METHOD 3585

WASTE DILUTION FOR VOLATILE ORGANICS

1.0 SCOPE AND APPLICATION

1.1 This method describes a solvent dilution of a non-aqueous waste sample prior to direct injection analysis. It is designed for use in conjunction with GC or GC/MS analysis of wastes that may contain organic chemicals at a concentration greater than 1 mg/kg and that are soluble in the dilution solvent. Method 3585 has adequate sensitivity to determine the regulatory concentrations of the Toxicity Characteristic (TC) Rule.

1.2 This method may be used with *n*-hexadecane for direct injection of target volatiles in oily matrices.

1.3 Use of a 1 - 2 µL injection of a 1:1 dilution can be used to provide detection limits of 0.5 ppm for volatile target analytes with a sensitive GC/MS.

1.4 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Highly contaminated or highly complex samples may be diluted prior to analysis for volatiles using direct injection.

2.2 One gram of sample is weighed into a capped tube or volumetric flask. The sample is diluted to 2.0 - 10.0 mL with *n*-hexadecane or other appropriate solvent.

2.3 Diluted samples are injected into the GC or GC/MS for analysis.

3.0 INTERFERENCES

3.1 Use of a direct injection procedure will result in considerable contamination of injection ports, injection port liners, GC columns, and detectors. A Pyrex® wool plug should be placed into the injection port liner and the liner should be changed after every 12 hours of sample analysis.

3.2 The solvent used for waste dilution may contain volatile contaminants that could interfere with analyses.

3.2.1 *n*-Hexadecane elutes after target volatiles. However, volatile impurities in n-hexadecane may interfere with analyses.

3.2.2 Each lot of *n*-hexadecane (or any other solvent used for dilution) must be analyzed for impurities prior to use.

3.3 The presence of methanol and other oxygenated solvents in samples may lead to baseline humps that interfere with qualitative and quantitative analysis of early eluting target analytes when direct injection is employed.

4.0 APPARATUS AND MATERIALS

4.1 Glass scintillation vials - At least 20-mL, with polytetrafluoroethylene (PTFE)- or aluminum foil-lined screw-cap, or equivalent.

4.2 Spatula - Stainless steel or PTFE.

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 - 2- or 10-mL (optional).

4.9 Direct injection liner (HP catalogue #18740-80200 or equivalent) - Modify with a 1-cm plug of Pyrex® wool placed approximately 50-60 mm down the length of the injection port (towards the oven). A 0.53 mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications. Figure 1 is an example of the placement of the glass wool plug in the liner.

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Figure 1 Modified Injector

5.0 REAGENTS

n-Hexadecane, *n*-$C_{16}H_{34}$ - Pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

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

7.0 PROCEDURE

7.1 Samples consisting of multiple phases must be prepared by the phase separation method (Chapter Two) before extraction. The oil phase is prepared as outlined below. An aqueous phase is prepared and analyzed following the guidance in Method 5030.

7.2 The sample dilution may be performed in a 2- or 10-mL volumetric flask. If disposable glassware is preferred, the 10-dram vial may be calibrated for use. Pipet 2.0 mL of methanol into the vial and mark the bottom of the meniscus. Discard this solvent. Dry the vial.

7.3 Transfer approximately 1 g of the oil phase of the sample to a vial or volumetric flask (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 Immediately dilute to volume with *n*-hexadecane or other appropriate solvent. The choice of solvents is dependent on the nature of the target analytes. *n*-Hexadecane is late eluting and, therefore, presents no solvent interference for the majority of volatile organics. An early eluting solvent, e.g., pentane or hexane, may be chosen if the target analytes are mid to late eluting.

7.5 Add surrogate spiking solution, if required, for the analytical method to be employed.

7.6 Cap and shake the sample for 2 minutes.

7.7 The extract is ready for analysis by GC Methods 8015 or 8021, or by GC/MS Method 8260.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One, Method 8000, and the analytical method to be employed, for specific quality control procedures.

8.2 Each time samples are prepared and analyzed, and when there is a change in reagents, a reagent blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. Any reagent blanks, matrix spike samples, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.3 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling technique. Each analysis batch of 20 or fewer samples must contain: a reagent blank; either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis; and a laboratory control sample, unless the determinative method provides other guidance.

8.4 Surrogates should be added to all samples when specified in the appropriate determinative method.

9.0 METHOD PERFORMANCE

Refer to the determinative methods for performance data.

10.0 REFERENCES

1. Marsden, P.J., Colby, B.N., and Helms, C.L., "Determining TCLP Volatiles at Regulatory Levels in Waste Oil", Proceedings of the Eighth Annual Waste Testing and Quality Assurance Symposium, July, 1992.

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