

## METHOD 3040A

### DISSOLUTION PROCEDURE FOR OILS, GREASES, OR WAXES

#### 1.0 SCOPE AND APPLICATION

1.1 This method is used for the preparation of samples containing oils, greases, or waxes for analysis by flame atomic absorption spectrometry (FLAA) or inductively coupled plasma atomic emission spectrometry (ICP-AES) for the following metals:

|           |           |
|-----------|-----------|
| Antimony  | Copper    |
| Arsenic   | Iron      |
| Barium    | Lead      |
| Beryllium | Manganese |
| Cadmium   | Nickel    |
| Chromium  | Vanadium  |

1.2 This method is a solvent dissolution procedure, not a digestion procedure. This procedure can be very useful in the analysis of crude oil, but with spent or used oil high in particulate material it is less effective; most particulate material is not dissolved, and therefore the analysis is not a "total" metal determination. Because the highest percentage of metals is expected to be contained in the particulate material, oil analysis using Method 3040A will not provide an adequate estimate of the total metals concentration.

Caution: Overheating of oils and solvents can result in an explosion or fire, caution should be taken.

1.3 This method is applicable for the dissolution of multi-phasic aqueous wastes containing either oils, greases, or waxes. If a waste is multi-phasic it can be determined by using Method 3040A in combination with one of the other sample preparation methods.

Caution: The analysis of solvents in an ICP-AES should only be conducted after consultation with the manufacturer.

1.4 This method is suitable for conducting analyses in support of TCLP determinations if the percent solids, as conducted according to the procedures specified in the Method 1311, are below 0.5%.

#### 2.0 SUMMARY OF METHOD

2.1 A representative sample is dissolved in an appropriate solvent (e.g., xylene, kerosene, or methyl isobutyl ketone). Organometallic standards are prepared using the same solvent, and the samples and standards are analyzed by FLAA or ICP-AES.

#### 3.0 INTERFERENCES

3.1 Diluted samples and diluted organometallic standards are often unstable. Once standards and samples are diluted, they should be analyzed as soon as possible.

3.2 Solvent blanks should be used to rinse nebulizers thoroughly following aspiration of high concentration standards or samples.

3.3 Viscosity differences can result in different rates of sample introduction; therefore, all analyses shall be performed by the method of standard addition or internal standardization (only for ICP-AES). Peristaltic pumps often prove useful when analysis is performed by ICP-AES. In addition, a mass-flow controller may also alleviate some viscosity problems.

#### 4.0 APPARATUS AND MATERIALS

4.1 Volumetric glassware or equivalent.

4.2 Analytical balance, 300 g capacity, minimum  $\pm 0.01\text{g}$ .

4.3 Atomic absorption spectrometer: With an auxiliary oxidant control and a mechanism for background correction.

4.4 Inductively coupled plasma emission spectrometer system: With a mechanism for background correction and interelement interference correction. A peristaltic pump is optional.

#### 5.0 REAGENTS

5.1 Methyl isobutyl ketone (MIBK).

5.2 Xylene.

5.3 Kerosene.

5.4 Organometallic standards - scandium and yttrium may be used as internal standards for most samples. Standards traceable to NIST Standard No. 1085, for wear metals in oil, may be used. (Two possible sources are Conostan Division, Conoco Speciality Products, Inc., P.O. Box 1267, Ponca City, OK 74601, and the U.S. Department of Commerce, National Institutes of Standards and Technology, Washington, DC 20234).

5.5 Base Oil.

#### 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

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

6.2 Samples shall be stored in an undiluted state at room temperature.

6.3 Samples should be processed and analyzed as soon as possible.

#### 7.0 PROCEDURE

7.1 Weigh out a 2 gram representative sample of the waste or extract. Separate and weigh the phases if more than one phase is present.

7.2 Internal standards should be added to all samples at the mid-point of the concentration range, prior to dilution (the internal standard concentration may vary depending on your instrument or instrument configuration). Weigh an aliquot of the organic phase and dilute the aliquot in the appropriate solvent. Warming facilitates the subsampling of crude-type oils and greases and wax-type wastes. Xylene or kerosene are usually the preferred solvent for longer-chain hydrocarbons and for most analyses performed by ICP-AES. The longer-chain hydrocarbons usually require a minimum of a 1:10 (W/W) dilution, and lighter oils may require only a 1:5 (W/W) dilution if low detection limits are required.

7.3 Prepare a series of standards using the base oil and diluting by the same factor used for the samples. Add the internal standard before diluting. The concentration of the internal standard should be in the middle of the concentration range (The internal standard concentration may vary depending on your instrument or instrument configuration).

7.4 If the sample contains particulates, the result may be variable depending on whether the particles are aspirated into the instrument. Samples may be centrifuged after dilution to remove particulates from the solution prior to analysis.

7.5 All metals must be analyzed by the method of standard additions if an internal standard is not used. Because the method of standard additions can account only for multiplicative interferences (matrix or physical interferences), the analytical program must account for additive interference (nonspecific absorption and scattering in FLAA and nonspecific emission and interelement interference in ICP-AES) by employing background correction when using the ICP-AES.

7.6 Sample preparation for the method of standard additions can be performed on a weight or volume basis. Sample aliquots of viscous wastes should be weighed. Weigh identical amounts of the sample into three wide-mouth vials. Dilute the first vial such that the final concentration falls on the lower end of the linear portion of the calibration curve and significantly above the detection limit. Add sufficient standard to the second aliquot to increase the sample concentration by approximately 50%. Adjust the third sample concentration so that it is approximately twice that of the first. The second and third aliquots are then diluted to the same final volume as the first aliquot. Because of the wide variability in waste samples, and the problems encountered with analyzing them, the analyst's best judgement must be used to permit efficient use of this method.

7.7 Set up and calibrate the analytical instrumentation according to the manufacturer's directions for nonaqueous samples.

7.8 Report data as the weighted average for all sample phases.

$$[(P_1 \times C_1) + (P_2 \times C_2)]/P_T = C_F$$

$P_1$  = weight of the first phase (kg)

$P_2$  = weight of the second phase (kg)

$C_1$  = concentration of the first phase (mg/kg)

$C_2$  = concentration of the second phase (mg/kg)

$P_T$  = weight of both phases (kg)

$C_F$  = final concentration of waste (mg/kg)

7.9 For nonaqueous TCLP extracts and other samples where results are reported in "mg/L" units, the conversion from "mg/kg" to "mg/L" units can be done by determining the density of the liquid and converting mathematically.

## 8.0 QUALITY CONTROL

8.1 Preparation blanks (e.g., Conostan base oil or mineral oil plus reagents) should be carried through the complete sample-preparation and analytical process on a routine basis. These blanks will be useful in detecting and determining the magnitude of any sample contamination. Refer to Chapter One.

8.2 Replicate samples should be processed on a routine basis. Replicate samples will be used to determine precision. Refer to Chapter One.

8.3 Samples and standards should be diluted as closely as possible to the time of analysis.

8.4 All analyses must be performed by the method of standard additions if an internal standard is not used. See Method 7000, Section 8.7, for further information.

8.5 Data must be corrected for background absorption and emission and interelement interferences.

## 9.0 METHOD PERFORMANCE

9.1 Refer to Tables 1 and 2 for a single lab study.

## 10.0 REFERENCES

1. Used Oil Characterization Sampling and Analysis Program. Draft Final Report. February 15, 1991.

TABLE 1  
METHOD PERFORMANCE DATA  
FROM SINGLE LAB STUDY: ASSESSMENT OF ACCURACY

| ANALYTE  | # ANALYSES | MEAN PERCENT SPIKE RECOVERY | STANDARD DEVIATION | OUTLIERS <sup>a</sup> |
|----------|------------|-----------------------------|--------------------|-----------------------|
| Arsenic  | 2          | 76                          | 39.6               | 1                     |
| Cadmium  | 20         | 100.6                       | 16.8               | 4                     |
| Chromium | 20         | 107.2                       | 13.1               | 2                     |
| Lead     | 20         | 97.4                        | 20.2               | 2                     |
| Barium   | 20         | 97.0                        | 30.7               | 4                     |

TABLE 2  
METHOD PERFORMANCE DATA  
FROM SINGLE LAB STUDY: ASSESSMENT OF PRECISION

| ANALYTE  | # REPLICATE PAIRS | RELATIVE % DIFFERENCE | STANDARD DEVIATION | OUTLIERS <sup>b</sup> |
|----------|-------------------|-----------------------|--------------------|-----------------------|
| Arsenic  | 1                 | 73                    | ---                | 1                     |
| Cadmium  | 10                | 1.8                   | 1.9                | 0                     |
| Chromium | 10                | 2.8                   | 1.9                | 0                     |
| Lead     | 10                | 4.1                   | 6.6                | 1                     |
| Barium   | 10                | 5.9                   | 12.1               | 1                     |

<sup>a</sup> Percent recovery outside of the laboratory's 80 - 120 % acceptance criteria. Outliers included in statistical analysis.

<sup>b</sup> RPD outside of the laboratory's 20 % acceptance criteria. Outliers included in statistical analysis.

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