IN THE COURT OF COMMON PLEAS
CUYAHOGA COUNTY, OHIO

STATE OF OHIO, ex rel. ANTHONY J. CELEBREZZE, JR. ATTORNEY GENERAL OF OHIO Plaintiff, vs. SPECIALIZED FINISHERS, INC., Defendants.	:	CASE NO. 126971 JUDGE JAMES J. McMONAGLE <u>CONSENT ORDER BETWEEN THE STATE OF OHIO AND THOMAS J. FOLEY, ROBERT W.HORN, JONATHAN TAYLOR, ROBERT BAUMGARTNER, AND B.A. CARRAN</u>

The Plaintiff, State of Ohio, ex rel. Anthony J. Celebrezze, Jr., Attorney General of Ohio ("State" or "Plaintiff"), filed the amended Complaint in this action on March 3, 1989 against Defendants Specialized Finishers, Inc., Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran, to enforce the State of Ohio's hazardous waste laws and water pollution laws and the rules promulgated thereunder concerning the Defendants' waste handling and disposal practices at the Specialized Finishers, Inc. facility located at 2133-2139 Hamilton Avenue, Cleveland, Cuyahoga County, Ohio (hereinafter the "facility"). The case against Specialized Finishers, Inc., was resolved through a default judgment. Plaintiff and Defendants Thomas J. Foley, Robert W. Horn, Johnathan Taylor, Robert Baumgartner, and B.A. Carran consent to entry of this Order;

THEREFORE, without trial or admission of any issue of law or of fact, and upon the consent of the Plaintiff and Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran hereto, it is hereby ORDERED, ADJUDGED and DECREED as follows:

I. PERSONS BOUND

The provisions of this Consent Order shall apply to and be binding upon Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran. These defendants shall provide a copy of this Consent Order to each consultant or contractor they employ to perform the work referenced herein.

II. SATISFACTION OF LAWSUIT

Compliance with the terms of this Consent Order shall constitute full satisfaction of any civil liability by Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner and B.A. Carran for all claims against said Defendants alleged in the Complaint. Nothing in this Consent Order shall be construed so as to limit the authority of the State of Ohio to seek relief for claims or conditions not alleged in the Complaint, including violations or conditions

which occur after the filing of the Complaint. Nothing in this Consent Order shall be construed so as to limit the authority of the State of Ohio to undertake any action against any person, including Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran, to eliminate or mitigate conditions arising after the date hereof which may present a threat to the public health, welfare or the environment.

III. JURISDICTION AND VENUE

The Court has both personal and subject matter jurisdiction over Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran. The Complaint states a claim upon which relief can be granted against Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, Robert Baumgartner, and B.A. Carran under Chapters 3734 and 6111 of the Ohio Revised Code and the rules promulgated thereunder. Venue is proper in this court.

IV. CLOSURE PLAN

Defendants Foley, Horn, Taylor, Baumgartner, and B.A. Carran shall fully implement the closure plan attached hereto as Attachment 1, which is incorporated herein as if fully restated.

Said Defendants are enjoined and ordered to fully implement the closure plan as approved by the Ohio EPA and comply with the rules contained in O.A.C. 3745-65-14, 3745-66-10, 3745-66-11, 3745-66-12, 3745-66-14, 3745-66-15, 3745-66-16 and (without admission by any Defendant of legal status as a "generator") the generator requirements of R.C. Chapter 3734 and O.A.C. 3745-52 et seq.

In addition, Defendant Foley shall determine the whereabouts of the fiberglass tank referenced in Item 2 of the closure plan that has been removed from the facility, and Defendants Foley, Horn, Taylor, and Baumgartner shall demonstrate decontamination of such tank by the methods provided in the approved closure plan. As part of the closure, Defendants shall specifically provide for closure of the additional sump unit as provided by Condition 4 of the approved closure plan.

Defendant shall complete waste removal from the facility as soon as possible but no later than ninety (90) days after entry of this consent order. Defendants shall complete closure of the facility within one hundred fifty (150) days after entry of this order and certify closure pursuant to O.A.C. 3745-66-15 no later than one hundred eight (180) days after the entry of this consent order.

Should implementation of the closure plan after the entry of this order reveal that amendments to the closure plan are required due to subsequent discoveries of contamination at the facility, Defendants shall amend the closure plan within the

time frames set forth in O.A.C. 3745-66-12(C)(2). Defendants shall comply with the amended closure plan as approved by Ohio EPA. Such amended closure plan shall be attached to this consent order as attachment 2 and incorporated herein as if fully restated. Defendants shall comply with the amended closure plan.

V. PERMANENT INJUNCTION

Except as provided in Section 4 above, Defendants Thomas J. Foley, Robert W. Horn, Jonathan Taylor, and Robert Baumgartner are permanently enjoined to comply with O.R.C. Chapter 6111 and the rules adopted thereunder and O.R.C. Chapter 3734 and the rules adopted therunder.

VI. CIVIL PENALTY

It is hereby ordered that Defendants Taylor and Baumgartner shall each pay a civil penalty of ten thousand ($10,000.00) dollars each. This civil penalty shall be paid by checks made payable to "Treasurer, State of Ohio," which checks shall be delivered by mail, or otherwise, to Timothy Kern, or his successor in office, at his office at the Ohio Attorney General's Office, Environmental Enforcement Section, 30 East Broad Street, 25th Floor, Columbus, Ohio 43266-0410, within

thirty (30) days of the Court's entry of this order. This penalty shall be paid into the hazardous waste clean-up fund created by R.C. 3734.28.

Defendant Foley shall pay a civil penalty of twenty-five thousand ($25,000.00) dollars. This civil penalty shall be paid by check made payable to "Treasurer, State of Ohio," which check shall be delivered by mail, or otherwise, to Timothy Kern, or his successor in office, at his office at the Ohio Attorney General's Office, Environmental Enforcement Section, 30 East Broad Street, 25th floor, Columbus, Ohio 43266-0410. Payment shall be made as follows:

An initial payment of not less than $675.00 on or before April 20, 1990, with monthly installments of at least $400.00 per month until paid in full.

Failure to pay the penalty in a timely fashion will result in the imposition of statutory interest that begins to accrue at the time the payment is delinquent. Defendant shall pay the statutory interest accrued by the next scheduled payment date for civil penalty.

VIII. RETENTION OF JURISDICTION

The Court will retain jurisdiction of this action for the purpose of overseeing that Defendants Foley, Horn, Taylor, Baumgartner and B.A. Carran, subject to further order of the

Court, carry out the terms and conditions of this Consent Order and comply with O.R.C. Chapter 3734. and the rules adopted thereunder.

IX. INSPECTIONS

Pursuant to O.R.C. 3734.07, Defendants Foley, Horn, Taylor, Baumgartner and B.A. Carran are ordered to allow employees, representatives, and agents of the Ohio EPA, upon proper identification, to enter upon the facility at reasonable times, to inspect, investigate, take samples and pictures and examine or copy records in order to determine compliance with the terms of this Consent Order and O.R.C. Chapter 3734. and the rules promulgated thereunder. Nothing in this Consent Order shall limit the rights of the Ohio EPA or U.S. EPA to conduct regular and routine inspections pursuant to statute or regulation at the Specialized Finishers, Inc. facility.

X. NOTICE

Any submission to the Ohio EPA as required by this Consent Order, unless otherwise indicated, shall be delivered to:

1. Ohio EPA
   Northeast District Office
   2110 East Aurora
   Twinsburg, Ohio 44087
   Attn: Gregory Taylor

2. Ohio EPA
Division of Solid and Hazardous Waste Management
P.O. Box 1049
1800 WaterMark Drive
Columbus, Ohio 43266-0149
Attn: Michael A. Savage

XI. COURT COSTS

Defendants Foley, Horn, Taylor, Baumgartner, and B.A. Carran shall pay the court costs of this action incurred to date.

XII.

For the purposes of effectuating the cleanup of the facility, the Defendants are jointly and severally liable. The remaining issues joined in this lawsuit shall be set for trial on February 6, 1990. Nothing herein shall be construed as a waiver of rights or defenses of parties involved in the remaining issues of this case.

DATE: 1/9/90

JUDGE

ANTHONY J. CELEBREZZE, JR.
ATTORNEY GENERAL OF OHIO

Dominic J. Hanket

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JAN 09 1990

GERALD E. FUERST, CLERK
BY _____ DEP.

-BERG

OhioEPA
State of Ohio Environmental Protection Agency
P.O. Box 1049, 1800 WaterMark Dr.
Columbus, Ohio 43266-0149

Toward A Cleaner Environment
15 Year Anniversary 1972 - 1987

Richard F. Celeste
Governor

CERTIFIED MAIL

January 26, 1988

Re: CLOSURE PLAN
SPECIALIZED FINISHERS, INC.
OHD013550371

Mr. Thomas J. Poley
Specialized Finishers, Inc.
P.O. Box 93893
Cleveland, Ohio 44101-5893

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OHIO EPA-N.E.D.O.

Dear Mr. Poley:

On July 3, 1987, Specialized Finishers, Inc. submitted to Ohio EPA a closure plan for three above-ground hazardous waste storage tanks and one hazardous waste sump unit located at 2139 Hamilton Avenue, Cleveland, Ohio. An addendum to the closure plan was received on October 9, 1987. The closure plan was submitted pursuant to Rule 3745-66-12 of the Ohio Administrative Code (OAC) in order to demonstrate that Specialized Finishers, Inc.'s proposal for closure complies with the requirements of OAC Rules 3745-66-11 and 3745-66-12.

The public was given the opportunity to submit written comments regarding the closure plan of Specialized Finishers, Inc. in accordance with OAC Rule 3745-66-12. No comments were received by Ohio EPA in this matter.

Based upon review of the company's submittal and subsequent revisions, I conclude that the closure plan for the hazardous waste facility at Specialized Finishers, Inc. meets the performance standard contained in OAC Rule 3745-66-11 and complies with the pertinent parts of OAC Rule 3745-66-12.

The closure plan submitted to Ohio EPA by Specialized Finishers, Inc. is hereby approved with the following modifications:

1. As stated in the plan, each of the three (3) waste tank units and the one (1) sump unit shall be cleaned using a series of rinses. The final rinseates from each of the four (4) units shall be collected separately. A representative sample of each of the final rinseates shall be collected individually and each of the samples shall be analyzed for the presence of cyanide, barium, cadmium, chromium, lead and mercury. If total available cyanide (using attached USEPA draft method) exceeds 250 milligrams per kilogram of waste, or if any of the aforementioned metals are detected in any of the samples in amounts exceeding their respective maximum concentrations for the characteristic of EP Toxicity, the unit(s) shall be considered to be still contaminated; in those instances, Specialized

I certify this to be a true and accurate copy of the official document as filed in the records of the Ohio Environmental Protection Agency.

By: Mary Carver Date 1-26-88

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Finishers should continue to repeat the rinsing and sampling/analysis procedure until the stated "clean" levels for these hazardous constituents are achieved.

2. All sample collection and analytical procedures shall be conducted in strict accordance with the appropriate SW-846 methods ("Test Methods for Evaluating Solid Wastes," 2nd or 3rd Edition), including sample chain-of-custody procedures, sample holding and preparation procedures, etc. Rinseate samples shall be analyzed using SW-846 Method #3010 (EP Toxicity) and the attached USEPA draft method for total available cyanide.

3. The three (3) above-ground waste tanks shall be visually inspected, prior to their removal from the site, to ensure that decontamination measures have effectively removed all visable signs of contamination from the interior and exterior of the units.

4. Flooring and remaining equipment in other areas of the facility shall also be inspected to ensure that hazardous residues from previous production activities do not remain. If residues are noted, the flooring and equipment in the previous production areas shall also be cleaned by rinsing.

5. Should signs of cracks or deterioration in the surfaces of the sump or concrete flooring under the tanks be noted, Specialized Finishers shall prepare and submit to Ohio EPA for review and approval a plan detailing how Specialized Finishers will assess the extent of any subsurface contamination which may have resulted from leaks or spills of hazardous constituents.

6. Deborah Berg, Northeast District Office (NEDO), Ohio EPA, should be notified at least five (5) business days in advance of critical activities, i.e., waste removal, final rinsing and inspection activities, so that Ohio EPA personnel may be present at the site to view the activities.

7. At the completion of closure activities, Specialized Finishers shall submit to Deborah Berg, NEDO, Ohio EPA: (a) uniform hazardous waste manifests completed for waste and rinseate shipments initiated from the facility; (b) shipping or invoice documents for any plating solutions sold from the facility; (c) copies of all rinseate sampling and analytical reports generated; and (d) properly worded closure certification statements. Copies of the certification statements also shall be submitted to Thomas Crepeau, Ohio EPA, DSHWM (address below).

Please be advised that approval of this closure plan does not release Specialized Finishers, Inc. from any responsibilities as required under the Hazardous and Solid Waste Amendments of 1984 regarding corrective action for all releases of hazardous waste or constituents from any solid waste management unit, regardless of the time at which waste was placed in the unit.

Due to the fact that the Ohio EPA is not currently authorized to conduct the federal hazardous waste program in Ohio, your closure plan also must be reviewed and approved by USEPA. Federal RCRA closure regulations (40 CFR 265.112) require that you submit a closure plan to George Hamper, Chief, Waste Management Division, Technical Programs Section, Ohio Unit, USEPA, Region V, 5HS-13, 230 South Dearborn Street, Chicago, Illinois 60604. Approval by both agencies is necessary prior to commencement of activities required by the approved closure plan.

You are notified that this action of the Director is final and may be appealed to the Environmental Board of Review pursuant to Section 3745.04 of the Ohio Revised Code. The appeal must be in writing and set forth the action complained of and the grounds upon which the appeal is based. It must be filed with the Environmental Board of Review within thirty (30) days after notice of the Director's action. A copy of the appeal must be served on the Director of the Ohio Environmental Protection Agency and the Environmental Enforcement Section of the Office of the Attorney General within three (3) days of filing with the Board. An appeal may be filed with the Environmental Board of Review at the following address: Environmental Board of Review, 236 East Town Street, Room 300, Columbus, Ohio 43266-0557.

When closure is completed, the Ohio Administrative Code Rule 3745-66-15 requires the owner or operator of a facility to submit to the Director of the Ohio EPA certification by the owner or operator and a registered professional engineer that the facility has been closed in accordance with the approved closure plan. The certification by the owner or operator shall include the statement found in OAC 3745-50-42(D). These certifications should be submitted to: Ohio Environmental Protection Agency, Division of Solid and Hazardous Waste Management, Attn: Thomas Crepeau, Program Planning and Management Section, P.O. Box 1049, Columbus, Ohio 43266-0149.

Sincerely,

Richard L. Shank, Ph.D.
Director

RLS/RM/ara

Attachment

cc: Thomas Crepeau/DSHWM Central File, Ohio EPA
Rebecca Strom, USEPA, Region V
Debby Berg, NEDO, Ohio EPA
Dave Wertz, NEDO, Ohio EPA
Randy Meyer, DSHWM, Ohio EPA
Brian O'Niell, Burk, Huber & Berick

I certify this to be a true and accurate copy of the official document as filed in the records of the Ohio Environmental Protection Agency.

By: Mary Cover Date 1-26-88

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ATTACHMENT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM #8

DATE: July 1985

SUBJECT: Notes on RCRA Methods and QA Activites

FROM: David Friedman, Manager
Methods Program (WH-562B)

TO: Addressees

Today's memo will cover the following subjects:

- Interim Thresholds for Toxic Gas Generation Reactivity (§261.23(a)(5))
- Test Method to Determine Hydrogen Cyanide Released from Wastes
- Test Method to Determine Hydrogen Sulfide Released from Wastes
- Revised RCRA Method 8280, Method of Analysis for Chlorinated Dibenzo-p-dioxins and Dibenzofurans

Over the past year, we have received many inquiries about how to evaluate wastes for reactivity (§261.23(a)(5)). We have initiated a number of studies in this area, and expect to propose a quantitative threshold for toxic gas generation reactivity in December of this year. On an interim basis, however, we feel stongly that wastes releasing more than the following levels of toxic gas should be regulated as hazardous wastes:

Total Available Cyanide: 250 mg HCN/Kg waste
Total Available Sulfide: 500 mg H_2S/Kg waste

The available cyanide or sulfide should be measured using the attached draft testing methods. Work currently being done on the agitation and waste introduction steps may result in significant changes in the subsequent proposed test. However, pending the conclusion of the investigations, we recommend use of this draft procedure.

I have attached a brief outline of the methodology we have employed to derive these interim thresholds. Work on estimating dispersion factors, however, is currently in progress. Any comments or suggestions you may have with respect to either the draft test method or the approach to establishing thresholds would be appreciated.

As a result of single laboratory evaluation of Method 8280 (reported in Memorandum #7), the method has been condensed and rewritten. The attached revised method is now being familarized in three laboratories and a three laboratory confirmation study will be initiated October 1, 1985. In the interim it should be used in lieu of Method 8280 published in Federal Register, April 4, 1983. The EMSL-Las Vegas group is concurrently documenting detection limits in "typical" matrix types.

While you may want to be flexible in your application of these levels and the attached test method, we believe the levels should apply in most cases. Should you have any specific questions, please call me at FTS 382-4770 (202-382-4770).

Attachment

Attachment

Mismanagement scenario:

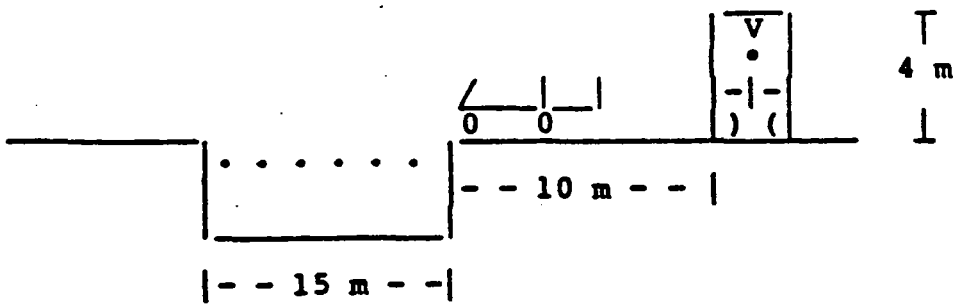
A truckload of waste is discharged into a pit containing acidic waste. As a result of the reaction of the waste with the acid, a rapid, high level release of toxic gas ensues. The objective of the characteristic is to identify those wastes which, if such an activity were to take place, pose a hazard to those persons in the general vicinity of the disposal site.

Assume:

1. The truckload of waste contains 6130 Kg of waste (about a 5 yd^3 dump truck @ 100 lbs/ft^3).
2. The velocity of the wind is 150 cm/sec (3.4 mph).
3. A person is standing 10 meters from the edge of the disposal pit.
4. Exposure to concentrations of:
   HCN above 10 mg/m^3 or
   H_2S above 20 mg/m^3
   pose an acute, immediate danger to human health.
5. The area of the pit over which the toxic gas is generated covers 225 m^2.
6. Before reaching an exposed individual the plume of contaminated air disperses, in a linear manner, to a height of 4 meters.

Then:

1. The minimum toxic gas release rate that would have to be present to exceed the danger level can be calculated using the following model:



2. Total Available Toxicant level then that poses a hazard can be calculated as follows:

V is a hypothetical volume of air to which an individual is exposed. Since the pit is 15 meters wide, and V is assumed to be 1.5 m thick, V = 15 m wide x 4 m high x 1.5 m thick = 90 m^3.

is the time it takes for a given volume of air to travel across the surface of the pit and become contaminated with toxic gas. Since the wind speed is 150 cm/sec, and the volume slice is assumed to be 1.5 m thick, T = 10 seconds.

C is concentration in mg/m^3 of toxicant that poses a danger.

A is the amount of toxicant contained in V when V is contaminated to a level that poses a health hazard. A = V x C. Since a given "slice" of air takes 10 seconds to move across the pit, this amount of toxicant can be generated over 10 seconds.

M is mass of waste dumped into the pit.

R is the total available toxicant necessary to pose a hazard as measured using the attached test protocol.

$$= \frac{\text{Amount of toxic gas that has to be released/length of test}}{\text{Mass of waste available to release } H_2S}$$

$$= \frac{(A)(1800/T)}{(M/\text{Percent of pit area available to contaminate air volume in any given unit of time})}$$

$$= \frac{(V)(C)(1800/T)}{(M/10)}$$

$$= \frac{(90)(C)(1800/10)}{(6130/10)}$$

$$= \frac{(90)(C)(180)}{(613)}$$

$$= 26.4\ (C)$$

= 264 mg/Kg total available cyanide

= 528 mg/Kg total available sulfide

3. As an added margin of safety, we accordingly recommend the action levels of:

Total Available Cyanide: 250 mg HCN/Kg waste
Total Available Sulfide: 500 mg H_2S/Kg waste

I certify this to be a true and accurate copy of the official document filed in the records of the Ohio Environmental Protection Agency.

By: Mary Cross Date 1-26-88

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TEST METHOD TO DETERMINE HYDROGEN CYANIDE RELEASED FROM WASTES

1. Scope and Application

1.1 This method is applicable to all wastes with the conditions that waste which are combined with acids do not form explosive mixtures.

1.2 This method provides a way to determine the specific rate of release of hydrocyanic acid upon contact with an aqueous acid.

1.3 This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

2. Summary of Method

2.1 An aliquot of the waste is acidified to pH 2 in a closed system. The gas generated is swept into a scrubber. The analyte is quantified. The procedure for quantifying the cyanide is Method 9010 starting with Step 7.3.5 of that method (attached).

3. Sample Handling and Preservation

3.1 Samples containing, or suspected of containing sulfide or a combination of sulfide and cyanide wastes, should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and stoppered. Analysis should commence as soon as possible; and samples should be kept in a cool, dark place until analysis begins.

3.2 It is suggested that sample cyanide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base, this will cause dilution of the sample, increase the ionic strength and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid. Storage of samples should be under refrigeration and in the dark.

3.3 Testing should be in a ventilated hood.

4. Apparatus (see Figure 1)

4.1 Three-neck, round-bottom flask with 24/40 ground-glass joints, 500 ml.

4.2 Stirring apparatus to achieve approximate 30 rpm. This may be a rotating magnet and stirring bar combination or an overhead motor driven propellor stirrer.

4.3 Separatory funnel with pressure equalizing tube and 24/40 ground glass joint and Teflon sleeve.

4.4 Flexible tubing for connection from nitrogen supply to apparatus.

4.5 Water pumped or oil pumped nitrogen gas with two-stage regulator.

5. Reagents

5.1 Sulfuric Acid 0.005 M

5.2 Cyanide reference solution: Dissolve approximately 2.5 gm KOH and 2.51 gm KCN in one liter of distilled water. Cyanide concentration in this solution is 1 mg/ml.

5.3 NaOH solution, 1.25N: Dissolve 50 gm NaOH in distilled water and dilute to 1 liter with distilled water.

5.4 NaOH solution, 0.25 N: Dilute 200 ml of sodium hydroxide solution to 1 liter with distilled water.

5.5 Stock cyanide solution, 1 mg/ml: Dissolve 2.51 gm KCN and 2 gm KOH in 1 liter of distilled water. Standardized with 0.0192 N $AgNO_3$. Dilute to appropriate concentration so that 1 ml = 1 mg CN.

5.6 Intermediate cyanide solution: Dilute 50 ml of stock solution to 1 liter with distilled water.

5.7 Standard cyanide solution, 5 mg/L: Prepare fresh daily by diluting 100 ml of intermediate solution to 1 liter with distilled water and store in a glass-stoppered bottle.

5.8 Silver nitrate solution: Prepare by crushing approximately 5 gm of $AgNO_3$ crystals and drying to constant weight at 40°C. Weigh 3.3 gm dried $AgNO_3$, dissolve in distilled water and dilute to 1 liter.

5.9 Rhodanine indicator: Dissolve 20 mg p-dimethylamino benzalrhodanine in 100 ml of acetone.

5.10 Methyl red indicator: Prepare 0.02 gm dissolved in 60 ml distilled water and 40 ml acetic acid.

6. System Check

6.1 The operation of the system can be checked using the cyanide reference solution. The reference solution can be used to verify system operation.

7. Procedure

7.1 Add 500 ml of 0.25N NaOH solution to a calibrated scrubber and dilute with distilled water to obtain an adequate depth of liquid.

7.2 Close the sytem and adjust the flow rate of nitrogen using the rotometer. Flow should be 60 ml/min.

7.3 Add 10 gm of the waste to be tested to the system.

7.4 With the nitrogen flowing, add enough acid to fill the system 1/2 full. While starting the 30-minute test period.

7.5 Begin stirring while the acid is entering the round bottomed flask.

7.6 After 30 minutes, close off the nitrogen and disconnect the scrubber. Determine the amount of cyanide in the scrubber by Method 9010 starting with step 7.3.5. of the method (attached).

8. Calculations

8.1 Determine the specific rate of release of HCN.

- Concentration of HCN in scrubber (mg/l) This is obtained from Method 9010 = A
- Volume of solution in scrubber (l) = L
- Weight of waste used (Kg) = W
- Time of measurement = Time N_2 stopped - Time N_2 started (seconds) = S

$$R = \text{specific rate of release} = \frac{A \cdot L}{W \cdot S}$$

Total available HCN = R·1800 mg/Kg

TEST METHOD TO DETERMINE HYDROGEN SULFIDE RELEASED FROM WASTES

1. Scope and Application

1.1 This method is applicable to all wastes with the conditions that waste which are combined with acids do not form explosive mixtures.

1.2 This method provides a way to determine the specific rate of release of hydrogen sulfide upon contact with an aqueous acid.

1.3 This procedure releases only the evolved hydrogen sulfide at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

2. Summary of Method

2.1 An aliquot of the waste is acidified to pH 2 in a closed system. The gas generated is swept into a scrubber. The analyte is quantified. The procedure for quantifying the sulfide is given in Method 9030.

3. Sample Handling and Preservation

3.1 Samples containing, or suspected of containing sulfide wastes, should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and stoppered. Analysis should commence as soon as possible; and samples should be kept in a cool, dark place until analysis begins.

3.2 It is suggested that samples of sulfide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base and addition of zinc acetate to the sample, this will cause dilution of the sample, increase the ionic strength and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrogen sulfide. Storage of samples should be under refrigeration and in the dark.

3.3 Testing should be in a ventilated hood.

4. Apparatus (See Figure 1)

4.1 Three-neck, round-bottom flask with 24/40 ground-glass joints, 500 ml.

4.2 Stirring apparaturs to achieve approximate 30 rpm. This may be a rotating magnet and stirring bar combination or an overhead motor driven propellor stirrer.

4.3 Separatory funnel with pressure equalizing tube and 24/40 ground glass joint and Teflon sleeve.

4.4 Flexible tubing for connection from nitrogen supply to apparatus.

4.5 Water pumped or oil pumped nitrogen gas with two-stage regulator.

4.6 Rotometer for monitoring nitrogen gas flow rate.

4.7 Industrial hygiene type detector tube for sulfide (100-2000 ppm range).

5. Reagents

5.1 Sulfuric Acid 0.005 M

5.2 Sulfide reference solution: Dissolve 4.02 gm of $Na_2S \cdot 9H_2O$ in 1.0 liters of distilled water. This is 680 ppm hydrogen sulfide. Dilute this stock solution to cover the analytical range required (100 ppm to 680 ppm).

5.3 NaOH solution, 1.25N: Dissolve 50 gm NaOH in distilled water and dilute to 1 liter with distilled water.

5.4 NaOH solution, 0.25N: Dilute 200 ml of sodium hydroxide solution to 1 liter with distilled water.

6. System Check

6.1 The operation of the system can be checked using the sulfide reference solution. The reference solution can be used to verify system operation.

7. Procedure

The procedure is dependent on the method chosen for quantification.

- If an adsorbent tube indicator is used for quantification, the analyst should start the procedure with Step 7.2.0
- If another procedure is chosen, the analyst should start the procedure with Step 7.1.0

7.1.0 Procedure employing scrubber solution with wet method quantification.

7.1.1 Add 500 ml of 0.25N NaOH solution to a calibrated scrubber and dilute with distilled water to obtain an adequate depth of liquid.

7.1.2 Assemble the system and adjust the flow rate of nitrogen using the rotometer. Flow should be 60 ml/min.

7.1.3 Add 10 gm of the waste to be tested to the system.

7.1.4 With the nitrogen flowing, add enough acid to fill the system 1/2 full, while starting the 30-minute test period.

7.1.5 Begin stirring while the acid is entering the round bottomed flask.

7.1.6 After 30 minutes close off the nitrogen and disconnect the scrubber. Determine the amount of sulfide in the scrubber by Method 9030 following methods.

7.1.7 Go to Section 8.1 for calculation of specific rate of release.

7.2.0 Procedure employing dry adsorbent indicator tube for quantification.

7.2.1 Assemble the system with the adsorber tube in place, making sure that the tube has the proper orientation (see manufacturer's literature).

7.2.2 Adjust the flow rate of nitrogen to be 60 ml/minute using the rotometer.

7.2.3 Add 10 gm of waste to the system.

7.2.4 Start the test by adding enough acid of pH 2 to fill the round bottom flask half full.

7.2.5 After 30 minutes, read the length of the stain on the indicator tube. Follow the manufacturer's directions in determining the concentration of sulfide in the gas using the length of the stain and the amount of gas passed through the tube.

7.2.6 Go to Section 8.2 to calculate the specific rate of release.

8. Calculations

8.1 Determine the specific rate of release of H_2S.

- Concentration of H_2S in scrubber (mg/l) = A
  This is obtained from Method 376.1 or 376.2.

- Volume of solution in scrubber (l) = L

RCRA METHOD 8280 WITH REVISIONS BASED ON SINGLE LABORATORY TESTING: METHOD OF ANALYSIS FOR CHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS[1,2,3]

Method 8280

1. Scope and Applications

[1]This method is appropriate for the analysis of tetra-, penta-, hexa-, hepta-, and octachlorinated dibenzo-p-dioxins and dibenzofurans.

[2]Analytical protocol for determination of TCDD's in phenolic chemical wastes and soil samples obtained from the proximity of chemical dumps. T. O. Tiernan and M. Taylor. Brehm Laboratory. Wright State University. Dayton, Ohio 45435.

[3]Analytical protocol for determination of chlorinated dibenzo-p-dioxins and chlorinated dibenzofurans in river water. T. O. Tiernan and M. Taylor. Brehm Laboratory. Wright State University. Dayton, Ohio 45435.

1.1 This method allows for the determination of chlorinated dibenzo-p-dioxins and chlorinated dibenzofurans in chemical wastes including still bottoms, filter aids, sludges, spent carbon, reactor residues, and in soils.

1.2 The sensitivity of this method is dependent upon the level of interferences.

1.3 This method is recommended for use only by analysts experienced with residue analysis and skilled in mass spectral analytical techniques.

1.4 Because of the extreme toxicity of these compounds, the analyst must take necessary precautions to prevent exposure to himself, or to others, of materials known to or believed to contain PCDD's or PCDF's. Typical infectious waste incinerators are probably not satisfactory devices for disposal of materials highly contaminated with PCDD's or PCDF's. Generators of 1 Kg or more of dioxin wastes must register as a generator. A laboratory planning to use these compounds should prepare a disposal plan to be reviewed and approved by EPA's Dioxin Task Force (Contact Conrad Kleveno, WH-548A, U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460). Additional safety instructions are outline in EPA Test Method 613.

2. Summary of the Method

2.1 This method is an analytical cleanup procedure and capillary column gas chromotography-low resolution mass spectrometry method, using capillary column GC/MS conditions and internal

standard techniques, which allow for the measurement of PCDD's and PCDF's in the extract.

2.2 If interferences are encountered, the method provides selected general purpose cleanup procedures to aid the analyst in their elimination. The analysis flow chart is shown in Figure 1.

3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Solvents distilled in all-glass systems are required.

3.2 Interferences co-extracted from the samples will vary considerably from source to source, depending upon the industrial process being sampled. PCDD and PCDF are often associated with other interfering chlorinated compounds such as PCB's and polychlorinated diphenyl ethers which may be at concentrations several orders of magnitude higher than that of the analytes. Retention times of analytes must be verified using standards. While general cleanup techniques are provided as part of this method, unique samples may require additional cleanup approaches such as HPLC, to achieve the sensitivity stated in Table 6.

3.3 Other isomers of tetrachlorodibenzo-p-dioxins may interfere with the measurement of 2,3,7,8-TCDD. Capillary column gas chromotography is required to resolve those isomers since they yield almost identical mass fragmentation patterns.

4. Apparatus and Materials

4.1 Sampling equipment for discrete or composite sampling.

4.1.1 Grab sample bottle--amber glass, 1-liter or 1-quart volume. French or Boston Round design is recommende The container must be washed and solvent rinsed before use to minimize interferences.

4.1.2 Bottle caps--threaded to screw onto the sample bottles. Caps must be lined with Teflon. Solvent washed foil, used with the shiny side toward the sample, may be substituted for the Teflon if sample is not corrosive.

4.1.3 Compositing equipment--automatic or manual compositing system. No tygon or rubber tubing may

be used, and the system must incorporate glass sample containers for the collection of a minimum of 250 mL. Sample containers must be kept refrigerated after sampling.

4.2 Water bath--heated, with concentric ring cover, capable of temperature control (+ or - 2°C). The bath should be in a hood.

4.3 Gas chromatograph/mass spectrometer data system.

4.3.1 Gas chromatograph: An analytical system with a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

4.3.2 Fused silica capillary columns are required. As shown in Table 1, four columns were evaluated using a column performance check mixture containing 1,2,3,4-TCDD, 1,2,3,4,7-PeCD, 1,2,3,4,7,8-HxCDD, 1,2,3,4,6,7,8-HpCDD, and 2,3,7,8-TCDF.

The columns include the following: (a) 50 m SP-Sil-88 programmed 60°-190° at 20°/minute, then 190°-240° at 5°/minute; (b) 30 m DB5 programmed 170° for 10 minutes, then 170°-280° at 8°/minute, hold at 280°C for 30 minutes; (c) 30 m SP-2240 programmed 70°-320° at 10°/minute. Column/conditions (a) provide good separation of 2,3,7,8-TCDD from the other TCDD's at the expense of longer retention times for higher homologs. Column/conditions (b) and (c) provide some separation of 2,3,7,8-TCDD. Resolution of 2,3,7,8-TCDD from the other TCDD's is better on column (c), but column (b) is more rugged, and may provide better separation of certain classes of interferences from the analytes and is recommended.

4.3.3 Mass spectrometer: Capable of scanning from 45 to 450 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the GC inlet. The system must also be capable of selected ion monitoring (SIM) for at least 5 ions simultaneously, with a cycle time of 1 sec or less. Minimum integration time for SIM is 50 ms. Selected ion monitoring is verified by injecting 0.15 ng of native TCDD to give a minimum signal-to-noise ratio of 5 to 1 at mass 320.

4.3.4 GC/MS interface: Any GC-to-MS interface that gives

acceptable calibration points for each compound of interest at concentration monitored and achieves acceptable tuning performance criteria (see Sections 6.1-6.3) may be used. GC-to-MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane. Inserting a fused silica column directly into the MS source is recommended.

4.3.5 Data system: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be able to integrate the abundance, in any EICP, between specified time or scan number limits.

4.3.6 High Performance Liquid Chromatography: HPLC pump with loop valve injector.

4.4 Apparatus Pipettes-Disposable, Pasteur, 150 mm long x 5 mm ID (Fisher Scientific Company, No. 13-678-6A or equivalent).

4.5 Amber glass bottle (500 mL, Teflon-lined screw cap).

4.6 Reacti-vial 1 mL, amber glass (silanized) (Pierce Chemical Company).

4.7 500 mL Erlenmeyer flask (American Scientific Products cat #f4295-500f0) fitted with Teflon stoppers (ASP #s 9058-8 or equivalent).

4.8 Wrist Action Shaker (VWR #57040-049 or equivalent).

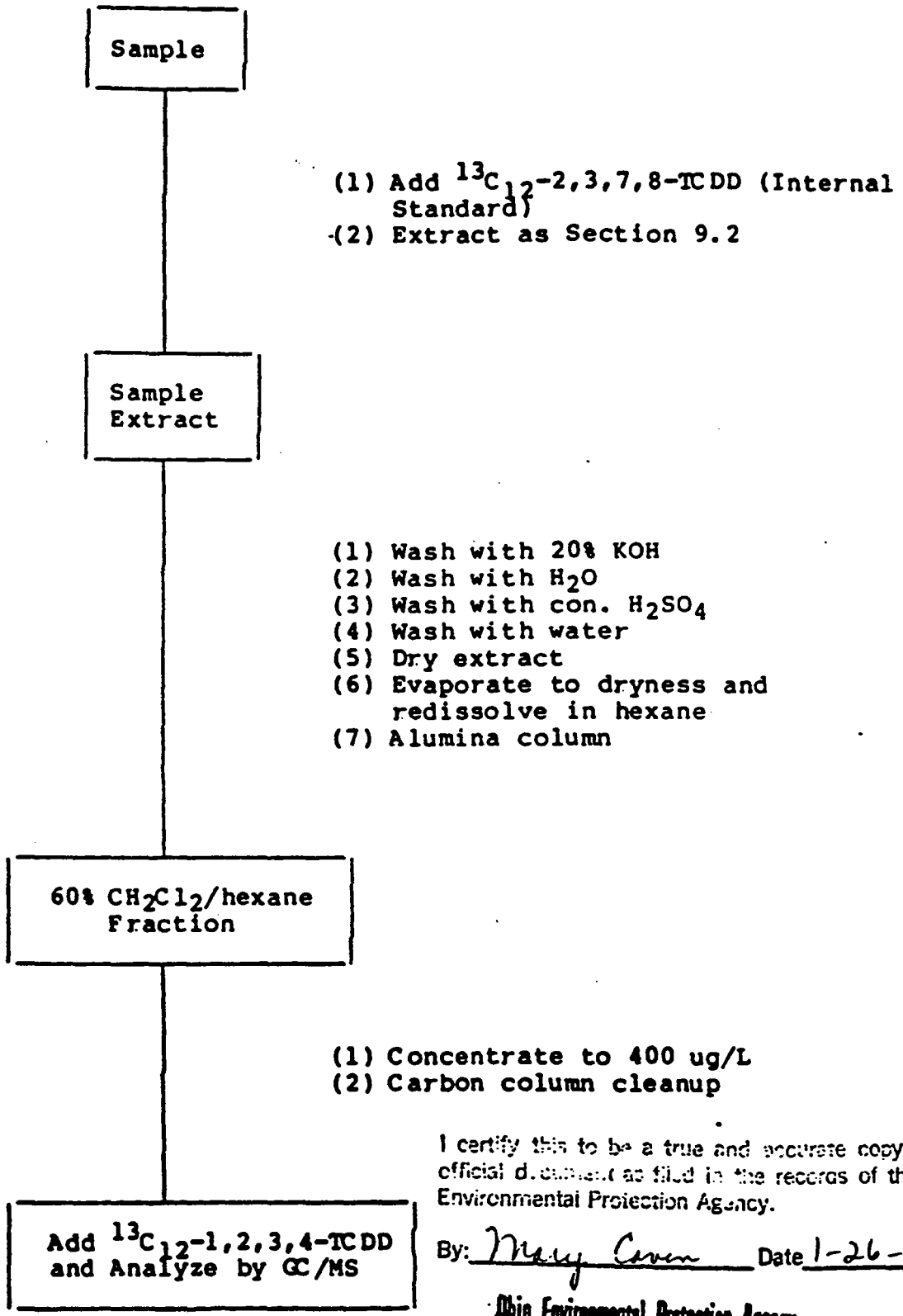
4.9 125 mL Separatory Funnels (Fisher (10-437-5b or equivalent).

4.10 500 mL Kuderna-Danish fitted with a 10 mL concentrator tube and 3-ball Snyder column (Ace Glass #6707-02, 6707-12, 6575-02 or equivalent).

4.11 Teflon boiling chips (Berghof American #15021-450 or equivalent). Wash with hexane prior to use.

4.12 300 mm x 10.5 mm glass chromatographic column fitted with Teflon stopcock.

Figure 1. Revised Method 8280 Analysis Flow Chart



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By: Mary Caven Date 1-26-88

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4.13 15 mL conical concentrator tubes (Kontes #K-288250 or equivalent).

4.14 Adaptors for concentrator tubes (14/20 to 19/22) (Ace Glass #9092-20 or equivalent).

4.15 2-Ball micro-Snyder columns (Ace Glass #6709-24 or equivalent).

4.16 Nitrogen evaporator (N-Evap #1156 or equivalent). Teflon tubing connection to trap and gas regulator is required.

4.17 Microflex conical vials (Kontes K-749000 or equivalent).

4.18 Filter paper (Whatman #54 or equivalent).

4.19 Carbon Column: An HPLC column (4.6 mm x 7 cm, stainless steel), prepared by mixing 5 percent (by weight) active carbon PX-21 (Amoco Research Corporation, Chicago, Illinois, or the equivalent active carbon AX-21, washed wtih methanol and dried in vacuo at 110°C, Anderson Development Co., Adrian, Michigan) and 10 um silica (Spherisorb S 10 W from Phase Separations, Inc., Norwalk, Connecticut). The materials must be stirred and sieved through a 40 um screen to remove any clumps.4/

4.20 Dean-Stark trap, 10 mL with T joints, condenser and 125 mL flask.

5. Reagents

5.1 Potassium hydroxide-(ACS), 20 percent (w/v) in distilled water.

5.2 Sulfuric acid-(ACS), concentrated.

5.3 Methylene chloride, hexane, benzene, petroleum ether, methanol, tetradecane, isoctane, toluene. Distilled in glass.

5.4 Prepare stock standards in a glovebox from cencentrates or neat materials. The stock solutions are stored in the dark at 4°C, and checked frequently for signs of degradation or evaporation, especially just prior to the preparation of working standards.

4/ The carbon column preparation and use is adapted from W. A. Korfmacher, L. G. Rushing, D. M. Nestorick, H. C. Thompson, Jr., R. K. Mitchum, and J. R. Kominsky, Journal of High Resolution Chromatography and Chromatography

5.5 Alumina, neutral, Super 1, Woelm, 80/200 mesh. Store at room temperature in a desiccator wtih $CaSO_4$ drying agent. Oven drying at 600°C overnight is acceptable, but alumina so processed should be checked for contamination by solvent rinsing and GC/ECD analysis.

5.6 Prepurified nitrogen gas.

5.7 Anhydrous sodium sulfate (reagent grade). Extracted overnight with hexane using a Soxhlet extraction apparatus and dried at 100°C.

6. Calibration

6.1 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from reagents. Both open column and carbon column performance must be checked. Routinely check the 8 percent CH_2Cl_2/hexane eluate of environmental extracts from the alumina column for presence of analytes.

6.2 Prepare multi-level calibration standards5/ keeping the recovery standard ($^{13}C_{12}$-1,2,3,4-TCDD) and the internal standard ($^{13}C_{12}$-2,3,7,8-TCDD) at fixed concentrations of 500 ng/mL. Recommended concentration levels for standard analytes are 200, 500, 1000, 2000, and 5000 ng/mL. Calculation of response factors is described in Section 11.1. Standards must be analyzed using the same solvent as used in the final extract, toluene is required.

6.3 Establish operating parameters of the GC.MS apparatus as indicated in Section 10.1 of this method. The instrument should be tuned as described in Table 2 by the use of decafluorotriphenyl phosphine (DFTPP). By injecting calibration standards, establish the standard response factors vs. $^{13}C_{12}$-2,3,7,8-TCDD (PCDF response factors are established, vs. $^{13}C_{12}$-TCDF is this standard is used). An adequate detection limit should be verified by injecting 0.15 ng of $^{13}C_{12}$-TCDD which should give a minimum signal to a noise ratio of 5 to 1 at mass 332 or 334. GC column performance should be checked for resolution and peak shape daily using a mixed standard such as the GC column performance check mixture described in Section 4.3.2.

5/ $^{13}C_{12}$-labeled TCDD is available from Cambridge Isotope Laboratory, Woburn, Massachusetts. Proper standardization requires the use of a specific labeled isomer for each congener to be determined. When labeled PCDD's and PCDF's of each homolog are available, their use will be required

removed. Cool the sample, filter the toluene solution through Whatman #54 filter paper (or equivalent) into a 100 mL round bottom flask. Concentrate the extract to just dryness using a rotary evaporator at 50°C. Proceed with Step 9.2.4.

9.2.2 Still bottom. Extract the still bottom sample by mixing 100 mg of sample wtih 10 mL of toluene and filtering the solution through Whatman #54 filter paper (or equivalent) into a 50 mL round bottom flask. Rinse the filter wtih 5 mL of toluene. Concentrate the combined toluene solution to just dryness using a rotary evaporator at 50°C. Proceed with Step 9.2.4.

9.2.3 Fly ash. Extract the fly ash sample by placing 10 g of sample and 10 g of anhydrous sodium sulfate in a Soxhlet extraction apparatus charged with toluene and extract 16 hours. Cool and filter the toluene extract through Whatman #54 filter paper (or equivalent) into a 500 mL round bottom flask. Rinse the filter with 5 mL of toluene. Concentrate the combined toluene solution just to dryness using a rotary evaporator at 50°C. Proceed with Step 9.2.4.

9.2.4 Transfer the residue to a 125 mL separatory funnel using 15 mL of hexane. Rinse the flask with 2-5 mL aliquots of hexane and add the rinses to the funnel. Shake 2 minutes with 50 mL of 5% NaCl solution, discard the aqueous layer and proceed with Step 9.3.

9.2.5 Soil. Extract soil samples by placing 10 grams of sample and 10 grams of anydrous sodium sulfate in a 500 mL Erlenmeyer flask fitted with a Teflon™ stopper. Add 70 mL of petroleum ether and 30 mL for methanol, in that order, to the Erlenmeyer flask. Shake on a wrist-action shaker for two hours. The solid portion of sample should mix freely. If a smaller soil aliquot is used, scale down the amount of methanol proportionally.

9.2.5.1 Filter the extract from Section 9.2.5 through a glass funnel fitted with filter paper (Whatman #54 or equivalent) and filled with a 10 mL concentrator tube. Add 50 mL of petroleum ether to the Erlenmeyer flask, restopper the flask and swirl the sample gently, remove the stopper carefully and decant the solvent through the funnel as above. Wash the sodium sulfate on the funnel with two

9.2.5.2 Add a Teflon boiling chip and a three-ball Snyder column to the KD flask. Concentrate in a 70°C steam bath to an apparent volume of 10 mL. Remove the apparatus from the steam bath and allow it to cool for 5 minutes.

9.2.5.3 Add 50 mL of hexane and a new boiling chip to the KD flask. Concentrate in a 100°C steam bath to an apparent volume of 10 mL. Remove the apparatus from the steam bath and allow to cool for 5 minutes.

9.2.5.4 Remove and invert the Snyder solumn and rinse it down into the KD with two, 1 mL portions of hexane. Decant the contents of the KD and concentrator tube into a 125 mL separatory funnel. Rinse the KD with two additional five mL portions of hexane, combine. Proceed with Step 9.3.

9.3 Partition the solvent against 40 mL of 20 percent (w/w) potassium hydroxide. Agitate for two minutes. Remove and discard the aqueous layer (bottom).

9.4 Partition the solvent against 40 mL of distilled water. Agitate for two minutes. Remove and discard aqueous layer (bottom).

9.5 Partition the solvent against 40 mL of concentrated sulfuric acid. Agitate for two minutes. Romove and discard the aqueous layer (bottom). Repeat the acid washings until no color is visible in the acid layer.

9.6 Partition the extract against 40 mL of distilled water. Agitate for two minutes. Remove and discard aqueous layer (bottom). Dry the organic layer by pouring through a funnel containing anhydrous sodium sulfate, wash with two 5 mL portions of hexane, and concentrate the hexane solution to near dryness with a rotary evaporator (35°C water bath).

9.7 Pack a gravity column (glass 300mm x 10.5mm), fitted with a Teflon stopcock, in the following manner:

Insert a glass-wool plug into the bottom of the column. Add a 4 gram layer of sodium sulfate. Add a 3.6 gram layer of Woelm super 1 neutral alumina. Tap the top of the column gently. Woelm super neutral alumina need not be activated or cleaned prior to use but should be stored and sealed in a desiccator. Add a 4 gram layer of sodium sulfate. Elute wtih 20 mL of hexane and close the stopcock just prior to the exposure of the sodium sulfate layer to air. Discard the effluent.

Check the column for channeling. If channeling is present discard the column. Do not tap on a wetted column.

9.8 Dissolve the residue from 9.6 in 2 mL of hexane and apply the hexane solution of the sample to the top of the column. Elute with enough hexane to transfer the sample cleanly to the surface of the alumina. Discard the effluent.

9.8.1 Elute with 10 mL of 8 percent methylene chloride by volume in hexane. Check that no PCDDs or PCDFs eluted in this fraction as a quality assurance step.

9.8.2 Elute the PCDDs and PCDFs from the column with 15 mL of 60 percent (v/v) methylene chloride in hexane and collect this fraction in a conical shaped (15 mL) concentrator tube.

9.9 Carbon column cleanup.

9.9.1 Using N_2, gently concentrate both fractions from the alumina column (Section 9.8) to about 1 mL. Wash sides of tube down wtih a small volume (100-300 uL) of hexane and reconcentrate to about 1 mL. Save the 8 percent fraction for GC/MS injection to check for any bleedthrough of PCDD and PCDFs (a quality assurance step). Evaporate the 60 percent CH_2Cl_2/hexane fraction to about 400 uL and transfer to HPLC injector loop (1.0 mL) for carbon column cleanup. Rinse the centrifuge tube with 500 uL hexane, and add this to HPLC injector loop.

9.9.2 Elute the column at 2 mL/minute, ambient temperature, with 30 mL of cyclohexane/hexane 1:1 (v/v). Discard eluate. Next elute the column with 10 mL of CH_2Cl_2/MeOH/Benzene 70:20:5 (v/v). Discard eluate. Backflush the column wtih 40 mL toluene to elute and collect PCDDs and PCDFs (entire fraction). The solumn is cleaned by pumping an additional 30 mL methanol followed by 40 mL of toluene in the back flush position. After returning the column to the original position, 30 mL of cyclohexane/hexane 1:1 (v/v) is pumped through the column to re-equilibriate it in preparation for the next sample.

9.9.3 Evaporate the toluene fraction to about 1 mL on a rotary evaporator using a water bath at 45°C. Transfer to a 2.0 mL reacti-vial using toluene and concentrate to the desired volume using a stream of N_2.

327, 372, 374, 376, 388, 390, 392 (HexCDDs and HexCDFs); and (4) 361, 379, 422, 424, 426, 440, 442, 444 (HepCDDs and Octa CDFs). Cycle time less than 1 second/descriptor.

10.5 In those circumstances where these procedures do not yield a definitive conclusion, the use of high resolution mass spectrometry or HRGC/MS/MS is suggested.

11. Calculations

11.1 Determine the concentration of individual compounds according to the formulas:

$$\text{Concentration, ng/g} = \frac{Q_{is} \times A_s}{G \times A_{is} \times R_f}$$

Where:

Q_{is} = ng of internal standard $^{13}C_{12}$-2,3,7,8-TCDD, added to the sample before extraction.

G = g of sample extracted.

A_s = area of characteristic ion of the compound of interest.

A_{is} = area of characteristic ion (m/z-334) of the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD.

R_f = response factor of the characteristic ion of the compound of interest relative to the m/z 334 of $^{13}C_{12}$-2,3,7,8-TCDD.

Response factors are calculated using data obtained from the analysis of standards according to the formula:

$$R_f = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

C_{is} = concentration of the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD.

C_s = concentration of the compound of interest.

Calculate recovery of the internal standard, R_{is}, $^{13}C_{12}$-2,3,7,8-TCDD, in the sample extract, using for formula:

$$R_{is} = \frac{A_{is} \times Q_{rs}}{A_{rs} \times R_{fr} \times Q_{is}} \times 100$$

Where:

A_{rs} = Area of characteristic ion (m/z-334) of the recovery standard, $^{13}C_{12}$-1,2,3,4-TCDD.

Q_{rs} = ng of recovery standard, $^{13}C_{12}$-1,2,3,4-TCDD added to extract.

The response factor for determination of recovery is calculated using data obtained from the analysis of standards according to the formula:

$$Rf_r = \frac{A_{is} \times C_{rs}}{A_{rs} \times C_{is}}$$

Where:

C_{rs} = Concentration of the recovery standard, $^{13}C_{12}$-1,2,3,4-TCDD.

11.2 Report results in nanograms per gram without correction for recovery data. When duplicate and spiked samples are reanalyzed, all data obtained should be reported.

11.3 Accuracy and Precision. Table 3 gives the precision data for revised Method 8280 for selected analytes in the matrices shown. Table 4 gives recovery data for the same analyses. Table 5 give the linear range and variation of response factors over the range for selected analyted standard. Table 6 gives estimated detection limits.

TABLE 1. REPRESENTATIVE GAS CHROMATOGRAPH RETENTION TIMES* OF ANALYTES

Analyte	50 m CP-Sil-88	30 m DB-5	30 m SP-2250	30 m DB-225
2,3,7,8-TCDD	23.6	22.8	26.7	37.3
1,2,3,4-TCDD	24.1	22.7	26.5	37.6
1,2,3,4,7-PeCDD	30.0	25.2	28.1	NM
1,2,3,4,7-HxCDD	39.5	28.0	30.6	NM
1,2,3,4,6,7,8-HpCDD	57.0	34.2	33.7	NM
2,3,7,8-TCDF	25.2	22.5	26.7	42.5

*Retention time in minutes, using temperature programs shown below.

NM - not measured.

Temperature Programs:

CP-Sil-88	60°C-190°C at 20°/minute; 190°-240° at 5°/minute.
30 m DB-5	170°, 10 minutes; then at 8°/minute to 280°C, hold at 280°C 30 minutes.
SP-2250	70°-320° at 10°/minute.
DB-225	70°-230° at 10°/minute.

Column Manufacturers

CP-Sil-88	Chrompack Incorporated, Bridgewater, New Jersey
DB-5, DB-225	J&W Scientific, Incorporated, Rancho Cordova, California
SP-2250	Supelco, Incorporated, Bellefonte, Pennsylvania

TABLE 2. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60 percent of mass 198
68	Less than 2 percent of mass 69
70	Less than 2 percent of mass 69
127	40-60 percent of mass 198
197	Less than 1 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5-9 percent of mass 198
275	10-30 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17-23 percent of mass 442

J. W. Eichelberger, L. E. Harris and W. L. Budde. 1975. Reference compound to calibrate ion abundance measurement in gas chromatography mass spectometry. Analytical Chemistry 47:995.

TABLE 3. PRECISION DATA FOR REVISED METHOD 8280

Compound	Matrix	Analyte Level (ng/g) Native	Native + Spike	N	Percent RSD

(DATA NOT AVAILABLE AT THIS TIME)

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By: Mary Cavin Date 1-26-88

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TABLE 4. RECOVERY DATA FOR REVISED METHOD 8280

Compound	Matrix[1]	Native[2] (ng/g)	Spiked[3] Level (ng/g)	Mean Percent Recovery

(DATA NOT AVAILABLE AT THIS TIME)

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TABLE 5. LINEAR RANGE AND VARIATION OF RESPONSE FACTORS

Analyte	Linear Range Tested (pg)	n**	Mean RF	%RSD
1,2,7,8-TCDF*	50-6000	8	1.634	12.0
2,3,7,8-TCDD*	50-7000	7	0.721	11.9
2,3,7,8-TCDF	300-4000	5	2.208	7.9

*Response factors for these analytes were calculated using 2,3,7,8-TCDF as the internal standard. The response factors for 2,3,7,8-TCDF were calculated vs. $^{13}C_{12}$-1,2,3,4-TCDD.

**Each value of n represents a different concentration level.

TABLE 6. DETECTION LIMITS (ppb) FOR RCRA METHOD 8280[1,2]

Analyte Class	Clay	Soil	Fly Ash	Still Bottom[3]	Sludge
TCDD	1.0	5.0	1.0	500	25
TCDF	0.5	2.5	0.5	250	12
PeCDD	1.5	7.5	1.5	750	38
PeCDF	1.0	5.0	1.0	500	25
HxCDD	2.0	10.0	2.0	1000	50
HxCDF	1.5	7.5	1.5	750	38

[1]The analytes of the class indicated were not quantified below this value. The instrument detection limit (S=3N) for 2,3,7,8-TCDD in standards is 0.5 ppb when extrapolated for a 10 g sample concentrated to 100 uL.

[2]Matrix types:

Clay: Pottery clay, Westwood Ceramic Supply Co., City of Industry, California.

Soil: Times Beach, Missouri, soil blended to form a homogeneous sample. This sample was analyzed as a performance evaluation sample for the Contract Laboratory Program (CLP) in April 1983. The results from EMSL-LV and 8 contract laboratories using the CLP protocol were 305.8 ng/g 2,3,7,8-TCDD with a standard deviation of 81.0. The 90 percent window was 143 to 469 ng/g.

Fly Ash: Ash from an incinerator in Tennessee, resource recovery ash #1, as described in Appendix B.

Still Bottom: Distillation bottoms (tar) from 2,4-dichlorophenol production ARB-12-14-04, from Arthur D. Little, Inc.

Sludge: Sample B-9b from J. H. Baxter, Eugene, Oregon, sludge from cooling tower which received both creosote and pentachlorophenolic wastewaters.

[3]The still bottom samples were not tested below this level due to high analyte levels found.

Yvonne C. Billingsley
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July 3, 1987

Ms. Debbie Berg
Ohio EPA
Northeast District Office
2110 E. Aurora Road
Twinsburge, Ohio 44087-1969

RE: Specialized Finishers Inc./
Thomas J. Foley

CLOSURE PLAN
ID. No. OH.D.O. 13550371

Dear Ms. Berg:

Enclosed please find a copy of the Closure Plan involving waste contained at the above-captioned facility.

Hopefully, the Closure Plan will be approved, or require very little revision. Upon your review of same, I would appreciate any comments or suggestions that you may have that will help to expedite approval of the Closure Plan.

Thank you for your courtesy and cooperation in this matter.

Sincerely,

Yvonne C. Billingsley

YVONNE C. BILLINGSLEY, ESQ.

YCB:an

Enclosure

CLOSURE PLAN

SPECIALIZED FINISHERS, INC.
2133-2139 Hamilton Avenue
Cleveland, Ohio 44114

ID No: OH. D. O. 13550371

1. DESCRIPTION OF FACILITY

Specialized Finishers, Inc. occupies two brick buildings, totalling 9,000 square feet, located at 2133-2139 Hamilton Avenue, Cleveland, Ohio 44114. The receptacles containing hazardous wastes are located at the 2139 Hamilton Avenue facility.

Specialized Finishers is an electroplating-metal finishing facility. The 2139 Hamilton Avenue facility processed customer parts for zinc plating, cadmium plating and cleaning. The plant closed its doors on March 20, 1987.

2. DESCRIPTION OF WASTE MANAGEMENT UNITS TO BE CLOSED

The hazardous waste tanks are located in the 2139 Hamilton Avenue facility. There are three above ground tanks and one concrete sump (pit) below the ground. The concrete sump contains 2,000 gallons filled to 24 inch freeboard. Two of the above ground tanks are made of steel and koroseal-lined, each containing 500 gallons of hazardous waste. The third above ground tank is made of fiberglass and contains 1,500 gallons filled to 24 inches freeboard.

3. MAP OF FACILITY

Attached and marked as Exhibit A is a map of the Hamilton Avenue facility in relationship to its location in the City of Cleveland. A copy of said map was taken from the Official Street Atlas of Cleveland and Cuyahoga County, 1986-1987 edition.

4. DRAWINGS OF UNITS TO BE CLOSED

Attached hereto and marked as Exhibits B, C and D are diagrams of the units and explanation of the locations and other points or structures on the facility property.

5. LISTS OF HAZARDOUS WASTES

D. The three tanks and one sump located at the 2139 Hamilton Avenue facility contain greenish liquids with the following composition: arsenic, selenium, lead, cadmium, chromium, silver, mercury, and barium. The foregoing is based upon a lab sample conducted on August 15, 1986 by Alchem Labs. See Exhibt E for additional details.

6. SCHEDULE FOR CLOSURE

Specialized Finishers, Inc. will dispose of all hazardous wastes in accordance with the approved closure plan within one week after approval of the closure plan. The transporter will be:

Alchem-Tron, Inc.
7415 Bessemer Avenue
Cleveland, Ohio 44127
(216) 441-5628

(See Exhibit F and G).

7. AIR EMISSIONS

There will be no air emissions nor nuisance problems such as dust or odors related to closure.

8. PERSONNEL SAFETY AND FIRE PREVENTION

As the Hamilton Avenue facility has been closed since March 20, 1987, no personnel other than Mr. Thomas Foley will be present at the time of closure. The other individuals on site will be the independent, registered engineer and employees of Alchem-Tron, Inc. The following safety and fire prevention precautions will be taken.

a. Spills: For chemical spills a non-reactive absorbent material will be utilized and readily available. Absorbed spills, if any, will then be transferred to a drum for proper disposal as hazardous waste.

b. Fire: Three (3) ABC fire extinguishers are on site.

c. Clothing: Workers will wear neoprene gloves, chemical resistant boots and clothing, safety goggles, and respirators.

d. Other: A first-aid kit and eye wash equipment will also be available.

There are no flammable solutions.

9. DECONTAMINATION EFFORTS

After removal of the hazardous waste from the three tanks and sump said containers will be decontaminated through a process of pressure washing. Numerous rinses will be conducted. A final rinse sample to ensure that the containers are free of any contaminants will be made by a registered, independent engineer. The engineer that will do sample

testing of the final rinse will be:

Mark Harransky
Eagle Technologies, Inc.
P.O. Box 1133
Mentor, Ohio 44061-1133

The three above ground tanks and the sump will be subjected to all reasonable means of decontamination. The above-mentioned independent engineer will certify the methods used and will ensure that the minimum amount of residue remains.

The plating baths have been contracted for sale to:

Accurate Plating Co.
6512 Carnegie Avenue
P.O. Box 03277
Cleveland, Ohio 44106
(216) 881-7400

(See Exhibit H).

The three (3) tanks above the ground will be sold to:

Frank Fanta
Fanta Equipment Co.
6521 Storer Avenue
Cleveland, Ohio 44102
(216) 281-1515

10. "CLEAN" LEVELS FOR SOIL

Based on prior inspections by Christine Frazier, Environmental Scientist, Division of Solid and Hazardous Waste Management of the Ohio EPA, there has been no soil contamination at the Hamilton Avenue facility.

The surfaces of the sump and the concrete flooring under the above ground tanks are inspected on a daily and weekly basis by Mr. Thomas Foley for signs of cracks and deterioration. In the event that any signs of cracks or deterioration are noted, Mr. Foley will immediately report same to the appropriate authorities.

11. SAMPLING PLAN AND ANALYTICAL METHODS

As stated in Item No. 10 there is no soil contamination. Frequent inspections are being made for leaks, spills or potential for waste or waste constituent migration.

Final rinses will be done, separately, to ensure that the tanks and sump are clean and free of any contaminants.

12. DESCRIPTION OF REMOVAL EFFORTS

As stated previously Specialized Finishers, Inc. has contracted the services of Alchem-Tron, Inc. to dispose of the hazardous waste materials located at the Hamilton Avenue facility.

Alchem-Tron will vacuum out the three above ground tanks and sump with a high-powered pump that is attached to a trailer tank. The waste will then be transported to Alchem-Tron's facility via the vacuumed trailer tank and blown out of the tanker into Alchem-Tron's treatment tanks.

13. SPECIFICS FOR LANDFILL CLOSURES

The Hamilton Avenue facility does not have any landfills or surface impoundments to be closed as landfills.

14. DESCRIPTION OF EQUIPMENT CLEANING

All equipment used by Alchem-Tron, Inc. for removal of the hazardous waste materials will be drained and cleaned out. Inasmuch as most plating materials are water-soluable, the cleaning process will primarily consist of high-pressured water rinsing.

15. CERTIFICATION

Closure activities at the Hamiliton Avenue facility will be supervised and certified by an independent, registered, professional engineer, and Mr. Thomas Foley, the owner/operator of Specialized Finishers, Inc.

The independent, registered engineer, Mark Harransky of Eagle Technologies, Inc., is not an employee of Specialized Finishers, Inc. Mr. Harransky is an "outside" consulting engineer.

The independent engineer will be present at all critical, major points (activities) during the closure, which include, but are not limited to, waste removal, final rinse sampling and clean-up. The frequency of inspections by the independent engineer will be sufficient to determine the adequacy of each critical activity.

The independent engineer or Mr. Thomas Foley will notify the Ohio EPA reviewer/inspector in advance of any critical activities, should this be required through the Closure Plan.

At the completion of closure, Mr. Foley and the registered, professional engineer will certify that all actions were done in accordance with the Closure Plan.

16. STATUS OF FACILITY AFTER CLOSURE

Specialized Finishers, Inc., closed its doors on March 20, 1987. Business will not resume after closure is completed. No units will remain in operation.

As stated in Item 9, the plating baths will be sold to Accurate Plating Company and the three above ground tanks will be sold to Fanta Equipment Company.

Upon completion of closure, a written withdrawal request will be made.

17. NUMBER OF COPIES OF PLAN

Three (3) copies of this Closure Plan were mailed this 3rd day of July, 1987 to:

Thomas E. Crepeau
Ohio EPA
Division of Solid and Hazardous Waste Management
Data Management Section
P.O. Box 1049
361 E. Broad Street
Columbus, Ohio 43266-1049

One copy was mailed this 3rd day of July, 1987 to each of the following:

Mr. David Wertz
Ms. Debbie Berg
Ohio EPA
Northeast District Office
2110 E. Aurora Road
Twinsburg, Ohio 44087-1969

and

Dominic J. Hanket
Assistant Attorney General
Environmental Enforcement Section
State Office Tower
30 East Broad Street
Columbus, Ohio 443266-0410

DOWNTOWN MAP

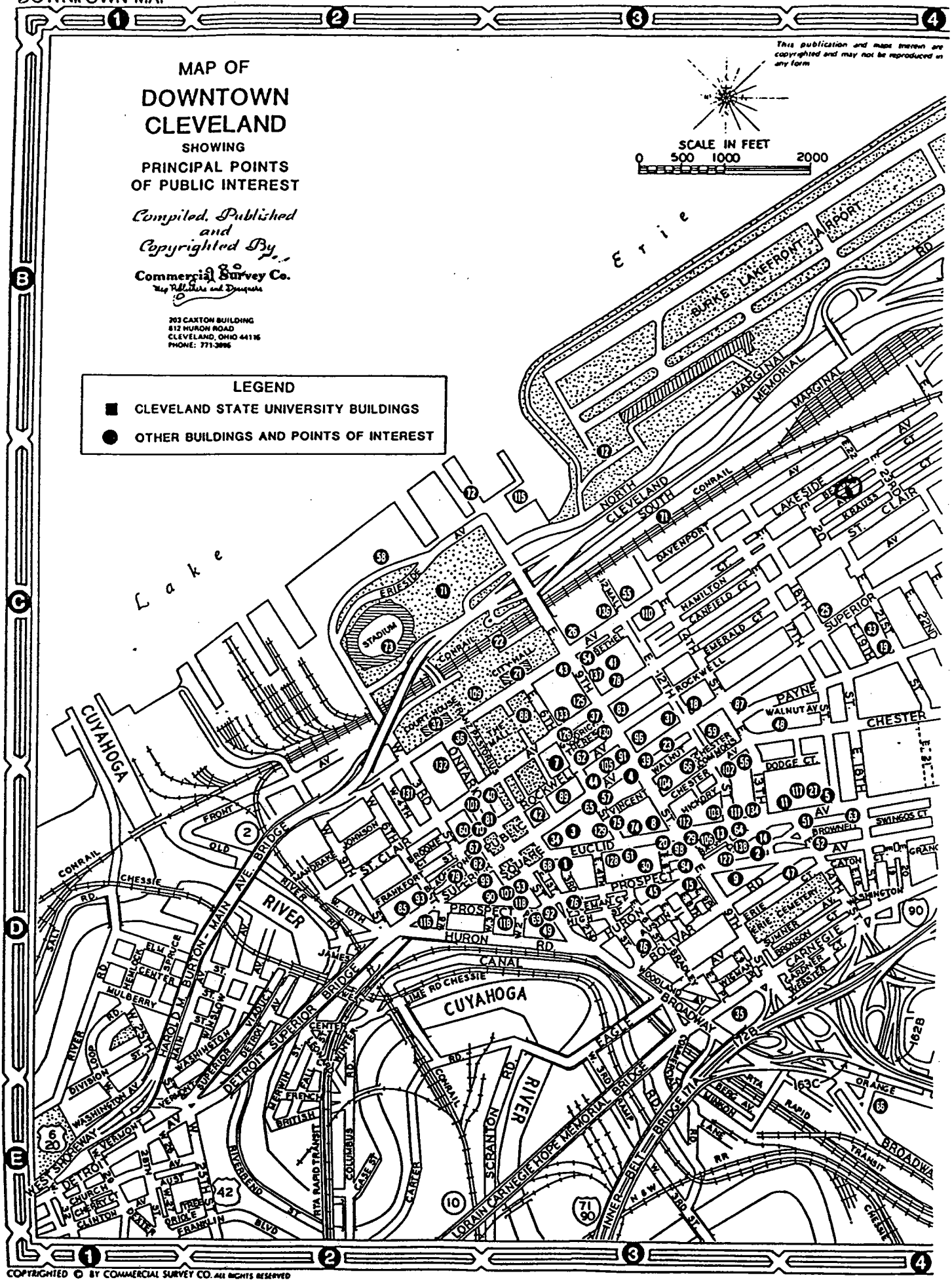
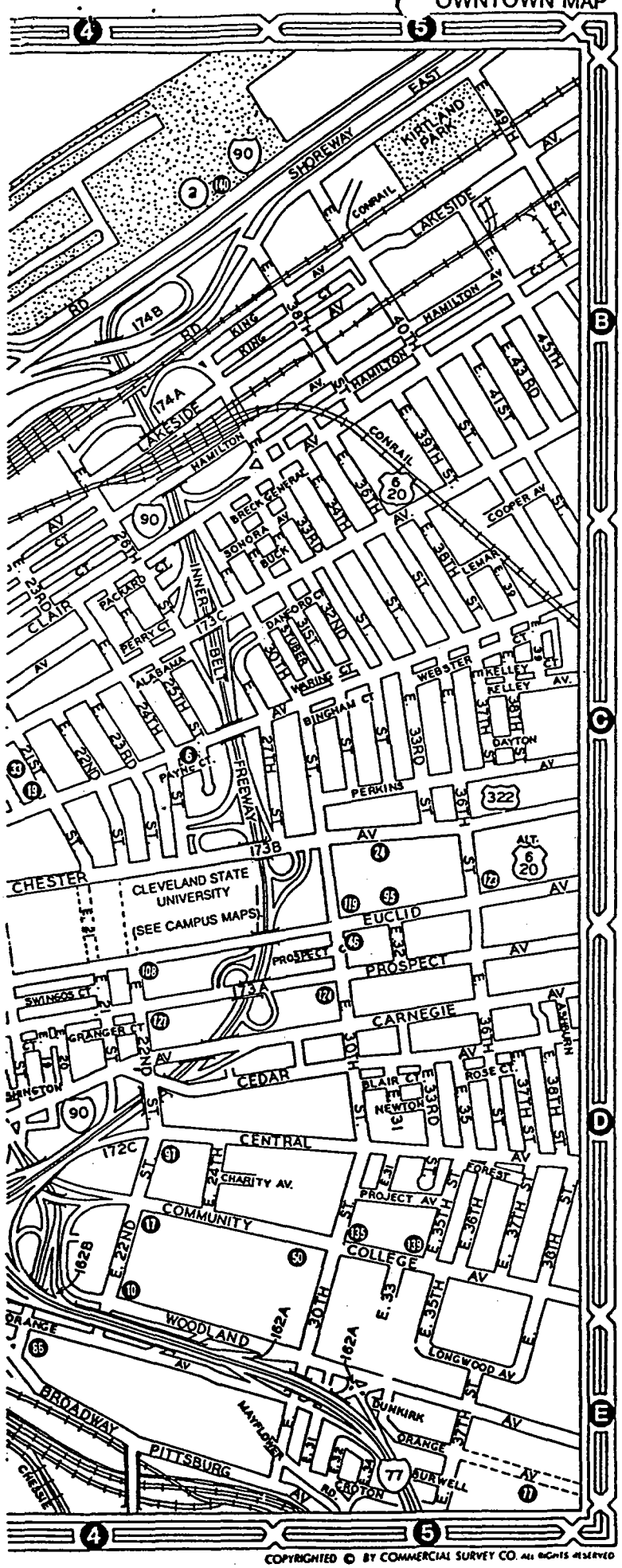
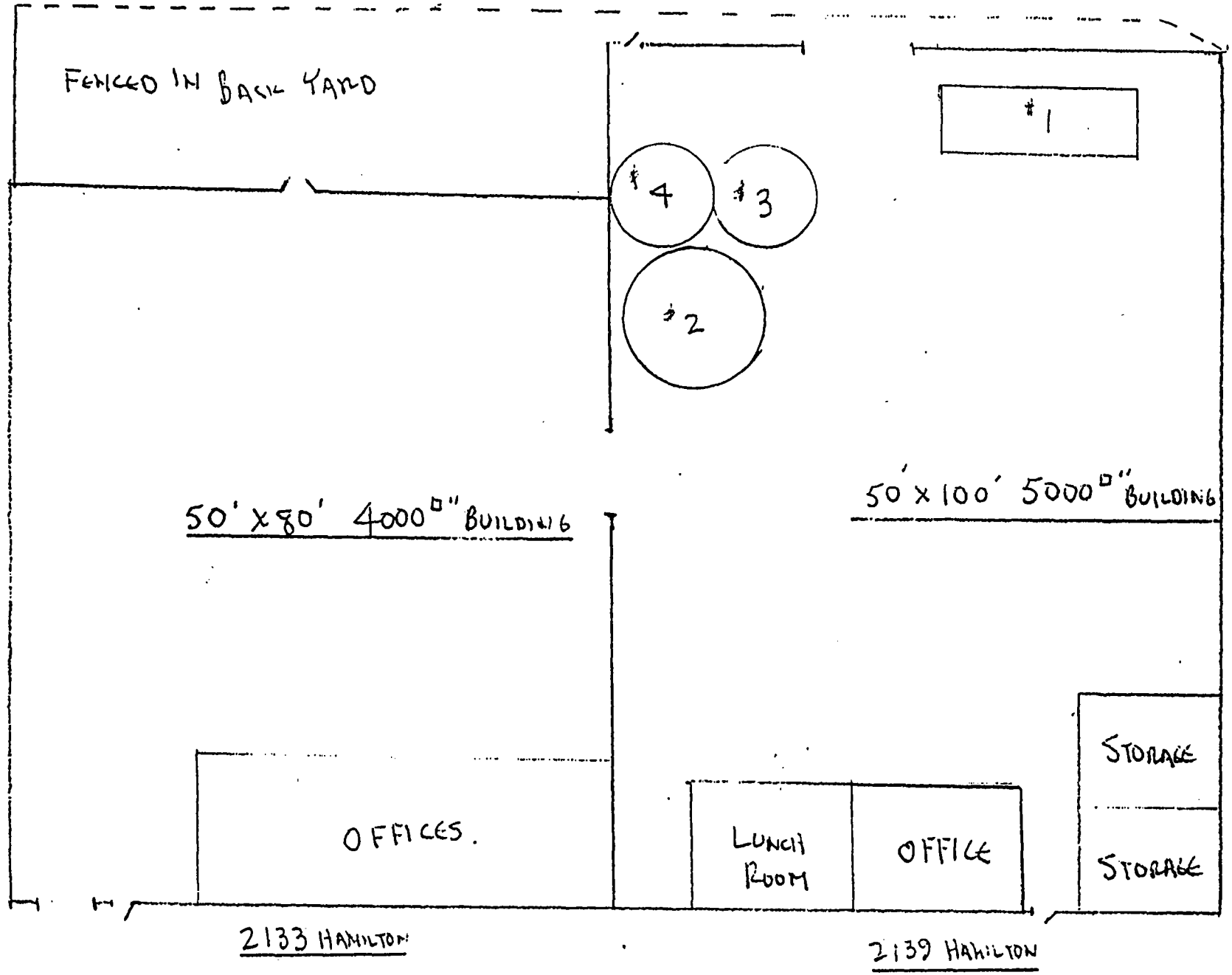


EXHIBIT "A"

OWNTOWN MAP





SEE NOTES: Exhibit "C"

SPECIALIZED FINISHERS	BOTH BUILDINGS	OVERHEAD VIEW

NOTES: SPECIALIZED FINISHERS, INC. - HAZARDOUS WASTE TANKS

Specialized Finishers occupies two brick buildings at 2133 and 2139 Hamilton Avenue, Cleveland, Ohio 44114

The Buildings are securely locked and have a fenced-in yard that is also kept locked.

The four hazardous waste tanks are located in the rear portion of the 2139 Building.

#1 Hazardous Waste Sump. Concrete pit below the ground containing 2,000 gallons filled to 24" freeboard, located three (3) feet from back wall.

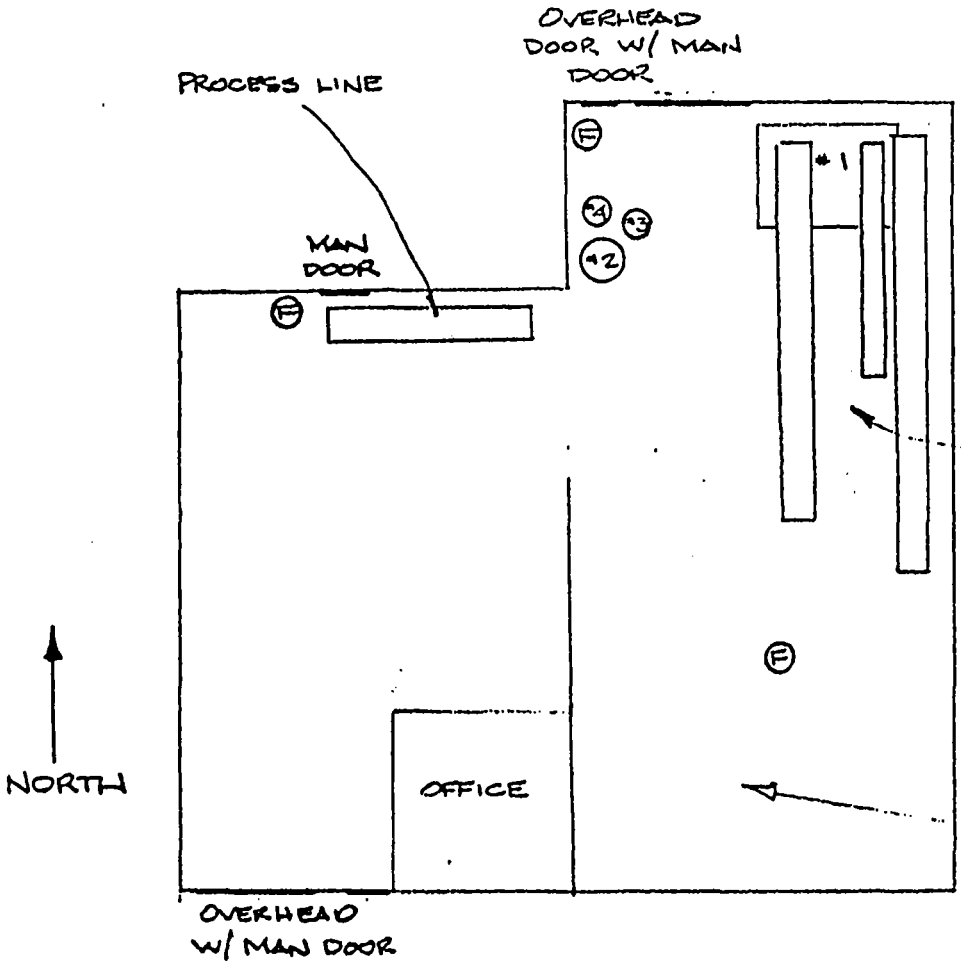
#2 Hazardous Waste Tank. Fiberglass above the ground tank containing 1,500 gallons filled to 24" freeboard, located 20 feet from back wall.

#3 and #4 Hazardous Waste Tanks. Koroseal lined steel tanks above the ground, both containing 500 gallons each located next to each other, fifteen (15) feet from back wall.

Total of 4,500 gallons hazardous waste contained in these four (4) tanks.

Approximately 1,500 gallons additional for cleanout, so that 6,000 gallons of hazardous waste will be hauled out by liquid tanker.

BUILDING LAYOUT / SAFETY EQUIPMENT LOCATION.



#1 THRU #4 - HAZARD WASTE STORAGE VESSELS.

PROCESS LINES (3)

Ⓕ - DENOTES FIRE EXTINGUISHER

WAREHOUSE AREA - LOCATION OF ABSORBANT MATERIAL & FIRST AID KIT.

HAMILTON AVE.

SPECIALIZED FINISHERS, INC
2139 HAMILTON AVE., CLEVELAND, OH

SCALE: NONE	APPROVED BY:	DRAWN BY MJH
DATE: 8-16-86		REVISED

EAGLE TECHNOLOGIES, INC.
P.O. BOX 1133
MENTOR, OHIO 44061-1133

DRAWING NUMBER
SF-1000-1

Yvonne C. Billingsley
ATTORNEY AND COUNSELOR AT LAW

614 SUPERIOR, N.W.
ROCKEFELLER BUILDING, SUITE 1310
CLEVELAND, OHIO 44113
(216) 861-0611

October 6, 1987

Ms. Debbie Berg
Ohio EPA
Northeast District Office
2110 E. Aurora Road
Twinsburg, Ohio 44087-1969

RE: Specialized Finishers, Inc.
and Thomas J. Foley

CLOSURE PLAN
I.D. No. OH.D.O. 13550371

Dear Ms. Berg:

Enclosed please find the results of the analysis of the contents of the three (3) tanks and one (1) sump for hazardous waste constituents at the Hamilton Avenue facility. Also enclosed please find another copy of the purchase agreement between Accurate Plating Company and Specialized Finishers, Inc., for the plating solutions.

I would like to clarify and confirm that the transporter and disposer of the hazardous wastes at the Hamilton Avenue facility will be:

Alchem-Tron, Inc.
7415 Bessemer Avenue
Cleveland, Ohio 44127
(216) 441-5628

Hopefully, we have met all of the requirements for approval of the Closure Plan. We await your further instructions.

Should you have additional questions or require additional information, please contact the undersigned.

Yvonne C. Billingsley

YVONNE C. BILLINGSLEY, ESQUIRE
Attorney for Thomas J. Foley

RECEIVED
OCT -8 1987
OHIO EPA-N.E.D.O.

YCB:an

cc: Thomas E. Crepeau, Ohio EPA, Columbus, Ohio
Mr. David Wertz, Ohio EPA, Northeast District Office
Dominic J. Hanket, Assistant Attorney General
Mr. Valdas Adamkus, Administrator, U.S. EPA, Region V

Enclosures: Analysis by Alchem Labs, dated September 28, 1987
Accurate Plating Company Purchase Order, dated September 18, 1987

OCT 1 1987

ALCHEM LABS

INCORPORATED

7415 Bessemer Ave., Cleveland, Ohio 44127 (216) 441-5628

Date Received 9-18-87

Specialized Finishers Inc.
2139 Hamilton Avenue
Cleveland, Ohio 44114

Customer I.D. Plating Waste

P.O.# WB

Date Reported (9-28-87)

Alchem-Lab # 870918-27

Description

~~Dark Liquids~~

ANALYSIS: Metal results in (ug / 1) ppb, all other results in (mg / 1) ppm, unless otherwise noted.

	1	2	3	4		1	2	3	4
Acidity					Mercury ppm	<.05	<.05	<.05	<.05
Alkalinity					Nickel	<0.10	<.10	<.10	<.10
Bacteria					Potassium				
T. Coliform / 100ml					Selenium ppm	<.02	<.02	<.02	<.02
F. Coliform / 100ml					Silver ppm	<.01	<.01	<.01	<.01
F. Strep / 100ml					Sodium				
Total Plate Count					Tin				
Chloride					Zinc	985	980	1150	1280
Chlorine: Res.					Nitrogen: Nitrate (N)				
Color					Nitrite (N)				
Conductivity					Ammonia (N)				
Cyanide: Total	136	138	155	140	Organic (N)				
Amenable					T Kjeldahl (N)				
Free					Odor				
Fluoride					Oil & Grease				
Hardness					Oxygen Demand: BODs				
MBAS					COD				
Metals: Aluminum					pH	11.6	10.5	10.5	11
Arsenic ppm	<0.01	<0.01	<0.01	<0.01	Phenols				
Barium ppm	.87	1.15	1.0	1.20	Phosphorus: Total (P)				
Cadmium ppm	264	380	285	300	Ortho (P)				
Calcium					Residue: Total				
Chromium: Total ppm	2.78	2.1	3.0	2.45	Total Vol				
Hexavalent ppm	<0.01	<0.01	<0.01	<0.01	Suspended				
Trivalent					Vol Suspended				
Cobalt					Settleable				
Copper	3.5	3.8	4.2	4.0	Sulfate				
Iron	95	85	78	75	TOC				
Lead ppm	.07	.10	.15	.12	Turbidity				
Manganese									
Magnesium									

Purchase Order Nº 2899

ACCURATE PLATING COMPANY

6512 Carnegie Ave.
P. O. Box 03277
Cleveland, Ohio 44103
216/432-1066

9-18-87

PURCHASE AGREEMENT

Accurate Plating agrees to purchase from Specialized Finishers the following plating solutions.

800 Gallons Chloride Zinc Plating Solution	.10 Gal	80.00
900 Gallons Cadmium Plating Solution	.20 Gal	180.00
		$260.00

ACCEPTED BY:

Colleen J. Stella

Thomas J. Foley