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**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Parts 261, 264, 265, and 775**

**[SWH-FRL 2315-5]**

# Hazardous Waste Management System: Identification and Listing of Hazardous Waste; Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities; Interim Status, Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities; and Storage and Disposal of Waste Material: Prohibition of Disposal of Tetrachlorodibenzo-p-Dioxin

**AGENCY:** Environmental Protection Agency.

**ACTION:** Proposed rule and request for comments.

**SUMMARY:** The Environmental Protection Agency (EPA) is today proposing to amend the regulations for hazardous waste management under the Resource Conservation and Recovery Act (RCRA), by listing additional hazardous wastes containing certain chlorinated dioxins, -dibenzofurans, and -phenols, and by specifying certain management standards for these wastes. These wastes are being listed as acutely hazardous. EPA is also proposing to delete several commercial chemical products from the list of hazardous wastes since these listings are duplicated in today's proposal. In addition, EPA is proposing to list these materials as solid wastes when they are recycled by being used or reused, so that these wastes remain subject to regulation when recycled in this manner. EPA also is proposing to revoke its regulation concerning the disposal of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD)-contaminated wastes under the Toxic Substances Control Act (TSCA), when this regulation under RCRA becomes effective. This action extends regulatory control to certain hazardous wastes not covered by the existing regulation. It requires handlers of such wastes to comply with the appropriate regulatory standards.

**DATE:** EPA will accept public comment on this amendment until June 3, 1983.

**ADDRESSES:** Comments should be sent to the Docket Clerk, Office of Solid Waste (WH-562), U.S. Environmental Protection Agency, 401 M Street S.W., Washington, D.C. 20460. Communications should identify the regulatory docket numbers "Section 3001/Dioxin" or "OPTS 62007".

Pursuant to provisions of RCRA and TSCA, requests for a hearing should be addressed to Eileen Claussen, Director, Office of Management, Information, and Analysis, Office of Solid Waste (WH-562), U.S. Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460.

Public Docket: The public docket for 40 CFR Parts 261, 264, and 265 is located in Room S-269C, U.S. Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460, and is available for viewing from 9:00 a.m. to 4:00 p.m., Monday through Friday, excluding holidays.

The public docket for 40 CFR Part 775 is located in Room E-107 at the same address, and is available for viewing during the same hours.

**FOR FURTHER INFORMATION CONTACT:** RCRA Hotline, toll free at (800) 424-9346 or at (202) 382-3000 or Judy Bellin (202) 382-4770.

**SUPPLEMENTARY INFORMATION:**

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## I. Background

On May 19, 1980, as part of the final and interim final regulations implementing Section 3001 of RCRA, EPA published a list of hazardous wastes that included hazardous wastes generated from non-specific sources. (See 40 CFR 261.31.) This list has been amended several times. In today's action, EPA is proposing to amend this section to add particular wastes containing certain contaminants that are, for certain animal species, among the most toxic known; these wastes consequently are of particular environmental concern. EPA has evaluated these wastes against the criteria for listing acutely hazardous and hazardous wastes (40 CFR 261.11 (a) (2) and (a) (3)), and has determined that they: (1) Are capable of causing or significantly contributing to an increase in serious irreversible or incapacitating reversible, illness, and (20) also pose a substantial present or potential threat to human health or the environment when improperly treated, stored, transported, disposed of, or otherwise managed, and therefore are acutely hazardous wastes.[1]

## II. Summary of the Proposed Listing[2]

This proposed regulation covers principally wastes from the production of certain chlorophenols and of chlorophenoxy pesticides, as well as discarded unused formulations containing tri-, tetra-, or pentachlorophenol and their derivatives. Specifically, this proposed regulation designates as hazardous certain wastes (including reactor residues, still bottoms, brines, spent filter aids, spent carbon from product purification, and sludges from wastewater treatment, but not including untreated wastewater or spent carbon from hydrogen chloride purification) resulting from the following processes:[3, 4]

[1] The RCRA definition of acutely hazardous waste is set forth at 40 CFR 261.11(a)(2). Under that definition, such a material is not necessarily "acutely toxic" in the way that term is used by toxicologists. Rather, the term is intended by EPA to identify wastes which are so hazardous that they may, either through acute or chronic exposure, "cause, or significantly contribute to an increase in serious irreversible, or incapacitating reversible, illness", regardless of how they are managed.

[2] The following acronyms and definitions are used in this document (and in the background document for this regulation):

PCDDs = all isomers of all chlorinated dibenzo-*p*-dioxins.

PCDFs = all isomers of all chlorinated dibenzo-furans

CDDs and CDFs = all isomers of the tetra-, penta-, and hexachlorodibenzo-*p*-dioxins and -dibenzofurans, repectively.

TCDDs and TCDFs = all isomers of the tetrachlorodibenzo-*p*-dioxins and -dibenzofurans, respectively.

TCDD and TCDF = the respective 2,3,7,8-isomers.

The prefixes D, Tr, T, Pe, and Hx denote the di-, tri-, tetra, penta-, and hexachloro-congeners, respectively.

[3] Not all of these wastes are generated by every process discussed in the text.

[4] We are not proposing to list untreated wastewaters or spent carbon from hydrogen chloride purification because these wastes are not expected to contain CDDs or CDFs at levels of concern.

(a) The production and manufacturing use [5] of tri-, tetra-, or pentachlorophenol and intermediates used to produce their derivatives;[6, 7]

(b) The manufacturing use of tetra-, penta-, or hexachlorobenzenes under alkaline conditions;

(c) the production of materials on equipment previously used for the production or manufacturing use of materials listed under (a) and (b) above; and

(d) discarded unused formulations containing tri-, tetra-, or pentachlorophenols, or discarded unused formulations containing compounds derived from these chlorophenols.[8]

[5] In the context of this listing, "manufacturing use" means the use of the named chemical as a reactant or chemical intermediate (for instance, as in the use of 2,4,5,-trichlorophenol (2,4,5,-TCP) as a feedstock for the synthesis of 2,4,5,-T), or as a component in a formulating process (as, for instance, in the formulation of a mixture of 2,4,5,-TCP and 1,2,4,5,-TeCP, in which these components retain their chemical identity). In the present context, the term "manufacturing use" does not include residues from the use of chlorophenoxy pesticide formulations, *e.g.*, in wood preservation.

[6] The principal manufacturing use of chlorophenols is in the synthesis of chlorophenoxy acids, esters, and amines. They are also used in the synthesis of phenolic resins, and of dye and pigment intermediates. However, only wastes from chlorophenoxy synthesis are listed as hazardous wastes, because the Agency has no data on the conditions of synthesis, generation of wastes, and the level of chlorinated dibenzo-*p*-dioxin or -dibenzofuran contamination of wastes from the synthesis of phenolic resins, dyes, and pigments. The Agency solicits data on the extent of CDD/CDF contamination of the latter wastes. We also are presently initiating sampling of some of these wastes in the course of our ongoing Industry Studies program.

[7] The 2,4,5,-TCP derivative Hexachlorophene is now synthesized from a purified 2,4,5,-TCP in an acid-catalyzed condensation reaction. Because the reaction occurs at rather low temperatures, and at acid pH, no CDD or CDF formation is expected to occur. Earlier production techniques resulted in TCDD contamination. Wastes resulting from Hexachlorophene production therefore are not included in this listing unless prepurified 2,4,5, TCP was not used, or the process took place on equipment contaminated with CDDs or CDFs.

[8] This category of listed wastes includes discarded pesticides and formulations containing tri-, tetra-, or pentachlorophenol as ingredients. Some of these materials, namely EPA Hazardous Wastes U212, 230, 231, 242, and the chlorophenoxy pesticides U232 and U233 already are hazardous wastes under 40 CFR 261.33(f) when discarded in commercial grade, technical grade, or off-specification form, or when present as the sole active ingredient in a formulation. However, discarded formulations containing these chlorophenols or chlorophenoxy compounds as one of a number of ingredients (for example, in a mixture of 2,4,5-T and 2,4,-D) are not presently considered to be hazardous wastes (unless they exhibit a characteristic of hazardous waste). These multi-ingredient formulations nevertheless are likely to be just as toxic as sole active ingredient mixtures, since the concentration of toxic ingredients is the same or higher. Today's action thus would remedy this gap in regulatory coverage by listing the multi-ingredient formulations containing the discarded listed compounds. In addition, we are amending the basis for listing these commercial chemical products to include certain chlorinated dioxins and -dibenzofurans as toxicants of concern.

[9] The statements on toxicity, persistence, and environmental contamination outlined in this preamble are more fully explained and substantiated in the background document for this listing which is available for review in the public docket.

## III. Basis for Listing

### *A. Toxicity of contaminants of concern*

*1. Toxicity of chlorinated dioxins and -dibenzofurans.* The contaminants of concern in these wastes are CDDs and CDFS, tri-, tetra-, and pentachlorophenols, and the chlorophenoxy derivatives of these chlorophenols. CDDs and CDFs are, for certain animal species, among the most potent toxic substances known.[9] TCDD and two HxCDD isomers are among the most potent animal carcinogens tested. Since each of these substances are carcinogenic in well-conducted tests in both rats and mice, they are also considered by the Agency to be potential human carcinogens (see 44 FR 39858–39879 (July 6, 1979)). In laboratory studies, TCDD has also been shown to be teratogenic, fetotoxic, and embryotoxic at extremely low doses (ng/kg/day). Based on structure-activity relationships, TCDF also may have reproductive effects at extremely low doses. Many CDDs and CDFs are acute toxicants as tested in laboratory animals at the μg/kg/day dose rate, and, even at these very low concentrations, have many observable physiologic effects. Although an EPA Scientific Advisory Panel determined 1 ng/kg body weight to be "for all practical purposes" a no observed effect level in rodents (44 FR 72337 (December 13, 1979)), several other scientists have concluded that a NOEL has not been conclusively demonstrated. Moreover, the Scientific Advisory Panel concluded that a NOEL had not been demonstrated for primates. In addition, the U.S. Food and Drug Administration has established a guideline suggesting limitation of human consumption of fish containing TCDD concentrations greater than 25–50 ng/kg (ppt). Furthermore, in several enforcement actions and in two site-specific risk assessments conducted by the Agency regarding Times Beach and Imperial in Missouri, environmental concentrations in the ppt to ppb range were determined to be levels of concern, and were used to define clean-up levels.

The Agency emphasizes that, for purposes of this regulation, it considers all CDDs and CDFs as toxicants of concern in these wastes. Many biochemical and toxicology studies have demonstrated that there are well-defined structure/activity correlations defining the acute and chronic toxic effects of PCDDs and PCDFs. Those isomers that have halogens in at least three of the four lateral ring positions (numbers 2,3,7, or 8), and that have at least one ring hydrogen atom, are the most toxic isomers. All the CDDs and CDFs substituted in this manner have extremely high acute toxicity, bind strongly to a cytosolic protein receptor, and are potent inducers of several liver enzymes. The Agency recognizes that, even within such congeneric groupings, there are differences in toxicity. There is, for instance, a 370-fold difference in acute toxicity between the 1,2,3,7,8- and the 1,2,4,7,8-PeCDD isomers; however, even the less toxic isomer has extremely high acute toxicity (oral $LD_{50}$ in the guinea pig = 1.1 mg/kg).

Only limited toxicity information is available on certain of the CDD and CDF isomers. However, many are structurally similar to other CDD and CDF isomers that are potent toxicants. The Agency may permissibly infer that certain waste constituents are toxic, based upon structural similarity to known toxicants. See *EDF v. EPA*, 598 F. 2d 62, 78–83 (D.C. Cir. 1978) (prohibition of discharge to navigable waters of less chlorinated PCBs is permissible in the absence of specific toxicologic data due to their structural similarity to the more chlorinated PCBs).

Consequently, because most of the isomers of the listed CDDs and CDFs are toxic, albeit to different degrees, because identification of individual isomers in the waste would be an excessive regulatory burden, and because the Agency believes that these wastes would contain a certain percentage of the more toxic component, the Agency has determined that it is a conservative public health assumption that all the isomers of TCDD should be considered in estimating its toxicity. We have therefore determined that all the CDDs and CDFs identified or proposed to be identified in Appendix VIII should be considered as toxicants of concern in these wastes. This decision is analogous to the finding adopted by the Agency in the case of municipal waste resource recovery facilities.[10]

*2. Toxicity of chlorophenols and their chlorophenoxy derivatives.* The other toxicants of concern in these wastes also have serious adverse effects. EPA's Carcinogen Assessment Group has

[10] *See* Interim evaluation of the health risks associated with emissions of tetrachlorinated dioxins from municipal waste resource recovery facilities. U.S. EPA, Office of the Administrator, November 16, 1981.

determined that 2,4,6-TCP is a potential human carcinogen.[11] In addition, chlorophenols may cause liver and kidney damage. Some chlorophenoxy compounds are also known or potential human carcinogens, and may have reproductive and teratogenic effects. Water Quality Criteria have been established for many of these compounds. For example, for 2,4,6-TCP, the criterion for the protection of people from excess risk of developing cancer ($10^{-5}$ risk level) from the lifetime consumption of contaminated fish and water is 12 ppb. The criterion for 2,4,5-TCP (based on its chronic systemic toxic effects) is 2.6 ppm.

For several other chlorophenols, the organoleptic water quality criteria are at the ppb level (see 45 FR 79318 November 28, 1980).

*B. Contaminant concentration levels in these wastes*

The toxicants of concern are likely to be present in the listed wastes at concentrations many orders of magnitude greater than the levels which, as cited above, are of concern in terms or human health. In some cases, the Agency has inferred the presence of these contaminants from knowledge of reaction chemistry and process operating conditions. In other cases, the contamination of chemical intermediates and commercial chemical products is analytically established. For example, analysis of distillation bottoms from manufacturing processes making or using trichlorophenols can contain several hundred ppm CDDs, filter aids may contain up to 6000 ppm TCDDs, and cooling pond muds were shown to contain as much as 1200 ppm CDDs. Still bottoms from 2,4,5-TCP and 2,4,5-T production generated by the Vertac Chemical Corporation contained up to 111 ppm TCDDs. (*U.S* v. *Vertac Chemical Corp.*, 489 F. Supp. 870, 879 (D. Ark. 1980)) (improper storage and disposal of dioxin-containing wastes results in imminent and substantial endangerment warranting injunctive relief).

Some process wastes may be contaminated with CDDs or CDFs because they were generated in the course of a manufacturing process performed on equipment that was previously used for a CDD or CDF-generating process. In the manufacture of chemicals on a production train previously used for a process generating, *e.g.*, CDDs, both the product and the wastes generated can be contaminated with CDDs. This was shown to be the case, for instance, for wastes resulting from the manufacture of 2,4-D. These wastes contained TCDDs at the ppb level, presumably because the equipment, used previously to produce 2,4,5-T, remained contaminated with TCDD after production shifted to 2,4-D (45 *FR* 32677, May 19, 1980).

The contamination of tri-, tetra-, and pentachlorophenols, and their phenoxy derivatives with CDDs (30–100 ppm) and CDFs (50–140 ppm) also results in the contamination of biocides and their formulations.

The concentration of higher chlorinated phenols and chlorophenoxy derivatives in these wastes also is likely to be considerable. In the case of wastes from cholorophenol production, cholorophenols (because of their solubility characteristics) are likely to be present in reactor residues, in still bottoms, and in the sludges from wastewater treatment. Wastes from the manufacturing use of these compounds likewise will contain these chlorophenols—since they are the principal raw material in the process—as well as various chlorophenoxy derivatives. Because of the nature of the purification and precipitation processes, the latter compounds will occur principally in reactor residues, on adsorbents used for product purification, and on filter aids. These compounds are known to be present in the wastes from these processes. For example, a study of the aqueous waste of one herbicide manufacturing facility found that it contained 13.5 kg/day of mixed chlorophenols, and about 32.7 kg/day of phenoxy acid. These are discharged in fairly concentrated form: a typical untreated aqueous waste stream from phenoxy acid manufacture contains 112 ppm of mixed chlorophenols and 235 ppm of chlorophenoxy acids.

Discarded pesticides and pesticide formulations containing these chlorophenols as active ingredients obviously will contain these toxicants in high (percent) concentrations.

*C. The wastes' potential to cause substantial harm if mismanaged*

Not only are the contaminants of concern present in significant concentrations, but they are capable of migrating from waste matrices and reaching environmental receptors in potentally dangerous concentrations. These contaminants are persistent[12]—CCDs and CDFs extremely so—and several can accumulate in the food chain. The measured bioaccumulation factor (BCF) for TCDD is species dependent, and varies from 2,000 to 48,000; structure/activity considerations make it reasonable to assume that the BCF for other CDDs and CDFs are within the same range. The calculated BCF for the chlorophenols ranges from 290 for 2,4,5-TCP to 610 for 2,4,6-TCP (however, the measured steady state BCF for PCP is only 13). Thus, if these toxicants migrate from these wastes, even in extremely low concentrations, they can accumulate in biological organisms at much higher levels, increasing the likelihood of substantial harm to human health and the environment.

These toxicants, moreover, are mobile in the environment, particularly as a result of water run-off or wind dispersion of contaminated particles, and can migrate from these wastes if they are improperly managed. Although CDDs and CDFs are relatively water insoluble, and bind strongly to organic soil constituents, improper land disposal could cause substantial harm to environmental receptors. Pollution of air and surface waters can occur, perhaps as a result of windblown dust, water run-off or erosion, or flooding of waste disposal sites. All of these scenarios have occurred. In the *Vertac* case cited earlier, improper storage and disposal of wastes from the manufacture of 2,4,5-TCP, 2,4-D, and 2,4,5-T resulted in significant environmental contamination. Fish and other aquatic life in a local stream accumulated TCDD at levels as high as 600 ppt. The court concluded:

> Dioxins * * * can and have been transported off the Vertac site on dust, by the action of landfill areas and equalization basin area, and when people and equipment move to and from the Vertac site. Samples show that dioxin has been transported off the Vertac site into fish and sediment in (a local stream), and also into the Jacksonville sewage treatment plant. (489 F. Supp. at 879)

TCDDs also have been detected at levels of concern in the sediments of streams, public sewers, and home sumps at other sites, including Love Canal. TCDD has been reported in fish and crayfish living in contaminated streams, in concentrations (600 ng/kg) up to fifteen times higher than that at which FDA advises that human consumption be limited. High ppt concentrations have been reported for other CDDs and CDFs in fish. Because of their insolubility in

[11] U.S. EPA, Ambient water quality criteria for chlorophenols. EPA 440/5-80-032.

[12] CDDs (and presumably CDFs) are highly resistant to microbial degradation. The Agency thus believes that these toxicants will be present in wastewater treatment sludges. The listed chlorophenols and their chlorophenoxy derivatives are biodegradable, but where overloading and inadequate treatment occur, and in the anaerobic environment of sludge disposal, they may persist.

water, and their strong binding to organic soil constituents, CDDs and CDFs are not ordinarily expected to leach to ground water if proper precautions are taken. However, if these wastes are co-disposed with solubilizing solvents, or disposed in situations where soil binding site are exhausted, ground water contamination could result.

Although chlorophenols and chlorophenoxy compounds are subject to environmental degradation, including biodegradation by adapted communities, environmental pollution from these constituents has occurred where wastes from the production and manfuacturing use of chlorophenols were mismanaged. More than twenty-five years after the improper disposal of chlorophenolic wastes at Love Canal, tri-, tetra-, and pentachlorophenols were identified in soil, water, and storm sewer sediments at concentrations ranging from 14 ppb (PCP in sump water) to 496 ppm (TCPs in storm sewer sediment).

A further risk to human health may be posed by improper incineration of these wastes. Improper incineration of chlorophenols are prediced to form CDDs and CDFs as products of incomplete combustion,[13] posing a further risk of substantial harm. Indeed, as discussed later in this preamble, the Agency is studying whether different criteria or management standards (*e.g.*, higher destruction and removal efficiency for the incineration of these wastes) are appropriate and practical.

D. Listing as acutely hazardous wastes.

It is clear from this discussion that these wastes have the potential to cause substantial harm, if mismanaged. The Agency is further convinced that these are acutely hazardous wastes under 40 CFR 261.11(a)(2), since they contain contaminants which, when tested in animals, are among the most toxic contaminants known, and thus are capable of causing, or significantly contributing to serious irreversible, or incapacitating reversible, illness.[14] This standard is taken directly from Section 1004(5) of RCRA, and is reserved for wastes particularly likely to pass a substantial risk to human health and the environment (see preamble to Part 261, 45 FR 33106, May 19, 1980).

Additional reasons for listing these wastes as acutely hazardous are that the wastes have been implicated in a series of damage incidents, among them the incidents of Love Canal and Times Beach. The Agency also has been compelled to exert regulatory control over many of these wastes under the Toxic Substances Control Act in the face of the unreasonable risk posed by ongoing and contemplated waste management practices (see further discussion in Part VI. below).

The practical consequences of such a listing are two-fold. First, these wastes will be subject to the 1 kg/month small quantity generator limitation contained in 40 CFR 261.5(e).[15]

The Agency deems 1 kg to be equivalent, for all practical purposes, to total control of the management of these wastes since they are generated in amounts far greater than 1 kg. The Agency solicits comments on the appropriateness of this limitation, however. Second, the residues in empty containers that contain these listed wastes are subject to control under Subtitle C of RCRA, unless the container has been triple-rinsed using a solvent capable of cleaning the container, or the container has been otherwise cleaned by a method that has been shown to achieve equivalent removal. In addition, as explained more fully below, we are proposing that these wastes be managed only at treatment, storage, and disposal facilities that have been fully permitted under RCRA (except as discussed in Section VII.). In the Agency's judgment, these wastes should be managed pursuant to the most stringent appropriate standards that are contained in the RCRA hazardous waste management regulations.

## IV. Removal of Certain Commercial Chemical Products Listed in 40 CFR 261.33(f)

As discussed in the previous section, the Agency is proposing to list as acutely hazardous those unused discarded formulations containing tri-, tetra-, or pentachlorophenol and discarded formulations containing as ingredients compounds derived from these chlorophenols. Some of these materials already are hazardous wastes under 40 CFR 261.33(f) when discarded or intended for discard in commercial grade, technical grade, or off-specification form, or when the toxicant is present in formulations as the sole active ingredient. They were originally listed as toxic (rather than acutely hazardous) because the Agency did not at that time consider the presence of CDDs and CDFs. However, as shown above, these formulations will contain chlorinated dioxins and -dibenzofurans, because the chlorophenol or chlorophenoxy derivatives will themselves be contaminated with CDDs and CDFs. For this reason, we are now listing them as actutely hazardous under 40 CFR 261.11(a)(2).

To avoid listing the same waste under two different (and inconsistent) provisions, we are proposing to remove EPA Hazardous Wastes U212, U230, U231, U232, U233, and U242 from 40 CFR 261.33(f). As a consequence, there should be no confusion that these wastes will be subject to a small quantity generator exclusion of 1 kg/mo.

## V. Regulatory Status of These Materials When Recycled by Being Used or Reused

On May 19, 1980, EPA promulgated a definition of solid waste which, among other things, states which materials are solid wastes when recycled. In promulgating this rule, EPA established broad jurisdiction over recycled materials and recycling operations. EPA is today proposing in a separate notice to revise this rule to state that certain types of activities involving secondary material use and reuse do not constitute solid waste management; in particular, the proposal states that secondary materials recycled by being used or reused as ingredients in new products, or by being used or reused directly as products, ordinarily are not solid wastes. The proposal provides that materials so used or reused may be listed individually in § 261.2(a)(3) as solid wastes if they meet two conditions: (1) They are ordinarily disposed of (on a nationwide basis), or contain toxic constituents which are not ordinarily found in raw materials or proudcts used for the same purpose which constituents are not used during the recycling process; and (2) the materials could pose a substantial hazard to human health or the environment when so used or reused.

Both these conditions are met here. These wastes are typically disposed of or incinerated, rather than recycled.

[13] Shaub, W. M. and W. Tsang, Physical and chemical properties of dioxins in relation to their disposal. Proceedings 2nd. International Symposium on Dioxins. Arlington, VA. October 1981.

[14] By means of a site-specific exclusion petition, a generator may be able to show that a waste does not contain CDDs and/or CDFs at levels sufficient to sustain regulatory concern as acutely hazardous waste. Such levels, however, as well as the presence of chlorophenols or chlorophenoxy compounds, may still render such wastes hazardous.

[15] The Agency is proposing today to amend this provision to apply to all acutely hazardous wastes, not just to the acutely hazardous wastes listed in 40 CFR 261.33(e). At the time § 261.5 was written, there were no acutely hazardous wastes other than those in § 261.33(e). Now that we are proposing to list wastes in § 261.31 as acutely hazardous, we are proposing to conform the reference to acutely hazardous waste in § 261.5. For the same reason, we are proposing to make the same type of conforming change to § 261.7(b)—the provision stating when containers that have held acutely hazardous wastes are "empty."

They also contain significant levels of hazardous constituents—CDDs and CDFs—not ordinarily found in raw materials or analogous commercial products, nor would these toxicants contribute to the efficacy of the recycling practice. In addition, in light of their toxicity and their environmental persistence, these wastes could pose the same potential for causing substantial harm when used or reused as when disposed. Since use or reuse would be unregulated, the potential for harm in fact is probably greater.[16]

Accordingly, we are proposing elsewhere in today's Federal Register to list these materials as solid wastes when they are used or reused. As a result, these wastes will remain subject to regulation when transported and stored under the Subtitle C regulations even when recycled by being used or reused as ingredients in new products, or by being used or reused directly as products.

## VI. Relation of Today's Proposal to Regulation of TCDD-Contaminated Wastes Under the Toxic Substances Control Act

Many wastes containing TCDD are presently regulated under 40 CFR Part 775, a regulation issued under Section 6 of the Toxic Substances Control Act (TSCA).[17] This regulation, promulgated on May 19, 1980 (45 FR 32676), prohibits the Vertac Chemical Company from disposing of certain wastes containing TCDD, and requires the company to store and monitor these wastes until a long-term management solution can be determined. The regulation also requires other persons intending to dispose of TCDD wastes (defined as those resulting from the production of 2,4,5-TCP or its pesticide derivatives, or substances produced on equipment that was previously used for the production of 2,4,5-TCP or its pesticide derivatives) to notify the Agency 60 days in advance of such disposal. The regulation does not apply, however, "to persons disposing of wastes containing TCDD at facilities permitted for disposal of TCDD under Section 3005(c) of RCRA." (See 40 CFR 775.197.)

On January 5, 1982, EPA issued an Advance Notice of Proposed Rulemaking (ANPRM) (47 FR 193), announcing the Agency's intent to review the TCDD disposal rule (40 CFR 775), and solicited comment to aid the Agency in determining the most appropriate long-term solution for TCDD-contaminated wastes. Several comments received on this ANPRM stated that the regulation of treatment and disposal of hazardous wastes properly belongs under RCRA, and that the Agency should avoid overlapping and potentially contradictory approaches to the same problem under different regulatory authority, *e.g.*, TSCA and RCRA. Section 1006(b) of RCRA in fact provides that, in implementing the Act, EPA "Shall avoid duplication" with other statutes administered by the Agency. Section 9(b) of TSCA provides that the Agency must utilize its authority under the other environmental laws it administers where these laws are adequate to protect against unreasonable risk, and where there is no stong public interest in taking action under TSCA.

EPA agrees that RCRA provides the appropriate long-term solution for controlling the management of TCDD-contaminated wastes. The disposal rule under TSCA was only meant as a temporary solution. *See* 45 FR 32682. EPA, in fact, acknowledged the advantages of using RCRA, by providing that final permits issued under RCRA for disposal of TCDD-contaminated wastes would supersede the TSCA rule. The rule proposed today under RCRA will provide the safeguards of a final permit, and will, therefore, render the TSCA rule unnecessary.

Accordingly, only the RCRA rule becomes effective, EPA proposes to revoke the TSCA rule that applies to disposal of TCDD-contaminated wastes. The basis for this revocation is stated in the following paragraphs.

EPA promulgated the TSCA rule under Section 6(a) of that Act. Section 6(a) provides that EPA may prohibit or otherwise regulate any manner or method of disposal of chemical substances or mixtures if the Agency finds that there is a reasonable basis to conclude that such activities present or will present "an unreasonable risk of injury to health or the environment." Determining unreasonable risk involves an administrative judgment which is reached by balancing "the probability that harm will occur and the magnitude and severity of that harm against the effect of proposed regulatory action on the availability to society of the benefits of the substance or mixture, taking into account the availability of substitutes for the substance or mixture which do not require regulation, and other adverse effects which such proposed action may have on society." (TSCA Legislative History at 422).

In the May 19, 1980 regulation, EPA determined that removal for disposal of certain TCDD wastes at Vertac's Jacksonville, Arkansas site would present unreasonable risks. The Agency found that maintaining drummed wastes on-site, with monitoring, presented a relatively known and correctable risk, while disposing of the wastes, as proposed by *Vertac*, posed a substantially greater risk, particularly where a case-specific assessment on the management of these wastes had not been performed. (*See* Preamble to Final Rule at 45 FR 32680; Preamble to Proposed Rule at 45 FR 15595.) Similar considerations led EPA to determine that disposal of TCDD wastes by other persons without prior notification to EPA would present unreasonable risks. The minimal costs of notifying EPA sixty days before disposal, so that EPA could evaluate the management scheme proposed by the notifier, was determined to be outweighed by the risks of harm that could occur from exposure to TCDD disposed of improperly.

We now propose to regulate these wastes under RCRA. On May 19, 1980, EPA believed that the then existing RCRA regulations for treatment and disposal of hazardous waste were not appropriate for TCDD-contaminated waste because EPA had not yet developed final permit standards for land disposal or incineration of hazardous wastes. These final regulations are not effective, and provide a means of properly evaluating the various management alternatives for TCDD-contaminated wastes to ensure that these wastes are managed in a manner that does not present an unreasonable risk. Thus, when the rules proposed today under RCRA become final, it will no longer present an unreasonable risk for these wastes to be treated and disposed of in RCRA facilities, and this will be the only legal waste management option. Since promulgation of these RCRA regulations will vitiate the unreasonable risk finding under TSCA, we will at the same time revoke the TSCA May 19, 1980 regulation.

It should be noted that by doing this, we are eliminating the 60-day notification requirement under TSCA for waste disposed at facilities not

[16] We note, however, that when secondary materials that are derived from commercial pesticides (such as rinse waters from pesticide containers) are put to use as pesticides, they are not considered to be RCRA solid wastes. The Agency considers this activity to constitute continued use of the pesticide, and so not to involve waste management. Any such use, of course, would have to comply with requirements for use of the pesticide imposed under the Federal Insecticide, Fungicide, and Rodenticide Act.

[17] The hazardous waste listings proposed today are more inclusive than those regulated under TSCA, including for example, wastes from the production of certain tetrachlorophenols and chlorobenzenes.

permitted under Section 3005(c) of RCRA. However, when this rule becomes effective, it will be illegal to dispose of these wastes at facilities that have not been fully permitted. Therefore, we believe the TSCA 60-day notification requirement is unnecessary; in addition, notification under Section 3010(b) will still be required and thus, the Agency will still be informed of who is handling these types of wastes.

We also believe that it will be less confusing for the regulated community, and more cost effective, both with respect to compliance and regulatory enforcement, for waste disposal to be regulated under RCRA alone, rather than under both statutes. Moreover, the technical expertise needed to issue permits for these wastes is chiefly within the Agency's office administering RCRA. We consequently believe that the public interest warrants rescission of the TSCA rule once this RCRA regulation becomes effective.

## VII. Proposed Management of These Wastes

### *A. Management at RCRA Interim Status Facilities*

As noted, the TSCA rule presently does not allow these wastes to be disposed or treated at interim status facilities without prior approval, because management of such waste at unscrutinized interim status facilities ordinarily presents an unreasonable risk (45 FR 32682). To avoid a decrease in regulatory coverage, and in light of the waste contaminants' high toxicity, persistence, and potential to bioaccumulate, we are proposing to amend the RCRA regulations, except as noted below, for landfills, waste piles, surface impoundments, land treatment facilities, and incinerators, to require that these wastes be managed only at fully permitted facilities. The reasons for the unreasonable risk finding still hold. Interim status incinerators need not perform at 99.99% destruction and removal efficiencies, or meet the other performance standards contained in Subpart O of Part 264. Interim status landfills, waste piles, surface impoundments, and land treatment facilities need not meet the monitoring requirements in Subpart F or many of the design and operating standards of Subparts K, L, M, and N of Part 264 until they are permitted. In addition, we believe that any facility that manages these wastes should be evaluated individually by EPA before accepting them in order to ensure that the facility is designed and operated properly. The proposed regulation consequently prohibits interim status facilities from managing these wastes.

We have proposed three exceptions to this prohibition. The first applies to surface impoundments in which wastewater treatment sludges are generated. The Agency has the authority to prohibit interim status surface impoundments from receiving these wastes. If we propose this action, however, the facilities now generating the listed wastewater treatment sludges would probably have to close down until they obtain permits for their impoundments, or build alternative treatment facilities that can efficiently treat these wastes. The Agency is not proposing this course of action, and notes that Section 3004 of RCRA (as amended by the Solid Waste Disposal Act Amendments of 1980) specifically allows the Administrator, in setting standards for hazardous waste management facilities, to distinguish between new and existing facilities. The legislative history indicates that Congress was concerned with the costs of modifying existing wastewater treatment impoundments installed to meet Clean Water Act requirements (although the Agency has the authority to require such modification where appropriate). See S. Rep. No. 96-172, 96th Cong. 1st Sess., at 3. We are drawing this distinction in today's proposal.

Allowing these wastewater treatment sludges at interim status surface impoundments in which they are generated should be environmentally acceptable for the period until a permit is issued. These sludges are expected to contain lower concentrations of CDDs, CDFs, and chlorophenols than the other waste we are listing. The CDDs and CDFs present also will be adsorbed to the organic matter present; in addition, we believe that there should be little chance that solubilizing solvents, such as benzene, toluene, xylene, or halogenated benzenes, will be present in significant concentrations (since these solvents have very limited water solubility). This situation therefore should not present a significant risk of leaching. Risk of wind dispersal, one of the principal exposure pathways for CDD and CDF-containing wastes which are stored in open piles or disposed in landfills, is not present for these wastewater treatment sludges when they are in an impoundment.

We are not proposing to allow other interim status surface impoundments to manage these sludges, however, because other impoundments could contain CDD- or CDF-solubilizing residues from processes not related to chlorophenol or chlorophenoxy manufacture. In addition, manufacturing operations will not be curtailed if these impoundments have to obtain permits before receiving these wastes.

For all of these reasons, therefore, we are proposing to allow the listed wastewater treatment sludges to be managed at the interim status surface impoundments in which they are generated. However, we expect, as a first priority, to evaluate the Part B permit applications of those interim status surface impoundments that manage these wastes, in order to minimize any potential risk. In addition, if monitoring data, or a review of site management make it apparent that the wastes cannot be prevented from migrating, the owner or operator of the facility will be required to remove the waste from the surface impoundment.

The second exception is for interim status tank and container facilities, which will be allowed to accept these wastes. These facilities, although not providing maximum protection, do provide control of these wastes to prevent them from posing a substantial environmental hazard or an unreasonable risk since tanks or containers at interim status facilities must meet most of the requirements (*e.g.*, storage in non-leaking units, periodic inspections) required for fully permitted tank and container facilities. Therefore, these facilities should provide adequate management of these wastes in the short term. However, we do expect to give highest priority to examining the Part B permit applications of those interim status tank and container storage facilities that store these wastes, in order to minimize any potential risk.

The final exception is for enclosed waste piles. An "enclosed waste pile" is defined in this proposed rule as a pile that meets the requirements of § 264.250(c)—namely, that the pile is inside a structure that provides protection from run-on, precipitation, and wind dispersal; does not generate leachate, and does not contain free liquids. Under existing regulations, waste piles meeting these requirements are exempt from the otherwise-applicable permitting provisions of Subpart L of Part 264 relating to containment. (See 46 FR 55112, November 6, 1981.)

We are proposing that enclosed waste piles be allowed to accept these wastes without first obtaining a permit because enclosure of this type will guard in the short-term against the means of exposure of concern—run-off, wind dispersal, and leaching. In addition,

allowing this type of interim status facility to accept these wastes should help provide management capacity until disposal facilities receive permits to manage these wastes.

We are proposing that interim status enclosed waste piles accepting these wastes still must meet the remaining applicable requirements of Subpart L of Part 265: waste analysis, special requirements for ignitable, reactive, or incompatible wastes, and closure requirements. (Post-closure requirements would not be applicable because we are assuming that these wastes will be removed from these piles.) We note, in addition, that to be eligible for interim status, the facility must have been in existence on November 19, 1980, submitted a Part A permit application, and (if required) submitted a notification of hazardous waste activity. (See § 122.23(a) and 45 FR 76636, November 19, 1980.) Enclosed piles added at interim status facilities after November 19, 1980, or accepting these wastes after that date, may be eligible for interim status provided they meet the requirements for adding waste, increasing design capacity, and (possibly) adding a new management process. (See § 122.23(c) (1), (2), and (3) (permissible changes during interim status)).

*B. Management at Fully Permitted RCRA Facilities*

1. *Management at fully permitted landfills, waste piles, surface impoundments, lagoons, and land treatment facilities.* Except as described in the previous sections, the storage, treatment, and disposal of these wastes will be allowed only at fully permitted RCRA landfills, surface impoundments, waste piles that are not enclosed, and land treatment facilities. Enclosed waste piles that are permitted under Part 264 would not require a waste management plan prior to accepting these wastes. (The Agency made this determination because it judged that the means of enclosure satisfies the concerns the plan would address.)

In addition, before any of these particular facilities can obtain a permit and, thus, before it can accept any of the wastes proposed to be listed today, it must have a "waste management plan" that is approved by the Regional Administrator. EPA believes that the inherent hazard of these wastes, and their mismanagement history, warrants regulatory controls on potential migration above those contained in the existing permit requirements. The management plan will be the vehicle for assuring individualized consideration that the wastes will be managed safely. The plan must be submitted by the owner or operator of the facility as part of the permit application; it must describe the potential for migration of toxicants from the site via any media, and, where migration is possible, it must address measures to be taken, over and above the applicable permitting requirement, to reduce migration of the wastes or waste constituents.

At a minimum, the proposed plan must address the volume and toxicant concentrations in the wastes to be managed at the facility, the propensity of toxicants to be emitted to the air through volatilization or as aerosols or dusts during placement of the wastes, whether toxicants may migrate from or with the wastes, whether the wastes will be co-disposed with other materials having mobilizing properties, and the potential for soils to attenuate migrating toxicants if the liner system (when one is required by the regulations) is damaged and breached. Where a potential for migration is identified, the proposed plan must identify design provisions and/or operating practices to be adopted to prevent that migration. These design and operating features are in addition to those that would otherwise be required by the regulations. For example, if the facility also disposes of dioxin solubilizing solvents, the applicant may propose to segregate the wastes to prevent contact. If leaching is possible, the applicant might propose lining the unit or mixing the waste with activated carbon, organic sorbents, or other materials designed to immobolize the migrating toxicants. Whatever is proposed by the applicant must be supported by data or a technical rationale. The Regional Administrator will evaluate whether these additional management and design features are adequate to prevent migration.

As a general matter, the additional measures required under a waste management plan will focus on control measures not currently specifically required by the Part 264 land disposal regulations. For example, the plan may include specific waste treatment processes that will reduce the likelihood that dioxin will migrate out of the unit. In addition, the plan may include a demonstration that siting factors (*e.g.*, the attenuative properties of the soil beneath the site) would operate to control migration of dioxin. In most cases, EPA does not believe that it will be necessary to impose additional structural requirements (*e.g.*, liner specifications) for the unit. The Agency intends to provide detailed guidance for the preparation of a waste management plan for managing these wastes prior to issuing this regulation in final form.

Waste management plans will be considered in the normal course of the permitting process, so that no special EPA review procedures are required.

2. *Management at fully permitted incinerators.* As stated, we also are proposing to allow incineration of these wastes only at fully permitted incinerators. It is the Agency's view that incineration often is a preferred management option for these wastes, because high temperature destorys the chlorinated dioxins and -dibenzofurans. If incineration is not properly performed, however, the original toxicants may be released undestroyed, or chlorinated -dioxins, -dibenzofurans, biphenyls, and -phenols can be formed from precursors such as chlorinated biphenyls, benzenes, and -phenols.

The proposed regulation, therefore, allows these wastes to be burned only at fully permitted RCRA incinerators which have proven capability to assure 99.99% DRE for principal organic hazardous constituents (POHCs) which are as difficult, or more difficult to incinerate than CDDs or CDFs.[18] The Agency judges that such a demonstration of DRE is sufficiently rigorous to ensure the proper management of these wastes, and therefore feels that it is not necessary to require an additional management plan for incinerator facilities treating these wastes.

3. *Management at fully permitted tank and container storage facilities.* We also have tentatively decided against requiring fully permitted tank and container storage facilities to have a waste management plan approved by the Regional Administrator, before storing these wastes. The current storage regulations provide the Agency with sufficient information to evaluate the storage facility's ability to contain these wastes. Therefore, an additional management plan for tank and container storage facilities storing these wastes appears unnecessary.

*C. Other Management Options Considered or These Wastes*

1. *Standards for Interim Status Landfills, Waste Piles, Surface Impoundments, Land Treatment Facilities, and Incinerators.* The Agency considered proposing a regulation that would allow interim status land disposal and incineration facilities to manage these wastes if they obtained prior

[18] The requirements for trial burn permits are described in 40 CFR 122.27. See also, "Guidance manual for hazardous waste incinerator permits. "U.S. EPA, Office of Solid Waste, November 1982.

approval from the Regional Administrator. It was felt that the Agency could provide interim status facilities the same opportunities to handle these wastes as are provided to fully permitted facilities if the Agency can be assured that such management can be accomplished safely. The vehicle to be used to assure individualized consideration of prospective waste management would be a waste management plan that would address the factors outlined in the previous section as well as other design and operating conditions contained in the Part 264 regulations, as deemed appropriate by the Regional or State officials.

The procedures we considered for approving a waste management plan would be the same as, or similar to, those for approving a closure plan. They would allow for public participation on the plan submitted by the facility, and on a tentative decision (and a rationale therefor) of the Regional Administrator. The Regional Administrator could hold a public hearing if he believed it would aid in elucidating the issues.

However, the Agency believes that most interim status facilities probably could not adequately manage these wastes without significant changes. In addition, the amount and detail of information to be provided in the management plan could be almost equivalent to the information needed to obtain a permit. For example, as part of the waste management plan for incineration, the owner or operator of the incinerator would need to conduct a trial burn to ensure that destruction and removal efficiencies could be met. EPA thinks it is unlikely that interim status facilities would go to the expense of preparing and receiving approval on a plan, only to have to go through a later permit proceeding when their Part B application is processed.

However, the Agency solicits comments on the desirability of allowing disposal and treatment of these wastes at interim status facilities having an approved management plan for these wastes.

2. *Additional Standards for Container and Tank Storage Facilities.* The Agency believes that container storage facilities storing these wastes should meet the most stringent requirements under Part 264. Present regulations (Subpart I) do not require secondary containment for non-liquid wastes (*e.g.* tarry materials) if the storage area slopes, or the container is elevated. However, the Agency believes that secondary containment might appropriately be required for container storage areas that store all non-liquid CDD and CDF-contaminated wastes, due to the toxicity of these wastes, their potential to ooze and to spill, and the long time periods these wastes may be stored before a disposal or incinerator treatment facility can be found that is willing to accept these wastes. (For example, the wastes at the Vertac site have been stored for many years, despite the repeated attempts by the company to find a disposal site.) This requirement may be necessary to ensure that any spillage or release of these wastes is contained and not released into the environment. The Vertac damage incident, where improper storage of these wastes was responsible for considerable harm, serves as an example of these wastes' potential for harm if stored improperly. The Agency therefore is considering a provision that would require secondary containment at container storage facilities that store non-liquid CDD- and CDF-contaminated wastes.

The Agency is also considering a provision that would require secondary containment at tank storage facilities that store CDD- and CDF-contaminated wastes, due to the wastes' toxicity and the long periods of time they might be stored. The damage incident at Neosho, Missouri, when a concrete tank holding chlorophenol production still bottoms (a waste covered by this proposal) and wastewater cracked, and caused considerable contamination, illustrates the potential for harm that secondary containment could address. The Agency solicits comment on the suitability of these two provisions.

3. *Optional Standards Considered for Permitted Incinerators.* Under current regulations, a facility which has shown that it can achieve 99.99% DRE for POHC's which are more resistant to thermal degradation than are CDDs or CDFs (such as carbon tetrachloride or pentachlorophenol), may be permitted to incinderate CDD or CDF-containing wastes without conducting an additional trial burn or modifying its permit (40 CFR 264.342 and 264.343). Because of their hazardousness, the Agency is considering proposing that a facility burning these wastes notify the Regional Administrator of that fact. We are considering this requirement because it is felt that Regional authorities might wish to prioritize compliance monitoring for facilities incinerating these wastes.

The Agency solicits comments on the desirability of requiring notification to the Regional Administrator on the part of a facility that is burning CDD or CDF wastes.

4. *Development of Special Management Standards.* The Agency is considering the development of special management standards for CDD/CDF-contaminated wastes. For some wastes, high temperature incineration might be the preferred method of treatment, whereas for other wastes land disposal might be a better alternative. For the latter, disposal at sites having particular hydrogeological and topographic or surface water characteristics might be needed. The Agency is presently reviewing these problems, and may, for example, propose incineration standards that could require levels of destruction and removal efficiency (DRE's) for these wastes that are greater than the 99.99% DRE presently required under RCRA. For some wastes, land disposal controls ensuring the prevention of dust formation could be imposed, and for some wastes the Agency could prescribe the application of special technologies, such as photodechlorination, or molten salt or critical water oxidation, which are known to cause the destruction of chlorinated aromatics such as CDDs.

The Agency solicits comment with respect to the regulatory alternatives discussed above, as well as any other approaches which might realistically be considered.

## VIII. Analytical Method for Tetra-, Penta-, and Hexachloro-Dibenzo-p-Dioxins and -Dibenzofurans

In order to assist generators in the determination of the contamination of wastes with the above compounds, (*e.g.*, for delisting purposes under §§ 260.20 and 260.22 of the RCRA regulations), the Agency is proposing a method of analysis for tetra-, penta-, and hexachlorodibenzo-*p*-dioxins and -dibenzofurans (see Appendix A). The method proposed in this regulation was largely developed by the workers at Wright State University, and has been used for the analysis of TCDDs in a variety of wastes.[19] If adopted, this method will replace the method for analysis of TCDD presently listed in the solid waste test manual ("Test Methods for Evaluating Solid Waste Physical/Chemical Methods", EPA publication number SW-846). The present method is inappropriate because it is not sufficiently sensitive, and does not sufficiently eliminate interfering substances. It also does not specify the procedure to be followed for the

[19] "Analytical protocol for determination of TCDDs in phenolic chemical wastes and soil samples obtained from the proximity of chemical dumps", and "Analytical protocol for determination of chlorinated dibenzo-*p*-dioxins and chlorinated dibenzofurans in river water", Brehm Laboratory, Wright State University, Dayton, OH 45435, January 7, 1982. These protocols are available in the Docket for this listing.

analysis of the chlorinated dibenzofurans.

The proposed method subjects the sample to extraction with petroleum ether (waste not amenable to petroleum ether as an extractant, such as tar-like or carbonaceous wastes, will require extraction with other organic solvents, such as toluene, hexane, or dichloromethane).[20] The extract is successively washed with alkali and acid, subjected to fractionation on alumina, and the eluate is analyzed by high-resolution gas chromatography, using a capillary glass column, and by low-resolution mass spectrometry. In case of interference, the alumina eluate is subjected to further cleanup with high pressure liquid chromatography (HPLC).

The Agency has chosen the proposed method because it is the one that has been most successfully applied to chemical wastes. In addition, its originators have indicated to the Agency that it can be used for the analysis of both CDDs and CDFs.[21-24] For example, the proposed method has been used for the analysis of TCDD in chlorophenol still bottoms, reactor residues, oxidation pond sediments, cooling pond muds, contaminated soils, and sludge samples. The detection limit for TCDD established in these different matrices varied from 15 ppt (soils, 0.5 ppb (cooling pond muds), and 1 ppb (sludges) to 0.1–1300 ppb (still bottoms, highly variable). The recovery of added TCDD varied from 14–111 percent, averaging 76 percent. In incinerator stack effluents, the minimum detectable quantity was 3.8 ng for CDDs and 3.5 ng for CDFs, and the recovery of added CDDs or CDFs averaged 80 percent.

## IX. Questions for Comment

The Agency welcomes public comment on all aspects of this proposed rule. However, public comment is especially solicited with respect to the following questions.

1. *Should EPA develop, for CDD or CDF-containing wastes, a "characteristic" definition of hazardousness under Subpart C of Section 261 of the RCRA regulations?* Instead of listing CDD/CDF-containing wastes as hazardous under Subpart D of the Part 261 regulations, EPA considered an alternative approach, namely, identifying such wastes as "characteristic" hazardous wastes under Subpart C of Part 261. This approach would oblige the Agency to make a generic determination as to the lower level of concern regarding CDD/CDF contamination, and would then require generators to either analyze or estimate the amount of CDD's or CDF's in a waste (by actual analysis or, for example, from a knowledge of reaction chemsistry, process technology, and chemical engineering principles).

The Agency judged that this approach, although at first glance appealing because of its apparent simplicity, would not be a suitable regulatory alternative. It would require that the Agency set a concentration (as in the EP hazardous waste characteristic) defining the level at which CDD's and CDF's constitute a minimum level of concern. Heretofore, EPA has not attempted to set a lower limit for the concentration of a toxicant of concern in a waste, except in a limited manner. Instead, EPA has made qualitative assessments in determining that certain wastes should be listed in the RCRA regulations because they present a potential threat to human health and the environment, if mismanaged.

Because of the high acute and chronic toxicity properties of many of the CDD's and CDF's, as evidenced in animal studies, the Agency considered that, if a lower limit of concern were to be developed it would be *very* low. Additionally, because biological availability of these toxicants is expected to be dependent on waste matrix characteristics, it was felt that a generic risk estimation for all wastes would be extremely difficult to perform. One alternative was to set the lower limit at the limit of detection of CDD's and CDF's in the waste. However, this is not a fixed concentration. As outlined above, the limit of detection is sample and matrix-dependent. Since industrial wastes are highly variable, it may not be realistic to establish generally applicable standards for the level of detection, recovery, and reproducibility for the analytical determination of CDD's and CDF's in these wastes.

Within the above limitations, the Agency could nevertheless set a lower level of concern for the concentration of CDD's and CDF's in these wastes. The Agency solicits comment on the advisability, practicality, and desirability of doing so. If a lower level of concern is to be established, at what level should it be set, and how could this level be justified?

2. *Analytical Methodology*—The Agency solicits comment on the proposed method of analysis for CDD's and CDF's; in particular, evidence that some extraction media may be more efficacious than others for particular wastes. The Agency considered whether it might be useful to develop a method of analysis that would be less detailed, and therefore less expensive, than that proposed, since a high degree of specificity with respect to isomeric content is not necessary in the present instance. For delisting purposes, for instance, it might be sufficient for a petitioner to show that a waste does not contain any CCD's or CDF's—even though, for example, dichloro- or hepta- and octachlorodioxins are present. The Agency solicits comments with respect to the usefulness, practicability, and cost, for instance, of a GC/MS analytical method which would detect *total* CDD's and CDF's at low levels in a waste in one analytical determination.

3. *Wastes resulting from manufacturing processes conducted on equipment contaminated with CDD's or CDF's*—The Agency is proposing to list as hazardous, wastes resulting from processes conducted on equipment previously used for a manufacturing process that generated CDD's or CDF's. A generator could legitimately question how this regulation can be enforced: how can they know whether the equipment in question was previously used for these processes? The Agency considers that a demonstration of historical knowledge would be deemed sufficient for this purpose (45 *FR* 32678 (May 19, 1980); see also the Listing Background Document). If historical records are not available, or inaccurate, analysis of the listed wastes on several occasions for total CDD's and CDF's would be sufficient to establish their absence. The Agency solicits comments on the appropriate historical records and time periods to be used, and the appropriate analytical detection limit to be used if historical records are not available.

4. *Identification of commercial chemical products subject to this listing.* The Agency is concerned that some users of commercial chemical products may not be able to identify which commercial chemical products contain tri-, tetra-, or pentachlorophenol, or their chlorophenoxy derivative acids, esters, and amine salts, and which, therefore, would be regulated (when discarded) as

[20] "Capabilities on methodology for the analysis of tetrachlorodibenzo-*p*-dioxin", Battelle Columbus Laboratories, Columbus, OH 43201, August 10, 1981.

[21] Dr. M. Taylor, Wright State University, to C. Gozde, U.S. EPA, Report of analytical data (December 6, 1979).

[22] Dr. T. O. Tiernan, Wright State University, to D. L. Rosengren, Viar and Company, Inc., Report on sample analyses (June 1, 1982).

[23] Dr. T. O. Tiernan, Wright State University, to L. Haas, Viar and Company, Inc., Report on sample analyses (August 6, 1982).

[24] T. O. Tiernan *et al.*, Incineration of chemical wastes containing polychlorinated biphenyl: assessment of tests conducted at Rollins Environmental Services, Deer Park, Texas, and Energy Systems Company, El Dorado, Arkansas. *In:* Detoxication of Hazardous Waste, J. H. Exner, Ed., Ann Arbor Science, 1982, pp 143–183.

EPA Hazardous Waste Nos. F023. Although the FIFRA regulations (40 CFR 162) do require that active ingredients be identified by their chemical name or by a usual common name, and an EPA publication ("Accepted common names and chemical names for the ingredient statement on pesticide labels", EPA 540/9-7-017) is available to aid in their chemical identification, these aids may not convey sufficient information to the unsophisticated user. Non-pesticide products may also be hard to identify. Therefore, the Agency is considering various mechanisms to solve this potential problem (*i.e.*, labeling requirements for manufacturers, publishing a list of all products which contain these compounds, etc.). The Agency solicits comment on this potential problem.

5. *Wastes which may contain CDDs and CDFs but which are not covered by the present regulation.* The Agency has some data indicating that wastes, other than those covered by this proposal, may contain CDDs and CDFs. This may be the case, for instance for residuals such as fly ash from low temperature combustion of certain industrial wastes (especially of wastes containing chlorophenols, or chlorobenzenes); residuals from dichlorophenol manufacture; and sludges from wood preserving using pentachlorophenols. In the case of the first two wastes, although the Agency, on the grounds of knowledge of reaction chemistry and process technology, believes this may be the case, it lacks sufficient data to support this supposition. For this reason, studies are being conducted in order to gather more data. These wastes may be listed at a future date if further evidence demonstrates that they indeed are hazardous.

With regard to waste from wood preserving, we are presently investigating whether additional wastes from this process should be listed as hazardous, and whether CDDs and CDFs should be constituents of concern in the process wastes already listed (EPA Hazardous Waste K001, Wastewater treatment sludges). Pending completion of those studies we may take further regulatory action.

## X. Economic, Environmental, and Regulatory Impacts

### *A. Regulatory Impact Analysis*

Under Executive Order 12291, EPA must determine whether a regulation is "major" and therefore subject to the requirement of a Regulatory Impact Analysis. The effect of the present amendment is judged not to be major, since it in part replaces regulation under a different statute (Section 6(d) of the Toxic Substances Control Act), and imposes an additional regulatory burden only on three manufacturers of chlorophenols, and five manufacturers of chlorophenols and their chlorophenoxy derivatives. In addition, some number of manufacturers who use equipment that may be contaminated with CDDs or CDFs may also have additional regulatory burden. However, we presume that this part of the regulation is unlikely to affect many additional manufacturers other than the sight referred to above. In addition, this regulation imposes a regulatory burden on persons or entities discarding some unused formulations containing tri-, tetra-, or pentachlorophenol or unused formulations containing compounds derived from these phenols. The disposal of many of these formulations, however, is already regulated under § 261.33 of RCRA. Additionally, because of their inherent value, we do not believe that the regulated community will usually discard substantial quantities of these materials, further minimizing any impact.

In addition, we believe that there will be no adverse impact on the ability of U.S.-based enterprises to compete with foreign-based enterprises in domestic or export markets. Therefore, since the Agency does not expect that the proposed action will result in either an effect on the economy of $100 million or more, or an adverse impact on U.S. based enterprises, this proposed regulation is not considered to be a major action. Because this proposed amendment is not a major regulation, no Regulatory Impact Analysis has been conducted.

This amendment was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291. Any comments from OMB to EPA, and any EPA responses to those comments are available for public inspection in Room S-269C at EPA.

### *B. Regulatory Flexibility Act*

Pursuant to the Regulatory Flexibility Act, 5 U.S.C. § 601 *et seq.*, whenever an agency is required to publish a general notice of rulemaking for any proposed or final rule, it must prepare and make available for public comment a regulatory flexibility analysis that describes the impact of the rule on small entities (*i.e.*, small businesses, small organizations, and small governmental jurisdictions). No regulatory flexibility analysis is required, however, if the head of the agency certifies that the rule will not have a significant economic impact on a substantial number of small entities.

Only one of the hazardous wastes proposed to be listed in § 261.31 is expected to be generated by small entities. The Agency anticipates that pesticide aerial applicators will constitute the main segment of small business entities affected by this regulation.[28] However, these persons are probably already regulated under RCRA since a large number of pesticides (both acutely hazardous and toxic) are currently covered by existing regulations. Therefore, we would not expect any aerial applicators to be newly regulated as a result of this rule. In addition, the Agency does not believe that small entities will dispose of significant quantities of the commercial chemical products proposed for regulation. Thus, today's amendment is unlikely to have a significant economic impact on a substantial number of small entities. This regulation therefore does not require a regulatory flexibility analysis.

### *C. Paper Reduction Act of 1980*

The reporting or recordkeeping (information) provisions in this rule will be submitted for approval to the Office of Management and Budget (OMB) under Section 3504(h) of the Paperwork Reduction Act of 1980, U.S.C. 3501 *et seq.* Any final rule will explain how its reporting or recordkeeping provisions respond to any OMB or public comments.

## XII. Rulemaking Record

The public docket for 40 CFR Part 775 is located in Room E-107 at the address listed for the U.S. Environmental Protection Agency in the address section of this preamble. The entire rulemaking docket for the rule being proposed today is included in the record for 40 CFR Part 775. EPA will identify the complete rulemaking record for 40 CFR part 775 on or before the date of repeal. EPA will consider any time between the publication of this notice and the date the Agency identifies the final record.

### *XII List of Subjects*

### *40 CFR Part 261*

Hazardous materials, Waste treatment and disposal, Recycling.

[28] Farmers may also generate some of these wastes—discarded unused formulations—however, farmers are exempt from regulation provided the waste pesticides are from their own use and are disposed of on their own farm in a manner consistent with the disposal instructions on the pesticide label. Therefore, we do not expect farmers to be severely impacted.

*40 CFR Part 264*

Hazardous materials, Packaging and containers, Reporting requirements, Security measures, Surety bonds, Waste treatment and disposal.

*40 CFR Part 265*

Hazardous materials, Packaging and containers, Reporting requirements, Security measures, Surety bonds, Waste treatment and disposal, Water supply.

*40 CFR Part 775*

Environmental protection, Hazardous materials, Pesticides and pests, Waste treatment and disposal.

Dated: March 21, 1983.

John W. Hernandez,

*Acting Administrator.*

For the reasons set out in the preamble, it is proposed to amend Title 40 of the Code of Federal Regulations as follows:

## PART 261—IDENTIFICATION AND LISTING OF HAZARDOUS WASTE

1. The authority citation for Part 261 reads as follows:

Authority: Secs. 1006, 2002(a), 3001, and 3002 of the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act of 1976, as amended (42 U.S.C. 6905, 6912(a), 6921, and 6922).

2. In § 261.5, paragraphs (e)(1) and (e)(2) are revised to read as follows:

**§ 261.5 Special requirements for hazardous waste generated by small quantity generators**

* * * * *

(e) * * *

(1) A total of one kilogram of acutely hazardous wastes listed in §§261.31, 261.32, or 261.33(e).

(2) A total of 100 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, of any acutely hazardous wastes listed in §§261.31, 261.32, or 261.33(e).

3. In §261.7, the introductory text of paragraphs (b)(1) and (b)(3) is revised to read as follows:

**§261.7 Residues of hazardous waste in empty containers.**

* * * * *

(b)(1) A container or an inner liner removed from a container that has held any hazardous waste, except a waste that is a compressed gas or that is identified as an acutely hazardous waste listed in §§261.31 261.32, or 261.33(e) of this chapter is empty if:

* * * * *

3. A container or an inner liner removed from a container that has held an acutely hazardous waste listed in §§261.31 261.32, or 261.33(e) is empty if:

* * * * *

4. In §261.31, add the following waste streams:

**§261.31 Hazardous waste from nonspecific sources.**

| Industry and EPA hazardous waste no. | Hazardous waste | Hazard code |
|---|---|---|
| * * * * * | | |
| Generic: | | |
| F020 | Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production or manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tri-, tetra-, or pentachlorophenol, or of intermediates used to produce their derivatives. (This listing does not include wastes from the production of Hexachlorophene from highly purified 2,4,5-trichlorophenol. | (H). |
| F021 | Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the manufacturing use (as a reactant, chemical intermediate, or component in a formulating process) of tetra-, penta-, or hexachlorobenzenes under alkaline conditions. | (H). |
| F022 | Wastes (except wastewater and spent carbon from hydrogen chloride purification) from the production of materials on equipment previously used for the production or manufacturing use (as a reactant, chemical intermediate or component in a formulating process) of materials listed under F020 and F021. | (H). |
| F023 | Discarded unused formulations containing tri-, tetra-, or pentachlorophenol or discarded unused formulations containing compounds derived from those chlorophenols. | (H). |
| * * * * * | | |

5. In § 261.33(f), remove the following waste streams:

**§ 261.33 Discarded commercial chemical products, off-specification species, container residues, and spill residues thereof.**

* * * * *

| Hazardous waste No. | Substance |
|---|---|
| * * * * * | |
| U212 | 2,3,4,6-Tetrachlorophenol. |
| U212 | Phenol, 2,3,4,6-tetrachloro-. |
| U230 | 2,4,5-Trichlorophenol. |
| U230 | Phenol, 2,4,5-trichloro-. |
| U231 | 2,4,6-Trichlorophenol. |
| U231 | Phenol, 2,4,6-trichloro-. |
| U232 | 2,4,5-Trichlorophenoxyacetic acid. |
| U232 | 2,4,5-T. |
| U233 | Silvex. |
| U233 | Propionic acid, 2-(2,4,5-trichlorophenoxy)-. |
| U242 | Pentachlorophenol. |
| U242 | Phenol, pentachloro-. |
| * * * * * | |

6. Amend Table I in Appendix III of Part 261, by removing the entry "chlorinated dibenzodioxins", and adding the following entries in alphabetical order:

## Appendix III—Chemical Analysis Test Methods

* * * * * * *

TABLE 1.—ANALYTICAL CHARACTERISTICS OF ORGANIC CHEMICALS

| Compound | Sample handling class/fraction | Non-GC methods | Measurement techniques | | |
|---|---|---|---|---|---|
| | | | GC/MS | Conventional | |
| | | | | GC | Detector |
| * * * | * * * | * * * | * | | |
| Chlorinated dibenzo-*p*-dioxins | Extractable/SN | | 8280 | | |
| Chlorinated dibenzofurans | Extractable/SN | | 8290 | | |
| * * * | * * * | * * * | * | | |

7. Add the following entries in numerical order to Appendix VII of Part 261:

## Appendix VII—Basis for Listing Hazardous Wastes

| EPA hazardous waste No. | Hazardous constituents for which listed |
|---|---|
| * * * | * * * |
| F020 | tetra-, penta-, and hexachlorodibenzo-*p*-dioxins; tetra-, penta-, and hexachlorodibenzofurans; and tri-, tetra-, and pentachlorophenols and their chlorophenoxy derivative acids, esters, and amine salts. |
| F021 | tetra-, penta-, and hexachlorodibenzo-*p*-dioxins; tetra-, penta-, and hexachlorodibenzofurans; and tri-, tetra-, and pentachlorophenols and their chlorophenoxy derivative acids, esters, and amine salts. |
| F022 | tetra-, penta-, and hexachlorodibenzo-*p*-dioxins; tetra-, penta-, and hexachlorodibenzofurans; and tri-, tetra-, and pentachlorophenols and their chlorophenoxy derivative acids, esters, and amine salts. |
| F023 | tetra-, penta-, and hexachlorodibenzo-*p*-dioxins; tetra-, penta-, and hexachlorodibenzofurans; and tri-, tetra-, and pentachlorophenols and their chlorophenoxy derivative acids, esters, and amine salts. |

8. Add the following constituents in alphabetical order to Appendix VIII of Part 261:

## Appendix VIII—Hazardous Constituents

* * * * *

hexachlorodibenzo-*p*-dioxins
hexachlorodibenzofurans
pentachlorodibenzo-*p*-dioxins
pentachlorodibenzofurans
tetrachlorodibenzo-*p*-dioxins
tetrachlorodibenzofurans

* * * * *

9. Appendix IX is added to Part 261 to read as follows:

## Appendix IX—Method of Analysis for Chlorinated dibenzo-p-dioxins and dibenzofurans [1 2 3 4]

*Method 8280*

1. Scope and Application.

1.1 This method covers the determination 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 must take necessary precautions to prevent exposure to himself, 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 chromatography-low 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/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstated 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 PCB's 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.

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 must be washed and solvent rinsed before use to minimize interferences.

4.1.2 Bottle caps—threaded to screw on to the sample bottles. Caps must 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 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 (± 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.

---

[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 practice. Typical infectious waste incinerators are probably not satisfactory devices for disposal of materials highly contaminated with CDDs or CDFs. 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).

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 decafluorotriphenyl-phosphine (DFTPP) is injected through the GC inlet. The system must 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 320.

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 6.1-6.3) 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 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.

4.4 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 percent 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 Stock standards in a glovebox, prepare stock standard solutions of TCDD and C1-TCDD (molecular weight 328). 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 must 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 three TCDD/$^{37}$C1-TCDD and TCDF/$^{37}$C1-TCDF ratios.[6] The $^{37}$C1-TCDD/F concentration in the standard should be fixed and selected to yield a reproducible response at the most sensitive setting of the mass spectrometer.

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. $^{37}$C1-TCDF. The detection limit provided in Table 1 should be verified by injecting .015 ng of $^{37}$C1-TCDD which should give a minimum signal to noise ratio of 5 to 1 at mass 320.

7. Quality Control.

7.1 Before processing any samples, the analyst should 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 should be processed as a safeguard against laboratory contamination.

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

8. Sample Collection, Preservation, and Handling.

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

8.2 The samples must be iced or refrigerated from the time of collection until extraction. Chemical preservatives should 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 49 hours of collection, the sample should be adjusted to a pH range of 6.0-8.0 with sodium hydroxide or sulfuric acid.

8.3 All samples must be extracted with 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 should be dried using a stream of prepurified nitrogen and pulverized in a ball-mill or similar device. Transfer the sample to a tared 125 ml flint glass bottle (Teflon-lined screw cap) and determine the weight of the sample. Add an appropriate quantity of $^{37}$C1-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 should be completely dissolved in any of the recommended neat solvents. Activated carbon samples must 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 #055-002-81001-2). 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 50° 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).

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[6] $^{37}$C1-labelled TCDD and TCDF are available from K.O.R. Isotopes, 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 $^{37}$C1-2,3,7,8-TCDD and $^{37}$C1-2,3,7,8-TCDF. This method therefore uses these isomers as surrogates for the CDDs and CDFs. When labelled CDDs and CDFs are available, their use will be required.

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 nitogren) 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 micro-reaction 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.

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 × 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, three injections of standard mixtures.

11.2 Calculate response factors for standards relative to $^{37}$Cl–TCDD/F (see Section 12).

11.3 Analyze samples with selected ion monitoring of at least two 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 must match that in the standard, within the performance specifications of the analytical system.

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

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

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

11.5 If a response is obtained for the appropriate set of ions, but is outside the expected ratio, a co-eluting impurity may be suspected. In this case, another set of ions characteristic of the CDD/CDF molecules should 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 of TCDD or TCDF. For analysis of TCDD good analytical technique would require using all four ions, m/e 257, 320, 322, 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 should 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 should employ cleanup procedures and reanalyze by GC/MS.

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:

$$\text{Concentration, ug/gm} = \frac{A \times A_s}{G \times A_{is} \times R_f}$$

Where:

A = ug 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

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

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

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

Where:

$C_{is}$ = concentration of the internal standard

$C_s$ = 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.

TABLE 1—Gas Chromatography of TCDD.

| Column | Retention time (min.) | Detection limit (ug/kg) [1] |
|---|---|---|
| Glass Capillary | 9.5 | 0.003 |

[1] Detection limit for liquid samples is 0.003 ug/l. This is calculated from the minimum detectable GC response being equal to five 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).

TABLE 2.—DFTPP KEY IONS AND ION ABUNDANCE CRITERIA [1]

| Mass | Ion abundance criteria |
|---|---|
| 51 | 30 to 60 percent of mass 198. |
| 69 | Less than 2 percent of mass 69. |
| 70 | Do. |
| 127 | 40 to 60 percent of mass 198. |
| 197 | Less than 1 percent of mass 198. |
| 198 | Base peak, 100 percent relative abundance. |
| 199 | 5 to 9 percent of mass 198. |
| 275 | 10 to 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 to 23 percent 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.

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 dibenzofurans $C_{12}H_{8-x}OCl_x$ | Monitored m/z for dibenzodioxins $C_{12}H_{8-x}O_2Cl_x$ | Approximate theoretical ratio expected on Basis of isotopic abundance |
|---|---|---|---|---|
| Tetra | 4 | [1]319.897 | [1]303.902 | 0.74 |
| | | 321.894 | 321.899 | 1.00 |
| | | [2]327.865 | | |
| | | [2]256.933 | | .21 |
| | | [2]258.930 | | .20 |
| Penta | 5 | [3]353.858 | [3]337.863 | .57 |
| | | 355.855 | 339.860 | 1.00 |
| Hexa | 6 | 389.816 | 373.821 | 1.00 |
| | | 391.813 | 375.818 | .87 |

[1] Molecular ion peak.

[2] $Cl_4$-labelled standard peaks.

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

## PART 264—STANDARDS FOR OWNERS AND OPERATORS OF HAZARDOUS WASTE TREATMENT, STORAGE, AND DISPOSAL FACILITIES

10. The authority citation for Part 264 reads as follows:

Authority: Secs. 1006, 2002(a), 3004, and 3005 of the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act of 1976, as amended (42 U.S.C. 6905, 6912(a), 6924, and 6925).

11. In Subpart K of Part 264, add the following § 264.231:

**§ 264.231 Special requirements for hazardous wastes F020, F021, F022, and F023.**

(a) Hazardous Wastes F020, F021, F033, and F023 must not be placed in a surface impoundment unless the owner or operator operates the surface impoundment in accordance with a management plan for these wastes that is approved by the Regional Administrator pursuant to the standards set out in this paragraph, and in accord with all other applicable requirements of this Part. The factors to be considered are:

(1) the volume, physical, and chemical characteristics of the wastes, including their potential to migrate through soil or to volatilize or escape into the atmosphere;

(2) the attenuative properties of underlying and surrounding soils or other materials;

(3) the mobilizing properties of other materials co-disposed with these wastes;

(4) the effectiveness of additional treatment, design, or monitoring techniques.

(b) The Regional Administrator may determine that additional design, operating, and monitoring requirements are necessary for surface impoundments managing hazardous wastes F020, F021, F022, and F023 in order to reduce the possibility of migration of these wastes to ground water, surface water, or air so as to protect human health and the environment.

12. In Subpart L of Part 264, add the following § 264.259:

**§ 264.259 Special requirements for hazardous wastes F020, F021, F022, and F023.**

(a) Hazardous Wastes F020, F021, F022, and F023 must not be placed in waste piles that are not enclosed (as defined in § 264.250(c)) unless the owner or operator operates the waste pile in accordance with a management plan for these wastes that is approved by the Regional Administrator pursuant to the standards set out in this paragraph, and in accord with all other applicable requirements of this Part. The factors to be considered are:

(1) the volume, physical, and chemical characteristics of the wastes, including their potential to migrate through soil or to volatilize or escape into the atmosphere;

(2) the attenuative properties of underlying and surrounding soils, or other materials;

(3) the mobilizing properties of other materials co-disposed with these wastes;

(4) the effectiveness of additional treatment, design, or monitoring techniques.

(b) The Regional Administrator may determine that additional design, operating, and monitoring requirements are necessary for piles managing hazardous wastes F020, F021, F022, and F023 in order to reduce the possibility of migration of these wastes to ground water, surface water, or air so as to protect human health and the environment.

13. In Subpart M of Part 264, add the following § 264.283:

**§ 264.283 Special requirements for hazardous wastes F020, F021, F022, and F023.**

(a) Hazardous wastes F020, F021, F022, and F023 must not be placed in a land treatment facility unless the owner or operator operates the facility in accordance with a management plan for these wastes that is approved by the Regional Administrator pursuant to the standards set out in this paragraph, and in accord with all other applicable requirements of the Part. The factors to be considered are:

(1) the volume, physical, and chemical characteristics of the wastes, including their potential to migrate through soil or to volatilize or escape into the atmosphere;

(2) the attenuative properties of underlying and surrounding soils or other materials;

(3) the mobilizing properties of other materials co-disposed with these wastes;

(4) the effectiveness of additional treatment, design, or monitoring techniques.

(b) The Regional Administrator may determine that additional design, operating, and monitoring requirements are necessary for land treatment facilities managing hazardous wastes F020, F021, F022, and F023 in order to reduce the possibility of migration of these wastes to ground water, surface water, or air so as to protect human health and the environment.

14. In Subpart N of Part 264, add the following § 264.317:

**§ 264.317 Special requirements for hazardous wastes F020, F021, F022, and F023.**

(a) Hazardous wastes F020, F021, F022, and F023 must not be placed in landfills unless the owner or operator operates the landfill in accordance with a management plan for these wastes that is approved by the Regional Administrator pursuant to the standards set out in this paragraph, and in accord with all other applicable requirements of this Part. The factors to be considered are:

(1) the volume, physical, and chemical characteristics of the wastes, including their potential to migrate through the soil or to volatilize or escape into the atmosphere;

(2) the attenuative properties of underlying and surrounding soils or other materials;

(3) the mobilizing properties of other materials co-disposed with these wastes;

(4) the effectiveness of additional treatment, design, or monitoring requirements.

(b) The Regional Administrator may determine that additional design, operating, and monitoring requirements are necessary for landfills managing hazardous wastes F020, F021, F022, and F023 in order to reduce the possibility of migration of these wastes to ground water, surface water, or air so as to protect human health and the environment.

## PART 265—INTERIM STANDARDS FOR OWNERS AND OPERATORS OF HAZARDOUS WASTE TREATMENT, STORAGE, AND DISPOSAL FACILITIES

15. The authority citation for Part 265 reads as follows:

Authority: Secs. 1006, 2002(a), 3004, and 3005 of the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act of 1976, as amended (42 U.S.C. 6905, 6912(a), 6924, and 6925).

16. § 265.1 is amended by adding paragraph (d).

**§ 265.1 Purpose, scope and applicability.**

* * * * *

(d) The following hazardous wastes must not be managed at facilities subject to regulation under this Part.

(1) EPA Hazardous Waste Nos. F020, F021, F022, and F023 unless:

(i) The waste is generated in a surface impoundment as part of the plant's wastewater treatment system.

(ii) The waste is stored in tanks or containers.

(iii) The waste is stored or treated in waste piles that meet the requirements of § 264.250(c) as well as all other applicable requirements of Subpart L of this Part.

## PART 775 [REMOVED]

17. The authority citation for Part 775 reads as follows:

Authority. Sec. 6 of the Toxic Substances Control Act (TSCA) Pub. L. 94–469, 90 Stat. 2020 [15 U.S.C. 2605].

18. Title 40 is amended by removing Part 775.

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