APPENDIX I

REPRESENTATIVE SAMPLING METHODS

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the following sampling protocols, for sampling waste with properties similar to the indicated materials, will be considered by the department to be representative of the waste:

(1) For extremely viscous liquid - ASTM Standard D140-70

(2) For crushed or powdered material - ASTM Standard D346-78

(3) For soil or rock-like material - ASTM Standard D420-69

(4) For soil-like material - ASTM Standard D1452-80

(5) For fly ash-like material - ASTM Standard D2234-76

Note: The publications containing these standards may be obtained from the:

American Society for Testing and Materials
1916 Race Street
Philadelphia, PA 19103

These publications are available for inspection at the offices of the department, the secretary of state, and the revisor of statutes.

(6) For containerized liquid wastes - "COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (SW-846).

(7) For liquid waste in pits, ponds, lagoons and similar reservoirs - "Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (SW-846).

Note: This publication may be obtained from:

Superintendent of Documents
U.S. Government Printing Office
Washington, DC 20402

This publication is available for inspection at the offices of the department, the secretary of state and the revisor of statutes.

APPENDIX II

CHEMICAL ANALYSIS TEST METHODS

Tables 1, 2 and 3 specify the appropriate analytical procedures, described in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW 846 which shall be used to determine whether a sample contains a given Appendix III or IV toxic constituent.

Table 1 identifies each Appendix III or IV organic constituent along with the approved measurement method. Table 2 identifies the corresponding methods for inorganic species. Table 3 summarizes the contents of SW-846 and supplies specific section and method numbers for sampling and analysis methods.

Prior to final sampling and analysis method selection the analyst should consult the specific section or method described in SW-846 for additional guidance on which of the approved methods should be employed for a specific sample analysis situation.

Table 1
Analysis Methods for Organic Chemicals Contained in SW-846

Compound	Method Numbers
Acetonitrile	8030, 8240
Acrolein	8030, 8240
Acrylamide	8015, 8240
Acrylonitrile	8030, 8240
2-Amino-l-methylbenzene (o-Toluidine)	8250
4-Amino-l-methylbenzene (p-Toluidine)	8250
Aniline	8250
Benzene	8020, 8024
Benz(a)anthracene	8100, 8250, 8310
Benzo(a)pyrene	8100, 8250, 8310
Benzotrichloride	8120, 8250
Benzyl chloride	8120, 8250
Benzo(b)fluoanthene	8100, 8250, 8310
Bis(2-chloroethoxymethane)	8010, 8240
Bis(2-chloroethyl)ether	8010, 8240
Bis(2-chloroisopropyl)ether	8010, 8240
Carbon disulfide	8015, 8240
Carbon tetrachloride	8010, 8240
Chlordane	8080, 8250
Chlorinated biphenyls	8080, 8250
Chlorinated dibenzo-p-dioxins	8280
Chlorinated dibenzofurans	8280
Chloroacetaldehyde	8010, 8240
Chlorobenzene	8020, 8240
Chloroform	8010, 8240
Chloromethane	8010, 8240
2-Chlorophenol	8040, 8250
Chrysene	8100, 8250, 8310
Creosote[1]	18100, 8250
Cresol(s)	8040, 8250
Cresylic Acid(s)	8040, 8250
Dichlorobenzene(s)	8010, 8120, 8250
Dichloroethane(s)	8010, 8240
Dichloromethane	8010, 8240
Dichlorophenoxyacetic acid	8150, 8250
Dichloropropanol	8120, 8250

Appendix

Compound	Method Numbers
2,4-Dimethylphenol	8040, 8250
Dinitrobenzene	8090, 8250
4,6-Dinitro-o-cresol	8040, 8250
2,4-Dinitrotoluene	8090, 8250
2,6-Dinitrotoluene	8060, 8250
Endrin	8080, 8250
2-Ethoxyethanol	8030, 8240
Ethyl ether	8015, 8240
Ethylene dibromide	8010, 8240
Ethylene thiourea	8250, 8330
Formaldehyde	8015, 8240
Formic acid	8250
Heptachlor	8080, 8250
Hexachlorobenzene	8120, 8250
Hexachlorobutadiene	8120, 8250
Hexachloroethane	8010, 8240
Hexachlorocyclopentadiene	8120, 8250
Lindane	8080, 8250
Maleic anhydride	8250
Methanol	8010, 8240
Methomyl	8250
Methyl ethyl ketone	8015, 8240
Methyl isobutyl ketone	8015, 8240
Napthalene	8100, 8250
Napthoquinone	8090, 8250
Nitrobenzene	8090, 8250
4-Nitrophenol	8040, 8240
2-Nitropropane	8030, 8240
Paraldehyde (trimer of acetaldehyde)	8015, 8240
Pentachlorophenol	8040, 8250
Phenol	8040, 8250
Phorate	8140
Phosphorodithioic acid esters	8140
Phthalic anhydride	8090, 8250
2-Picoline	8090, 8250
Pyridine	8090, 8250
Tetrachlorobenzene(s)	8120, 8250
Tetrachloroethane(s)	8010, 8240
Tetrachloroethene	8010, 8240
Tetrachlorophenol	8040, 8250
Toluene	8020, 8024
Toluene diisocyanate(s)	8250
Toluenediamine	8250
2,4-Toluenediamine	8250
2,6-Toluenediamine	8250
3,4-Toluenediamine	8250
Toxaphene	8080, 8250
Trichloroethane	8010, 8240
Trichloroethene(s)	8010, 8240
Trichlorofluoromethane	8010, 8240
Trichlorophenol(s)	8040, 8250
2,4,5-Trichlorophenoxy propionic acid	8150, 8250
Trichloropropane	8010, 8240

Compound	Method Numbers
Vinyl chloride	8010, 8240
Vinylidene chloride	8010, 8240
Xylene	8020, 8240

[1] Analyne for phenanthrene and carbazole; if these are present in a ratio between 1.4:1 and 5:1 creosote should be considered present.

Table 2
Analysis Methods for Inorganic Chemicals Contained in SW-846

Compound	First edition method(s)	Second edition method(s)
Antimony	8.50	7040, 7041
Arsenic	8.51	7060, 7061
Barium	8.52	7080, 7081
Cadmium	8.53	7090, 7091
Chromium	8.54	7190, 7191
Chromium: Hexavalent	8.545, 8.546, 8.547	7195, 7196, 7197
Lead	8.56	7420, 7421
Mercury	8.57	7470, 7471
Nickel	8.58	7520, 7521
Selenium	8.59	7740, 7741
Silver	8.60	7760, 7761
Cyanides	8.55	9010
Total Organic Halogen	8.66	9020
Sulfides	8.67	9030

Table 3
Sampling and Analysis Methods Contained in SW-846

Title	First edition		Second edition	
	Section No.	Method No.	Section No.	Method No.
Sampling of Solid Wastes	1.0	—	1.0	—
Development of Appropriate Sampling Plans	1.0	—	1.1	—
Regulatory and Scientific Objectives	1.0-2	—	1.1.1	—
Fundamental Statistical Concepts	1.0-3	—	1.1.2	—
Basic Statistical Strategies	1.0-7	—	1.1.3	—
Simple Random Sampling	—	—	1.1.3.1	—
Stratified Random Sampling	—	—	1.1.3.2	—
Systematic Random Sampling	—	—	1.1.3.3	—
Special Considerations	1.0-7	—	—	—
Composite Sampling	—	—	1.1.4.1	—
Subsampling	-	—	1.1.4.2	—
Cost and Loss Functions	—	—	1.1.4.3	—
Implementation of Sampling Plan	1.0-7	—	1.2	—
Selection of Sampling Equipment	—	—	1.2.1	—
Composite Liquid Waste Sampler	3.2.1	—	1.2.1.1	—
Weighted Bottle	3.2.2	—	1.2.1.2	—
Dipper	3.2.3	—	1.2.1.3	—
Thief	3.2.4	—	1.2.1.4	—
Trier	3.2.5	—	1.2.1.5	—
Auger	3.2.6	—	1.2.1.6	—
Scoop and Shovel	3.2.7	—	1.2.1.7	—
Selection of Sample Containers	3.3	—	1.2.2	—
Processing and Storage of Samples	3.3	—	1.2.3	—
Documentation of Chain of Custody	2.0	—	1.3	—
Sample Labels	2.0-1	—	1.3.1	—

Appendix

Title	First edition		Second edition	
	Section No.	Method No.	Section No.	Method No.
Sample Seals	2.0-3	—	1.3.2	—
Field Log Book	2.0-5	—	1.3.3	—
Chain-of-Custody Record	2.0-6	—	1.3.4	—
Sample Analysis Request Sheet	2.0-9	—	1.3.5	—
Sample Delivery to Laboratory	2.0-10	—	1.3.6	—
Shipping of Samples	2.0-10	—	1.3.7	—
Receipt and Logging of Sample	2.0-12	—	1.3.8	—
Assignment of Sample for Analysis	2.0-13	—	1.3.9	—
Sampling Methodology	3.0	—	1.4	—
Containers	3.2-2	—	1.4.1	—
Tanks	3.2-2	—	1.4.2	—
Waste Piles	3.2-2	—	1.4.3	—
Landfills and Lagoons	3.2-2	—	1.4.4	—
Waste Evaluation Procedures	—	—	2.0	—
Characteristics of Hazardous Waste	—	—	2.1	—
Ignitability	4.0	—	2.1.1	—
Pensky-Martens Closed-Cup Method	4.1	—	2.1.1	1010
Setaflash Closed-Cup Method	4.1	—	2.1.1	1020
Corrosivity	5.0	—	2.1.2	—
Corrosivity Toward Steel	5.3	—	2.1.2	1110
Reactivity	6.0	—	2.1.3	—
Extraction Procedure Toxicity	7.0	—	2.1.4	—
Extraction Procedure Toxicity Test	7.1, 7.2, 7.5	—	—	—
Method and Structural Integrity Test	7.4	—	2.1.4	1310
Sample Workup Techniques	—	—	4.0	—
Inorganic Techniques	8.49	—	4.1	—
Acid Digestion for Flame AAS	1	—	4.1	3010
Acid Digestion for Furnace AAS	1	—	4.1	3020
Acid Digestion of Oil, Grease, or Wax	8.49-9	—	4.1	3030
Dissolution Procedure for Oil, Grease or Wax	8.49-8			
Alkaline Digestion	8.0	8.458	4.1	3060
Organic Techniques	8.0	—	4.2	—
Separatory Funnel Liquid-Liquid Extraction	9.0	9.1	4.2	3510
Continuous Liquid-Liquid Extraction	9.0	9.01	4.2	3520
Acid-Base Cleanup Extraction	8.0	8.84	4.2	3530
Soxhlet Extraction	8.0	8.86	4.2	3540
Sonication Extraction	8.0	8.85	4.2	3550
Sample Introduction Techniques	—	—	5.0	—
Headspace	8.0	8.82	5.0	5020
Purge-and-Trap	8.0	8.83	5.0	5030
Inorganic Analytical Methods	8.0	—	7.0	—
Antimony, Flame AAS	8.0	8.50	7.0	7470
Antimony, Furnace AAS	8.0	8.50	7.0	7471
Arsenic, Flame AAS	8.0	8.51	7.0	7060
Arsenic, Furnace AAS	8.0	8.51	7.0	7061
Barium, Flame AAS	8.0	8.52	7.0	7080
Barium, Furnace AAS	8.0	8.52	7.0	7081
Cadmium, Flame AAS	8.0	8.53	7.0	7130

Title	First edition		Second edition	
	Section No.	Method No.	Section No.	Method No.
Cadmium, Furnace AAS	8.0	8.53	7.0	7131
Chromium, Flame AAS	8.0	8.54	7.0	7090
Chromium, Furnace AAS	8.0	8.54	7.0	7191
Chromium, Hexavalent, Coprecipitation	8.0	8.545	7.0	7195
Chromium, Hexavalent, Colorimetric	8.0	8.546	7.0	7196
Chromium, Hexavalent, Chelation	8.0	8.547	7.0	7197
Lead, Flame AAS	8.0	8.56	7.0	7420
Lead, Furnace AAS	8.0	8.56	7.0	7421
Mercury, Cold Vapor, Liquid	8.0	8.57	7.0	7470
Mercury, Cold Vapor, Solid	8.0	8.57	7.0	7471
Nickel, Flame AAS	8.0	8.58	7.0	7520
Nickel, Furnace AAS	8.0	8.58	7.0	7521
Selenium, Flame AAS	8.0	8.59	7.0	7740
Selenium, Gaseous Hydride AAS	8.0	8.59	7.0	7741
Silver, Flame AAS	8.0	8.60	7.0	7760
Silver, Furnace AAS	8.0	8.60	7.0	7761
Organic Analytical Methods	8.0	—	8.0	—
Gas Chromatographic Methods	8.0	—	8.1	—
Halogenated Volatile Organics	8.0	8.01	8.1	8010
Nonhalogenated Volatile Organics	8.0	8.01	8.1	8015
Aromatic Volatile Organics	8.0	8.02	8.1	8020
Acrolein, Acrylonitrile, Acetonitrile	8.0	8.03	8.1	8030
Phenols	8.0	8.04	8.1	8040
Phthalate Esters	8.0	8.06	8.1	8060
Organochlorine Pesticides and PCBs	8.0	8.08	8.1	8080
Nitroaromatics and Cyclic Ketones	8.0	8.09	8.1	8090
Polynuclear Aromatic Hydrocarbons	8.0	8.10	8.1	8100
Chlorinated Hydrocarbons	8.0	8.12	8.1	8120
Organophosphorus Pesticides	8.0	8.22	8.1	8140
Chlorinated Herbicides	8.0	8.40	8.1	8150
Gas Chromatographic/Mass Spectroscopy Methods (GC/MS)	8.0	—	8.2	—
GC/MS Volatiles	8.0	8.24	8.2	8240
GC/MS Semi-Volatiles, Packed Column	8.0	8.25	8.2	8250
GC/MS Semi-Volatiles, Capillary	8.0	8.27	8.2	8270
Analysis of Chlorinated Dioxins and Dibenzofurans	—	—	8.28	8280
High Performance Liquid Chromatographic Methods (HPLC)	8.0	—	8.3	—
Polynuclear Aromatic Hydrocarbons	8.0	8.10	8.3	8310
Miscellaneous Analytical Methods	8.0	—	9.0	—
Cyanide; Total and Amenable to Chlorination	8.0	8.55	9.0	9010
Total Organic Halogen (TOX)	8.0	8.66	9.0	9020
Sulfides	8.0	8.67	9.0	9030
pH Measurement	5.0	5.2	9.0	9040
Quality Control/Quality Assurance	10.0	—	10.1	—

Appendix

Title	First edition		Second edition	
	Section No.	Method No.	Section No.	Method No.
Introduction	10.0	—	10.1	—
Program Design	10.0	—	10.2	—
Sampling	10.0	—	10.3	—
Analysis	10.0	—	10.4	—
Data Handling	10.0	—	10.5	—

[1] See specific metal.

APPENDIX III

BASIS FOR LISTING HAZARDOUS WASTES

BASIS FOR LISTING HAZARDOUS WASTES

Hazardous Waste Number	Hazardous Constituents for Which Listed
F001	tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorinated fluorocarbons, carbon tetrachloride
F002	tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloroethylene, 1,1, 2-trichloro-1,2,2-trifluoroethane, o-dichlorobenzene, trichlorofluoromethane
F003	N.A.
F004	cresols and cresylic acid, nitrobenzene
F005	toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine, 2-ethoxyethanol, benzene, 2-nitropropane
F006	cadmium, hexavalent chromium, nickel, cyanide (complexed)
F007	cyanide (salts)
F008	cyanide (salts)
F009	cyanide (salts)
F010	cyanide (salts)
F011	cyanide (salts)
F012	cyanide (complexed)
F019	hexavalent chromium, cyanide (complexed)
F020	Tetra- and pentachlorodibenzo-p-dioxins; tetra and pentachlorodi-benzofurans; tri- and tetrachlorophenols and their chlorophenoxy derivative acids, esters, ethers, amine and other salts.
F021	Penta- and hexachlorodibenzo-p-dioxins; penta- and hexachlorodibenzofurans; pentachlorophenol and its derivatives.
F022	Tetra-, penta- and hexachlorodibenzo-p-dioxins; tetra-, penta- and hexachlorodibenzofurans.
F023	Tetra- and pentachlorodibenzo-p-dioxins; tetra- and pentachlorodibenzofurans; tri- and tetrachlorophenols and their chlorophenoxy derivative acids, esters, ethers, amine and other salts.
F024	chloromethane, dichloromethane, trichloromethane, carbon tetrachloride, chloroethylene, 1,1-dichloroethane, 1,2-dichloroethane, trans-1-2-dichloroethylene, 1,1-dichloroethylene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethylene, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, tetrachloroethylene, pentachloroethane, hexachloroethane, allyl chloride (3-chloropropene), dichloropropane, dichloropropene, 2-chloro-1,3-butadiene, hexachloro-1,3-butadiene, hexachlorocyclopentadiene, benzene, chlorobenzene, dichlorobenzene, 1,2,4-trichlorobenzene, tetrachlorobenzene, pentachlorobenzene, hexachlorobenzene, toluene, naphthalene
F026	Tetra-, penta- and hexachlorodibenzo-p-dioxins; tetra-, penta- and hexachlorodibenzofurans.
F027	Tetra-, penta- and hexachlorodibenzo-p-dioxins; tetra-, penta- and hexachlorodibenzofurans; tri-, tetra- and pentachlorophenols and their chlorophenoxy derivative acids, esters, ethers, amine and other salts.
F028	Tetra-, penta- and hexachlorodibenzo-p-dioxins; tetra-, penta- and hexachlorodibenzofurans; tri-, tetra- and pentachlorophenols and their chlorophenoxy derivative acids, esters, ethers, amine and other salts.
F500	Same as F001 and F002
K001	benz(a)anthracene, benzo(a)pyrene, chrysene, naphthalene, phenol, 2-chlorophenol, 2,4-dimethylphenyl, trichlorphenols, pentachlorophenol, tetrachlorophenols, p-chloro-m-cresol, 2,4-dinitrophenol, creosote, fluoranthene, benzo(b)fluoranthene, indeno (1,2,3-cd) pyrene, dibenz(a)anthracene, acenaphthalene, pentachlorophenol
K002	hexavalent chromium, lead
K003	hexavalent chromium, lead

Appendix

Hazardous Waste Number	Hazardous Constituents for Which Listed
K004	hexavalent chromium
K005	hexavalent chromium, lead
K006	hexavalent chromium
K007	cyanide (complexed), hexavalent chromium
K008	hexavalent chromium
K009	chloroform, formaldehyde, methylene chloride, methyl chloride, paraldehyde, formic acid
K010	chloroform, formaldehyde, methylene chloride, methyl chloride, paraldehyde, formic acid, chloroacetaldehyde
K011	acrylonitrile, acetonitrile, hydrocyanic acid
K013	hydrocyanic acid, acrylonitrile, acetonitrile
K014	acetonitrile, acrylamide
K015	benzyl chloride, chlorobenzene, toluene, benzotrichloride
K016	hexachlorobenzene, hexachlorobutadiene, carbon tetrachloride, hexachloroethane, perchloroethylene
K017	epichlorohydrin, chloroethers (bis(chloromethyl) ether and bis (2-chloroethyl) ethers), trichloropropane, dichloropropanols
K018	1,2-dichloroethane, trichloroethylene, hexachlorobutadiene, hexachlorobenzene
K019	ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, tetrachloroethanes (1,1,2,2-tetrachloroethane and 1,1,1,2-tetrachloroethane), trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride
K020	ethylene dichloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, tetrachloroethanes (1,1,2,2-tetrachloroethane and 1,1,1,2-tetrachloroethane), trichloroethylene, tetrachloroethylene, carbon tetrachloride, chloroform, vinyl chloride, vinylidene chloride
K021	antimony, carbon tetrachloride, chloroform
K022	phenol, tars (polycyclic aromatic hydrocarbons)
K023	phthalic anhydride, maleic anhydride
K024	phthalic anhydride, 1,4 napthoquinone
K025	meta-dinitrobenzene, 2,4-dinitrotoluene
K026	paraldehyde, pyridines, 2-picoline
K027	toluene diisocyanate, toluene-2,4-diamine
K028	1,1,1-trichloroethane, vinyl chloride
K029	1,2-dichloroethane, 1,1,1-trichloroethane, vinyl chloride, vinylidene chloride, chloroform
K030	hexachlorobenzene, hexachlorobutadiene, hexachloroethane, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, ethylene dichloride
K031	arsenic
K032	hexachlorocyclopentadiene
K033	hexachlorocyclopentadiene
K034	hexachlorocyclopentadiene
K035	creosote, benzo(b)fluoroanthene, benzo(a)pyrene, chrysene, naphthalene, fluoranthene, indeno(1,2,3-cd)pyrene, benzo(a)anthracene, dibenzo(a)anthracene, acenaphthalene
K036	toluene, phosphorodithioic and phosphorothioic acid esters
K037	toluene, phosphorodithioic and phosphorothioic acid esters
K038	phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K039	phosphorodithioic and phosphorothioic acid esters
K040	phorate, formaldehyde, phosphorodithioic and phosphorothioic acid esters
K041	toxaphene
K042	hexachlorobenzene; ortho-dichlorobenzene
K043	2,4-dichlorophenol, 2,6-dichlorophenol, 2,4,6-trichlorophenol
K044	N.A.
K045	N.A.
K046	lead
K047	N.A.

Hazardous Waste Number	Hazardous Constituents for Which Listed
K048	chromium (VI), lead
K049	chromium (VI), lead
K050	chromium (VI)
K051	chromium (VI), lead
K052	lead
K060	cyanide, naphthalene, phenolic compounds, arsenic
K061	chromium (VI), lead, cadmium
K062	chromium (VI), lead
K069	chromium (VI), lead, cadmium
K071	mercury
K073	chloroform, carbon tetrachloride, hexachloroethane, trichloroethane, tetrachlorethylene, dichloroethylene, 1,1,2,2-tetrachloroethane
K083	aniline, nitrobenzene, diphenylamine, phenylenediamine
K084	arsenic
K085	benzene, dichlorobenzenes, trichlorobenzenes, tetrachlorobenzene, pentachlorobenzene, hexachlorobenzene, benzyl chloride
K086	chromium (VI), lead
K087	phenol, naphtalene
K093	Phthalic anhydride, maleic anhydride
K094	Phthalic anhydride
K095	1,1,2-trichloroethane, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane
K096	1,2-dichloroethane, 1,1,1-trichloroethane, 1,1,2-trichloroethane
K097	Chlordane, heptachlor
K098	Toxaphene
K099	2,4-dichlorophenol, 2,4,6-trichlorophenol
K100	Hexavalent chromium, lead, cadmium
K101	Arsenic
K102	Arsenic
K103	Aniline, nitrobenzene, phenylenediamine
K104	Aniline, benzene, diphenylamine, nitrobenzene, phenylenediamine
K105	Benzene, monochlorobenzene, dichlorobenzene, 2,4,6-trichlorophenol
K106	Mercury
K111	2,4-Dinitrotoluene
K112	2,4-Toluenediamine, o-toluidine, p-toluidine, aniline
K113	2,4-Toluenediamine, o-toluidine, p-toluidine, aniline
K114	2,4-Toluenediamine, o-toluidine, p-toluidine
K115	2,4-Toluenediamine
K116	Carbon tetrachloride, tetrachloroethylene, chloroform, phosgene
K117	Ethylene dibromide
K118	Ethylene dibromide
K123	Ethylene thiourea
K124	Ethylene thiourea
K125	Ethylene thiourea
K126	Ethylene thiourea
K136	Ethylene dibromide

N.A. - Waste is hazardous because it meets either the ignitability, corrosivity or reactivity characteristics.

APPENDIX IV

HAZARDOUS CONSTITUENTS

A solid waste which contains any of the hazardous constituents listed in this appendix shall be listed in s. NR 605.09 as a hazardous waste unless the department concludes, after considering the factors in s. NR 605.08 (6) (a) 3., that the waste is not capable of posing a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed or otherwise managed.

Note: Section NR 605.08 (6) (a) 3. identifies criteria for listing hazardous waste. A waste containing any of the constituents in this appendix is examined by the department using these criteria. If the department determines the waste should be listed, it will be included under: Table II, Hazardous Waste from Nonspecific Sources; Table III, Hazardous Waste from Specific Sources; Table IV, Acute Hazardous Commercial Chemical Products and Manufacturing Chemical Intermediates; or Table V, Toxic Commercial Chemical Products and Manufacturing Chemical Intermediates. One shall not assume that a waste containing one or more of the constituents in this appendix will automatically be a hazardous waste. In this appendix, the abbreviation N.O.S. (not otherwise specified) signifies those members of the general class not specifically listed by name.

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Acetonitrile	Same	75-05-8	U003
Acetophenone	Ethanone, 1-phenyl-	98-86-2	U004
2-Acetylaminefluarone	Acetamide, N-9H-fluoren-2-yl-	53-96-3	U005
Acetyl chloride	Same	75-36-5	U006
1-Acetyl-2-thiourea	Acetamide, N-(aminothioxomethyl)-	591-08-2	P002
Acrolein	2-Propenal	107-02-8	P003
Acrylamide	2-Propenamide	79-06-1	U007
Acrylonitrile	2-Propenenitrile	107-13-1	U009
Aflatoxins	Same	1402-68-2	—
Aldicarb	Propanal, 2-methyl-2-(methylthio)-, O-[(methylamino)carbonyl]oxime	116-06-3	P070
Aldrin	1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8ahexahydro-, (1alpha,4alpha,4abeta,5alpha, 8alpha,8abeta)-	309-00-2	P004
Allyl alcohol	2-Propen-1-ol	107-18-6	P005
Aluminum phosphide	Same	20859-73-8	P006
4-Aminobiphenyl	[1,1'-Biphenyl]-4-amine	92-67-1	—
5-(Aminomethyl)-3-isoxazolol	3(2H)-Isoxazolone, 5-(aminomethyl)-	2763-96-4	P007
4-Aminopyridine	4-Pyridinamine	504-24-5	P008
Amitrole	1H-1,2,4-Triazol-3-amine	61-82-5	U011
Ammonium vanadate	Vanadic acid, ammonium salt	7803-55-6	P119
Aniline	Benzenamine	62-53-3	U012
Antimony	Same	7440-36-0	—
Antimony compounds, N.O.S.[1]	—	—	—
Aramite	Sulfurous acid, 2-chloroethyl 2-[4-(1,1dimethylethyl) phenoxy]-1-methylethyl ester	140-57-8	—
Arsenic	Same	7440-38-2	—

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Arsenic compounds, N.O.S.[1]	—	—	—
Arsenic acid	Arsenic acid H3AsO4	7778-39-4	P010
Arsenic pentoxide	Arsenic oxide As2O5	1303-28-2	P011
Arsenic trioxide	Arsenic oxide As2O3	1327-53-3	P012
Auramine	Benzenamine, 4,4'-carbonimidoylbis [N,Ndimethyl	492-80-8	U014
Azaserine	L-Serine, diazoacetate (ester)	115-02-6	U015
Barium	Same	7440-39-3	—
Barium compounds, N.O.S.[1]	—	—	—
Barium cyanide	Same	542-62-1	P013
Benz[c]acridine	Same	225-51-4	U016
Benz[a]anthracene	Same	56-55-3	U018
Benzal chloride	Benzene, (dichloromethyl)-	98-87-3	U017
Benzene	Same	71-43-2	U019
Benzenearsonic acid	Arsonic acid, phenyl-	98-05-5	—
Benzidine	[1,1'-Biphenyl]-4,4 ' -diamine	92-87-5	U021
Benzo[b]fluoranthene	Benz[e]acephenanthrylene	205-99-2	—
Benzo[j]fluoranthene	Same	205-82-3	—
Benzo[a]pyrene	Same	50-32-8	U022
p-Benzoquinone	2,5-Cyclohexadiene-1,4-dione	106-51-4	U197
Benzotrichloride	Benzene, (trichloromethyl)-	98-07-7	U023
Benzyl chloride	Benzene, (chloromethyl)-	100-44-7	P028
Beryllium	Same	7440-41-7	P015
Beryllium compounds, N.O.S.[1]	—	—	—
Bromoacetone	2-Propanone, 1-bromo-	598-31-2	P017
Bromoform	Methane, tribromo-	75-25-2	U225
4-Bromophenyl phenyl ether	Benzene, 1-bromo-4-phenoxy-	101-55-3	U030
Brucine	Strychnidin-10-one, 2,3-dimethoxy-	357-57-3	P018
Butyl benzyl phthalate	1,2-Benzenedicarboxylic acid, butyl phenylmethyl ester	85-68-7	—
Cacodylic acid	Arsinic acid, dimethyl-	75-60-5	U136
Cadmium	Same	7440-43-9	—
Cadmium compounds, N.O.S.[1]	—	—	—
Calcium chromate	Chromic acid H2CrO4, calcium salt	13765-19-0	U032
Calcium cyanide	Calcium cyanide Ca(CN)2	592-01-8	P021
Carbon disulfide	Same	75-15-0	P022
Carbon oxyfluoride	Carbonic difluoride	353-50-4	U033
Carbon tetrachloride	Methane, tetrachloro-	56-23-5	U211
Chloral	Acetaldehyde, trichloro-	75-87-6	U034
Chlorambucil	Benzenebutanoic acid, 4-[bis(2-chloroethyl) amino]-	305-03-3	U035
Chlordane	4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-	57-74-9	U036
Chlordane (alpha and gamma isomers)	—	—	U036
Chlorinated benzenes, N.O.S.[1]	—	—	—
Chlorinated ethane, N.O.S.[1]	—	—	—
Chlorinated fluorocarbons, N.O.S.[1]	—	—	—
Chlorinated naphthalene, N.O.S.[1]	—	—	—

Appendix

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Chlorinated phenol, N.O.S.[1]	—	—	—
Chlornaphazin	Naphthalenamine, N,N'-bis(2-chloroethyl)-	494-03-1	U026
Chloroacetaldehyde	Acetaldehyde, chloro-	107-20-0	P023
Chloroalkyl ethers, N.O.S.[1]			
p-Chloroaniline	Benzenamine, 4-chloro-	106-47-8	P024
Chlorobenzene	Benzene, chloro-	108-90-7	U037
Chlorobenzilate	Benzeneacetic acid, 4-chloro-alpha-(4-chlorophenyl)-alpha-hydroxy-, ethyl ester	510-15-6	U038
p-Chloro-m-cresol	Phenol, 4-chloro-3-methyl-	59-50-7	U039
2-Chloroethyl vinyl ether	Ethene, (2-chloroethoxy)-	110-75-8	U042
Chloroform	Methane, trichloro-	67-66-3	U044
Chloromethyl methyl ether	Methane, chloromethoxy-	107-30-2	U046
beta-Chloronaphthalene	Naphthalene, 2-chloro-	91-58-7	U047
o-Chlorophenol	Phenol, 2-chloro-	95-57-8	U048
1-(o-Chlorophenyl)thiourea	Thiourea, (2-chlorophenyl)-	5344-82-1	P026
Chloroprene	1,3-Butadiene, 2-chloro-	126-99-8	—
3-Chloropropionitrile	Propanenitrile, 3-chloro-	542-76-7	P027
Chromium	Same	7440-47-3	—
Chromium compounds, N.O.S.[1]	—	—	—
Chrysene	Same	218-01-9	U050
Citrus red No. 2	2-Naphthalenol, 1-[(2,5-dimethoxyphenyl)azo]-	6358-53-8	—
Coal tar creosote	Same	8007-45-2	—
Copper cyanide	Copper cyanide CuCN	544-92-3	P029
Creosote	Same	—	U051
Cresol (Cresylic acid)	Phenol, methyl-	1319-77-3	U052
Crotonaldehyde	2-Butenal	4170-30-3	U053
Cyanides (soluble salts and complexes) N.O.S.[1]	—	—	P030
Cyanogen	Ethanedinitrile	460-19-5	P031
Cyanogen bromide	Cyanogen bromide (CN)Br	506-68-3	U246
Cyanogen chloride	Cyanogen chloride (CN)Cl	506-77-4	P033
Cycasin	beta-D-Glucopyranoside, (methyl-ONN-azoxy)methyl	14901-08-7	—
2-Cyclohexyl-4,6-dinitrophenol	Phenol, 2-cyclohexyl-4, 6-dinitro-	131-89-5	P034
Cyclophosphamide	2H-1,3,2-Oxazaphosphorin-2-amine, N,Nbis(2-chloroethyl)tetrahydro-, 2oxide	50-18-0	U058
2,4-D	Acetic acid, (2,4-dichlorophenoxy)-	94-75-7	U240
2,4-D, salts, esters	—	—	U240
Daunomycin	5,12-Naphthacenedione, 8-acetyl-10-[(3-amino-2,3,6-trideoxy-alpha- L-lyxohexopyranosyl)oxy]-7,8,9, 10-tetrahydro-6,8,11-trihydroxy-1-methoxy-, (8S-cis)-	20830-81-3	U059
DDD	Benzene, 1,1'-(2,2-dichloroethylidene) bis[4-chloro-	72-54-8	U060
DDE	Benzene, 1,1'-(dichloroethenylidene) bis[4-chloro-	72-55-9	—

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
DDT	Benzene, 1,1'-(2,2,2-trichloroethylidene) bis[4-chloro-	50-29-3	U061
Diallate	Carbamothioic acid, bis(1-methylethyl)-, S(2,3-dichloro-2-propenyl) ester	2303-16-4	U062
Dibenz[a,h]acridine	Same	226-36-8	—
Dibenz[a,j]acridine	Same	224-42-0	—
Dibenz[a,h]anthracene	Same	53-70-3	U063
7H-Dibenzo[c,g]carbazole	Same	194-59-2	—
Dibenzo[a,e]pyrene	Naphtho[1,2,3,4-def]chrysene	192-65-4	—
Dibenzo[a,h]pyrene	Dibenzo[b,def]chrysene	189-64-0	—
Dibenzo[a,i]pyrene	Benzo[rst]pentaphene	189-55-9	U064
1,2-Dibromo-3-chloropropane	Propane, 1,2-dibromo-3-chloro-	96-12-8	U066
Dibutyl phthalate	1,2-Benzenedicarboxylic acid, dibutyl ester	84-74-2	U069
o-Dichlorobenzene	Benzene, 1,2-dichloro-	95-50-1	U070
m-Dichlorobenzene	Benzene, 1,3-dichloro-	541-73-1	U071
p-Dichlorobenzene	Benzene, 1,4-dichloro-	106-46-7	U072
Dichlorobenzene, N.O.S.[1]	Benzene, dichloro-	25321-22-6	—
3,3'-Dichlorobenzidine	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro-	91-94-1	U073
1,4-Dichloro-2-butene	2-Butene, 1,4-dichloro-	764-41-0	U074
Dichlorodifluoromethane	Methane, dichlorodifluoro-	75-71-8	U075
Dichloroethylene, N.O.S.[1]	Dichloroethylene	25323-30-2	—
1,1-Dichloroethylene	Ethene, 1,1-dichloro-	75-35-4	U078
1,2-Dichloroethylene	Ethene, 1,2-dichlrolo-, (E)-	156-60-5	U079
Dichloroethyl ether	Ethane, 1,1'oxybis[2-chloro-	111-44-4	U025
Dichloroisopropyl ether	Propane, 2,2'-oxybis[2-chloro-	108-60-1	U027
Dichloromethoxy ethane	Ethane, 1,1'-[methylenebis(oxy)] bis[2-chloro-	111-91-1	U024
Dichloromethyl ether	Methane, oxybis[chloro-	542-88-1	P016
2,4-Dichlorophenol	Phenol, 2,4-dichloro-	120-83-2	U081
2,6-Dichlorophenol	Phenol, 2,6-dichloro-	87-65-0	U082
Dichlorophenylarsine	Arsonous dichloride, phenyl-	696-28-6	P036
Dichloropropane, N.O.S.[1]	Propane, dichloro-	26638-19-7	—
Dichloropropanol, N.O.S.[1]	Propanol, dichloro-	26545-73-3	—
Dichloropropene, N.O.S.[1]	1-Propene, dichloro-	26952-23-8	—
1,3-Dichloropropene	1-Propene, 1,3-dichloro-	542-75-6	U084
Dieldrin	2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro- 1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2aalpha, 3beta,6beta,6aalpha, 7beta,7aalpha)-	60-57-1	P037
1,2:3,4-Diepoxybutane	2,2[one-fourth]-Bioxirane	1464-53-5	U085
Diethylarsine	Arsine, diethyl-	692-42-2	P038
1,4-Diethyleneoxide	1,4-Dioxane	123-91-1	U108
Diethylhexyl phthalate	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester	117-81-7	U028
N,N'-Diethylhydrazine	Hydrazine, 1,2-diethyl-	1615-80-1	U086
O,O-Diethyl S-methyl dithiophosphate	Phosphorodithioic acid, O,O-diethyl S-methyl ester	3288-58-2	U087
Diethyl-p-nitrophenyl phosphate	Phosphoric acid, diethyl 4-nitrophenyl ester	311-45-5	P041

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Diethyl phthalate	1,2-Benzenedicarboxylic acid, diethyl ester	84-66-2	U088
O,O-Diethyl O-pyrazinyl phosphorothioate	Phosphorothioic acid, O,O-diethyl Opyrazinyl ester	297-97-2	P040
Diethylstilbesterol	Phenol, 4,4'-(1,2-diethyl-1, 2-ethenediyl) bis-, (E)-	56-53-1	U089
Dihydrosafrole	1,3-Benzodioxole, 5-propyl-	94-58-6	U090
Diisopropylfluorophosphate (DFP)	Phosphorofluoridic acid, bis(1-methylethyl) ester	55-91-4	P043
Dimethoate	Phosphorodithioic acid, O,O-dimethyl S-[2-(methylamino)-2-oxoethyl] ester	60-51-5	P044
3,3'-Dimethoxybenzidine	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethoxy-	119-90-4	U091
p-Dimethylaminoazobenzene	Benzenamine, N,N-dimethyl-4-(phenylazo)-	60-11-7	U093
7,12-Dimethylbenz[a]anthracene	Benz[a]anthracene, 7,12-dimethyl-	57-97-6	U094
3,3'-Dimethylbenzidine	[1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethyl-	119-93-7	U095
Dimethylcarbamoyl chloride	Carbamic chloride, dimethyl-	79-44-7	U097
1,1-Dimethylhydrazine	Hydrazine, 1,1-dimethyl-	57-14-7	U098
1,2-Dimethylhydrazine	Hydrazine, 1,2-dimethyl-	540-73-8	U099
alpha,alpha-Dimethylphenethylamine	Benzeneethanamine, alpha,alpha-dimethyl-	122-09-8	P046
2,4-Dimethylphenol	Phenol, 2,4-dimethyl-	105-67-9	U101
Dimethyl phthalate	1,2-Benzenedicarboxylic acid, dimethyl ester	131-11-3	U102
Dimethyl sulfate	Sulfuric acid, dimethyl ester	77-78-1	U103
Dinitrobenzene, N.O.S.[1]	Benzene, dinitro-	25154-54-5	
4,6-Dinitro-o-cresol	Phenol, 2-methyl-4,6-dinitro-	534-52-1	P047
4,6-Dinitro-o-cresol salts	—	—	P047
2,4-Dinitrophenol	Phenol, 2,4-dinitro-	51-28-5	P048
2,4-Dinitrotoluene	Benzene, 1-methyl-2,4-dinitro-	121-14-2	U105
2,6-Dinitrotoluene	Benzene, 2-methyl-1,3-dinitro-	606-20-2	U106
Dinoseb	Phenol, 2-(1-methylpropyl)-4, 6-dinitro-	88-85-7	P020
Di-n-octyl phthalate	1,2-Benzenedicarboxylic acid, dioctyl ester	117-84-0	U017
Diphenylamine	Benzenamine, N-phenyl-	122-39-4	—
1,2-Diphenylhydrazine	Hydrazine, 1,2-diphenyl-	122-66-7	U109
Di-n-propylnitrosamine	1-Propanamine, N-nitroso-N-propyl-	621-64-7	U111
Disulfoton	Phosphorodithioic acid, O,O-diethyl S-[2-(ethylthio)ethyl] ester	298-04-4	P039
Dithiobiuret	Thioimidodicarbonic diamide [(H2N)C(S)]2NH	541-53-7	P049
Endosulfan	6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro- 1,5,5a,6,9,9a-hexahydro-, 3-oxide	115-29-7	P050
Endothall	7-Oxabicyclo[2.2.1] heptane-2,3-dicarboxylic acid	145-73-3	P088

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Endrin	2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9- hexachloro-1a,2,2a,3,6,6a, 7,7aocta-hydro-, (1aalpha,2beta, 2abeta,3alpha,6alpha, 6abeta,7beta,7aalpha)-	72-20-8	P051
Endrin metabolites	—	—	P051
Epichlorohydrin	Oxirane, (chloromethyl)-	106-89-8	U041
Epinephrine	1,2-Benzenediol, 4-[1-hydroxy-2-(methylamino)ethyl]-, (R)-	51-43-4	P042
Ethyl carbamate (urethane)	Carbamic acid, ethyl ester	51-79-6	U238
Ethyl cyanide	Propanenitrile	107-12-0	P101
Ethylenebisdithiocarbamic acid	Carbamodithioic acid, 1,2-ethanediylbis-	111-54-6	U114
Ethylenebisdithiocarbamic acid, salts and esters	—	—	U114
Ethylene dibromide	Ethane, 1,2-dibromo-	106-93-4	U067
Ethylene dichloride	Ethane, 1,2-dichloro-	107-06-2	U077
Ethylene glycol monoethyl ether	Ethanol, 2-ethoxy-	110-80-5	U359
Ethyleneimine	Aziridine	151-56-4	P054
Ethylene oxide	Oxirane	75-21-8	U115
Ethylenethiourea	2-Imidazolidinethione	96-45-7	U116
Ethylidene dichloride	Ethane, 1,1-dichloro-	75-34-3	U076
Ethyl methacrylate	2-Propenoic acid, 2-methyl-, ethyl ester	97-63-2	U118
Ethyl methanesulfonate	Methanesulfonic acid, ethyl ester		
62-50-0	U119		
Famphur	Phosphorothioic acid, O-[4-[(dimethylamino) sulfonyl]phenyl] O,Odimethyl ester	52-85-7	P097
Fluoranthene	Same	206-44-0	U120
Fluorine	Same	7782-41-4	P056
Fluoroacetamide	Acetamide, 2-fluoro-	640-19-7	P057
Fluoroacetic acid, sodium salt	Acetic acid, fluoro-, sodium salt	62-74-8	P058
Formaldehyde	Same	50-00-0	U122
Formic acid	Same	64-18-6	U123
Glycidylaldehyde	Oxiranecarboxyaldehyde	765-34-4	U126
Halomethanes, N.O.S.[1]	—	—	—
Heptachlor	4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-	76-44-8	P059
Heptachlor epoxide	2,5-Methano-2H-indeno [1,2-b]oxirene, 2,3,4,5,6,7,7-heptachloro- 1a,1b,5,5a,6,6a-hexa- hydro-, (1aalpha, 1bbeta, 2alpha, 5alpha, 5abeta, 6beta, 6aalpha)-	1024-57-3	
Heptachlor epoxide (alpha, beta, and gamma isomers)	—	—	—
Hexachlorobenzene	Benzene, hexachloro-	118-74-1	U127
Hexachlorobutadiene	1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	87-68-3	U128
Hexachlorocyclopentadiene	1,3-Cyclopentadiene, 1,2,3,4,5,5hexachloro-	77-47-4	U130
Hexachlorodibenzo-p-dioxins	—	—	—

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Hexachlorodibenzofurans	—	—	—
Hexachloroethane	Ethane, hexachloro-	67-72-1	U131
Hexachlorophene	Phenol, 2,2'-methylenebis [3,4,6-trichloro-	70-30-4	U132
Hexachloropropene	1-Propene, 1,1,2,3,3,3-hexachloro-	1888-71-7	U243
Hexaethyl tetraphosphate	Tetraphosphoric acid, hexaethyl ester	757-58-4	P062
Hydrazine	Same	302-01-2	U133
Hydrogen cyanide	Hydrocyanic acid	74-90-8	P063
Hydrogen fluoride	Hydrofluoric acid	7664-39-3	U134
Hydrogen sulfide	Hydrogen sulfide H2S	7783-06-4	U135
Indeno[1,2,3-cd]pyrene	Same	193-39-5	U137
Iron dextran	Same	9004-66-4	U139
Isobutyl alcohol	1-Propanol, 2-methyl-	78-83-1	U140
Isodrin	1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexachloro-1,4,4a,5,8, 8a-hexahydro-, (1alpha, 4alpha, 4abeta, 5beta, 8beta, 8abeta)	465-73-6	P060
Isosafrole	1,3-Benzodioxole, 5-(1-propenyl)-	120-58-1	U141
Kepone	1,3,4-Metheno-2H-cyclobuta [cd]pentalen-2-one, 1,1a,3,3a,4,5,5,5a,5b, 6-decachlorooctahydro-	143-50-0	U142
Lasiocarpine	2-Butenoic acid, 2-methyl-,7-[[2,3dihydroxy-2- (1-methoxyethyl)-3-methyl-1-@xl oxobutoxy]methyl] -2,3,5,7atetrahydro- 1H-pyrrolizin-1-yl ester, [1S- [1alpha(Z),7(2S*,3R*), 7aalpha]]-	303-34-1	U143
Lead	Same	7439-92-1	—
Lead compounds, N.O.S.[1]	—	—	—
Lead acetate	Acetic acid, lead(2+) salt	301-04-2	U144
Lead phosphate	Phosphoric acid, lead(2+) salt (2:3)	7446-27-7	U145
Lead subacetate	Lead, bis (acetato-O) tetrahydroxytri-	1335-32-6	U146
Lindane	Cyclohexane, 1,2,3,4,5,6- hexachloro-, (1alpha, 2alpha, 3beta, 4alpha, 5alpha, 6beta)-	58-89-9	U129
Maleic anhydride	2,5-Furandione	108-31-6	U147
Maleic hydrazide	3,6-Pyridazinedione, 1,2-dihydro-	123-33-1	U148
Malononitrile	Propanedinitrile	109-77-3	U149
Melphalan	L-Phenylalanine, 4-[bis (2-chloroethyl)aminol]-	148-82-3	U150
Mercury	Same	7439-97-6	U151
Mercury compounds, N.O.S.[1]	—	—	—
Mercury fulminate	Fulminic acid, mercury(2+) salt	628-86-4	P065
Methacrylonitrile	2-Propenenitrile, 2-methyl-	126-98-7	U152
Methapyrilene	1,2-Ethanediamine, N,N-dimethyl- N'-2pyridinyl-N'- (2-thienylmethyl)-	91-80-5	U155
Methomyl	Ethanimidothioic acid, N[[(methylamino) carbonyl]oxy]-, methyl ester	16752-77-5	P066

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Methoxychlor	Benzene, 1,1'-(2,2,2trichloroethylidene) bis[4-methoxy-	72-43-5	U247
Methyl bromide	Methane, bromo-	74-83-9	U029
Methyl chloride	Methane, chloro-	74-87-3	U045
Methyl chlorocarbonate	Carbonochloridic acid, methyl ester	79-22-1	U156
Methyl chloroform	Ethane, 1,1,1-trichloro-	71-55-6	U226
3-Methylcholanthrene	Benz[j]aceanthrylene, 1,2-dihydro-3-methyl-	56-49-5	U157
4,4'-Methylenebis(2-chloroaniline)	Benzenamine, 4,4'-methylenebis [2-chloro	101-14-4	U158
Methylene bromide	Methane, dibromo-	74-95-3	U068
Methylene chloride	Methane, dichloro-	75-09-2	U080
Methyl ethyl ketone (MEK)	2-Butanone	78-93-3	U159
Methyl ethyl ketone peroxide	2-Butanone, peroxide	1338-23-4	U160
Methyl hydrazine	Hydrazine, methyl-	60-34-4	P068
Methyl iodide	Methane, iodo-	74-88-4	U138
Methyl isocyanate	Methane, isocyanato-	624-83-9	P064
2-Methyllactonitrile	Propanenitrile, 2-hydroxy-2-methyl-	75-86-5	P069
Methyl methacrylate	2-Propenoic acid, 2-methyl-, methyl ester	80-62-6	U162
Methyl methanesulfonate	Methanesulfonic acid, methyl ester	66-27-3	—
Methyl parathion	Phosphorothioic acid, O,O-dimethyl O- (4-nitrophenyl) ester	298-00-0	P071
Methylthiouracil	4(1H)-Pyrimidinone, 2,3-dihydro- 6-methyl-2-thioxo-	56-04-2	U164
Mitomycin C	Azirino[2',3':3,4] pyrrolo [1,2a] indole-4,7-dione, 6-amino-8-[[(aminocarbonyl) oxy]methyl]-1,1a,2,8,8a,8b - hexahydro-8a-methoxy-5-methyl-, [1aS (1aalpha, 8beta, 8aalpha, 8balpha)]-	50-07-7	U010
MNNG	Guanidine, N-methyl-N'-nitro-N-nitroso	70-25-7	U163
Mustard gas	Ethane, 1,1'-thiobis [2-chloro-	505-60-2	—
Naphthalene	Same	91-20-3	U165
1,4-Naphthoquinone	1,4-Naphthalenedione	130-15-4	—
alpha-Naphthylamine	1-Naphthalenamine	134-32-7	U167
beta-Naphthylamine	2-Naphthalenamine	91-59-8	U168
alpha-Naphthylthiourea	Thiourea, 1-naphthalenyl-	86-88-4	P072
Nickel	Same	7440-02-0	—
Nickel compounds, N.O.S.[1]	—	—	—
Nickel carbonyl	Nickel carbonyl Ni(CO)4, (T-4)-	13463-39-3	P073
Nickel cyanide	Nickel cyanide Ni(CN)2	557-19-7	P074
Nicotine	Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)- 54-11-5	P075	
Nicotine salts	—	—	P075
Nitric oxide	Nitrogen oxide NO	10102-43-9	P076
p-Nitroaniline	Benzenamine, 4-nitro-	100-01-6	P077
Nitrobenzene	Benzene, nitro-	98-95-3	U169
Nitrogen dioxide	Nitrogen oxide NO2	10102-44-0	P078

Appendix

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Nitrogen mustard	Ethanamine, 2-chloro-N- (2-chloroethyl)-N-methyl-	51-75-2	—
Nitrogen mustard, hydrochloride salt	—	—	—
Nitrogen mustard N-oxide	Ethanamine, 2-chloro-N- (2-chloroethyl)-N-methyl-, N-oxide	126-85-2	—
Nitrogen mustard, N-oxide, hydro- chloride salt	Nitroglycerin 1,2,3-Propanetriol, trinitrate	55-63-0	P081
p-Nitrophenol	Phenol, 4-nitro-	100-02-7	U170
2-Nitropropane	Propane, 2-nitro-	79-46-9	U171
Nitrosamines, N.O.S.[1]	—	35576-91-1D	—
N-Nitrosodi-n-butylamine	1-Butanamine, N-butyl-N-nitroso-	924-16-3	U172
N-Nitrosodiethanolamine	Ethanol, 2,2'-(nitrosoimino) bis-	1116-54-7	U173
N-Nitrosodiethylamine	Ethanamine, N-ethyl-N-nitroso-	55-18-5	U174
N-Nitrosodimethylamine	Methanamine, N-methyl-N-nitroso-	62-75-9	P082
N-Nitroso-N-ethylurea	Urea, N-ethyl-N-nitroso-	759-73-9	U176
N-Nitrosomethylethylamine	Ethanamine, N-methyl-N-nitroso-	10595-95-6	—
N-Nitroso-N-methylurea	Urea, N-methyl-N-nitroso-	684-93-5	U177
N-Nitroso-N-methylurethane	Carbamic acid, methylnitroso-, ethyl ester	615-53-2	U178
N-Nitrosomethylvinylamine	Vinylamine, N-methyl-N-nitroso-	4549-40-0	P084
N-Nitrosomorpholine	Morpholine, 4-nitroso-	59-89-2	—
N-Nitrosonornicotine	Pyridine, 3-(1-nitroso-2-pyrrolidinyl)-, (S)-	16543-55-8	—
N-Nitrosopiperidine	Piperidine, 1-nitroso-	100-75-4	U179
N-Nitrosopyrrolidine	Pyrrolidine, 1-nitroso-	930-55-2	U180
N-Nitrososarcosine	Glycine, N-methyl-N-nitroso-	13256-22-9	—
5-Nitro-o-toluidine	Benzenamine, 2-methyl-5-nitro-	99-55-8	U181
Octamethylpyrophosphoramide	Diphosphoramide, octamethyl-	152-16-9	P085
Osmium tetroxide	Osmium oxide OsO4, (T-4)-	20816-12-0	P087
Paraldehyde	1,3,5-Trioxane, 2,4,6-trimethyl-	123-63-7	U182
Parathion	Phosphorothioic acid, O,O-diethyl O-(4-nitrophenyl) ester	56-38-2	P089
Pentachlorobenzene	Benzene, pentachloro-	608-93-5	U183
Pentachlorodibenzo-p-dioxins	—	—	—
Pentachlorodibenzofurans	—	—	—
Pentachloroethane	Ethane, pentachloro-	76-01-7	U184
Pentachloronitrobenzene (PCNB)	Benzene, pentachloronitro-	82-68-8	U185
Pentachlorophenol	Phenol, pentachloro-	87-86-5	See F027
Phenacetin	Acetamide, N-(4-ethoxyphenyl)-	62-44-2	U187
Phenol	Same	108-95-2	U188
Phenylenediamine	Benzenediamine	25265-76-3	—
Phenylmercury acetate	Mercury, (acetato-O)phenyl-	62-38-4	P092
Phenylthiourea	Thiourea, phenyl-	103-85-5	P093
Phosgene	Carbonic dichloride	75-44-5	P095
Phosphine	Same	7803-51-2	P096

Appendix

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Phorate	Phosphorodithioic acid, O,O-diethyl S-[(ethylthio) methyl] ester	298-02-2	P094
Phthalic acid esters, N.O.S.[1]	—	—	—
Phthalic anhydride	1,3-Isobenzofurandione	85-44-9	U190
2-Picoline	Pyridine, 2-methyl-	109-06-8	U191
Polychlorinated biphenyls, N.O.S.[1]	—	—	—
Potassium cyanide	Potassium cyanide K(CN)	151-50-8	P098
Potassium silver cyanide	Argentate(1-), bis(cyano-C)-, potassium	506-61-6	P099
Pronamide	Benzamide, 3,5-dichloro-N-(1,1-dimethyl-2-propynyl)-	23950-58-5	U192
1,3-Propane sultone	1,2-Oxathiolane, 2,2-dioxide	1120-71-4	U193
n-Propylamine	1-Propanamine	107-10-8	U194
Propargyl alcohol	2-Propyn-1-ol	107-19-7	P102
Propylene dichloride	Propane, 1,2-dichloro-	78-87-5	U083
1,2-Propylenimine	Aziridine, 2-methyl-	75-55-8	P067
Propylthiouracil	4(1H)-Pyrimidinone, 2,3-dihydro-6-propyl-2-thioxo-	51-52-5	—
Pyridine	Same	110-86-1	U196
Reserpine	Yohimban-16-carboxylic acid, 11,17-dimethoxy-18- [(3,4,5-trimethoxybenzoyl)oxy]-smethyl ester, (3beta, 16beta, 17alpha, 18beta, 20alpha)	50-55-5	U200
Resorcinol	1,3-Benzenediol	108-46-3	U201
Saccharin	1,2-Benzisothiazol-3(2H)-one, 1,1dioxide	81-07-2	U202
Saccharin salts	—	—	U202
Safrole 1,3-Benzodioxole, 5-(2-propenyl)-	94-59-7	U203	
Selenium	Same	7782-49-2	—
Selenium compounds, N.O.S.[1]	—	—	—
Selenium dioxide	Selenious acid	7783-00-8	U204
Selenium sulfide	Selenium sulfide SeS2	7488-56-4	U205
Selenourea	Same	630-10-4	P103
Silver	Same	7440-22-4	—
Silver compounds, N.O.S.[1]	—	—	—
Silver cyanide	Silver cyanide Ag(CN)	506-64-9	P104
Silvex (2,4,5-TP)	Propanoic acid, 2-(2,4,5-trichlorophenoxy)	93-72-1	See F027
Sodium cyanide	Sodium cyanide Na(CN)	143-33-9	P106
Streptozotocin	D-Glucose, 2-deoxy-2-[[(methylnitrosoamino) carbonyl]amino]-	18883-66-4	U206
Strontium sulfide	Strontium sulfide SrS	1314-96-1	P107
Strychnine	Strychnidin-10-one	57-24-9	P108
Strychnine salts	—	—	P108
TCDD	Dibenzo[b,e][1,4]dioxin, 2,3,7,8-tetrachloro-	1746-01-6	—
1,2,4,5-Tetrachlorobenzene	Benzene, 1,2,4,5-tetrachloro-	95-94-3	U207
Tetrachlorodibenzo-p-dioxins	—	—	—
Tetrachlorodibenzofurans	—	—	—
Tetrachloroethane, N.O.S.[1]	Ethane, tetrachloro-, N.O.S.	25322-20-7	—
1,1,1,2-Tetrachloroethane	Ethane, 1,1,1,2-tetrachloro-	630-20-6	U208
1,1,2,2-Tetrachloroethane	Ethane, 1,1,2,2-tetrachloro-	79-34-5	U209

Appendix

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
Tetrachloroethylene	Ethene, tetrachloro-	127-18-4	U210
2,3,4,6-Tetrachlorophenol	Phenol, 2,3,4,6-tetrachloro-	58-90-2	See F027
Tetraethyldithiopyrophosphate	Thiodiphosphoric acid, tetraethyl ester	3689-24-5	P109
Tetraethyl lead	Plumbane, tetraethyl-	78-00-2	P110
Tetraethyl pyrophosphate	Diphosphoric acid, tetraethyl ester	107-49-3	P111
Tetranitromethane	Methane, tetranitro-	509-14-8	P112
Thallium	Same	7440-28-0	—
Thallium compounds, N.O.S.[1]	—	—	—
Thallic oxide	Thallium oxide Tl2O3	1314-32-5	P113
Thallium(I) acetate	Acetic acid, thallium(1) salt	563-68-8	U214
Thallium(I) carbonate	Carbonic acid, dithallium(1) salt	6533-73-9	U215
Thallium(I) chloride	Thallium chloride TlCl	7791-12-0	U216
Thallium(I) nitrate	Nitric acid, thallium(1) salt	10102-45-1	U217
Thallium selenite	Selenious acid, dithallium(1) salt	12039-52-0	P114
Thallium(I) sulfate	Sulfuric acid, dithallium(1) salt	7446-18-6	P115
Thioacetamide	Ethanethioamide	62-55-5	U218
Thiofanox	2-Butanone, 3,3-dimethyl-1-(methylthio)-, 0-[(methylamino)carbonyl] oxime	39196-18-4	P045
Thiomethanol	Methanethiol	74-93-1	U153
Thiophenol	Benzenethiol	108-98-5	P014
Thiosemicarbazide	Hydrazinecarbothioamide	79-19-6	P116
Thiourea	Same	62-56-6	U219
Thiram	Thioperoxydicarbonic diamide [(H2N)C(S)]2S2, tetramethyl-	137-26-8	U244
Toluene	Benzene, methyl-	108-88-3	U220
Toluenediamine	Benzenediamine, ar-methyl-	25376-45-8	U221
Toluene-2,4-diamine	1,3-Benzenediamine, 4-methyl-	95-80-7	—
Toluene-2,6-diamine	1,3-Benzenediamine, 2-methyl-	823-40-5	—
Toluene-3,4-diamine	1,2-Benzenediamine, 4-methyl-	496-72-0	—
Toluene diisocyanate	Benzene, 1,3-diisocyanatomethyl-	26471-62-5	U223
o-Toluidine	Benzenamine, 2-methyl-	95-53-4	U328
o-Toluidine hydrochloride	Benzenamine, 2-methyl-, hydrochloride	636-21-5	U222
p-Toluidine	Benzenamine, 4-methyl-	106-49-0	U353
Toxaphene	Same	8001-35-2	P123
1,2,4-Trichlorobenzene	Benzene, 1,2,4-trichloro-	120-82-1	—
1,1,2-Trichloroethane	Ethane, 1,1,2-trichloro-	79-00-5	U227
Trichloroethylene	Ethene, trichloro-	79-01-6	U228
Trichloromethanethiol	Methanethiol, trichloro-	75-70-7	P118
Trichloromonofluoromethane	Methane, trichlorofluoro-	75-69-4	U121
2,4,5-Trichlorophenol	Phenol, 2,4,5-trichloro-	95-95-4	See F027
2,4,6-Trichlorophenol	Phenol, 2,4,6-trichloro-	88-06-2	See F027
2,4,5-T	Acetic acid, (2,4,5-trichlorophenoxy)	93-76-5	See F027
Trichloropropane, N.O.S.[1]	—	25735-29-9	—
1,2,3-Trichloropropane	Propane, 1,2,3-trichloro-	96-18-4	—

Appendix

Common name	Chemical abstracts name	Chemical abstracts No.	Hazardous waste No.
O,O,O-Triethyl phosphorothioate	Phosphorothioic acid, O,O,O-triethyl ester	126-68-1	—
1,3,5-Trinitrobenzene	Benzene, 1,3,5-trinitro-	99-35-4	U234
Tris(1-aziridinyl) phosphine sulfide	Aziridine, 1,1',1 "phosphinothioylidynetris-	52-24-4	—
Tris(2,3-dibromopropyl) phosphate	1-Propanol, 2,3-dibromo-, phosphate (3:1)	126-72-7	U235
Trypan blue	l2,7-Naphthalenedisulfonic acid, 3,3' [(3,3'-dimethyl [1,1'-biphenyl]-4,4'-diyl) bis(azo)]-bis [5-amino-4-hydroxy-, tetrasodium salt	72-57-1	U236
Uracil mustard	2,4-(1H,3H)- Pyrimidinedione, 5-[bis(2-chloroethyl) amino]-	66-75-1	U237
Vanadium pentoxide	Vanadium oxide V2O5	1314-62-1	P120
Vinyl chloride	Ethene, chloro-	75-01-4	U043
Warfarin	2H-1-Benzopyran-2-one, 4-hydroxy-3- (3-oxo-1-phenylbutyl)-, when present at concentrations less than 0.3%	81-81-2	U248
Warfarin	2H-1-Benzopyran-2-one, 4-hydroxy-3- (3-oxo-1-phenylbutyl)-, when present at concentrations greater than 0.3%	81-81-2	P001
Warfarin salts, when present at concentrations less than 0.3%	—	—	U248
Warfarin salts, when present at concentrations greater than 0.3%	—	—	P001
Zinc cyanide	Zinc cyanide Zn(CN)2	557-21-1	P121
Zinc phosphide	Zinc phosphide Zn3P2, when present at concentrations greater than 10%	1314-84-7	P122
Zinc phosphide	Zinc phosphide Zn3P2, when present at concentrations of 10% or less	1314-84-7	U249

[1] The abbreviation N.O.S. (not otherwise specified) signifies those members of the general class not specifically listed by name in this appendix.

APPENDIX V

METHOD OF ANALYSIS FOR CHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS[1,2,3,4]

Method 8280

1. Scope and Application

1.1 This method measures the concentration of chlorinated dibenzo-p-dioxins and chlorinated dibenzofurans in chemical wastes including still bottoms, filter aids, sludges, spent carbon, and 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 shall take necessary precautions to prevent exposure to the analyst, or to others, of materials known or believed to contain CDDs or CDFs.

2. Summary of the Method

2.1 This method is an analytical extraction cleanup procedure, and capillary column gas chromatographlow resolution mass spectrometry method, using capillary column GC/MS conditions and internal standard techniques, which allow for the measurement of PCDDs and PCDFs in the extract.

2.2 If interferences are encountered, the method provides selected general purpose cleanup procedures to aid the analyst in their elimination.

3. Interferences

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

3.2 Interferences co-extracted from the samples will vary considerably from source to source, depending upon the diversity of the industry being sampled. PCDD is often associated with other interfering chlorinated compounds such as PCBs which may be at concentrations several orders of magnitude higher than that of PCDD. While general cleanup techniques are provided as part of this method, unique samples may require additional cleanup approaches to achieve the sensitivity stated in Table 1.

Table 1
Gas Chromatography of TCDD

Column	Retention time (min.)	Detection limit (g/kg) [1]
Glass capillary	9.5	0.003

[1] Detection limit for liquid samples is 0.003 µg/l. This is calculated from the minimum detectable GC response being equal to 5 times the GC background noise assuming a 1 ml effective final volume of the 1 liter sample extract, and a GC injection of 5 microliters. Detection levels apply to both electron capture and GC/MS detection. For further details see 44 FR 69526, December 3, 1979.

3.3 The other isomers of tetrachlorodibenzo-p-dioxin may interfere with the measurement of 2,3,7,8-TCDD. Capillary column gas chromatography is required to resolve those isomers that yield virtually 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 recommended. The container shall be washed and solvent rinsed before use to minimize interferences.

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

4.1.3. Compositing equipment-automatic or manual composing system. No tygon or rubber tubing may be used, and the system shall 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, ± 2° C. The bath should be used 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 Column: SP-2250 coated on a 30 m long X 0.25 mm I.D. glass column, Supelco No. 2-3714 or equivalent. Glass capillary column conditions: Helium carrier gas at 30 cm/sec linear velocity run splitless. Column temperature is 210° C.

4.3.3 Mass spectrometer: Capable of scanning from 35 to 450 amu every 1 sec or less, utilizing 70 volts, nominal, electron energy in the electron impact 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 shall also be capable of selected ion monitoring (SIM) for at least 4 ions simultaneously, with a cycle time of 1 sec or less. Minimum integration time for SIM is 100 ms. Selected ion monitoring is verified by injecting .015 ng of TCDD Cl^{37} to give a minimum signal to noise ratio of 5 to 1 at mass 328.

Table 2
DFTPP Key Ions and Ion Abundance Criteria[1]

Mass	Ion abundance criteria
51	30-60% of mass 198.
68	Less than 2% of mass 69.
70	Less than 2% of mass 69.

127 ... 40-60% of mass 198.
197 ... Less than 1% of mass 198.
198 ... Base peak, 100% relative abundance.
199 ... 5-9% of mass 198.
275 ... 10-30% of mass 198.
365 ... Greater than 1% of mass 198.
441 ... Present but less than mass 443.
442 ... Greater than 40% of mass 198.
443 ... 17-23% of mass 442.

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

4.3.4 GC/MS interface: Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria (see Sections 6.1 to 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. The interface must be capable of transporting at least 10 ng of the components of interest from the GC to the MS.

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

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

4.5 Flint glass bottle (Teflon-lined screw cap).

4.6 Reacti-vial (silanized) (Pierce Chemical Co.).

5. Reagents

5.1 Potassium hydroxide-(ACS), 2% in distilled water.

5.2 Sulfuric acid-(ACS), concentrated.

5.3 Methylene chloride, hexane, benzene, petroleum ether, methanol, tetradecane-pesticide quality or equivalent.

5.4 Prepare stock standard solutions of TCDD and ^{37}Cl-TCDD (molecular weight 328) in a glove box. The stock solutions are stored in a glovebox, and checked frequently for signs of degradation or evaporation, especially just prior to the preparation of working standards.

5.5 Alumina-basic, Woelm; 80/200 mesh. Before use activate overnight at 600° C, cool to room temperature in a dessicator.

5.6 Prepurified nitrogen gas

6.0 Calibration

6.1 Before using any cleanup procedure, the analyst shall process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from reagents.

6.2 Prepare GC/MS calibration standards for the internal standard technique that will allow for measurement of relative response factors of at least 3 CDD/37CDD ratios. Thus, for TCDDs, at least 3 TCDD/37Cl-TCDD and TCDF/37Cl-TCDF shall be determined.[5] The ^{37}Cl-TCDD/F concentration in the standard shall be fixed and selected to yield a reproducible response at the most sensitive setting of the mass spectrometer. Response factors for PCDD and HxCDD may be determined by measuring the response of the tetrachloro-labelled compounds relative to that of the unlabelled 1,2,3,4- or 2,3,7,8-TCDD, 1,2,3,4,7PCDD or 1,2,3,4,7,8-HxCDD, which are commercially available.[6]

6.3 Assemble the necessary GC/MS apparatus and establish operating parameters equivalent to those indicated in Section 11.1 of this method. Calibrate the GC/MS system according to Eichelberger, et al. (1975) by the use of decafluorotriphenyl phosphine (DFTPP). By injecting calibration standards, establish the response factors for CDDs vs. ^{37}Cl-TCDD, and for CDFs vs. ^{37}Cl-TCDF. The detection limit provided in Table 1 should be verified by injecting .015 ng of ^{37}Cl-TCDD which shall give a minimum signal to noise ratio of 5 to 1 at mass 328.

7. Quality Control

7.1 Before processing any samples, the analyst shall demonstrate through the analysis of a distilled water method blank, that all glassware and reagents are interference-free. Each time a set of samples is extracted, or there is a change in reagents, a method blank shall be processed as a safeguard against laboratory contamination.

7.2 Standard quality assurance practices shall be used with this method. Field replicates shall be collected to measure the precision of the sampling technique. Laboratory replicates shall be analyzed to establish the precision of the analysis. Fortified samples shall be analyzed to establish the accuracy of the analysis.

8. Sample Collection, Preservation, and Handling

8.1 Grab and composite samples shall be collected in glass containers. Conventional sampling practices should be followed, except that the bottle shall not be prewashed with sample before collection. Composite samples shall be collected in glass containers in accordance with the requirements of the RCRA program. Sampling equipment shall be free of tygon and other potential sources of contamination.

8.2 The samples shall be iced or refrigerated from the time of collection until extraction. Chemical preservatives shall not be used in the field unless more than 24 hours will elapse before delivery to the laboratory. If an aqueous sample is taken and the sample will not be extracted within 48 hours of collection, the sample shall be adjusted to a pH range of 6.0-8.0 with sodium hydroxide or sulfuric acid.

8.3 All samples shall be extracted within 7 days and completely analyzed within 30 days of collection.

9. Extraction and Cleanup Procedures

9.1 Use an aliquot of 1-10 g sample of the chemical waste or soil to be analyzed. Soils shall be dried using a stream of prepurified nitrogen and pulverized in a ball-mill or similar device. Perform this operation in a clear area with proper hood space. Transfer the sample to a tared 125 ml flint glass bottle (Teflonlined screw cap) and determine the weight of the sample. Add an appropriate quantity of ^{37}Cl-labelled 2,3,7,8-TCDD (adjust the quantity according to the required minimum detectable concentration), which is employed as an internal standard.

9.2 Extraction

9.2.1 Extract chemical waste samples by adding 10 ml methanol, 40 ml petroleum ether, 50 ml doubly distilled water, and then shaking the mixture for 2 minutes. Tars shall be completely dissolved in any of the recommended neat solvents. Activated carbon samples shall be extracted with benzene using method 3540 in SW-846 (Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, available from G.P.O. Stock 1B055-022-81001-2). Quantitatively transfer the organic extract or dissolved sample to a clean 250 ml flint glass bottle (Teflon lined screw cap), add 50 ml doubly distilled water and shake for 2 minutes. Discard the aqueous layer and proceed with Step 9.3.

9.2.2 Extract soil samples by adding 40 ml of petroleum ether to the sample, and then shaking for 20 minutes. Quantitatively transfer the organic extract to a clean 250 ml flint glass bottle (Teflon-lined screw cap), add 50 ml doubly distilled water and shake for 2 minutes. Discard the aqueous layer and proceed with Step 9.3.

9.3 Wash the organic layer with 50 ml of 20% aqueous potassium hydroxide by shaking for 10 minutes and then remove and discard the aqueous layer.

9.4 Wash the organic layer with 50 ml of doubly distilled water by shaking for 2 minutes, and discard the aqueous layer.

9.5 Cautiously add 50 ml concentrated sulfuric acid and shake for 10 minutes. Allow the mixture to stand until layers separate (approximately 10 minutes), and remove and discard the acid layer. Repeat acid washing until no color is visible in the acid layer.

9.6 Add 50 ml of doubly distilled water to the organic extract and shake for 2 minutes. Remove and discard the aqueous layer and dry the organic layer by adding 10g of anhydrous sodium sulfate.

9.7 Concentrate the extract to incipient dryness by heating in a 55° C water bath and simultaneously flowing a stream of prepurified nitrogen over the extract. Quantitatively transfer the residue to an alumina microcolumn fabricated as follows:

9.7.1 Cut off the top section of a 10 ml disposable Pyrex pipette at the 4.0 ml mark and insert a plug of silanized glass wool into the tip of the lower portion of the pipette.

9.7.2 Add 2.8g of Woelm basic alumina (previously activated at 600° C overnight and then cooled to room temperature in a desiccator just prior to use).

9.7.3 Transfer sample extract with a small volume of methylene chloride.

9.8 Elute the microcolumn with 10 ml of 3% methylene chloride-in-hexane followed by 15 ml of 20% methylene chloride-in-hexane and discard these effluents. Elute the column with 15 ml of 50% methylene chloride-in-hexane and concentrate this effluent (55° C water bath, stream of prepurified nitrogen) to about 0.3-0.5 ml.

9.9 Quantitatively transfer the residue (using methylene chloride to rinse the container) to a silanized Reacti-Vial (Pierce Chemical Co.). Evaporate, using a stream of prepurified nitrogen, almost to dryness, rinse the walls of the vessel with approximately 0.5 ml methylene chloride, evaporate just to dryness, and tightly cap the vial. Store the vial at 5° C until analysis, at which time the sample is reconstituted by the addition of tridecane.

9.10 Approximately 1 hour before GC-MS (HRGC-LRMS) analysis, dilute the residue in the microreaction vessel with an appropriate quantity of tridecane. Gently swirl the tridecane on the lower portion of the vessel to ensure dissolution of the CDDs and CDFs. Analyze a sample by GC/EC to provide insight into the complexity of the problem, and to determine the manner in which the mass spectrometer should be used. Inject an appropriate aliquot of the sample into the GC-MS instrument, using a syringe.

9.11 If, upon preliminary GC-MS analysis, the sample appears to contain interfering substances which obscure the analyses for CDDs and CDFs, high performance liquid chromatographic (HPLC) cleanup of the extract is accomplished, prior to further GC-MS analysis.

10. HPLC Cleanup Procedure[7]

10.1 Place approximately 2 ml of hexane in a 50 ml flint glass sample bottle fitted with a Teflon-lined cap.

10.2 At the appropriate retention time, position sample bottle to collect the required fraction.

10.3 Add 2 ml of 5% (w/v) sodium carbonate to the sample fraction collected and shake for one minute.

10.4 Quantitatively remove the hexane layer (top layer) and transfer to a micro-reaction vessel.

10.5 Concentrate the fraction to dryness and retain for further analysis.

11. GC/MS Analysis

11.1 The following column conditions are recommended: Glass capillary column conditions: SP-2250 coated on a 30 m long x 0.25 mm I.D. glass column (Supelco No. 2-3714, or equivalent) with helium carrier gas at 30 cm/sec linear velocity, run splitless. Column temperature is 210° C. Under these conditions the retention time for TCDDs is about 9.5 minutes. Calibrate the system daily with, a minimum, 3 injections of standard mixtures.

11.2 Calculate response factors for standards relative to 37Cl-TCDD/F (see Section 12).

11.3 Analyze samples with selected ion monitoring of at least 2 ions from Table 3. Proof of the presence of CDD or CDF exists if the following conditions are met:

11.3.1 The retention time of the peak in the sample shall match that in the standard, within the performance specifications of the analytical system.

11.3.2 The ratio of ions shall agree within 10% with that of the standard.

11.3.3 The retention time of the peak maximum for the ions of interest shall exactly match that of the peak.

Table 3
List of Accurate Masses Monitored Using GC Selected-Ion Monitoring, Low Resolution, Mass Spectrometry for Simultaneous Determination of Tetra-, Penta- and Hexachlorinated Dibenzo-p-Dioxins and Dibenzofurans

Class of chlorinated dibenzodioxin or dibenzofuran	Number of chlorine substituents (x)	Monitored m/z for dibenzodioxins $C_{12}H_{8-x}O_2I_x$	Monitored m/z for dibenzofurans $C_{12}H_{8-x}OI_x$	Approximate theoretical ratio expected on basis of isotopic abundance
Tetra	4	[1] 319.897	[1] 303.902	0.74
—	—	321.894	305.903	1.00
—	—	[1] 327.885	[2] 311.894	—
—	—	[3] 256.933	—	0.21
—	—	[3] 258.930	—	0.20
Penta	5	[1] 353.858	[1] 337.863	0.57
—	—	355.855	339.860	1.00
Hexa	6	389.816	373.821	1.00
—	—	391.813	375.818	0.87

[1] Molecular ion peak.

[2] Cl4-labelled standard peaks.

[3] Ions which can be monitored in TCDD analyses for confirmation purposes.

11.4 Quantitate the CDD and CDF peaks from the response relative to the 37Cl-TCDD/F internal standards. Recovery of the internal standard should be greater than 50%.

11.5 If a response is obtained for the appropriate set of ions, but is outside the expected ratio, a coeluting impurity may be suspected. In this case, another set of ions characteristic of the CDD/CDF molecules shall be analyzed. For TCDD a good choice of ions is m/e 257 and m/e 259. For TCDF a good choice of ions is m/e 241 and 243. These ions are useful in characterizing the molecular structure to TCDD or TCDF. For analysis of TCDD good analytical technique would require using all 4 ions, m/e 257, 320, 322, and 328, to verify detection and signal to noise ratio of 5 to 1. Suspected impurities such as DDE, DDD or PCB residues can be confirmed by checking for their major fragments. These materials can be removed by the cleanup columns. Failure to meet criteria shall be explained in the report, or the sample reanalyzed.

11.6 If broad background interference restricts the sensitivity of the GC/MS analysis, the analyst shall employ cleanup procedures and reanalyze by GC/MS. See section 10.0.

11.7 In those circumstances where these procedures do not yield a definitive conclusion, the use of high resolution mass spectrometry is suggested.

12. Calculations

12.1 Determine the concentration of individual compounds according to the formula:

$$\text{Concentration, } \mu g / gm = \frac{A_x \times A_s}{G \times A_{is} \times R_f}$$

where:

A = μg of internal standard added to the sample[8]

G = gm of sample extracted

A_s = area of characteristic ion of the compound being quantified.

A_{is} = area of characteristic ion of the internal standard

R_f = response factor[9]

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

C_c = concentration of the standard compound

12.2 Report results in micrograms per gram without correction for recovery data. When duplicate and spiked samples are analyzed, all data obtained should be reported.

12.3 Accuracy and Precision. No data are available at this time.

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

[2] Analytical protocol for determination of TCDDs 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, OH 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, OH 45435.

[4] In general, the techniques that should be used to handle these materials are those which are followed for radioactive or infectious laboratory materials. Assistance in evaluating laboratory practices may be obtained from industrial hygienists and persons specializing in safe laboratory practices. Typical infectious waste incinerators are probably not satisfactory devices for disposal of materials highly contaminated with CDDs or CDFs. Safety instructions are outlined in EPA Test Method 613 (4.0)

See also: (1) "Program for monitoring potential contamination in the laboratory following the handling and analyses of chlorinated dibenzo-p-dioxins and dibenzofurans" by F. D. Hileman et al., In: Human and Environmental Risks of Chlorinated Dioxins and Related Compounds, R.E. Tucker, et al, eds., Plenum Publishing Corp., 1983.(2) Safety procedures outlined in EPA Method 613, Federal Register volume 44, No. 233, December 3, 1979.

[5] ^{37}Cl-labelled 2,3,7,8-TCDD and 2,3,7,8-TCDF are available from K.O.R. Isotopes, and Cambridge Isotopes, Inc., Cambridge, MA. Proper standardization requires the use of a specific labelled isomer for each congener to be determined. However, the only labelled isomers readily available are ^{37}Cl-2,3,7,8-TCDD and ^{37}Cl-2,3,7,8-TCDF. This method therefore uses these isomers as surrogates for the CDDs and the CDFs. When other labelled CDDs and CDFs are available, their use will be required.

[6] This procedure is adopted because standards are not available for most of the CDDs and CDFs, and assumes that all the congeners will show the same response as the unlabelled congener used as a standard. Although this assumption may not be true in all cases, the error will be small.

[7] For cleanup see also method #8320 or #8330, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (1982).

[8] The proper amount of standard to be used is determined from the calibrations curve (See section 6.0).

[9] If standards for PCDDs/Fs and HxCDDs/Fs are not available, response factors for ions derived from these congeners are calculated relative to ^{37}Cl-TCDD/F. The analyst may use response factors for 1,2,3,4- or 2.3.7.8-TCDD, 1,2,3,4,7-PeCDD, or 1,2,3,4,7,8-HxCDD for quantitation of TCDDs/Fs, PeCDDs/Fs and HxCDDs/Fs, respectively. Implicit in this requirement is the assumption that the same response is obtained from PCDDs/Fs containing the same number of chlorine atoms.