Designation: D7572 – 15

Standard Guide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil[1]

1. Scope*

1.1 This guide is applicable for the collection, extraction, and preservation of extracts from mine rock and soil samples for the analysis of cyanide in the extracts. Responsibilities of field sampling personnel and the laboratory are indicated.

1.2 The sampling, preservation, and extraction procedures described in this guide are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods D2036, D4282, D4374, D6888, D6994, D7237, and D7284. The information supplied in this guide can also be applied to other analytical methods for cyanide, for example, US EPA Method 335.4.

1.3 The procedure options methods appear in the following order:

Procedure Option	Sections
Option A Laboratory Processing of Field Preserved Samples	11 and 12
Option B Laboratory Processing of Moist Field Samples	13 and 14

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
D2036 Test Methods for Cyanides in Water
D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents
D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
D4282 Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion
D4374 Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate (Withdrawn 2012)[3]
D4840 Guide for Sample Chain-of-Custody Procedures
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D6696 Guide for Understanding Cyanide Species
D6888 Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection
D6994 Test Method for Determination of Metal Cyanide Complexes in Wastewater, Surface Water, Groundwater and Drinking Water Using Anion Exchange Chromatography with UV Detection
D7237 Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection
D7284 Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection
D7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide
D7511 Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection

***A Summary of Changes section appears at the end of this standard**

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[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

2.2 *U.S. EPA Methods:*[4]

EPA OIA-1677 Available Cyanide in Water

EPA Method 335.2 Cyanide, Total (Titrimetric; Spectrophotometric)

EPA Method 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology D1129 and Guide D6696.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mine rock, n*—ore, waste rock or overburden excavated in order to construct an ore-processing site, or recover metals or minerals during mining operations; or coarse processed ore such as heap-leach spoils.

3.2.2 *nominal size, n*—in sampling, for a screen of the standard series, the opening that would pass 95 % of a representative sample.

3.2.3 *refrigeration, n*—storing the sample between its freezing point and 6°C.

4. Summary of Guide

4.1 Samples are collected in appropriate containers at the sampling site, optionally field preserved, refrigerated, and transported to the laboratory where they are weighed, optionally sub-sampled, the moisture is determined or aqueous mass estimated, and cyanides are extracted prior to analysis. Results of the analysis of the extract are applied to the original solid sample to determine the apparent content of cyanides on the basis of dry weight.

5. Significance and Use

5.1 This guide is intended as a means for obtaining an extract from mine rock and soil samples to measure cyanide content in the aqueous portion of the sample on a dry weight basis. Cyanide is analyzed in mine rock and soil extracts for measurement of cyanide concentration; however, improper sample collection and extraction can result in significant positive or negative bias.

5.2 This guide is designed to mobilize aqueous cyanides present in the solids, so that the resulting extract can be used to assess leachate that could potentially be produced from mine rock or soil.

5.3 This guide is not intended to simulate actual site leaching conditions.

5.4 This guide produces extracts that are amenable to the determination of trace cyanides. When trace cyanides are being determined, it is especially important that precautions be taken in sample preservation, storage, and handling to avoid possible contamination of the extracts.

5.5 This guide uses a comparative test method and is intended for use as a routine method for monitoring mine rock and soils. It is assumed that all who use this guide will be trained analysts capable of performing it skillfully and safely. It is expected that work will be performed in a properly equipped laboratory applying appropriate quality control practices such as those described in Guide D3856.

5.6 This guide identifies proper methods for obtaining mine rock and soil samples for the specific purpose of measuring cyanide concentrations.

6. Interferences

6.1 Many interferences are known for the analysis of cyanide and could effect the results of the analysis of extracts produced using this guide. Refer to Practice D7365 for proper handling of the extracts during sampling, mitigation of interferences, and preservation prior to cyanide analysis.

6.2 Unless otherwise specified, samples must be extracted as soon as possible after sampling and the extracts must be analyzed within 14 days; however, it is recommended to estimate the actual holding time for each new sample matrix as described in Practice D4841. Certain sample matrices may require immediate analysis to avoid cyanide degradation due to interferences. A holding time study is required if there is evidence that cyanide degradation occurs from interferences which would cause the holding time to be less than specified in this guide or Practice D7365. Potential interferences for cyanide analytical methods are shown in Table 1.

7. Apparatus

7.1 *Agitation Equipment,* of any type that rotates the extraction vessel in an end-over-end fashion at a rate of 30 ± 2 r/min such that the axis of rotation is horizontal and it passes through the center of the bottle (see Fig. 1).

7.2 *Drying Pans or Dishes,* for moisture content determinations, 500 g to 8 kg capacity.

7.3 *Drying Oven*—Any thermostatically controlled drying oven capable of maintaining a steady temperature of ±2°C in a range of 100 to 110°C.

7.4 *Extraction Vessels,* cylindrical, wide-mouth, of a composition suitable to the nature of the mine rock or soil and cyanide analyses to be performed, constructed of materials that will not allow sorption of the constituents of interest, and sturdy enough to withstand the impact of the falling sample fragments. The size of the container should be selected so that the sample, plus extraction fluid occupy approximately 50–95 % of the container in order to provide good mixing without overfilling. The containers must have water-tight closures of sufficient diameter to fill with the samples.

7.5 *Filtration Device,* pressure or vacuum of a composition suitable to the nature of the analyses to be performed and equipped with a pre-washed glass wool or equivalent filter. An assembly for pre-filtration or a centrifuge may be required if filtration is difficult. (**Warning—**Avoid passing excessive amounts of air through the sample during filtration to prevent liberation of toxic hydrogen cyanide or cyanogen chloride gas.)

7.6 *Laboratory Balance,* capable of weighing to 1.0 g.

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[4] Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

TABLE 1 Examples of Potential Interferences if not Mitigated in Standard Cyanide Methods

NOTE 1—Alkyl halides were identified as a potential interference for the distillation methods, however they are not normally found in soil and mine rock.

Method	Description	Measurement	Interferences	Number
Total Cyanide	Automated UV	Colorimetric	Aldehydes Color Fatty Acids Ketones Mercury Nitrate Nitrite Oxidants Sulfides Turbidity Sulfur Compounds Thiocyanate	CFR Kelada-01 D4374
Total Cyanide	Manual Distillation $MgCl_2$	Amperometric	Aldehydes Carbonates Ketones Nitrite Nitrate Oxidants Sulfide Sulfur Compounds Thiocyanate	D7284 D2036, Test Method A
Total Cyanide	Manual Distillation $MgCl_2$	Manual or Automated Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Color Turbidity	D2036, Test Method A Standard Methods 4500-CN C/E EPA Method 335.2 EPA Method 335.4
Total Cyanide	Manual Distillation $MgCl_2$	ISE	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sulfide Sulfur Compounds Thiocyanate Color Turbidity	D2036, Test Method A
Total Cyanide	Manual Distillation $MgCl_2$	Titrimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036, Test Method A
Total Cyanide	Micro distillation	Amperometric	Carbonates Oxidants Sulfide	D7284
Total Cyanide	UV/FIA	Amperometric	Carbonates Oxidants Sulfide	D7511

TABLE 1 *Continued*

Method	Description	Measurement	Interferences	Number
Available Cyanide	Flow Injection Ligand Exchange	Amperometric	Carbonates Oxidants Sulfide	D6888 EPA OIA-1677
Cyanide Amenable to Chlorination	Alkaline Chlorination and Manual Distillations	Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sulfide Sulfur Compounds Thiocyanate Color Turbidity Unknowns that cause negative results	D2036, Test Method B
Weak Acid Dissociable Cyanide	Buffered Distillation	Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036, Test Method C
Weak Acid Dissociable Cyanide	Automated Method	Automated Colorimetric	Aldehydes Color Fatty Acids Ketones Mercury Nitrate Nitrite Oxidants Sulfides Turbidity	D4374
Weak Acid Dissociable Cyanide	Buffered Distillation	ISE	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036, Test Method C
Weak Acid Dissociable Cyanide	Buffered Distillation	Titrimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrate Nitrite Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Turbidity	D2036, Test Method C

TABLE 1 *Continued*

Method	Description	Measurement	Interferences	Number
Weak Acid Dissociable Cyanide		Manual Colorimetric	Aldehydes Carbonates Fatty Acids Ketones Nitrite Nitrate Oxidants Sugars Sulfide Sulfur Compounds Thiocyanate Volatile Compounds	D2036, Test Method B
Metal Cyanide Complexes	Ion Chromatography	UV	Carbonate Dissolved Solids Metal Anions Metal Cations Oxidants Photodecomposition	D6994
Free Cyanide	Flow Injection	Amperometric	Carbonate Oxidants Sulfide	D7237
Free Cyanide	Microdiffusion	Colorimetric	Aldehydes Ketones Oxidants Sulfide Sulfur Compounds	D4282

7.7 *pH Meter,* with a readability of 0.01 units and an accuracy of at least ±0.1 units at 25°C.

7.8 *Riffle Splitter*—A stationary sampler comprising an even number of equally-sized, adjacent chutes discharging in opposite directions. For use with this practice, there must be a minimum of twelve contained chutes (not bars) with an opening width of at least 3 times the nominal size.

NOTE 1—For riffle splitting finer materials (<3 mm) the 3 times nominal size should be increased to the point where the plugging of chutes is eliminated. For riffle splitting coarser materials (>12.5 mm) it is recommended not to exceed 3½ times nominal size as it is required that the full width of the riffle be used since the accuracy of the split increases with the number of chutes. For free-flowing materials, the 3 times top size may be reduced to 1½ times provided it is ascertained that there is no chute plugging for a particular material.

7.9 *Rotary Sample Divider*—A rotating sampler fed from a single point comprising equally-sized or adjustable proportional collection pans. For use with this guide, there must be a minimum opening width of at least 3 times the nominal size.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this guide. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets the purity specifications of Type I or Type II water, presented in Specification D1193.

8.3 *Concentrated Sodium Hydroxide Solution (0.4 M)*—In a 1-L volumetric flask, dissolve 16 g NaOH in reagent water and dilute to volume.

8.4 *Dilute Sodium Hydroxide Solution Extraction Fluid (0.04 M)*—In a 1-L volumetric flask, add 100 mL of 0.4 M NaOH in reagent water and dilute to volume.

8.5 *Sample Bottles*—See Section 10.4.1 for further information about sample bottles.

9. Hazards

9.1 **Warning—**Because of the toxicity of cyanide, great care must be exercised in its handling. Acidification of cyanide solutions produces toxic hydrocyanic acid (HCN). Adequate ventilation is necessary when handling cyanide solutions and a fume hood should be utilized whenever possible.

9.2 **Warning—**Many of the reagents used in these test methods are highly toxic. These reagents and their solutions and extracted solids must be disposed of properly.

10. Sampling

10.1 Obtain a representative sample of the mine rock or soil to be tested by using, where available, ASTM sampling

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[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD. (http://uk.vwr.com)

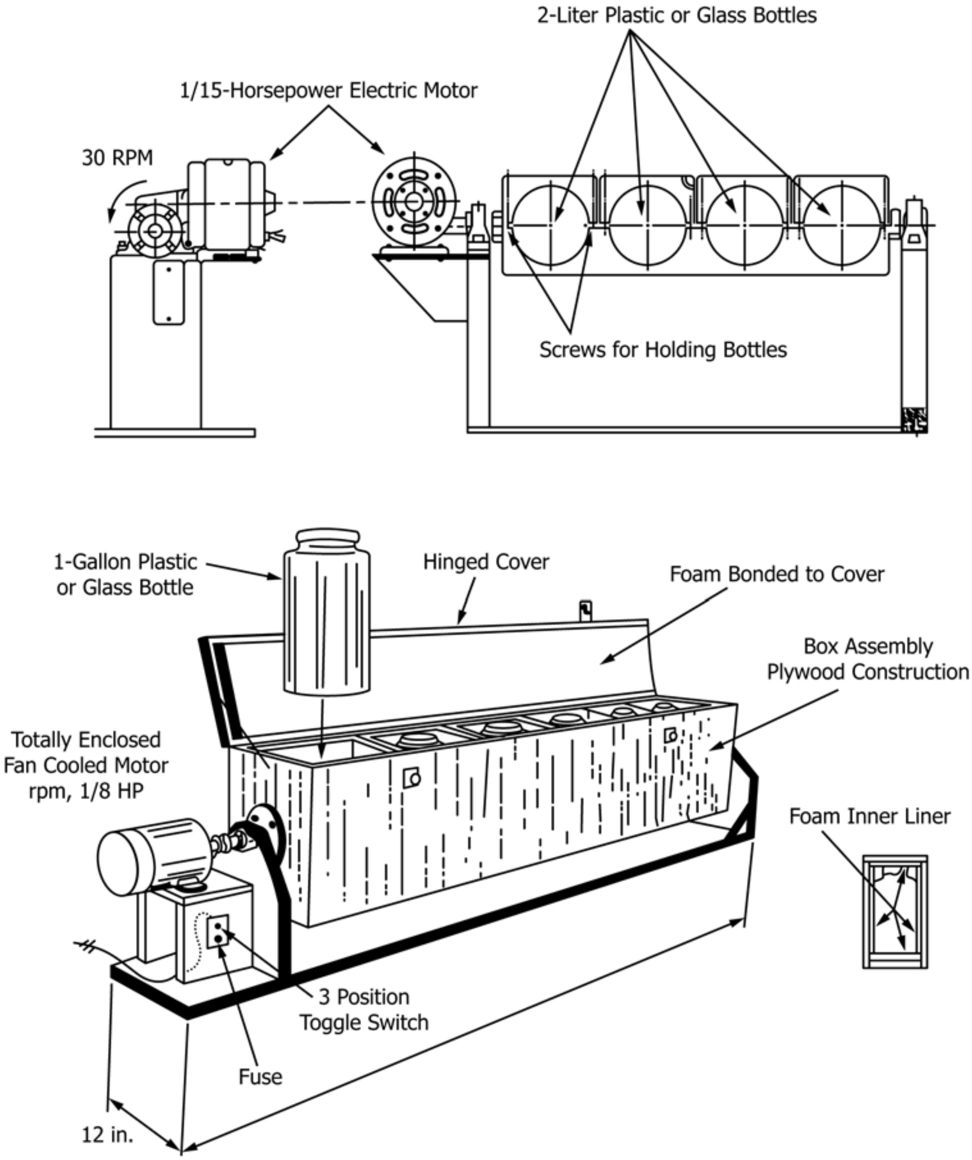

FIG. 1 Extractors

methods, or industry standards methods developed for use in the cyanide processing industry.

10.2 Sampling methodology for materials of similar physical form shall be used where no specific methods are available.

10.3 Laboratory personnel and field samplers should follow the industry best practice or acceptable metallurgical methods for sampling and sample preparation of mine rock and soil.

10.3.1 *Fine Rock and Soil Sampling*—For fine mine rock and soils, appropriate sized containers are listed in Table 2.

10.3.2 *Coarse Rock and Soil Sampling*—Samples with nominal size in excess of 0.94 cm should be collected by filling a nominal 20-L tare weighted sample bucket lined with a vapor barrier bag. It is recommended to consult with the analytical laboratory prior to collecting samples to ensure the proper sample volume, containers, etc., as these parameters may vary depending on the analytical methods used to measure cyanide. Collected samples must be sealed in vapor barrier bags in the shipping container and refrigerated as soon as practicable and transported to the laboratory. Refer to Guide D4840 for chain-of-custody procedures. If field preservation of the sample is not desired, proceed with Option B.

TABLE 2 Required Field Sample Weights and Sample Container Volumes as a Function of Particle Top Size

Nominal Sample Weight	Top Size, cm	Tared Field Container Size with Vapor Barrier Bag	Top Size, inches
30 kg	Run-of-mine	20 L (5 gal)	Run-of-mine
2 kg	>0.94	4 L (1 gal)	⅜ in.
500 g	>0.63	1 L (1 qt)	¼ in.

10.4 *Sample Containers:*

10.4.1 Sample and extract containers shall be made of materials that will not contaminate the sample, vapor barrier bags and bottles need to be cleaned thoroughly to remove all extraneous surface contamination prior to use, if necessary. Chemically resistant rigid plastic containers, such as those made of high density polyethylene (HDPE) are suitable. Samples should be collected and stored in opaque containers and sealed in vapor barrier bags to minimize exposure to ultraviolet radiation and loss of moisture and hydrogen cyanide.

10.4.2 Virgin or commercially cleaned containers certified to be free of contamination are recommended for handling extracts; otherwise, wash containers with soap or biodegradable detergent if required, then dry by draining. For further information on sample containers, see Practices D3694.

10.5 *Field Preservation Option*—If field preservation of the cyanide in the sample is desired, fill the field container with sample no more than half of its capacity, perform the weighing procedure in the field, add an equal volume of the dilute sodium hydroxide extraction fluid (8.4) to the wet weight of the sample in the field and seal the slurry in a vapor barrier bag for transport to the laboratory. Use multiple field collection containers if necessary to obtain a representative sample volume. Record the tare, field gross weight, gross weight with preservative, and transport to the laboratory for processing according to Option A. Collect a reserve sample using the same procedure.

NOTE 2—Replicate field samples needed for QA/QC, umpire, or reserve should be collected under the same conditions and a portion of the preservation solution provided to the laboratory as a field blan.

10.6 *Refrigeration*—Refrigerate during transport to the laboratory.

OPTION A
LABORATORY PROCESSING OF FIELD PRESERVED SAMPLES

11. Procedure

11.1 Collect the field tare, wet mass, volume of sodium hydroxide added and gross contained preserved slurry mass. Weigh the sample as received and verify the gross mass received in the laboratory represents the field gross mass.

11.2 Tare weigh the extraction container and record the mass.

11.3 Transfer the entire sample to the extraction container and proceed with extraction or resolve the causes of the mass differences before proceeding. Use multiple containers if necessary to contain the entire sample volume. If the mixture has absorbed all the liquid such that the mixture is not freely flowing and inhibits the ability of an analyst to obtain a pH measurement, add additional dilute 0.04 M NaOH solution until a free flowing mixture is obtained and a pH measurement is possible. Record any additional dilute NaOH volume added and determine the initial pH, in accordance with Test Methods D1293 and record. Seal extraction vessel that contains the slurry, weigh and record the mass.

11.4 Place the extraction vessel in the agitation equipment and rotate end-over-end at 30 ± 2 rpm for 2 hours.

11.4.1 After approximately 10 minutes of agitation, remove the extraction vessel from the agitation equipment, open, and measure and record the slurry pH.

11.4.2 If slurry pH < 12, add concentrated 0.4 M NaOH in 10-mL increments and mix until the pH exceeds 12.0.

11.4.3 Return the extraction vessel to the agitation equipment. Record the volume of concentrated 0.4 M NaOH added.

11.4.4 Repeat steps 11.4.1 and 11.4.3 until the pH stabilizes at or above pH 12.0 for two consecutive time periods.

11.5 At the end of two hours from the beginning of the extraction, record the final pH. If the pH is 11.9 or less, discard the sample and repeat the extraction with the reserve sample portion, adding the volume of concentrated 0.4 M NaOH used at the beginning of the extraction and make up the rest of the initial volume with the dilute 0.04 M NaOH extraction fluid. Recover and analyze the extract.

11.6 Weigh the final slurry and determine the wet slurry net weight by subtracting the extraction vessel tare weight and combine slurries if use of multiple extraction containers was necessary.

11.7 Filter the slurry and collect sufficient extract to perform cyanide analysis by the specified method. See Guide D6696 to assist with proper method selection for the intended use of this guide.

11.8 *Drying*—Transfer the residue to a tared pan and dry to a constant weight, at 104 ± 2°C, cool and weigh. Record the mass values to ±0.1 g. Record the temperature and time of the drying period.

11.9 *Aqueous Fraction Estimation*—Total the volumes of solution added to the sample in the field and during extraction for use in estimation of the aqueous fraction in the original sample.

11.10 Sample the extract, mitigate interferences, preserve and submit the sample for cyanide analysis in accordance with Practice D7365.

12. Calculation

12.1 When the results of the cyanide analysis are completed, calculate the concentration of the cyanide extracted from the field sample as follows:

12.1.1 Calculate the net slurry mass:

$$A = C - B \tag{1}$$

where:

A = net slurry mass (11.6), g,
B = extraction vessel tare mass (11.2), g, and
C = gross slurry mass (11.6).

12.1.2 Calculate the dry residue mass:

$$D = F - E \tag{2}$$

where:

D = dry residue mass (11.8), g,
E = drying pan tare mass (11.8), g, and
F = dry residue mass, g.

12.1.3 Calculate the extract mass:

$$G = A - D \tag{3}$$

where:

G = extract mass, g,

A = net slurry mass (11.6), g,
D = dry residue mass, g.

12.1.4 Calculate the aqueous mass of the field sample:

$$H = G - I \tag{4}$$

where:

H = aqueous mass in the field sample, g,
G = extract mass, g, and
I = sum of extraction solutions added to the sample, g.

12.1.5 Calculate the dilution factor for the aqueous mass:

$$J = G/H \tag{5}$$

where:

J = aqueous mass dilution factor,
G = extract mass, g, and
H = aqueous mass in the field sample, g.

12.1.6 Calculate the concentration of the cyanide in the aqueous mass of the field sample:

$$I = JK \tag{6}$$

where:

K = cyanide measured in extract, mg/L,
J = aqueous mass dilution factor, and
I = cyanide concentration extracted from the aqueous mass of the field sample, mg/L CN^-.

12.1.7 Calculate the cyanide content in the dry field sample:

$$L = IH/F \tag{7}$$

where:

L = aqueous cyanide extracted, µg/g CN^-,
I = cyanide concentration extracted from the aqueous mass of the field sample, mg/L CN^-,
H = aqueous mass in the field sample, g, and
F = dry residue mass, g.

OPTION B
LABORATORY PROCESSING OF MOIST FIELD SAMPLES

13. Procedure

13.1 *Weighing*—At the laboratory, record the pre-tared container weight, the gross sample weight, and calculate the net wet sample weight by difference.

13.2 *Sub-Sampling Options*—Sub-sample for laboratory processing by selecting one of the following options. Make sure that the sub-sample is representative of the gross sample for the purpose of the testing

13.2.1 *Fine Rock and Soil Sub-Sampling*—Weigh three nominal 100 ± 5 g sample weights, one for moisture determination and two for extraction and analysis, mixing the sample well between weighing from the field container as received at the laboratory. Proceed with moisture determination on one of the samples selected at random (13.3).

13.2.2 *Coarse Rock Sample Screening and Sub-Sampling*—If the sample nominal exceeds 2.54 cm, the sample is screened at 2.54 cm and the coarse fraction is separated from the fine fraction and each fraction is weighed and recorded.

13.2.2.1 The coarse fraction can be removed or extracted entirely without sub-sampling, if necessary, and the dry weight determined by drying after extraction.

13.2.2.2 *Sub-Sampling*—Prepare four clean tared sample pans each capable of containing at least one quarter of the volume of the gross sample after any screening. Proceed using one of the following options:

(1) Incremental Method—Using a scoop or spoon, transfer at least 50 approximately equal increments to the four pans, alternating pans in order until the sample or fine fraction is exhausted. Proceed with weighing step 13.2.3.

(2) Rotary Sample Divider Method—Divide the sample into multiple increments using a rotary sample divider, randomly select increments to fill the four pans and proceed with weighing step 13.2.3.

(3) Riffle Splitter Method—Divide the sample in half using a riffle splitter with fixed chutes at least twice as large as the largest particle. The chutes must be fed equally across the full width of the splitter at a rate which does not plug the chutes with material. Riffle split each half split again and place the quarters in pans.

13.2.3 Weigh each pan and select one pan at random for moisture analysis, one for cyanide extraction and reserve the other two fractions for quality assurance or umpire testing under refrigeration.

13.3 *Moisture Content*—Dry to a constant weight, at 104 ± 2°C, cool in a desiccator and weigh. Record the values to ±0.1 g. Record the temperature and time of the drying period.

13.3.1 Calculate the percent moisture as follows:

$$C = 100(B - A)B \tag{8}$$

where:

A = mass of sample after drying, g,
B = original mass of sample, g, and
C = moisture, %.

13.4 *Extraction*—Place the randomly selected, wet preweighed, sample in the extraction container and add the volume of the dilute 0.04 M NaOH solution approximating the wet weight of the sample portion, record the volume used and mix.

13.4.1 If the mixture absorbs all the liquid such that the mixture is not freely flowing and inhibits the ability of an analyst to obtain a pH measurement, add additional dilute 0.04 M NaOH solution until a free flowing mixture is obtained and a pH measurement is possible. Record any additional NaOH volume added and determine the initial pH, in accordance with Test Methods D1293 and record. Seal extraction vessel that contains the slurry and weigh.

13.5 Place the extraction vessel in the agitation equipment and rotate end-over-end at 30 ± 2 rpm for 2 hours.

13.5.1 After approximately 10 minutes of agitation, remove the extraction vessel from the agitation equipment, open and measure and record the slurry pH.

13.5.2 If slurry pH < 12, add concentrated 0.4 M NaOH in 10-mL increments and mix until the pH exceeds 12.0.

13.5.3 Return the extraction vessel to the agitation equipment. Record the volume of concentrated 0.4 M NaOH added.

13.5.4 Repeat steps 13.5.1 – 13.5.3 until the pH stabilizes at or above pH 12.0 for two consecutive time periods.

13.6 At the end of two hours from the beginning of the extraction, record the final pH. If the pH is 11.9 or less, discard the sample and repeat the extraction with a reserve sample portion, adding the volume of concentrated 0.4 M NaOH used at the beginning of the extraction and make up the rest of the initial volume with the dilute 0.04 M NaOH extraction fluid. Recover and analyze the extract.

13.6.1 Weigh the final slurry and determine the wet slurry weight by subtracting the extraction vessel tare weight.

13.6.2 Filter the slurry and collect sufficient extract to perform cyanide analysis by the specified method. See Guide D6696 to assist with proper method selection for the intended use of this guide.

13.6.3 Sample the extract, mitigate interferences, preserve, and submit the sample for cyanide analysis in accordance with Practice D7365.

14. Calculation

14.1 When the results of the cyanide analysis are completed, calculate the concentration of the cyanide in the sample as follows:

$$D = E(100 - C) \tag{9}$$

where:

D = mass of dry sample, g,
E = original mass of extraction sub-sample, g, and
C = moisture, %.

$$F = (G - D)/D \tag{10}$$

where:

F = dilution factor for slurry,
G = final weight of extract slurry, and
D = mass of dry sample in slurry, g.

$$I = FH \tag{11}$$

where:

F = dilution factor for slurry,
H = cyanide measured in extract, mg/L, and
I = aqueous cyanide extracted, µg/g CN^-

14.2 The cyanide concentration in the aqueous mass of the field sample may also be calculated as follows:

$$J = 100\ I/C \tag{12}$$

where:

J = cyanide concentration of the aqueous mass of the field sample, mg/L CN^-,
C = moisture, %, and
I = aqueous cyanide extracted, µg/g CN^-.

15. Quality Control and Reporting Requirements

15.1 Report cyanide as CN^- (usually in µg/g) and correct for any dilutions that may have been performed in the field or laboratory.

15.2 Indicate the type of cyanide (for example, free, available by FILE, WAD cyanide by distillation, or total) and standard method used for the determination. Make note of any specific mitigation of interference that was performed in the laboratory; if interference removal or suppression is not documented in this guide, provide a reference or supporting data to justify the action.

15.3 In addition to the quality control requirements specified in the analytical method, it is recommended to perform a laboratory matrix spike and matrix spike duplicate on the extract to evaluate precision and recovery for unknown matrices; although acceptable recoveries do not necessarily rule out the possibility of interference. For further information evaluating matrix spike duplicates, see Practice D5847. If interference is still suspected, sample characterization may be necessary to identify and mitigate the potential interference(s).

16. Keywords

16.1 aqueous cyanide; extraction; mine rock; soil

SUMMARY OF CHANGES

Committee D19 has identified the location of selected changes to this standard since the last issue (D7572 – 13) that may impact the use of this standard. (Approved Feb. 1, 2015.)

(1) Revised Eq 8.

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