## Conclusion

The AutoTrace is specifically designed for extraction of large volume water samples. The extraction of silica gel treated hexane extractable material (SGT-HEM) is efficient using Isolute SPE column TPH. The positive flow feature of the AutoTrace ensures that each extraction is uniform and optimizes EPA Method 1664.


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Application Note

# Extraction of Total Petroleum Hydrocarbons Using the AutoTrace


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## Overview


*This method was developed for the extraction of total petroleum hydrocarbons (or silica gel treated-hexane extractable material (SGT-HEM) as described in EPA method 1664) from water samples. The analytes are determined by gravimetric analysis.*


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## *Extraction Procedure*

| | |
|---|---|
| *ISOLUTE® SPE Column: | TPH 1 g / 6 ml |
| Pre-treatment: | Add 10 mL of methanol to 1 L of sample. Acidifiy the sample with 6 M HCl to a pH ~2. |
| Solvation: | Program the AutoTrace to WASH SYRINGE with 5 mL of methanol, followed by a CONDITION COLUMN with 10 mL of methanol. |
| Equilibration: | Program the AutoTrace to WASH SYRINGE with 10 mL of distilled deionised water pH~2, followed by a CONDITION COLUMN with 10 mL of distilled deionised water pH~2. |
| Sample application: | Program AutoTrace to LOAD 1100 mL SAMPLE. Following the sample load step, include a PAUSE AND ALERT step. |
| Interference elution: | During pause step, remove sample line from sample bottle. Manually rinse sample bottle with 10 mL of acetone, swirl well to cover sides of bottle. Dilute acetone in bottle with 40 mL of distilled deionised water, pH~2. Return sample line to bottle, and press CONT on AutoTrace. Program AutoTrace to LOAD 60 mL SAMPLE. Following this step, include a PAUSE AND ALERT step. Repeat this step if necessary until the rinse solvent appears clear. Press CONT on AutoTrace. Dry cartridge assembly for 30 min. |
| Analyte elution: | Rinse AutoTrace syringe with 5 mL of hexane. ELUTE TO SOAK with 4 mL of hexane. Follow this step with a TIMED PAUSE for 2 min. ELUTE TO COLLECT with 4 mL of hexane into tared collection tubes. Concentrate solvent to near dryness in a gentle stream of nitrogen. Collection tubes may be placed in a heating block held at 35 C to expediate evaporation. Once solvent is almost gone, weigh tubes in one minute intervals until weight loss is less than 1 mg. Total petroleum hydrocarbons is the weight of residue. |

***Setup parameters on AutoTrace should be as follows:***

| | | | |
|---|---|---|---|
| **Cond Flow:** | 40.0 mL / min | Push Delay | 5 sec |
| **Load Flow:** | 30.0 mL / min | Air Factor | 1.0 |
| **Rinse Flow:** | 40.0 mL / min | Autowash Vol | 1.00 mL |
| **Elute Flow:** | 20.0 mL / min | | |
| **Cond Air Push:** | 40.0 mL / min | | |
| **Rinse Air Push:** | 40.0 mL / min | | |
| **Elute Air Push:** | 40.0 mL / min | | |

** ISOLUTE column part numbers represent the product configuration of choice for use with an AutoTrace SPE Workstation.*

***The AutoTrace procedure should be written as follows:***

| | |
|---|---|
| Step 1: | Process 6 samples using the following procedure: |
| Step 2: | Wash syringe with 5 mL of methanol |
| Step 3: | Condition column with 10 mL of methanol in SOLVENT WASTE |
| Step 4: | Wash syringe with 10 mL of water pH=2 |
| Step 5: | Condition column with 10 mL of water to AQUEOUS WASTE |
| Step 6: | Load 1100 mL of sample on column |
| Step 7: | Pause and Alert operator, resume when CONTinue is pressed |
| Step 8: | Load 60 mL of sample onto column |
| Step 9: | Pause and Alert operator, resume when CONTinue is pressed |
| Step 10: | Dry column with gas for 30 minutes |
| Step 11: | Wash syringe with 5 mL of hexane |
| Step 12: | Soak and Collect 4 mL fraction using hexane |
| Step 13: | Pause for 2 minutes |
| Step 14: | Collect 3 mL fraction into sample tube using hexane |
| Step 15: | Clean each sample path with 10 mL into SOLVENT WASTE |
| Step 16: | Clean each sample path with 50 mL into AQUEOUS WASTE |
| Step 17: | Wash syringe with 10 mL of water |
| Step 18: | END |

*NOTE: Place sample lines into methanol for step 15 and reagent water for step 16 to flush out lines.*

| | |
|---|---|
| Structural | Non-polar hydrocarbons. |
| Various | |
| Structural Considerations | This method is suitable for the non-polar petroleum hydrocarbons (SGT-HEM). |
| Matrix Considerations | The matrix is polar, and the analytes are extracted by a non-polar retention mechanism. |
| Analytical Method | Gravimetric analysis using an analytical balance having precision to 0.1 mg. |
| General Comments | 1. This method describes an automated procedure for the determination of silica gel treated-hexane extractable material (SGT-HEM) from an aqueous sample. This column can also be used for Total Oil and Grease type extractions (EPA 1664 HEM) if the concentration of polar components is low. In this situation, the the hexane elution step should be followed by an elution (into a second tared vial) using 2 x 4 mL THF/hexane (1:1, v/v).Combination of these two fractions will give a determination of Total Oil and Grease, as described in EPA method 1664.However, for higher concentrations of polar components, we would recommend reference to application note IST1018 (TPH/Total Oil and Grease determination on the AutoTrace) which utilises a column specifically optimised for EPA 1664 extractions on the AutoTrace system.<br>2. Due to the nature of the analytes, the bottle washing steps after sample loading are very important, as analytes do stick to the walls of the sample bottle. For this reason, sample splitting is not recommended for TPH or Oil and Grease samples. |