Designation: D6052 – 97 (Reapproved 2016)

# Standard Test Method for Preparation and Elemental Analysis of Liquid Hazardous Waste by Energy-Dispersive X-Ray Fluorescence[1]

This standard is issued under the fixed designation D6052; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of trace and major element concentrations by energy-dispersive X-ray fluorescence spectrometry (EDXRF) in liquid hazardous waste (LHW).

1.2 This test method has been used successfully on numerous samples of aqueous and organic-based LHW for the determination of the following elements: Ag, As, Ba, Br, Cd, Cl, Cr, Cu, Fe, Hg, I, K, Ni, P, Pb, S, Sb, Se, Sn, Tl, V, and Zn.

1.3 This test method is applicable for other elements (Si-U) not listed in 1.2.

1.4 *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]

C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems (Withdrawn 2008)[3]

D1193 Specification for Reagent Water

2.2 *Other ASTM Documents:*

ASTM Data Series DS 46 X-ray Emission Wavelengths and KeV Tables for Nondiffractive Analysis[4]

## 3. Summary of Test Method

3.1 A weighed portion of activated alumina and sample are combined in a mixing vessel and shaken until well mixed. The sample mixture is transferred into a disposable sample cup and placed in the spectrometer for analysis.

3.2 The *K* spectral emission lines are used for elements Si-Ba.

3.3 The *L* spectral emission lines are used for elements with atomic numbers greater than Ba.

## 4. Significance and Use

4.1 The elemental analysis of liquid hazardous waste is often important for regulatory and process specific requirements. This test method provides the user an accurate, rapid method for trace and major element determinations.

## 5. Interferences

5.1 *Spectral Overlaps (Deconvolution):*

5.1.1 Samples containing a mixture of elements often exhibit X-ray emission line overlap. Modern Si (Li) detectors generally provide adequate resolution to minimize the effects of spectral overlap. In cases where emission line overlap exists, techniques of peak fitting exist for extracting corrected analyte emission line intensities. For example, the PbLα "line overlaps with the AsKα." The PbLβ line can be used to avoid this overlap and the AsK lines can then be resolved from the PbLα overlap. The actual lines used for any particular element should be such that overlaps are minimized. Follow the EDXRF manufacturer's recommendation concerning spectral deconvolution. Reference should be made to ASTM Data Series DS 46 for detailed information on potential line overlaps.

5.2 *Matrix Interferences (Regression):*

5.2.1 Matrix interference in the measurement of "as received" LHW samples using EDXRF has been the principle limitation in the development and expanding use of this instrumental technique. Using well understood XRF principles for controlling matrix effects, for example, dilution and matrix modification using lithium borate fusion and addition of heavy absorbers, a matrix can be stabilized. Using calcined alumina and the above principles matrices are stabilized for quantitative EDXRF analysis.

5.2.2 The response range of this test method should be linear with respect to the elements of interest and their regulatory or process control, or both, action thresholds. Large

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

Current edition approved Sept. 1, 2016. Published September 2016. Originally approved in 1997. Last previous edition approved in 2008 as D6052 – 97 (2008). DOI: 10.1520/D6052-97R16.

[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.

[4] Available from ASTM Headquarters, Customer Service.

concentration variations of element or matrix, or both, components in LHW samples can result in non-linear X-ray intensity response at increasing element concentrations.

## 6. Apparatus

6.1 *Energy-dispersive X-ray Fluorescence Spectrometer,* capable of measuring the wavelengths of the elements listed in 1.2. Refer to Guide C982 for system specifications.

6.2 *Analytical Balance,* capable of weighing to 0.001 g.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean meeting the numerical requirements of Type II water as defined by Specification D1193.

7.3 *Aluminum Oxide, $Al_2O_3$*—pre-calcined at 1500°C, approximately 100 to 125 mesh.

7.4 *Aqueous or organic-based Atomic Absorption Standards (AAS),* 1000 mg/L for the elements Ag, As, Ba, Cd, Cr, Cu, Fe, Hg, K, Ni, Pb, Sb, Se, Sn, Tl, V, and Zn. Standard solutions for elements not listed are also available.

NOTE 1—AAS standards are typically presented in mass/vol units. The density of these solutions can be considered as unity (that is, 1) thus they can be considered as % mass/mass (m/m).

7.5 *1-bromonaphthalene, trichlorobenzene, iodobenzoic acid, triethyl phosphate and dithiodiglycol* are the recommended standards for the elements Br, Cl, I, P and S, respectively.

7.6 *Low Molecular Weight Polyethylene Glycol (PEG 400, or equivalent) or Water* is used for producing method blank.

7.7 *High-Density Polyethylene (HDPE) Wide-mouth, Round, Screw-Cap Bottles,* 50 to 60 mL capacity.

7.8 *Mixing Balls,* approximately 1 cm diameter, stainless steel or equivalent.

NOTE 2—Potential low level Cr, Fe or Ni (<20 mg/kg[1]) contamination due to the use of stainless steel may exist. Other suitable materials would be tungsten carbide, Zr or Ta.

7.9 *Thin-film Support.*

NOTE 3—The user should select a thin-film support that provides for maximum transmittance and is resistant to typical components in LHW. The thin-film supports used in the development of this test method were a polypropylene base and a high-purity, 4 µm polyester film.

**TABLE 1 Recommended Standards Ranges**

| Analyte | Low Concentration Range, mg/kg | High Concentration Range, mg/kg | Analyte | Low Concentration Range, mg/kg | High Concentration Range, mg/kg |
|---|---|---|---|---|---|
| Ag | 5 | 600 | Zn | 5 | 600 |
| Ba | 5 | 600 | As | 5 | 600 |
| P | 0.1 % | 5 % | Se | 5 | 600 |
| S | 0.05 % | 5 % | Br | 10 | 5000 |
| Cl | 0.05 % | 5 % | Cd | 5 | 600 |
| K | 0.1 % | 5 % | Sb | 5 | 600 |
| V | 5 | 600 | Sn | 5 | 600 |
| Cr | 5 | 600 | I | 5 | 600 |
| Fe | 5 | 600 | Hg | 5 | 600 |
| Ni | 5 | 600 | Tl | 5 | 600 |
| Cu | 5 | 600 | Pb | 5 | 600 |

7.10 *Sample Cups,* vented.

7.11 *Helium, He*—minimum 99.99 purity for use as a chamber purge gas for the analysis of Cl, P and S. This numerical purity is intended to specify a general grade of helium. Ultra-high purity helium is not required for this test method.

## 8. Sample

8.1 Because of the potential heterogeneous nature of LHW, all possible efforts should be made to ensure that representative samples are taken.

## 9. Preparation of Apparatus

9.1 Follow the manufacturer's instructions for set-up, conditioning, preparation and maintenance of the XRF spectrometer.

9.2 When required, reference spectra should be obtained from pure element standards for all deconvoluted elements.

9.3 Spectral and matrix interferences as listed in the Interferences section must be addressed per the manufacturer's recommendations.

## 10. Calibration and Standardization

10.1 The spectrometer must be calibrated using an appropriate reference element(s) at a minimum frequency as recommended by the manufacturer.

10.2 Analytical standards should be prepared gravimetrically by blending the solution or pure element standards with $Al_2O_3$ to suitable standard concentrations as determined by the user's analytical requirements. Table 1 gives recommended concentration ranges for regression. Standards can be single or multi-element mixtures. Standard solutions are generally mixed with $Al_2O_3$ at a ratio of 3:1.

NOTE 4—More than one standard element(s) solution can be added to a single 15 g $Al_2O_3$ mass provided the total mass of standard is 5 g. This will maintain the proper 3:1 ratio while allowing mixtures of potentially incompatible elements to be combined in a single standard.

10.2.1 The number of standards required to produce calibrations is dependent on the number of elements to be determined. Generally, two calibrations are produced, the first is to determine potentially major elements such as halogens, S

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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 *Analar 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.

& P. The second is to determine trace elements, typically toxic metals and heavy elements. The minimum number of standards required can be determined from the following equation: minimum standards required = number elements determined plus two. Both of the above calibrations should use a minimum of ten standards each to cover the element concentration ranges shown in Table 1 and to ensure that adequate data is available to assess spectral overlaps as described in 5.1.

10.3 The $Al_2O_3$ + element(s) specimen is placed into an XRF sample cup supported by a suitable thin-film. The sample is gently tapped on a flat, hard surface to settle the powder against the thin-film support and ensure there are no air gaps.

10.3.1 The standard specimen in the sample cups is placed in the spectrometer's sample holder avoiding any contact with the film or rough handling that may disturb the standards.

10.4 Two methods of calibration are available.

10.4.1 *Method A*—Empirical calibration method using a suit of standard concentrations. Standard concentrations are limited to 600 mg/kg for Ag, As, Ba, Br, Cd, Cr, Cu, Fe, Hg, I, K, Ni, Pb, Sb, Se, Sn, Tl, V, and Zn. Standard concentrations are limited to 5 % for Cl, P, S, and other light elements (that is, $<z = 22$ and <0.5 % for Br). The limits ensure staying within the linear range and due to the limited concentration range of available traceable standards. The standards should provide a linear response of element intensity to concentration. Serial dilutions of analyte standards can be used to set up the calibration for each element. Multi-element standards can then be used to assess the deconvolution requirements of the spectrometer and check for calibration linearity.

NOTE 5—Standards may be diluted into the linear range using low molecular weight polyethylene glycol (PEG) or water. The choice of diluent is dependent upon whether the original standard solution is aqueous- or organic-based. For example, a 5000 mg/kg organic-based Pb standard solution can be diluted into the 0–600 mg/kg range by combining and mixing 15 g of $Al_2O_5$ + 0.5 g of 5000 mg/kg Pb standard solution + 4.5 g PEG. This yields a ten-fold dilution yielding a prepared standard concentration of 500 mg/kg.

10.4.1.1 *Drift Correction Monitors*—To correct for instrumental drift, use physically stable, solid disks or pressed pellets containing at least one element measured under each instrumental condition used.[6] At least two disks are necessary to correct both sensitivity and base-line drifts. One should provide a high net count-rate similar to standards from the upper end of the calibration range and the other should provide a low net count-rate similar to the blank. Measure the net count-rate for each element in the high concentration disk in such a way that the counting statistical error due to random fluctuation of the X-ray flux is less than 0.5 % relative to the net count-rate. Counting times must be long enough to collect 40 000 net counts for each element in the high-concentration disk. Use the same counting times when measuring the low concentration or blank disk.

10.4.2 *Method B*—Fundamental Parameters method. Most EDXRF manufacturers provide software capable of estimating the composition of materials without the use of a suite of standards. The setup of a particular manufacturer's fundamental parameters method may require a high and low concentration or mid-range concentration for each element present to determine the initial sensitivity for the elements in the alumina matrix. Other manufacturers provide the initial sensitivity with the added option to align the sensitivity to a specific matrix type for more accurate determinations using a single similar standard containing the elements of interest. By measuring the X-ray intensity (cps) for each element and using the above determined sensitivity factor for each element plus various equations to account for X-ray absorption and enhancement effects, the concentration of all elements present can be estimated. The exact equations used will differ for each manufacturer.

10.4.2.1 Follow the manufacturer's fundamental parameters set-up recommendations. The stoichiometric set-up of the fundamental parameters method for the analysis of the LHW mixed with alumina should allow for the manual input of a fixed 75 % $Al_2O_3$ concentration and the use of carbon as a balance estimate of the solvent/aqueous phase with the elements of interest determined directly according to the principles of 10.4.2.

10.4.3 Two control samples are needed for monitoring instrument stability. One control sample is a blank preparation using PEG or the low concentration drift correction monitor used in 10.4.1.1. The other sample is a stable mixture containing a suitable range and number of elements (for example, S, V, Zn, Pb, and Ba) at concentrations near the middle of the calibration ranges. A mixture of leftover samples/standards, spiked with element concentrations as needed and carefully mixed may be used.

10.4.4 Restandardization should be carried out whenever quality control results defined in Section 14 are outside data quality objectives as determined by the user. Method A: The initial linear regressions are performed only once as per 10.4.1. A day zero measurement of the drift correction monitors, 10.4.1.1 during the set-up of the initial regression allows for subsequent re-calibration to be performed using the two standards defined in 10.4.1.1, via a restandardization procedure in order to check the values of the slope and intercept for each regressed element. NOTE: Restandardization using drift correction monitors is often part of instrumental software. Follow the manufacturer's recommendations for the set-up of restandardization using two standards. Method B: Follow the manufacturer's recommendations for the set-up of initial element sensitivities and the appropriate fundamental parameters methods using the principles outlined in 10.4.2.

## 11. Procedure

11.1 Thoroughly mix the LHW sample before withdrawing a portion with a pipet, or equivalent device for preparation. See the Sample section.

11.2 Weigh 15.00 g $Al_2O_3$ ± 0.05 g directly into the HDPE bottle.

11.3 Weigh a 5.00 g ± 0.05 g portion of the sample directly into the blending vial/vessel containing the $Al_2O_3$ from 11.1.

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[6] Pressed aluminum powder doped with the elements of interest has been found to be satisfactory. Preparation guidelines can be found in: Forte, M., "Fabrication and Use of Permanent Monitors and Standards," *X-ray Spectrometry*, 1983, Vol 3, pp. 115.

11.4 Add two mixing balls to the HDPE bottle from 11.3 and seal.

11.5 Shake vigorously for approximately 30 s. Tapping the bottle on a hard surface will aid in proper mixing. The user may feel a mild exothermic reaction.

11.6 Empty the $Al_2O_3$/sample mixture from the bottle to an XRF sample cup as described in 10.3.

11.7 Place the sample cup into the spectrometer sample holder and initiate data acquisition. If re-analysis of the sample is required, the user should obtain a new portion from the HDPE bottle following the instructions in 11.6.

11.8 After analysis is completed by the instrument, process analytical results according to Section 12.

## 12. Calculation

12.1 Concentration units normally reported are mg/kg. A number of corrections may be required for samples.

12.1.1 *Conversion of Analysis Units to Mass/Volume*—A density will be required. This is determined by ensuring the sample is well mixed. Record the mass in grams of a 1 $cm^3$ volume to calculate the density. The conversion calculations are as follows:

$$\text{mass/vol}\ (\text{mg/l}) = \text{Density}\ (\text{g/cm}^3) \times \text{reported concentration}\ (\text{mg/kg}) \quad (1)$$

12.1.2 *Re-calculation of Concentration if Sample Dilution has been Used*—For samples that have been diluted, that is further than the 15 g alumina: 5 g sample ratio according to 10.4.1. The following calculations are required to give correct sample concentrations:

$$\text{Corrected sample concentration} = 5 \times \frac{\text{reported concentration}}{\text{Actual mass of sample used, g}} \quad (2)$$

## 13. Quality Control

13.1 Each laboratory using this test method should operate a formal quality control program.

13.2 Before using this test method, it is strongly recommended that the user fully investigate and implement any specific regulatory quality control requirements.

13.3 A measurement of the two control samples defined in 10.4.3 at a frequency of not less than one per day should be carried out.

13.4 Results of quality control sample measurements made in 13.3 must be evaluated.

13.4.1 Should values fall outside data quality objectives when using Method A in 10.4.1 then the regression must be restandardized following the procedure in 10.4.4, Method A.

13.4.2 Should values fall outside data quality objectives when using Method B in 10.4.2 then the instrument must be restandardized following the procedure in 10.4.4, Method B.

## 14. Precision and Bias

14.1 *Precision*—No statement is made about the precision at this time. Appendix X1 provides limited precision information to the user.

14.2 *Bias*—No statement is made about bias at this time. This information will be acquired in the future.

14.3 Tables X1.1-X1.6 in Appendix X1 contain analytical/quality control results for most of the elements in 1.2 following this test method on typical LHW, check standard, MS/MSD and method blank. Lower limits of detection are also given.

## 15. Keywords

15.1 EDXRF; halides; liquid hazardous waste; spectrometry; spectroscopy; toxic metals; trace elements; waste-derived fuel; XRF

# APPENDIX

## (Nonmandatory Information)

## X1. BLANK, ACCURACY AND MATRIX SPIKE RESULTS

TABLE X1.1 Blank

NOTE 1—A sample containing only PEG was used to check for any bias in the calibrations at the zero concentration level.

| Element | V | Cr | Fe | Ni | Cu | Zn | As | Se | Cd | Sn | Sb | I | Hg | Tl | Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| mg $kg^1$ | 1.2 | 0.0 | 17.3 | 0.0 | 0.0 | 1.6 | 1.9 | 2.6 | 0.0 | 1.3 | 0.0 | 3.9 | 1.9 | 2.2 | 0.0 |
| mg $kg^1$ error | 0.5 | 0.7 | 0.2 | 3.6 | 0.04 | 0.4 | 7.6 | 2.5 | 2.5 | 0.4 | 0.9 | 10 | 2.9 | 3.9 | 1.0 |

X1.1 Errors shown in Tables X1.1-X1.3 are taken from the results output of the instrument and nominally ± 2 sigma.[7] These represent the total error attributed to spectrum processing and counting statistics. See Stratham[12] for details of the calculations used in the error calculations.

X1.2 Table X1.4 gives the lower limit of detection for each analyte. This is based on the following equation:

$$LLD = \frac{3x\sqrt{bg}}{\text{net peak}} \times \frac{1}{\sqrt{T}} \times \text{concentration} \tag{X1.1}$$

where:

bg = background intensity under analyte peak cps,
net peak = fitted peak intensity of analyte cps,
$T$ = count time, s, and
conc. = concentration of analyte.

X1.3 *Matrix/Matrix Spike Recoveries:*

X1.3.1 A series of experiments was conducted to test the performance of the alumina method. Three types of actual waste solutions were selected from routine test samples taken at an incineration plant. These samples were:

X1.3.1.1 Clear solutions,

X1.3.1.2 Turbid solutions, that is, contained significant solids not in suspension, and

X1.3.1.3 Biphasal solutions, that is, contained two distinctly immiscible liquid phases.

X1.3.2 For each matrix type a sub-sample was spiked with a known concentration of analyte. The spiked sample was prepared using the alumina technique and measured. Using Eq X1.2, a recovery figure for each analyte in each matrix type was determined. The results, referred to as a matrix spike/matrix spike duplicate (MS/MSD), are shown below.

$$\%\ \text{MS/MSD recovery} - ((C2\ (D1 \times C1))/C3) \times 100 \tag{X1.2}$$

where:

$D1$ = dilution factor due to matrix spike addition = 1 − (mass of spike)/(total mass of sample + spike),
$C1$ = calculation concentration of matrix without spike,
$C2$ = calculation concentration of matrix + spike, and
$C3$ = given concentration of matrix spike.

X1.4 *Precision of measurement:*

X1.4.1 A number of repeat measurements were made on a waste sample.

X1.4.1.1 A single measurement from each of ten repeat sample preparations was made for Cl content. A repeat of this process was made on newly prepared samples 48 h later.

X1.4.1.2 A single measurement from each of ten repeat sample preparations was made for a waste sample spiked with 54.3 mg $kg^{-1}$ Cd.

X1.4.2 A single Cl analysis of the same waste sample used in X1.4.1.1 was measured by a second laboratory using the alumina sample preparation technique and is shown in the last column of Table X1.6.

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[7] Stratham, P., *Analytical Chemistry*, 1977, Vol 49, pp. 2149.

### TABLE X1.2 Accuracy

NOTE 1—Standards were run against the calibrations to assess accuracy and to check for bias in the calibrations due to either matrix or spectral effects. The % accuracy figure shows the match of given versus calculated concentration for each element.

| Element | P<br>% m/m | S<br>% m/m | Cl<br>% m/m | Se<br>mg/kg | As<br>mg/kg | Br<br>mg/kg | Cd<br>mg/kg | Sn<br>mg/kg | Sb<br>mg/kg | I<br>mg/kg |
|---|---|---|---|---|---|---|---|---|---|---|
| Calculation concentration | 0.96 | 2.09 | 0.33 | 93 | 393 | 4779 | 179 | 47 | 201 | 587 |
| Error | 0.02 | 0.02 | 0.005 | 4 | 10 | 15 | 4 | 3 | 7 | 13 |
| Given concentration | 1.024 | 2.095 | 0.598 | 100 | 400 | 4838 | 200 | 50 | 200 | 600 |
| % accuracy | 96 | 100 | 89 | 93 | 98 | 99 | 98 | 94 | 100 | 98 |

| Element | Hg | Tl | Pb | V | Cr | Fe | Ni | Cu | Zn |
|---|---|---|---|---|---|---|---|---|---|
| Calculation concentration, mg kg$^{1}$ | 99 | 96 | 94 | 109 | 6 | 589 | 53 | 55 | 95 |
| Error | 9 | 13 | 7 | 7 | 3 | 0.2 | 8 | 3 | 5 |
| Given concentration | 100 | 100 | 100 | 100 | 10 | 600 | 50 | 50 | 100 |
| % accuracy | 99 | 96 | 94 | 109 | 60 | 98 | 106 | 110 | 93 |

### TABLE X1.3 Light Elements and Halides

| Single Phase Solvent | P | S | Cl | Br | I |
|---|---|---|---|---|---|
| MS/MSD recovery (%) | 111.3 | 120 | 110 | 95 | 100 |
| error, % m/m | 0.02 | 0.01 | 0.007 | 0.0007 | 0.0032 |
| spike concentration, % m/m | 0.9156 | 0.926 | 1.279 | 0.0599 | 0.856 |
| Turbid Waste | | | | | |
| MS/MSD recovery, % | 98 | 91 | 89 | 104 | 100 |
| error, % m/m | 0.01 | 0.009 | 0.005 | 0.0006 | 0.0034 |
| spike concentration, % m/m | 0.849 | 0.805 | 0.858 | 0.042 | 0.845 |
| Biphasal Waste | | | | | |
| MS/MSD recovery, % | | | 98 | | |
| error, % m/m | | | 0.008 | | |
| spike concentration, % m/m | | | 1.654 | | |

### TABLE X1.4 3 Sigma Lower Limits of Detection

| Element | LLD mg/kg | Element | LLD mg/kg | Element | LLD mg/kg |
|---|---|---|---|---|---|
| P | n/a[A] | Cu | 17 | Se | 6 |
| S | n/a[A] | Zn | 11 | I | 7 |
| Cl | n/a[A] | As | 5 | Hg | 7 |
| V | 8 | Se | 5 | Tl | 4 |
| Cr | 8 | Br | 5 | Pb | 4 |
| Fe | 14 | Cd | 3 | | |
| Ni | 16 | Sn | 5 | | |

[A] n/a = these elements were calibrated at concentrations significantly higher than their respective detection limits.

### TABLE X1.5 Toxic Elements

| Biphasal Waste | Cd | Hg | Tl | Se | As | Pb | V | Cr | Fe | Ni | Cu | Zn | Sb | Sn |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ms/MSD recovery,% | 108 | 103 | 98 | 109 | 109 | 99 | 96 | 111 | 84 | 86 | 73 | 105 | 84 | 103 |
| error mg kg$^{-1}$ | 3 | 9 | 10 | 4 | 9 | 7 | 6 | 5 | 1 | 9 | 4 | 5 | 6 | 3 |
| spike concentration mg kg$^{-1}$ | 47.1 | 101.8 | 102.5 | 104.6 | 109.7 | 183.7 | 97.4 | 98.3 | 97.1 | 98.2 | 99.3 | 102.0 | 33.3 | 53.5 |
| Turbid Waste | Cd | Hg | Tl | Se | As | Pb | V | Cr | Fe | Ni | Cu | Zu | Sb | Sn |
| MS/MSD recovery,% | 92 | 114 | 43 | 90 | 121 | 108 | 102 | 110 | 104 | 118 | 94 | 105 | 108 | 103 |
| error mg kg$^{-1}$ | 3 | 11 | 13 | 5 | 10 | 10 | 6 | 6 | 2 | 11 | 5 | 5 | 6 | 3 |
| spike concentration mg kg$^{-1}$ | 44.8 | 96.8 | 47.7 | 93.6 | 94.8 | 177 | 103.4 | 102 | 106.4 | 104.5 | 103.5 | 106.6 | 53.2 | 53.2 |
| Single Phase Solvent | Cd | Hg | Tl | Se | As | Pb | V | Cr | Fe | Ni | Cu | Zn | Sb | Sn |
| Ms/MSD recovery,% | 100 | 98 | 77 | 108 | 108 | error[A] | 110 | 114 | 109 | 104 | 105 | 90 | 112 | 95 |
| error mg kg$^{-1}$ | 3 | 9 | 10 | 4 | 9 | | 6 | 6 | 2 | 10 | 5 | 5 | 6 | 3 |
| spike concentration mg kg$^{-1}$ | 45.5 | 97.0 | 96 | 103.8 | 105 | | 116.8 | 109.5 | 100 | 105.7 | 109.9 | 105.3 | 54.6 | 55.5 |

[A] Spike not added.

**TABLE X1.6 Precision**

| Element | 10 repeats—Day 1 | | | 10 repeats—Day 3 | | | second lab |
|---|---|---|---|---|---|---|---|
| | mean | S.D.[A] | %R.S.D.[B] | mean | S.D.[A] | %R.S.D.[B] | single result |
| Cl % m/m | 1.45 | 0.027 | 1.9 | 1.43 | 0.037 | 2.6 | 1.38 |
| Cd mg/kg | 56 | 1.3 | 2.2 | | | | |

[A] S.D. = 1 sigma standard deviation.

[B] % R.S.D. = % relative standard deviation.

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