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Standard Guide for Use of Coal Combustion Products for Solidification/ Stabilization of Inorganic Wastes¹

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1. Scope

1.1 This guide covers methods for selection and application of coal combustion products (CCPs) for use in the chemical stabilization of trace elements in wastes and wastewater. These elements include, but are not limited to, arsenic, barium, boron, cadmium, chromium, cobalt, lead, molybdenum, nickel, selenium, vanadium, and zinc. Chemical stabilization may be accompanied by solidification of the waste treated. Solidification is not a requirement for the stabilization of many trace elements, but does offer advantages in waste handling and in reduced permeability of the stabilized waste.

1.1.1 Solidification is an important factor in treatment of wastes and especially wastewaters. Solidification/Stabilization (S/S) technology is often used to treat wastes containing free liquids. This guide addresses the use of CCPs as a stabilizing agent (with or without the addition of other materials. Stabilization may be achieved by using combinations of CCPs and other products such as lime, lime kiln dust, cement kiln dust, cement, and others. CCPs used alone or in combination with other reagents promote stabilization of many inorganic constituents through a variety of mechanisms. These mechanisms include precipitation as hydrates, carbonates, silicates, sulfates, and so forth; microencapsulation of the waste particles through pozzolanic reactions; formation of metal precipitates; and formation of hydrated phases (1-4).² Long-term performance of the stabilized waste is an issue that must be addressed in considering any S/S technology. In this guide, several tests are recommended to aid in evaluating the long-term performance of the stabilized wastes.

1.2 The CCPs that are suited for this application include fly ash, dry flue gas desulfurization (FGD) material, and and fluidized-bed combustion (FBC) ash.

1.3 The wastes or wastewater, or both, containing the inorganic species may be highly variable, so the chemical

characteristics of the waste or wastewater to be treated must be determined and considered in the selection and application of any stabilizing agent, including CCPs. In any waste stabilization process, laboratory-scale tests for compatibility between the candidate waste or wastewater for stabilization with one or more selected CCPs and final waste stability are recommended prior to pilot-scale and full-scale application of the stabilizing agent.

1.4 This guide does not intend to recommend pilot-scale or full-scale processes or procedures for waste stabilization. Full-scale processes should be designed and carried out by qualified scientists, engineers, and environmental professionals. It is recommended that stabilized materials generated at the full-scale stabilization site be subjected to testing to verify laboratory test results.

1.5 The utilization of CCPs under this guide is a component of a pollution prevention program. Utilization of CCPs in this manner conserves land, natural resources, and energy.

1.6 This guide applies only to CCPs produced primarily from the combustion of coal. It does not apply to ash or other combustion products derived from the burning of waste; coal coking byproducts; municipal, industrial, or commercial garbage; sewage sludge or other refuse, or both; derived fuels; wood waste products; rice hulls; agricultural waste; or other noncoal fuels.

1.7 Regulations governing the use of CCPs vary by nation, state and locality. The user of this guide has the responsibility to determine and comply with applicable regulations.

1.8 It is recommended that work performed under this guide be designed and carried out by qualified scientists, engineers, and environmental professionals.

1.9 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.10 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the

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 $^{^{2}}$ The boldface numbers in parentheses refer to the list of references at the end of the text.

Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:³

- C114 Test Methods for Chemical Analysis of Hydraulic Cement
- C311 Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
- C400 Test Methods for Quicklime and Hydrated Lime for Neutralization of Waste Acid
- D75 Practice for Sampling Aggregates
- D558 Test Methods for Moisture-Density (Unit Weight) Relations of Soil-Cement Mixtures
- D1556 Test Method for Density and Unit Weight of Soil in Place by Sand-Cone Method
- D1633 Test Methods for Compressive Strength of Molded Soil-Cement Cylinders
- D1635 Test Method for Flexural Strength of Soil-Cement Using Simple Beam with Third-Point Loading
- D2166 Test Method for Unconfined Compressive Strength of Cohesive Soil
- D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
- D2937 Test Method for Density of Soil in Place by the Drive-Cylinder Method
- D3441 Test Method for Mechanical Cone Penetration Testing of Soils
- D3877 Test Methods for One-Dimensional Expansion, Shrinkage, and Uplift Pressure of Soil-Lime Mixtures (Withdrawn 2017)⁴
- D3987 Practice for Shake Extraction of Solid Waste with Water ASTM F2
- D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils
- D4832 Test Method for Preparation and Testing of Controlled Low Strength Material (CLSM) Test Cylinders
- D4843 Test Method for Wetting and Drying Test of Solid Wastes
- D4972 Test Methods for pH of Soils
- D5084 Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter
- D5907 Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water
- D6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis
- D6938 Test Methods for In-Place Density and Water Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth)

D7928 Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis

E2201 Terminology for Coal Combustion Products

3. Terminology

3.1 Definitions:

3.1.1 Definitions are in accordance with Terminology E2201.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 BDAT-best demonstrated available technology.

3.2.1.1 *Discussion*—The treatment technology that best minimizes the mobility or toxicity (or both) of the hazardous constituents for a particular waste.

3.2.2 *ettringite*—a mineral with the nominal composition $Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$ (1)

3.2.3 S/S—solidification/stabilization.

3.2.4 XRD-x-ray diffraction.

4. Significance and Use

4.1 *General*—CCPs can have chemical and mineralogical compositions that are conducive to use in the chemical stabilization of trace elements in wastes and wastewater. These elements include, but are not limited to, arsenic, barium, boron, cadmium, chromium, cobalt, lead, molybdenum, nickel, selenium, vanadium, and zinc. Chemical stabilization may be accompanied by solidification of the waste treated. Solidification is not a requirement for the stabilization of many trace elements, but does offer advantages in waste handling and in reduced permeability of the stabilized waste. This guide addresses the use of CCPs as a stabilizing agent with or without addition of other materials.

NOTE 1—In the United States, S/S is considered the BDAT for the disposal of some wastes that contain metals since they cannot be destroyed by other means (2).

4.1.1 Advantages of Using CCPs-Advantages of using CCPs for waste stabilization include their availability in high volumes, and generally good product consistency from a single source. In addition, in some instances certain CCPs can partly or entirely replace other expensive stabilization materials such as Portland cement. CCPs vary depending on the combustion or emission control process and the coal or sorbents used, or both, and CCPs contain trace elements, although usually at very low concentrations. CCPs are generally an environmentally suitable materials option for waste stabilization, but the compatibility of a specific CCP must be evaluated with individual wastes or wastewater through laboratory-scale tests followed by full-scale demonstration and verification. CCPs suitable for the chemical stabilization have the ability to incorporate large amounts of free water via hydration reactions. These same hydration reactions frequently result in the formation of mineral phases that stabilize or chemically immobilize the trace elements of concern. CCPs that exhibit high pHs (>11.5) offer advantages in stabilizing trace elements that exist as oxyanions in nature (such as arsenic, boron, chromium, molybdenum, selenium, and vanadium) and trace

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

elements that form oxyhydroxides, carbonates or other lowsolubility precipitates at high pH (such as cadmium, barium, nickel, and zinc).

4.2 Chemical/Mineralogical Composition-Since many CCPs are generated at higher temperature, reactions with water during contact with aqueous solutions can be expected. Mineral formation may contribute to the chemical stabilization and/or solidification achieved in the waste treatment process. One example of this type of chemical stabilization is achieved by ettringite formation. Reduced leachability of several trace elements has been correlated with ettringite formation in hydrated high-calcium CCPs typically from U.S. lignite and subbituminous coal, and dry FGD materials. These materials worthy candidates for use in this chemical stabilization process. Lower-calcium CCPs in presence of sulfate sources, may also be effective with the addition of a calcium source that maintains the pH above 11.5. Ettringite forms as a result of hydration of many high-calcium CCPs in presence of sulfate sources, so adequate water must be available for the reaction to occur. The mineral and amorphous phases of CCPs contribute soluble elements required for ettringite formation, and the ettringite formation rate can vary based on the mineral and amorphous phase compositions.

4.3 Regulatory Framework:

4.3.1 *Waste Management Framework*—Waste stabilization activities most often occur within a regulatory waste management framework. This regulatory framework will generally establish minimum waste sampling and characterization requirements as well as establish documentation, qualification, and performance criteria for waste management activities. The framework may also prescribe or prohibit certain waste management practices. The applicable requirements of the regulatory framework may be formalized in a permit. This guide is intended to be applied within the context of a regulatory waste management framework.

NOTE 2—The U. S. regulatory framework is briefly described in Stabilization/Solidification of CERCLA and RCRA Wastes: Physical Tests, Chemical Testing Procedures, Technology Screening, and Field Activities (2).

4.3.2 *Beneficial Use Framework*—Beneficial use activities often occur within a regulatory framework. In some locations, new beneficial uses require prior regulatory approval as part of a beneficial use determination. Beneficial use determinations may require specific characterization of the material and the beneficial use. Jurisdictions that require approval of beneficial use may also maintain exemptions or predeterminations for certain materials or beneficial uses.

5. CCP Characterization

5.1 *General*—Characterization of the CCP(s) under consideration for use as a stabilizing agent is required to determine bulk chemical and mineralogical composition. Sampling CCPs for testing purposes should conform to Practice D75 or Test Method C311 as appropriate. Proper laboratory protocols for handling fine material should be followed.

5.2 *Chemical Composition*—Test Method C311 is often used to determine the major chemical constituents of CCP

samples. The most critical constituents requiring quantitation are calcium, aluminum, and sulfur.

5.3 *pH*—Test Method D4972 U. S. Environmental Protection Agency (USEPA) Method SW-9045 (**5**) can be used to determine CCP pH. In assessing the test results, consideration should be given to the possibility that the pH of the CCP may differ with age, water content, and other conditions.

5.4 *Buffer Capacity*—Test Method C400 can be applied to evaluate the buffer capacity of the CCP, and to determine the basicity factor for the CCP. It is important that the buffer capacity of the CCP is sufficient to maintain a high pH so that conditions are favorable to allow the stabilization mechanisms of interest to occur (for eample, hydroxide formation, precipitation, etc.), and so that the waste remains stable over time and under environmental stresses.

5.5 *Swelling*—Test Method D3877 can be used to determine the swelling potential of self-hardening (high-calcium) CCPs and FGD material. The reactions producing the expansive properties generally do not begin to occur until at least 30 days after initial CCP hydration. To address this delayed reaction the test method must address this delayed reaction. The procedure should be modified to extend the wetting and drying cycle to 60 days. Expansive reactions, including the formation of ettringite, may have an impact on the permeability of the stabilized waste. Following completion of hydration at 60 days, the mineral composition should be determined.

5.6 *Mineral Composition*—XRD can be used to determine the mineral content of CCPs. Fly ash, bottom ash, and boiler slag contain significant amorphous phases that cannot be identified through the use of XRD techniques. Many dry FGD materials have a higher calcium content compared to that of CCPs. XRD evaluation can be performed on the hydrated CCP collected from the swelling test, summarized in 5.5, to determine whether or not the CCP(s) formed hydrated minerals.

6. Selection of CCP for Waste Solidification/Stabilization

6.1 *General*—Many CCPs have the potential to stabilize inorganic trace elements; however, those with the highest potential are finely divided powdered materials, including fly ash, dry FGD materials, and FBC ashes. Specifically, CCPs that are known to undergo hydration reactions that result in the formation of certain minerals, including ettringite, carbonate minerals, and insoluble sulfates, have high potential to serve as stabilizing agents for many trace elements that are not organically associated. A CCP exhibiting high pH also offers advantages in the stabilization of trace elements.

6.2 *Chemical Properties*—The chemical properties of CCPs used in waste stabilization applications may include a wide range of chemical compositions, pHs, buffering capacities, and reactivities.

6.2.1 *Chemical Composition*—CCPs of differing chemical composition can offer advantages for solidification/ stabilization applications. Most coal fly ashes are pozzolanic; however, certain subbituminous and lignite coal ashes may contain higher amounts of calcium oxide and exhibit greater cementitious properties. Cementitious CCPs offer additional

advantages in solidification/stabilization applications; the potential formation of monolithic structures of CCP-waste combinations without addition of cement or other additives; appropriate chemical components that allow the formation of ettringite for stabilization of oxyanions and the precipitation of low-solubility metal hydroxides and carbonates. Sulfate containing CCPs can can reduce the mobility of barium through barium sulfate formation. The presence of iron and/or manganese species can enhance the formation of low-solubility metal oxyhydroxides and, through co-precipitation, can further reduce the soluble amounts of other metals.

6.2.2 *pH*—It is recommended that the pH of the CCP be determined using methods noted in 5.3. CCPs exhibiting high pH (>8) offer advantages in numerous solidification/ stabilization mechanisms.

6.2.3 *Hydrated Mineral Formation*—The CCP selected for hydration purposes should be evaluated using Test Method D3877 to determine swelling potential. Thirty (30) days is recommended as the minimum hydration time; however, 60 days may provide more reliable results because ettringite formation is a slow reaction. As noted in 5.6, XRD is the best analytical tool to determine the presence of hydration products.

6.2.4 *Buffer Capacity*—The buffer capacity of the CCP should be determined as noted in 5.4. As an example of the use of the calculated basicity factor, to ensure that the CCP has the capability to neutralize significant acid while maintaining the pH above 11.5 for ettringite stability, a BF >0.1 would be appropriate. The limit for the BF of the CCP should be evaluated on the basis of the expected mechanism of solidification/stabilization, the candidate waste, and/or the site conditions expected for the stabilized material (see 7.5.2).

6.3 *Final CCP Selection*—One or more CCP(s) that meet Section 6 criteria can be selected for the laboratory-scale demonstration of the stabilization process using the candidate waste as noted in Section 8.

7. Waste Characterization and Stabilized Material Testing

7.1 *General*—Specific characteristics of the waste or wastewater requiring stabilization must be determined. The concentrations of the elements expected to participate in the chemical stabilization reactions are required to determine the quantity of stabilization agent or CCP to use. The pH and buffer capacity of the waste must be known. All waste characterization tests and procedures must be performed on representative samples of the specific waste to be treated. The final stabilized material form must also be evaluated for specific parameters to verify that the stabilization process has been effective.

7.2 *Physical Tests for Wastes*—Physical testing of the waste prior to treatment provides information on treatability, transport, storage, and mixing considerations. Physical testing of the stabilized material form provides information on the relative success of the stabilization process.

7.2.1 *Particle-Size Analysis*—The results of particle-size analysis provide information on the relative proportions of gravel, sand, silts, and clay-sized particles within the waste, uniformity, concavity, average grain size, and maximum and

minimum particle sizes and can be determined using Test Methods D6913 and D7928.

7.2.2 Atterberg Limits—The Atterberg Limits are a series of tests originally developed to determine the characteristics of clays used in ceramics. The recommended test procedure is Test Method D4318, which is used to determine the liquid limit (the moisture content at which the material flows as a viscous liquid), plastic limit (the moisture content at the boundary between the plastic and brittle states), and plasticity index (the difference between the liquid and plastic limits).

7.2.3 *Moisture Content*—The moisture content of the waste can be determined using Test Method D2216, which will indicate the amount of free water in the waste (waters of hydration are not included as part of the moisture content). Water may not be the only liquid phase in a waste requiring stabilization, so care must be taken to remove other liquids or otherwise account for them in determining the moisture content.

7.2.4 *Density*—The bulk density of a material is the ratio of the total weight of the material to the total volume and is frequently used to convert weight to volume for materials-handling calculations. Several methods are appropriate for determining the bulk density of the waste prior to stabilization. These include laboratory Test Methods D1556 and D4832 and in situ, Test Methods D2937 and D6938.

7.2.5 Suspended Solids—In the case of stabilizing oxyanionic species in a wastewater or liquid waste, it is recommended to determine the suspended solids in the wastewater using Test Method D5907. A liquid waste is defined as having less than 1 % suspended solids. Suspended solids can be removed from liquid waste or wastewater by filtering.

7.2.6 *Paint Filter Liquids Test*—The U.S. regulatory test (Title 40 of the Code of Federal Regulations [40 CFR] Sections 264.314 and 265.314) (6) is used to determine if a waste contains free liquids. For wastes treated by S/S to address free liquids, the EPA generally requires attainment of 50 psi unconfined compressive strength in the treated waste. This is to demonstrate that the free liquids were bound chemically rather than absorbed.

7.3 *Physical Tests for Stabilized Materials*—The physical nature of the waste may or may not change on application of the CCP for chemical stabilization depending on the CCP selected, the character of the waste, and the stabilization process used. Solidification is not a criterion for stabilization of inorganic species, but solidification generally reduces the permeability of the stabilized waste, which offers the advantages of limiting contact with air and water (or other gases and liquids). If solidification is desirable, it must be factored into the selection criteria for the CCP.

7.3.1 *Compaction Testing*—The moisture-density relationship can be determined on the stabilized waste using Test Method D558. This test determines the moisture content that allows maximum compaction, which provides maximum density of the stabilized waste. It is cautioned here that the moisture content must be adequate to provide the waters of hydration needed for the ettringite formation reaction to occur and that the ettringite formation is slow and can require 30 days