

METHOD 3580A

WASTE DILUTION

1.0 SCOPE AND APPLICATION

1.1 This method describes a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. It is designed for wastes that may contain organic chemicals at a concentration greater than 20,000 mg/kg and that are soluble in the dilution solvent.

1.2 It is recommended that an aliquot of the diluted sample be cleaned up. See this chapter, Organic Analytes, Section 4.2.2 (Cleanup).

2.0 SUMMARY OF METHOD

2.1 One gram of sample is weighed into a capped tube, and the sample is diluted to 10.0 mL with an appropriate solvent.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Glass scintillation vials: At least 20 mL, with Teflon or aluminum foil lined screw-cap, or equivalent.

4.2 Spatula: Stainless steel or Teflon.

4.3 Balance: Capable of weighing 100 g to the nearest 0.01 g.

4.4 Vials and caps: 2 mL for GC autosampler.

4.5 Disposable pipets: Pasteur.

4.6 Test tube rack.

4.7 Pyrex glass wool.

4.8 Volumetric flasks, Class A: 10 mL (optional).

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous), Na_2SO_4 . Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride,

a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.2 Methylene chloride, CH_2Cl_2 - Pesticide quality or equivalent.

5.3 Hexane, C_6H_{14} - Pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Samples consisting of multiphases must be prepared by the phase separation method (Chapter Two) before extraction.

7.2 The sample dilution may be performed in a 10 mL volumetric flask. If disposable glassware is preferred, the 20 mL scintillation vial may be calibrated for use. Pipet 10.0 mL of extraction solvent into the scintillation vial and mark the bottom of the meniscus. Discard this solvent.

7.3 Transfer approximately 1 g of each phase of the sample to separate 20 mL vials or 10 mL volumetric flasks (record weight to the nearest 0.1 g). Wipe the mouth of the vial with a tissue to remove any sample material. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

7.4 Add 2.0 mL surrogate spiking solution to all samples and blanks. For the sample in each analytical batch selected for spiking, add 2.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 200 ng/ μL of each base/neutral analyte and 400 ng/ μL of each acid analyte in the extract to be analyzed (assuming a 1 μL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column. See Method 3500 and the determinative method to be used for details on the surrogate standard and matrix spiking solutions.

7.5 Immediately dilute to 10 mL with the appropriate solvent. For compounds to be analyzed by GC/ECD, e.g., organochlorine pesticides and PCBs, the dilution solvent should be hexane. For base/neutral and acid semivolatile priority pollutants, use methylene chloride. If the dilution is to be cleaned up by gel permeation chromatography (Method 3640), use methylene chloride as the dilution solvent for all compounds.

7.6 Add 2.0 g of anhydrous sodium sulfate to the sample.

7.7 Cap and shake the sample for 2 min.

7.8 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect 5 mL of the extract in a tube or vial.

7.9 The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.0 QUALITY CONTROL

8.1 Any reagent blanks and matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

10.1 None applicable.

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