

## METHOD 1330

EXTRACTION PROCEDURE FOR OILY WASTE1.0 Scope and Application

- 1.1 Method 1330 is used to determine the mobile metal concentration (MMC) in oily wastes.
- 1.2 Method 1330 is applicable to API separator sludges, rag oils, slop oil emulsions and other oil wastes derived from petroleum refining.

2.0 Summary of Method

- 2.1 The sample is separated into solid and liquid components by filtration.
- 2.2 The solid phase is placed in a Soxhlet extractor, charged with tetrahydrofuran, and extracted. The THF is removed and the extractor is then charged with toluene and the sample is re-extracted.
- 2.3 The EP (Method 1310) is run on the dry solid residue.
- 2.4 The original liquid, combined extracts, and EP leachate are analyzed for the EP metals.

3.0 Interferences

- 3.1 Matrix interferences will be coextracted from the sample. The extent of these interferences will vary considerably from waste to waste depending on the nature and diversity of the particular refinery waste being analyzed.

4.0 Apparatus and Materials

- 4.1 Soxhlet extraction apparatus
- 4.2 Vacuum pump or other source of vacuum
- 4.3 Buchner funnel 12
- 4.4 Electric heating mantle
- 4.5 Paper extraction thimble
- 4.6 Filter paper
- 4.7 Muslin cloth disks
- 4.8 Evaporative flask 250 ml
- 4.9 Analytical Balance capable of weighing to  $\pm$  0.5 mg.

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## 5.0 Reagents

5.1 Tetrahydrofuran - ACS Reagent grade

5.2 Toluene

## 6.0 Sampling

6.1 Samples must be collected in glass containers having a total volume of at least 150 ml. No solid material should interfere with sealing the sample container.

6.2 Sampling devices should be wiped clean with paper towels or absorbent cloth and rinsed with a small amount of hexane followed by acetone rinse and dried between samples. Alternatively, samples can be taken with disposable sampling devices in beakers.

## 7.0 Procedure

7.1 Separate the sample (minimum 100 gm) into its solid and liquid components, using the filtration steps 7.1-7.6 in Method 1310.

7.2 Determine the quantity of liquid (ml) and the concentration of the metals of concern in the liquid phase (mg/l) using Method 3030, 3040, or 3050.

7.3 Place the solid phase into a Soxhlet extractor, charge the concentration flask with 300 ml tetrahydrofuran, and extract for three hours.

7.4 Remove the flask containing tetrahydrofuran and replace it with one containing 300 ml toluene.

7.5 Extract the solid for a second time, for three hours, with the toluene.

7.6 Combine the tetrahydrofuran and toluene extracts.

7.7 Determine the quantity of liquid (ml) and the concentration of the metals of concern in the combined extracts (mg/l) using either Method 3030 or 3040.

7.8 Take the solid material remaining in the Soxhlet thimble and dry it at 100°C for 30 minutes.

7.9 Run the EP (Method 1310) on the dried solid.

7.10 Calculate the mobil metal concentration (MMC in mg/l) using the following formula:

$$MMC = 1000 ([Q_1 + Q_2 + Q_3] / [L_1 + L_2])$$

$Q_1$  = Amount of metal in initial liquid phase of sample (Amt. of Liquid X Conc. of Metal) (mg)

$Q_2$  = Amount of metal in combined organic extracts of sample (Amt. of

$Q_3$  = Amount of metal in EP Extract of solid (Amt. of extract X Conc. of Metal) (mg)

$L_1$  = Amount of Initial Liquid (ml)

$L_2$  = Amount of liquid in EP = weight of dried solid from step 9 x 20 (ml)

## 8.0 Quality Control

8.1 Standard quality assurance practices should be used with this method. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity accuracy of the analysis.