL
HG	0.0	4.0	6.0	8.0	10.0

PERFORM PROCEDURE.

PROCEDURE;

COLD VAPOR TECHNIQUE - HG;

WEIGH 2.0G SOIL SAMPLES AND QUANTITATIVELY TRANSFER TO 300ML BOD BOTTLES.

ADD 25ML OF DEIONIZED WATER FOLLOWED BY 2.5ML OF CONCENTRATED NITRIC ACID, COVER WITH STOPPERS, AND LET STAND FOR 45 TO 60 MINUTES.

ADD 75.0ML OF DEIONIZED WATER TO EACH BOD BOTTLE AND PLACE IN A 75C+-5C WATER BATH FOR AT LEAST 2 HOURS.

REMOVE SAMPLES FROM WATER BATH AND ALLOW THEM TO COOL COMPLETELY.

TRANSFER 100ML OF WATER SAMPLE TO A 300ML BOD BOTTLE. ADD 5ML OF CONC SULFURIC ACID AND 2.5ML OF NITRIC ACID, MIXING AFTER EACH ADDITION.

ADD 15ML OF POTASSIUM PERMANGANATE SOLUTION TO

EACH BOTTLE. ADD 6ML OF POTASSIUM PERSULFATE SOLUTION TO EACH BOTTLE AND HEAT FOR 2 HR IN A WATER BATH AT 95C.

COOL AND ADD 6ML OF SODIUM CHLORIDE-HYDROXYLAMINE SULFATE SOLUTION TO REDUCE EXCESS PERMANGANATE. AFTER AT LEAST 30 SEC, ADD 5ML OF STANNOUS SULFATE SOLUTION AND IMMEDIATELY ATTACH BOTTLE TO AERATION APPARATUS.

ALLOW SAMPLE TO STAND WITHOUT MANUAL AGITATION. THE CIRCULATING PUMP (1L/MIN) IS ALLOWED TO RUN CONTINUOUSLY.

ABSORBANCE WILL INCREASE AND REACH MAXIMUM WITHIN 30 SEC. AS SOON AS RECORDER PEN LEVELS OFF (1 MIN) OPEN BYPASS VALVE AND CONTINUE AERATION UNTIL ABSORBANCE RETURNS TO ITS MINIMUM VALUE.

CALCULATIONS;

CONSTRUCT A STANDARD CURVE OF EACH PARTICULAR METAL BY PLOTTING ABSORBANCE X 1000 VERSUS MICROGRAMS OF METAL. DETERMINE ABSORBANCE X 1000 OF EACH PARTICULAR METAL, AND READ VALUE FROM STANDARD CURVE. DETERMINE CONCENTRATION OF METAL FROM CALIBRATION CURVE ACCORDING TO FOLLOWING FORMULA;

$$\text{CONCENTRATION (UG/L)} = \frac{C \times VF}{VI}$$

WHERE; C = CONCENTRATION OF METAL FROM CALIBRATION CURVE (UG/L)
VF = VOLUME OF FINAL SAMPLE (L)
VI = VOLUME OF INITIAL SAMPLE (L)

DETERMINE CONCENTRATION OF METAL IN SOIL MATRIX (ON A DRY-WEIGHT BASIS) ACCORDING TO THE FOLLOWING FORMULA;

$$\text{CONCENTRATION (UG/G)} = \frac{\text{UG/L METAL} \times VE}{WD}$$

WHERE; WD = DRY WEIGHT OF SAMPLE IN EXTRACT (G)
VE = VOLUME OF EXTRACT (L)

REFERENCES;

U.S. ENVIRONMENTAL PROTECTION AGENCY, 1979. METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES, EPA-600/4-79-020, CINCINNATI, OHIO.

APPENDIX B

MAP FILE

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INSAGMACD   BG01        2091754971          M12.5YR 4/8
INSAGMACD   BG02        2095954915          M110 YR 7/4
INSAGMACD   BG03        2098054957          M17.5YR 6/8
INSAGMACD   BG04        2098754967          M110 YR 7/6
INSAGMACD   BG05        2099454941          M110 YR 7/6
INSAGMACD   BG06        2100054957          M110 YR 6/6
INSAGMACD   BG07        2101154967          M110 YR 6/6
INSAGMACD   BG08        2102254915          M17.5YR 6/6
INSAGMACD   BG09        2103154957          M17.5 YR 6/6
INSAGMACD   BG10        2104254971          M17.5 YR 5/8
INSAGMACD   BG11        2105354915          M17.5 YR 6/8
INSAGMADTCHBG12         2105555000          M17.5 YR 6/8
INSAGMADTCHBG13         2095954832          M17.5 YR 6/6
INSAGMACD   BG14        2097954890          M110 YR 7/4
INSAGMACD   BG15        2097954832          M110 YR 5/6
INSAGMACD   BG16        2104254873          M110 YR 6/6
INSAGMACD   BG17        2106354895          M110 YR 7/6
INSAGMACD   BG18        2106354832          M110 YR 6/4
INSAGMACD   BG19        2104254737          M110 YR 7/4
INSAGMACD   BG20        2103554809          M110 YR 7/6
INSAGMACD   BG21        2102854790          M110 YR 7/8
INSAGMACD   BG22        2102154749          M110 YR 7/6
INSAGMACD   BG23        2101454800          M110 YR 7/6
INSAGMACD   BG24        2100754790          M110 YR 7/4
INSAGMACD   BG25        2100054800          M110 YR 6/4
INSAGMACD   BG26        2100354749          M110 YR 7/6
INSAGMACD   BG27        2099554790          M110 YR 7/6
INSAGMACD   BG28        2099054787          M110 YR 7/8
INSAGMACD   BG29        2097954809          M110 YR 7/4
INSAGMADTCHBG30         2108354729          M110 YR 7/3
INSAGMACD   BKG1        2112554967          M110 YR 7/4
INSAGMACD   BKG2        2112554967          M110 YR 7/4
INSAGMACD   BG06D       2100054957          M1
INSAGMADTCHBG12D        2105355000          M1
INSAGMACD   BG24D       2100754790          M1
INSAGMAMAHOMH01         2035155463          M1STATIC TEST AREA
INSAGMAMAHOMH02         2029755432          M1STATIC TEST AREA
INSAGMABLDG2750-1       2026355644          M1PAINT
INSAGMABLDG2750-2       2033355513          M1 NC
INSAGMABLDG27502A       2028355673          M1 NC
INSAGMABLDG2750-3       2025455555          M1PAINT NC
INSAGMABLDG2750-4       2032955607          M1PAINT
INSAGMABLDG2750-6       2037855450          M1PAINT NC
INSAGMABLDG2750-7       2019955603          M1PAINT
INSAGMABLDG2750-8       2029555567          M1PAINT
INSAGMACREKSE01         1814155822          M1LICK CREEK
INSAGMACREKSE02         1934256822          M114MI STEELBRIDGE
INSAGMACREKSE03         2066155040          M114MI BG ST DRAIN
INSAGMACREKSE04         2028253500          M114MI OHIO RIVER
OK.
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APPENDIX C

CHEMICAL FILE

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPRO HAP# 1043

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

		SAMPLE NUMBERS									
PARAMETERS	STORET #	BG01 117800	BG03 117801	BG02 117802	BG04 117803	BG05 117804	BG06 117805	BG06D 117806	BG07 117807	BG08 117808	BG09 117809
DATE		7/27/81	7/27/81	7/27/81	7/27/81	7/27/81	7/27/81	7/27/81	7/27/81	7/27/81	7/27/81
TIME		1542	1547	1551	1600	1606	1614	1617	1623	1632	1627
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
SAMPLE TYPE	71999	SO	SO	SO	SO	SO	SO	SO	SO	SO	SO
SITE TYPE 1	99759	CD	CD	CD	CD	CD	CD	CD	CD	CD	CD
SAMPLE DEPTH(CM)	99758	30	30	30	30	30	30	30	20	20	25
SAMPLING TECHNIQUE	72005	U	U	U	U	U	U	U	U	U	U
EXTRACTION DATE,JULI AN(NICELL)	99597	81230	81231	81230	81230	81231	81230	81231	81231	81231	81230
ZN,SOIL(MG/KG)	99586	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
PB,SOIL(MG/KG)	99581	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CD,SOIL(MG/KG)	99582	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CR,SOIL(MG/KG)	99584	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
HG,SOIL(MG/KG)	99580	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CU,SOIL(MG/KG)	99585	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
MOISTURE CONTENT(% W ET WT)	70320	5	4	4	4	4	3	3	4	4	4

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPSO MAP# 1043

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS BG10 117810	BG12 117811	BG12D 117812	BG11 117813	BG13 117814	BG14 117815	BG16 117816	BG17 117817	BG18 117818	BG15 117819
DATE		7/27/81	7/27/81	7/27/81	7/27/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81
TIME		1647	1655	1658	1704	1538	1548	1600	1609	1615	1626
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
SAMPLE TYPE	71999	SO	SO	SO	SO	SO	SO	SO	SO	SO	SO
SITE TYPE 1	99759	CD	DTCH	DTCH	CD	DTCH	CD	CD	CD	CD	CD
SAMPLE DEPTH(CM)	99758	30	30	30	30	30	30	30	30	30	30
SAMPLING TECHNIQUE	72005	U	U	U	U	U	U	U	U	U	U
EXTRACTION DATE,JULIAN(NICELL)	99597	81231	81230	81230	81231	81231	81230	81231	81231	81231	81231
ZN,SOIL(MG/KG)	99586	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
PB,SOIL(MG/KG)	99581	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CD,SOIL(MG/KG)	99582	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CR,SOIL(MG/KG)	99584	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
HG,SOIL(MG/KG)	99580	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CU,SOIL(MG/KG)	99585	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
MOISTURE CONTENT(% WET WT)	70320	7	4	4	4	5	3	3	3	3	3

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPSO NAF# 1043

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

		SAMPLE NUMBERS									
PARAMETERS	STORET #	BG29 117820	BG28 117821	BG27 117822	BG26 117823	BG25 117824	BG24 117825	BG24D 117826	BG23 117827	BG21 117828	BG22 117829
DATE		7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81
TIME		1647	1659	1709	1717	1723	1731	1735	1739	1745	1750
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
SAMPLE TYPE	71999	SO	SO	SO	SO	SO	SO	SO	SO	SO	SO
SITE TYPE 1	99759	CD	CD	CD	CD	CD	CD	CD	CD	CD	CD
SAMPLE DEPTH(CM)	99758	30	30	30	30	30	30	30	30	30	30
SAMPLING TECHNIQUE	72005	U	U	U	U	U	U	U	U	U	U
EXTRACTION DATE,JULIAN(NICELL)	99597	81230	81231	81231	81230	81230	81230	81230	81231	81231	81231
ZN,SOIL(MG/KG)	99586	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
PB,SOIL(MG/KG)	99581	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CD,SOIL(MG/KG)	99582	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CR,SOIL(MG/KG)	99584	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
HG,SOIL(MG/KG)	99580	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
CU,SOIL(MG/KG)	99585	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
MOISTURE CONTENT(% WET WT)	70320	3	5	4	3	6	3	4	3	3	3

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPSO MAP# 1043

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS BG19 117830	BG30 117831	BG20 117832	BKG1 117833	BKG2 117834
DATE		7/28/81	7/28/81	7/28/81	7/28/81	7/28/81
TIME		1759	1809	1815	1837	1837
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0	<2.0	<2.0	NA
INSTALLATION CODE	99720	IN	IN	IN	IN	IN
SAMPLE TYPE	71999	SO	SO	SO	SO	SO
SITE TYPE 1	99759	CD	DTCH	CD	CD	CD
SAMPLE DEPTH(CM)	99758	30	30	30	30	30
SAMPLING TECHNIQUE	72005	U	U	U	U	U
EXTRACTION DATE,JULIAN(NICELL)	99597	81230	81230	81230	81230	NA
ZN,SOIL(MG/KG)	99586	NA	NA	NA	NA	<142
PB,SOIL(MG/KG)	99581	NA	NA	NA	NA	<12
CD,SOIL(MG/KG)	99582	NA	NA	NA	NA	<3
CR,SOIL(MG/KG)	99584	NA	NA	NA	NA	<13
HG,SOIL(MG/KG)	99580	NA	NA	NA	NA	<0.2
CU,SOIL(MG/KG)	99585	NA	NA	NA	NA	<6.18
MOISTURE CONTENT(% WET WT)	70320	3	6	4	3	NA

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPSE MAP# 1042

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS SE03 117701	SE04 117702	SE02 117703	SE01 117704
DATE		7/29/81	7/29/81	7/29/81	7/29/81
TIME		1036	1100	1326	1330
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0	<2.0	<2.0
INSTALLATION CODE	99720	IN	IN	IN	IN
SAMPLE TYPE	71999	SE	SE	SE	SE
SITE TYPE 1	99759	CREK	CREK	CREK	CREK
SAMPLE DEPTH(CM)	99758	152	168	38	38
SAMPLING TECHNIQUE	72005	C	C	C	C
EXTRACTION DATE,JULI AN(NICELL)	99597	81237	81237	81237	81237

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INFSR MAP# 1044

PROJECT NUMBER 81424400

PROJECT MANAGER LYNN WIESE

PROJECT NAME INAAP

FIELD GROUP LEADER

SAMPLE NUMBERS

PARAMETERS	STORET #	MH01 117900	MH02 117901
DATE		7/28/81	7/28/81
TIME		917	930
NITROCELLULOSE,SOIL(UG/G)	99574	<2.0	<2.0
INSTALLATION CODE	99720	IN	IN
SAMPLE TYPE	71999	SR	SR
SITE TYPE 1	99759	MAHO	MAHO
SAMPLE DEPTH(CM)	99758	249	249
SAMPLING TECHNIQUE	72005	U	U
EXTRACTION DATE,JULIAN(NICELL)	99597	81237	81237

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPB1 MAP# 1038

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS 2750-2 117308	2750-2 117309	2750-2 117310	2750-2 117311	2750-2 117312	2750-2 117313	2750-2 117314	2750-2 117315	2750-2 117304	2750-3 117302
DATE		7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81
TIME		1305	1305	1305	1305	1305	1305	1305	1305	1155	1155
NITROCELLULOSE,TOTAL UG	99770	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04
NITROGLYCERIN(TOTAL UG)	99808	<15	<15	<15	<15	<15	<15	<15	<15	<15	<15
SAMPLE TYPE	71999	BI	BI	BI	BI	BI	BI	BI	BI	BI	BI
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
SAMPLING TECHNIQUE	72005	0	0	0	0	0	0	0	0	0	0
SAMPLE DEPTH(CM)	99738	0	0	0	0	0	0	0	0	0	0
SITE TYPE 1	99759	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPB1 MAP# 1038

PROJECT NUMBER 81424400 PROJECT NAME INAAP

PROJECT MANAGER LYNN WIESE FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS 2750-3 117303	2750-3 117305	2750-3 117306	2750-3 117307	2750-6 117300	2750-6 117301
DATE		7/30/81	7/30/81	7/30/81	7/30/81	7/30/81	7/30/81
TIME		1155	1155	1155	1155	1030	1030
NITROCELLULOSE,TOTA. UG	99770	<5.04	<5.04	<5.04	<5.04	<5.04	<5.04
NITROGLYCERIN(TOTAL UG)	99808	<15	<15	<15	<15	<15	<15
SAMPLE TYPE	71999	BI	BI	BI	BI	BI	BI
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN
SAMPLING TECHNIQUE	72005	0	0	0	0	0	0
SAMPLE DEPTH(CM)	99758	0	0	0	0	0	0
SITE TYPE 1	99759	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPUT MARS 1041

PROJECT NUMBER 81424400

PROJECT MANAGER LYNN WIESE

PROJECT NAME INAAP

FIELD GROUP LEADER

PARAMETERS	STORET #	SAMPLE NUMBERS									
		2750-1 117600	2750-1 117609	2750-3 117601	2750-3 117604	2750-3 117606	2750-3 117614	2750-4 117603	2750-7 117605	2750-7 117607	2750-8 117610
DATE		7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81	7/28/81
TIME		1028	1028	940	1028	940	942	957	1028	1027	1025
SAMPLE TYPE	71999	BX	BX	BX	BX	BX	BX	BX	BX	BX	BX
INSTALLATION CODE	99720	IN	IN	IN	IN	IN	IN	IN	IN	IN	IN
SAMPLING TECHNIQUE	72005	Z	Z	Z	Z	Z	Z	Z	Z	Z	Z
SAMPLE DEPTH(CM)	99758	0	0	0	0	0	0	0	0	0	0
SITE TYPE 1	99759	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG	BLDG
PB,SOIL(MG/KG)	99581	68	<12	<12	41	24	<12	<12	23	43	97
CD,SOIL(MG/KG)	99582	3	7	<3	4	5	11	5	5	5	6
CU,SOIL(MG/KG)	99585	28800	49800	49100	58300	55200	60900	57000	54600	54100	48000
CR,SOIL(MG/KG)	99584	33	37	26	45	50	133	52	723	43	78
ZN,SOIL(MG/KG)	99586	378	397	326	567	414	478	468	549	419	834
HG,SOIL(MG/KG)	99580	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2

ENVIRONMENTAL SCIENCE & ENGINEERING 09/15/81 COMPUTER ANALYSIS REPORT INPPT MAP# 1041

PROJECT NUMBER 81424400

PROJECT MANAGER LYNN WIESE

PROJECT NAME INAAP

FIELD GROUP LEADER

SAMPLE NUMBERS

PARAMETERS	STORET #	2750-8 117611
DATE		7/28/81
TIME		1005
SAMPLE TYPE	71999	BX
INSTALLATION CODE	99720	IN
SAMPLING TECHNIQUE	72005	Z
SAMPLE DEPTH(CM)	99758	0
SITE TYPE 1	99759	BLDG
PB,SOIL(MG/KG)	99581	160
CD,SOIL(MG/KG)	99582	7
CU,SOIL(MG/KG)	99585	40400
CR,SOIL(MG/KG)	99584	38
ZN,SOIL(MG/KG)	99586	644
HG,SOIL(MG/KG)	99580	<0.2


N
DATE
ILME