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Wednesday, August 3, 1994

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METHOD 3611A

ALUMINA COLUMN CLEANUP AND SEPARATION OF PETROLEUM WASTES

1.0 SCOPE AND APPLICATION

1.1 Method 3611 was formerly Method 3570 in the Second Edition of this manual.

1.2 Specific application: This method includes guidance for separation of petroleum wastes into aliphatic, aromatic, and polar fractions.

2.0 SUMMARY OF METHOD

2.1 The column is packed with the required amount of adsorbent, topped with a water adsorbent, and then loaded with the sample to be analyzed. Elution of the analytes is effected with a suitable solvent(s), leaving the interfering compounds on the column. The eluate is then concentrated (if necessary).

3.0 INTERFERENCES

3.1 A reagent blank should be performed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method may be necessary for reagent purification.

3.3 Caution must be taken to prevent overloading of the chromatographic column. As the column loading for any of these types of wastes approaches 0.300 g of extractable organics, separation recoveries will suffer. If overloading is suspected, an aliquot of the base-neutral extract prior to cleanup may be weighed and then evaporated to dryness. A gravimetric determination on the aliquot will indicate the weight of extractable organics in the sample.

3.4 Mixtures of petroleum wastes containing predominantly polar solvents, i.e., chlorinated solvents or oxygenated solvents, are not appropriate for this method.

4.0 APPARATUS AND MATERIALS

4.1 Chromatography column: 300 mm x 10 mm ID, with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

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4.2 Beakers: 500 mL.

4.3 Reagent bottle: 500 mL.

4.4 Muffle furnace.

4.5 Kuderna-Danish (K-D) apparatus:

4.5.1 Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

4.5.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

4.5.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

4.5.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

4.5.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

4.6 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.7 Water bath: Heated with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.8 Erlenmeyer flasks: 50 and 250 mL.

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 Eluting solvents:

5.2.1 Methanol, CH_3OH - Pesticide quality or equivalent.

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

5.2.3 Methylene chloride, CH_2Cl_2 - Pesticide quality or equivalent.

5.3 Alumina: Neutral 80-325 MCB chromatographic grade or equivalent. Dry alumina overnight at 130°C prior to use.

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 It is suggested that Method 3650, Acid-Base Partition Cleanup, be performed on the sample extract prior to alumina cleanup.

7.2 Place approximately 10 g of alumina into a chromatographic column, tap to settle the alumina, and add 1 cm of anhydrous sodium sulfate to the top.

7.3 Pre-elute the column with 50 mL of hexane. Discard the eluate and, just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1 mL sample extract onto the column using an additional 1 mL of hexane to complete the transfer. To avoid overloading the column, it is suggested that no more than 0.300 g of extractable organics be placed on the column (see Section 3.3).

7.4 Just prior to exposure of the sodium sulfate to the air, elute the column with a total of 15 mL of hexane. If the extract is in 1 mL of hexane, and if 1 mL of hexane was used as a rinse, then 13 mL of additional hexane should be used. Collect the effluent in a 50 mL flask and label this fraction "base/neutral aliphatics." Adjust the flow rate to 2 mL/min.

7.5 Elute the column with 100 mL of methylene chloride and collect the effluent in a 250 mL flask. Label this fraction "base/neutral aromatics."

7.6 Elute the column with 100 mL of methanol and collect the effluent in a 250 mL flask. Label this fraction "base/neutral polars."

7.7 Concentrate the extracts (if necessary) by the standard K-D technique to the volume (1-10 mL) required in the appropriate determinative method (Chapter Four). Analyze the fractions containing the analytes of interest.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3600 for cleanup procedures.

8.2 The analyst should demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples.

8.3 For sample extracts that are cleaned up using this method, the associated quality control samples must also be processed through this cleanup method.

9.0 METHOD PERFORMANCE

9.1 The precision and accuracy of the method will depend upon the overall performance of the sample preparation and analysis.

9.2 Rag oil is an emulsion consisting of crude oil, water, and soil particles. It has a density greater than crude oil and less than water. This material forms a layer between the crude oil and water when the crude oil is allowed to gravity separate at the refinery. A rag oil sample was analyzed by a number of laboratories according to the procedure outlined in this method. The results of these analyses by GC/MS for selected components in the rag oil are presented in Table 1. Reconstructed ion chromatograms from the GC/MS analyses are included as Figures 1 and 2.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

Figure 1

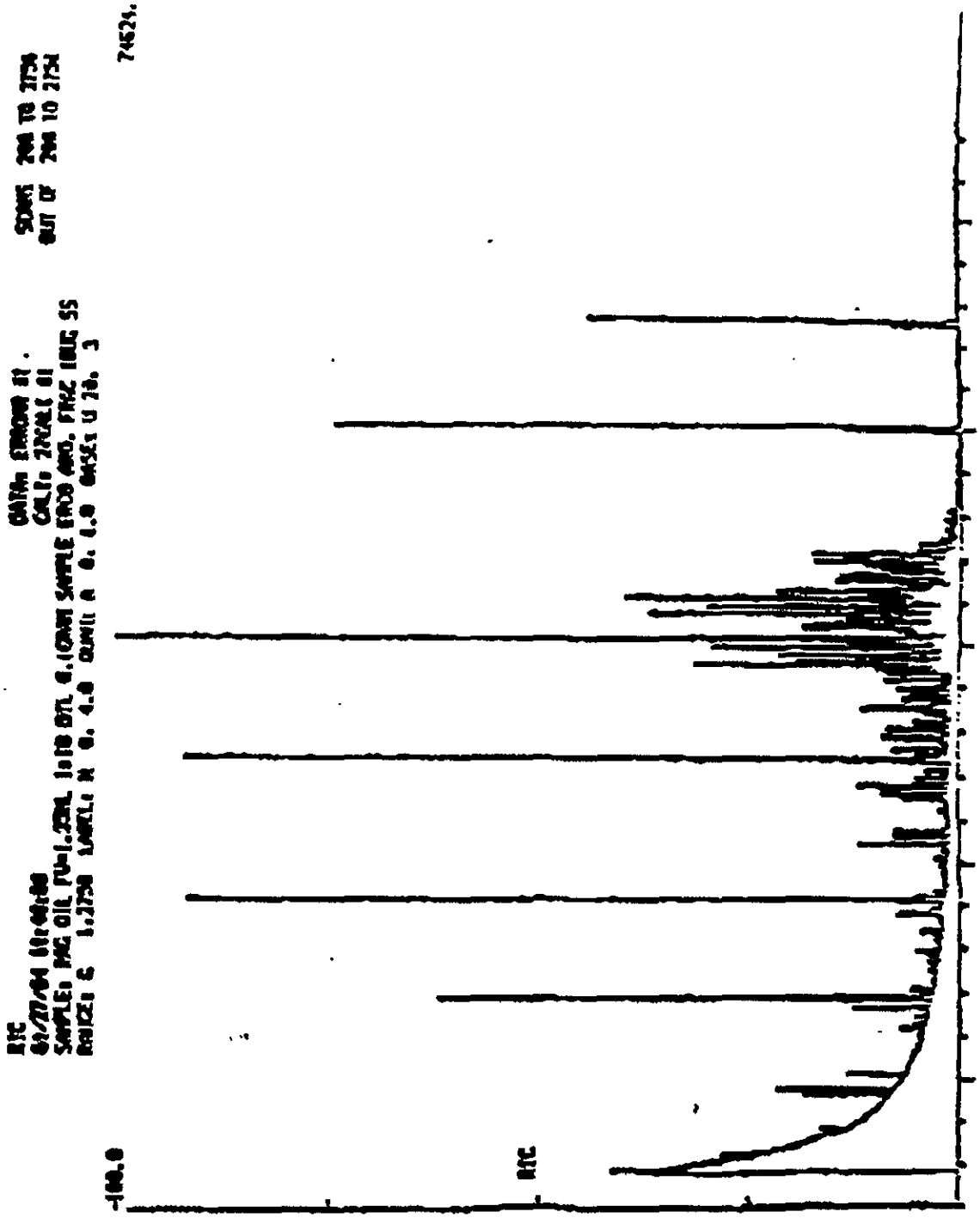

Figure 1. Reconstructed ion chromatogram from GC/MS analysis of the aromatic fraction from Rag Oil

Figure 2

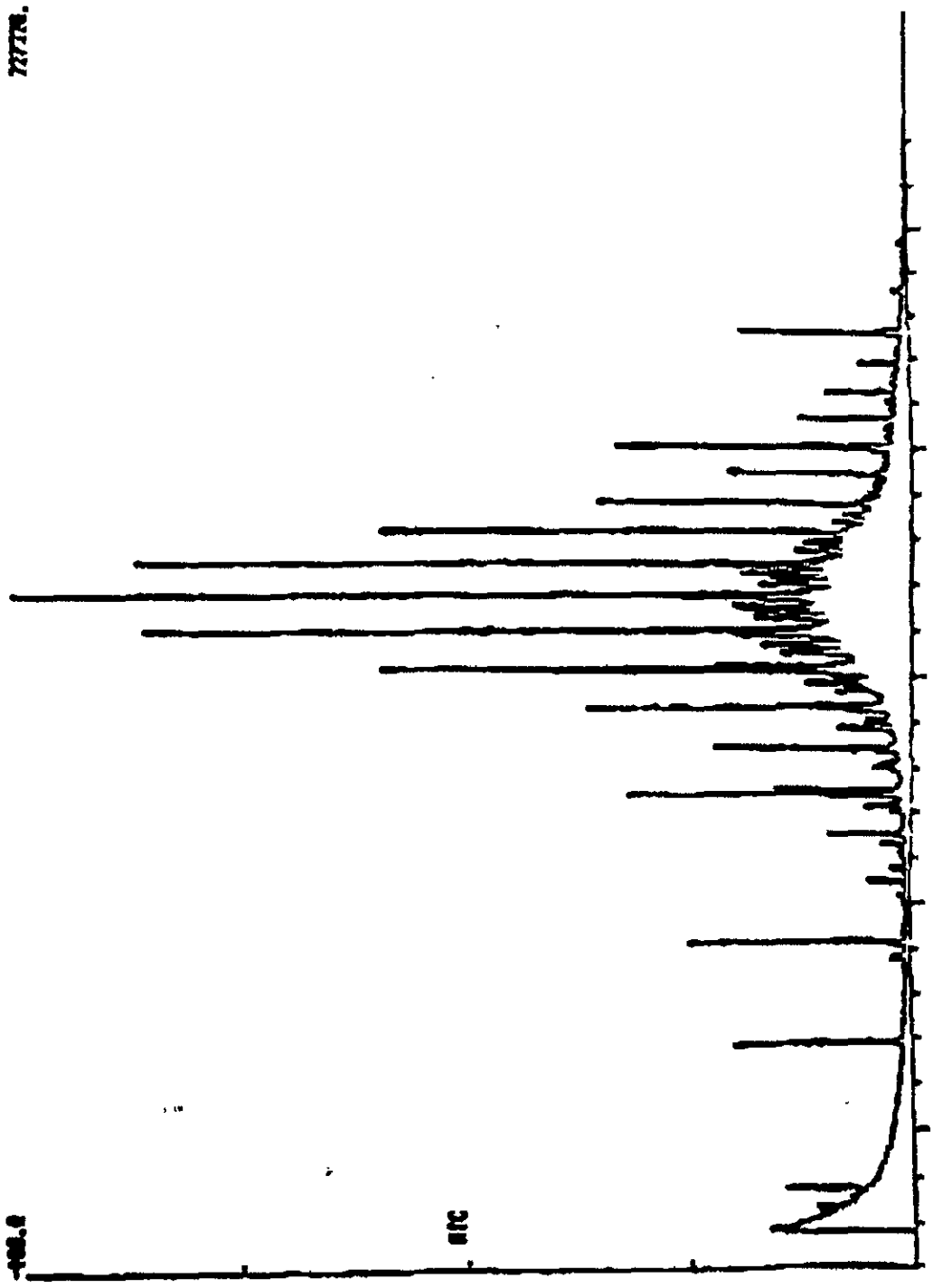

Figure 2. Reconstructed ion chromatogram from GC/MS analysis of the aliphatic fraction from Rag Oil

Table 1.
RESULTS OF ANALYSIS FOR SELECTED COMPONENTS IN RAG OIL

Analyte	Mean Conc. (mg/kg)[a]	Standard Deviation	%RSD[b]
Naphthalene	216	42	19
Fluorene	140	66	47
Phenanthrene	614	296	18
2-Methylnaphthalene	673	120	18
Dibenzothiophene	1084	286	26
Methylphenanthrene	2908	2014	69
Methyldibenzothiophene	2200	1017	46
	Average Surrogate Recovery		
Nitrobenzene-d_5	58.6	11	
Terphenyl-d_{14}	83.0	2.6	
Phenol-d_6	80.5	27.6	
Naphthalene-d_8	64.5	5.0	

[a] Based on five determinations from three laboratories.

[b] Percent Relative Standard Deviation.

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