

METHOD 3060A

ALKALINE DIGESTION FOR HEXAVALENT CHROMIUM

1.0 SCOPE AND APPLICATION

1.1 Any reference in this method to "Method 3060" refers to this version of that method, and does not refer to previously published versions (e.g., in the Second Edition of this manual). When published as a new method to SW-846, a method's number does not include a letter suffix. Each time a method is revised and made a part of SW-846 update, it receives a suffix. However, a method reference found within the text of SW-846 methods always refers to the latest version of that method published in SW-846, even if the method number at that location does not include the appropriate letter suffix.

1.2 Method 3060 is an alkaline digestion procedure for extracting hexavalent chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. To quantify total Cr(VI) in a solid matrix, three criteria must be satisfied: (1) the extracting solution must solubilize all forms of Cr(VI), (2) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and (3) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060 meets these criteria for a wide spectrum of solid matrices. Under the alkaline conditions of the extraction, minimal reduction of Cr(VI) or oxidation of native Cr(III) occurs. The addition of Mg^{2+} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation, if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g., $K_2Cr_2O_7$ and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is usually not observed except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$.

1.3 The quantification of Cr(VI) in Method 3060 digests should be performed using a suitable technique with appropriate accuracy and precision, for example Method 7196 (colorimetrically by UV-VIS spectrophotometry) or Method 7199 (colorimetrically by ion chromatography (IC)). Analytical techniques such as IC with inductively coupled plasma - mass spectrometric (ICP-MS) detection, high performance liquid chromatography (HPLC) with ICP-MS detection, capillary electrophoresis (CE) with ICP-MS detection, etc. may be utilized once performance effectiveness has been validated.

2.0 SUMMARY OF METHOD

2.1 This method uses an alkaline digestion to solubilize both water-insoluble (with the exception of partial solubility of barium chromate in some soil matrices, see Reference 10.9) and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. Failure to meet the pH specifications will necessitate redigestion of the samples.

2.2 The sample is digested using 0.28M Na_2CO_3 /0.5M NaOH solution and heating at 90-95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.3 The Cr(VI) reaction with diphenylcarbazide is the most common and reliable method for analysis of Cr(VI) solubilized in the alkaline digestate. The use of diphenylcarbazide has been well established in the colorimetric procedure (Method 7196), in rapid-test field kits, and in the ion chromatographic method for Cr(VI) (Method 7199). It is highly selective for Cr(VI) and few interferences are encountered when it is used on alkaline digestates.

2.4 For additional information on health and safety issues relating to chromium, refer to References 10.7 and 10.10.

3.0 INTERFERENCES

3.1 When analyzing a sample digest for total Cr(VI), it is appropriate to determine the reducing/oxidizing tendency of each sample matrix. This can be accomplished by characterization of each sample for additional analytical parameters, such as pH (Method 9045), ferrous iron (ASTM Method D3872-86), sulfides (Method 9030), and Oxidation Reduction Potential (ORP) (ASTM Method D 1498-93 - aqueous samples). Method 9045 (Section 7.2 of Method 9045) is referenced as the preparatory method for soil samples. The ORP and temperature probes are inserted directly into the soil slurry. The displayed ORP value is allowed to equilibrate and the resulting measurement is recorded. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD). Analysis of these additional parameters establishes the tendency of Cr(VI) to exist or not exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

3.2 Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentrations of these interfering substances are high and the Cr(VI) concentration is low. Refer to Methods 7196 and 7199 for a discussion of the specific agents that may interfere with Cr(VI) quantification. Analytical techniques that reduce bias caused by co-extracted matrix components may be applicable in correcting these biases after validation of their performance effectiveness.

3.3 For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation. If an analytical method for Cr(VI) is used that can correct for possible method induced oxidation/reduction, then the Mg^{2+} addition is optional. The presence of soluble Cr(III) can be approximated by extracting the sample with deionized water (ASTM methods D4646-87, D5233-92, or D3987-85) and analyzing the resultant leachate for both Cr(VI) and total Cr. The difference between the two values approximates soluble Cr(III).

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessel: borosilicate glass or quartz with a volume of 250 mL.

4.2 Graduated Cylinder: 100-mL or equivalent.

4.3 Volumetric Flasks: Class A glassware, 1000-mL and 100-mL, with stoppers or equivalent.

- 4.4 Vacuum Filtration Apparatus.
- 4.5 Filter membranes (0.45 μm). Preferably cellulosic or polycarbonate membranes. When vacuum filtration is performed, operation should be performed with recognition of the filter membrane breakthrough pressure.
- 4.6 Heating Device - capable of maintaining the digestion solution at 90-95°C with continuous auto stirring capability or equivalent.
- 4.7 Volumetric pipettes: Class A glassware, assorted sizes, as necessary.
- 4.8 Calibrated pH meter.
- 4.9 Calibrated balance.
- 4.10 Temperature measurement device (with NIST traceable calibration) capable of measuring up to 100°C (e.g. thermometer, thermistor, IR sensor, etc.).
- 4.11 An automated continuous stirring device (e.g. magnetic stirrer, motorized stirring rod, etc.), one for each digestion being performed.

5.0 REAGENTS

5.1 Nitric acid: 5.0 M HNO_3 , analytical reagent grade or spectrograde quality. Store at 20-25°C in the dark. Do not use concentrated HNO_3 to make up 5.0 M solution if it has a yellow tinge; this is indicative of photoreduction of NO_3^- to NO_2 , a reducing agent for Cr(VI).

5.2 Sodium carbonate: Na_2CO_3 , anhydrous, analytical reagent grade. Store at 20-25°C in a tightly sealed container.

5.3 Sodium hydroxide: NaOH , analytical reagent grade. Store at 20-25°C in a tightly sealed container.

5.4 Magnesium Chloride: MgCl_2 (anhydrous), analytical reagent grade. A mass of 400 mg MgCl_2 is approximately equivalent to 100 mg Mg^{2+} . Store at 20-25°C in a tightly sealed container.

5.5 Phosphate Buffer:

5.5.1 K_2HPO_4 : analytical reagent grade.

5.5.2 KH_2PO_4 : analytical reagent grade.

5.5.3 0.5M K_2HPO_4 /0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09 K_2HPO_4 and 68.04 g KH_2PO_4 into 700 mL of reagent water. Transfer to a 1L volumetric flask and dilute to volume.

5.6 Lead Chromate: PbCrO_4 , analytical reagent grade. The insoluble matrix spike is prepared by adding 10-20 mg of PbCrO_4 to a separate sample aliquot. Store under dry conditions at 20-25°C in a tightly sealed container.

5.7 Digestion solution: Dissolve 20.0 ± 0.05 g NaOH and 30.0 ± 0.05 g Na_2CO_3 in reagent water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20-25°C and prepare fresh monthly. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater, if not, discard.

5.8 Potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, spiking solution (1000 mg/L Cr(VI)): Dissolve 2.829 g of dried (105°C) $\text{K}_2\text{Cr}_2\text{O}_7$ in reagent water in a one-liter volumetric flask and dilute to the mark. Alternatively, a 1000 mg/L Cr(VI) certified primary standard solution can be used (Fisher AAS standard or equivalent). Store at 20-25°C in a tightly sealed container for use up to six months.

5.8.1 Matrix spiking solution (100 mg/L Cr(VI)): Add 10.0 mL of the 1000 mg Cr(VI)/L made from $\text{K}_2\text{Cr}_2\text{O}_7$ spiking solution (Section 5.8) to a 100 mL volumetric flask and dilute to volume with reagent water. Mix well.

5.9 Reagent Water - Reagent water will be free of interferences. Refer to Chapter One for a definition of reagent water.

6.0. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Samples should be collected using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass).

6.3 Samples should be stored field-moist at $4 \pm 2^\circ\text{C}$ until analysis.

6.4 Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection. In addition, Cr(VI) has also been shown to be stable in the alkaline digestate for up to 168 hours after extraction from soil.

6.5 Hexavalent chromium solutions or waste material that are generated should be disposed of properly. One approach is to treat all Cr(VI) waste materials with ascorbic acid or other reducing agent to reduce the Cr(VI) to Cr(III). For additional information on health and safety issues relating to chromium, the user is referred to References 10.7 and 10.10.

7.0 PROCEDURE

7.1 Adjust the temperature setting of each heating device used in the alkaline digestion by preparing and monitoring a temperature blank [a 250 mL vessel filled with 50 mLs digestion solution (Section 5.7)]. Maintain a digestion solution temperature of 90-95°C as measured with a NIST-traceable thermometer or equivalent.

7.2 Place 2.5 ± 0.10 g of the field-moist sample into a clean and labeled 250 mL digestion vessel. The sample should have been mixed thoroughly before the aliquot is removed.

For the specific sample aliquot that is being spiked (Section 8.5), the spike material should be added directly to the sample aliquot at this point. (Percent solids determination, U.S. EPA CLP SOW for Organic Analysis, OLM03.1, 8/94 Rev.) should be performed on a separate aliquot in order to calculate the final result on a dry-weight basis).

7.3 Add 50 mL \pm 1 mL of digestion solution (Section 5.7) to each sample using a graduated cylinder, and also add approximately 400 mg of MgCl₂ (Section 5.4) and 0.5 mL of 1.0M phosphate buffer (Section 5.5.3). For analytical techniques that can correct for oxidation/reduction of Cr, the addition of Mg²⁺ is optional. Cover all samples with watch glasses.

7.4 Stir the samples continuously (unheated) for at least five minutes using an appropriate stirring device.

7.5 Heat the samples to 90-95°C, then maintain the samples at 90-95°C for at least 60 minutes with continuous stirring.

7.6 Gradually cool, with continued agitation, each solution to room temperature. Transfer the contents quantitatively to the filtration apparatus; rinsing the digestion vessel with 3 successive portions of reagent water. Transfer the rinsates to the filtration apparatus. Filter through a 0.45µm membrane filter. Rinse the inside of the filter flask and filter pad with reagent water and transfer the filtrate and the rinses to a clean 250-mL vessel.

NOTE: The remaining solids and filter paper resulting from filtration of the matrix spike in Section 7.6 should be saved for possible use in assessing low Cr(VI) matrix spike recoveries. See Section 8.5.2. for additional details. Store the filtered solid at 4 \pm 2°C.

7.7 Place an appropriate stirring device into the sample digest beaker, place the vessel on a stirrer, and, with constant stirring, slowly add 5.0 M nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 \pm 0.5 if the sample is to be analyzed using Method 7196 (adjust the pH accordingly if an alternate analytical method is to be used; i.e. 9.0 \pm 0.5 if Method 7199 is to be used) and monitor the pH with a pH meter. If the pH of the digest should deviate from the desired range, discard the solution and redigest. If overshooting the desired pH range occurs repeatedly, prepare diluted nitric acid solution and repeat digestion procedure. If a flocculent precipitate should form, the sample should be filtered through a 0.45 µm membrane filter. If the filter becomes clogged using the 0.45 µm filter paper, a larger size filter paper (Whatman GFB or GFF) may be used to prefilter the samples.

CAUTION: CO₂ will be evolved. This step should be performed in a fume hood.

7.8 Remove the stirring device and rinse, collecting the rinsate in the beaker. Transfer quantitatively the contents of the vessel to a 100 mL volumetric flask and adjust the sample volume to 100 mL (to the mark for the volumetric flask) with reagent water. Mix well.

7.9 The sample digestates are now ready to be analyzed. Determine the Cr(VI) concentration in mg/kg by a suitable technique with appropriate accuracy and precision, for example Method 7196 (colorimetrically by UV-VIS spectrophotometry) or Method 7199 (colorimetrically by ion chromatography (IC)). Another analytical technique such as IC with inductively coupled plasma - mass spectrometric (ICP-MS) detection, high performance liquid chromatography (HPLC) with ICP-

MS detection, capillary electrophoresis (CE) with ICP-MS detection, etc. may be utilized once performance effectiveness has been validated.

7.10 CALCULATIONS

7.10.1 Sample Concentration

$$\text{Concentration} = \frac{A \times D \times E}{B \times C}$$

where: A = Concentration observed in the digest ($\mu\text{g}/\text{mL}$)
B = Initial moist sample weight (g)
C = % Solids/100
D = Dilution Factor
E = Final digest volume (mL)

7.10.2 Relative Percent Difference

$$\text{RPD} = \frac{(S - D)}{[(S + D)/2]}$$

where: S = Initial sample result
D = Duplicate sample result

7.10.3 Spike Recovery

$$\text{Percent Recovery} = \frac{(\text{SSR} - \text{SR})}{\text{SA}} \times 100$$

where: SSR = Spike sample result
SR = Sample (unspiked) result
SA = Spike added

8.0 QUALITY CONTROL

8.1 The following Quality Control (QC) analyses must be performed per digestion batch as discussed in Chapter One.

8.2 A preparation blank must be prepared and analyzed with each digestion batch, as discussed in Chapter One and detected Cr(VI) concentrations must be less than the method detection limit or one-tenth the regulatory limit or action level, whichever is greater or the entire batch must be redigested.

8.3 Laboratory Control Sample (LCS): As an additional determination of method performance, utilize the matrix spike solution prepared in Section 5.8.1 or the solid matrix spiking agent PbCrO_4 (Section 5.6) to spike into 50 mL of digestion solution (Section 5.7). Alternatively, the use of a certified solid reference material (if available) is recommended. Recovery must be within the certified acceptance range or a recovery range of 80% to 120% or the sample batch must be reanalyzed.

8.4 A separately prepared duplicate soil sample must be analyzed at a frequency of one per batch as discussed in Chapter One. Duplicate samples must have a Relative Percent Difference (RPD) of $\leq 20\%$, if both the original and the duplicate are \geq four times the laboratory reporting limit. A control limit of \pm the laboratory reporting limit is used when either the original or the duplicate sample is $<$ four times the laboratory reporting limit.

8.5 Both soluble and insoluble pre-digestion matrix spikes must be analyzed at a frequency of one each per batch of ≤ 20 field samples. The soluble matrix spike sample is spiked with 1.0 mL of the spiking solution prepared in Section 5.8.1 (equivalent to 40 mg Cr(VI)/Kg) or at twice the sample concentration, whichever is greater. The insoluble matrix spike is prepared by adding 10-20 mg of PbCrO_4 (Section 5.6) to a separate sample aliquot. It is used to evaluate the dissolution during the digestion process. Both matrix spikes are then carried through the digestion process described in Section 7.0. More frequent matrix spikes must be analyzed if the soil characteristics within the analytical batch appear to have significant variability based on visual observation. An acceptance range for matrix spike recoveries is 75-125%. If the matrix spike recoveries are not within these recovery limits, the entire batch must be rehomogenized/redigested/reanalyzed. If upon reanalysis, the matrix spike is not within the recovery limits, but the LCS is within criteria specified in Section 8.3, information such as that specified on Figures 1 and 2 and in Section 3.1 should be carefully evaluated. The Cr(VI) data may be valid for use despite the perceived "QC failure." The information shown on Figure 1 and discussed below is provided to interpret ancillary parameter data in conjunction with data on spike recoveries.

8.5.1 First measure the pH (Method 9045) and Oxidation Reduction Potential (ORP) (ASTM Method D 1498-93 - aqueous samples, Method 9045 preparatory for soil samples), in the field if possible. If not possible, the measurements are to be made in the laboratory prior to the determination of the spike recovery data. When and where the measurements are taken must be noted by the analyst. Adjust the ORP measurement based on reference electrode correction factor to yield Eh values. The pH and Eh values should be plotted on Figure 2 in order to give an initial indication of the sample's reducing/oxidizing nature. Upon completion of the analysis of the analytical batch, the LCS should be evaluated. If the LCS is not within 80 - 120% recovery or the certified acceptance range, then the entire analytical batch (plus the QC samples) should be redigested and reanalyzed. If the LCS was within acceptance criteria and the pre-digestion matrix spike recoveries for Cr(VI) were less than the acceptance range minimum (75%), this indicates that the soil samples reduced Cr(VI) (e.g., anoxic sediments), and no measurable native Cr(VI) existed in the unspiked sample (assuming the criteria in Section 8.3 are met). Such a result indicates that the combined and interacting influences of ORP, pH and reducing agents (e.g., organic acids, Fe^{2+} and sulfides) caused reduction of Cr(VI) spikes. Characterize each matrix spike sample for additional analytical parameters, such as ferrous iron (ASTM Method D3872-86), and sulfides (Method 9030). Laboratory measurements of pH and ORP should also be performed to confirm the field measurements. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD). Analysis of these additional parameters assists in evaluating the tendency of Cr(VI)

to exist or not exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

A value of Eh-pH below the bold diagonal line on Fig. 2 indicates a reducing soil for Cr(VI). The downward slope to the right indicates that the Eh value, at which Cr(VI) is expected to be reduced, decreases with increasing pH. The solubility and quantity of organic constituents influence reduction of Cr(VI). The presence of H₂S or other strong odors indicates a reducing environment for Cr(VI). In general, acidic conditions accelerate reduction of Cr(VI) in soils, and alkaline conditions tend to stabilize Cr(VI) against reduction. If pre-digestion matrix spike recovery is not within the recovery limits, the reductive nature of the sample must be documented. This is done by plotting the Eh and pH data on the Eh-pH diagram (Fig. 2) to see if spike recovery is or is not expected in the soil. If the data point falls below the Cr(VI)-Cr(III) line on the diagram, then the data is not qualified or rejected. The sample is reducing for Cr(VI). If the data point falls above the line, then the sample is capable of supporting Cr(VI). In this case, technical error may be responsible for the poor spike recovery, and the extraction should be repeated, along with the Eh and pH measurements. If re-extraction results in a poor spike recovery again, then the data is qualified. At this point, review of other soil characteristics, such as levels of pH, Eh, TOC, sulfides, Fe(II), is appropriate to understand why poor spike recovery occurred. This extra review of these soil properties is only necessary if the unspiked sample contains detectable Cr(VI).

8.5.2 If a low or zero percent pre-digestion matrix spike recovery is obtained, an alternate approach can be used to determine the potential contribution of the sample matrix to Cr(VI) reduction. This approach consists of performing a mass balance, whereby total chromium is analyzed (Method 3052) for two samples: (1) a separate unspiked aliquot of the sample previously used for spiking, and (2) the digested solids remaining after the alkaline digestion and filtration of the matrix spike (i.e., the filtered solids from the matrix spike in Section 7.6).

The difference between the total chromium measurements should be approximately equal to the amount of the spike added to the matrix spike. If the LCS (Section 8.3) met the acceptance criteria and the Cr(VI) spike is accounted for in the filtered solids as total chromium, it is likely that the reduction of the Cr(VI) to insoluble Cr(III) resulted from the reducing matrix of the original sample subjected to Cr(VI) spiking.

8.6 A post-digestion Cr(VI) matrix spike must be analyzed per batch as discussed in Chapter One. The post-digestion matrix spike concentration should be equivalent to 40 mg/kg or twice the sample concentration observed in the unspiked aliquot of the test sample, whichever is greater.

8.6.1 Dilute the sample aliquot to a minimum extent, if necessary, so that the absorbance reading for both the unspiked sample aliquot and spiked aliquot are within the initial calibration curve.

8.6.2 A guideline for the post-digestion matrix spike recovery is 85-115%. If not achieved, consider the corrective actions/guidance on data use specified in Section 8.5 or the Method of Standard Additions (MSA) as specified in Section 8.0 of Method 7000. If the MSA technique is applied post digestion and no spike is observed from the MSA, these results indicate that the matrix is incompatible with Cr(VI) and no further effort on the part of

the laboratory is required. These digestates may contain soluble reducing agents for Cr(VI), such as fulvic acids.

9.0 METHOD PERFORMANCE

9.1 A commercial laboratory analyzed soil/sediment samples containing Cr(VI) with the results found in Table 1.

10.0 REFERENCES

10.1 United States Environmental Protection Agency, 1982. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, Second Edition. Office of Solid Waste and Emergency Response, Washington, D.C.

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10.3 Vitale, R., G. Mussoline, J. Petura, B. James, 1993. A Method Evaluation Study of an Alkaline Digestion (Modified Method 3060) Followed by Colorimetric Determination (Method 7196) for the Analysis for Hexavalent Chromium in Solid Matrices. Environmental Standards, Inc. Valley Forge, PA 19482.

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10.7 U.S. Department of Health and Human Services - Agency for Toxic Substances and Disease Registry. Toxicological Profile for Chromium. April, 1993.

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10.12 ASTM (American Society for Testing and Materials), 1981. Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soil and Sediments. ASTM Designation:D4646-87.

10.13 ASTM (American Society for Testing and Materials), 1981. Standard Test Method for Single Batch Extraction Method for Waters. ASTM Designation:D5233-92.

10.14 ASTM (American Society for Testing and Materials), 1981. Standard Test Method for Shake Extraction of Solid Waste with Water. ASTM Designation:D3987-85.

10.15 U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multimedia Multiconcentration Document, OLM03.1, 8/94 Rev.

TABLE 1
SINGLE LABORATORY METHOD EVALUATION DATA

<u>Sample Type</u>	<u>Eh</u> <u>(mV)_b</u>	<u>pH_d</u>	<u>S²⁻</u> <u>(ppm)^c</u>	<u>Mean Native</u> <u>Cr(VI) Conc.</u> <u>(mg/kg)</u>	<u>Mean Cr(VI)</u> <u>Spike Conc.</u> <u>(mg/kg)</u>	<u>Matrix Spike</u> <u>Recovery</u> <u>Range.%</u>
COPR ^a /Soil Blends	550	7.4	<10.0	4.1	42.0	89.8-116
Loam	620	6.4	<10.0	ND	62.5	65.0-70.3
Clay	840	3.0	<10.0	ND	63.1	37.8-71.1
COPR ^a	460	7.4	<10.0	759	813	85.5-94.8
Anoxic Sediment	-189	7.2	25.0	ND	381	0
Quartz Sand	710	5.3	<10.0	ND	9.8	75.5-86.3

Source: Reference 10.3

Notes:

- ND - Not detected
- a - COPR - chromite ore processing residue
- b - Corrected for the reference electrode, laboratory field moist measurement
- c - Field measurement
- d - Laboratory field moist measurement

FIGURE 1
 QUALITY CONTROL FLOW CHART (Continued)

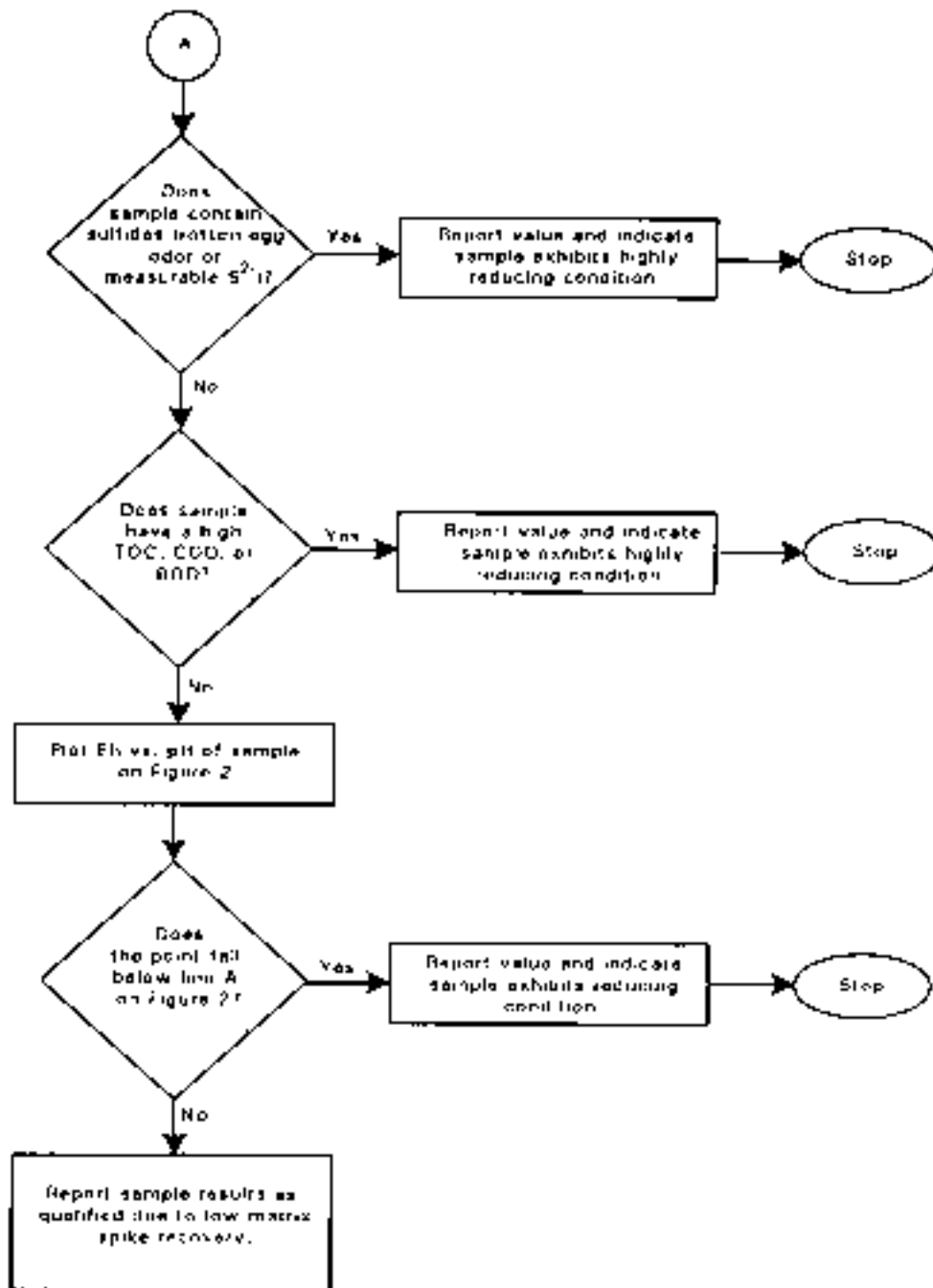
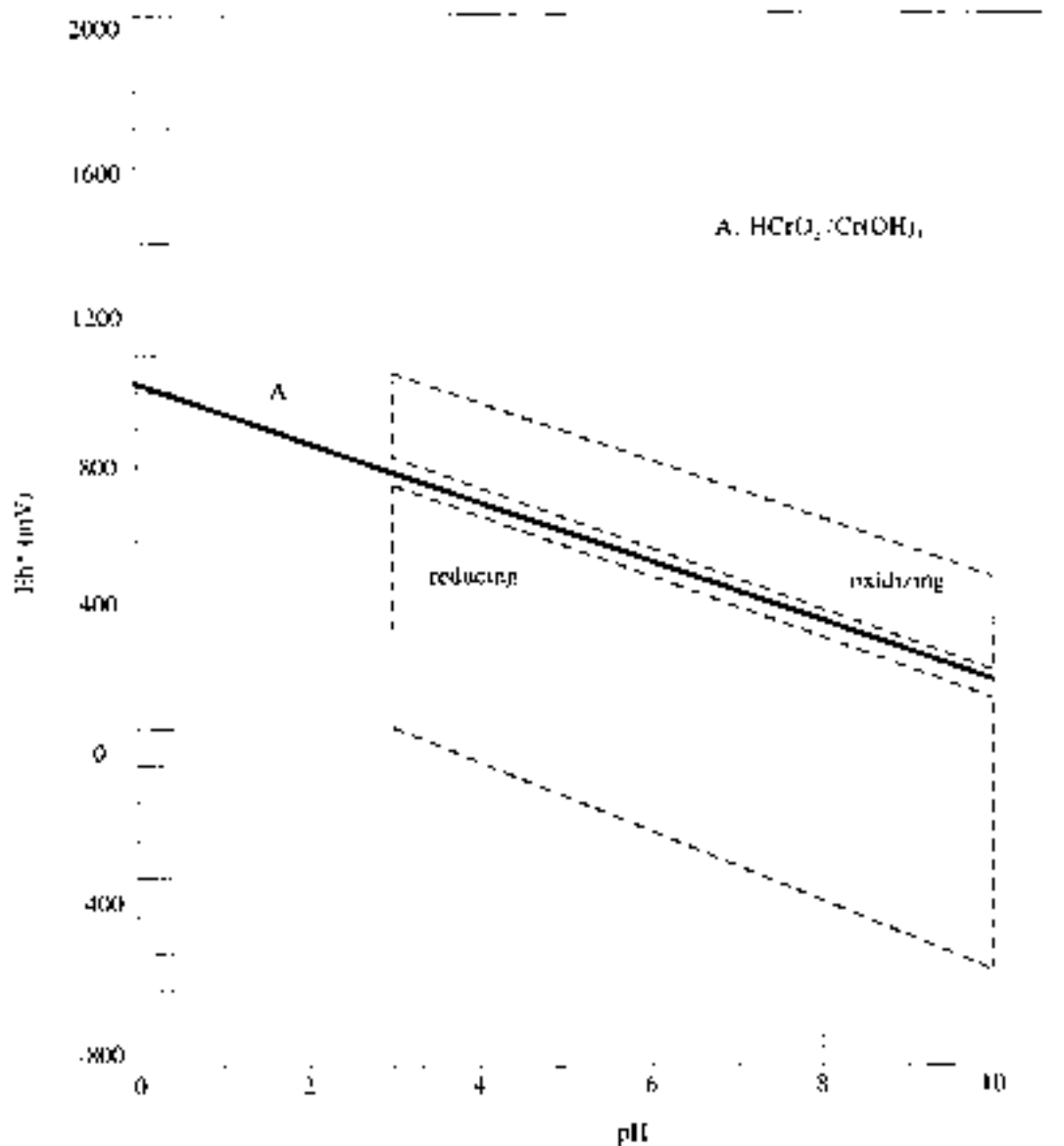


FIGURE 2
Eh/pH PHASE DIAGRAM

The dashed lines define Eh/pH boundaries commonly encountered in soils and sediments



* Note: the Eh values plotted on this diagram are corrected for the reference electrode voltage. 244 mV units must be added to the measured value when a separate calomel electrode is used, or 190 mV units must be added if a combination platinum electrode is used.

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