



A Honeywell Company

Soxhlet Extraction of Catalyst

UOP Method 602-15

Scope

This method is for separating hydrocarbons from catalyst samples by extraction with a volatile solvent. This extraction is used to obtain a hydrocarbon and water free catalyst sample for carbon determination by UOP Method 703, “Carbon on Catalysts by Induction Furnace Combustion and Infrared Detection.” It can also be used to determine the non-volatile hydrocarbon content of the sample, or to provide an extract for subsequent analysis. The use of an automated system is described in the *Appendix*. For self-heating samples, refer to the drying procedure in the *Appendix*.

References

ASTM Method G120, “Determination of Soluble Residual Contamination by Soxhlet Extraction,”
www.astm.org

UOP Method 703, “Carbon on Catalysts by Induction Furnace Combustion and Infrared Detection,”
www.astm.org

UOP Method 918, “Collection and Grinding of Adsorbents and Catalysts for Analysis,”
www.astm.org

Outline of Method

A ground sample is placed in an extraction thimble and Soxhlet extracted with methylene chloride for four hours, or until the solvent in the thimble chamber is clear. The thimble with sample is then drained and dried at 105°C in a nitrogen-purged oven for four hours. If quantitative results for hydrocarbon separated by this method are required, the thimble must be dried and weighed with the sample before and after extraction.

To obtain a sample of the non-volatile hydrocarbons for subsequent organic type identification, the extracted material is made up to a specified volume with the extraction solvent. When the mass of the non-volatile residue is desired, the solvent is volatilized in a tared beaker and reweighed.

The Appendix contains an alternative procedure using an automated extraction system.

<p>IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).</p>

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Apparatus

Reference to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, readability 0.0001 g

Beaker, 250-mL, borosilicate glass, Fisher Scientific, Cat. No. FB-100-250

Clamps, high pressure, 6- to 11-mm, Fisher Scientific, Cat. No. 14-198-5B

Cylinder, graduated, borosilicate glass, 250-mL, Fisher Scientific, Cat. No. 08-554F

Desiccator, borosilicate glass, large diameter, Fisher Scientific, Cat. No. 08-595E

Dessicator plate, polypropylene, 230-mm, Fisher Scientific, Cat. No. 08-643-2C

Extraction heater, 6-unit, 28-cm long x 76-cm wide x 23-cm high, includes brackets and rods, Fisher Scientific, Cat. No. 09-678-1

Flask, volumetric, 250-mL, Fisher Scientific, Cat. No. 10-208J

Forceps, extra long, 300-mm, Fisher Scientific, Cat. No. 10-316C

Hotplate, laboratory

Mortar and pestle, porcelain, 130-mm OD, Fisher Scientific, Cat. Nos. 12-961C and 12-961-5C, respectively

Oven, drying, capable of operation at 105°C, Fisher Scientific, Cat. No. 13-247-637G or 13-247-737G. The oven requires proper ventilation for the solvent vapors. Purging the oven with nitrogen is required and placing the oven inside of a fume hood is recommended.

Regulator, gas, two-stage, Matheson Tri-Gas, Cat. No. 81-580

Soxhlet extraction assembly, borosilicate glass, comprised of a 40-mm ID extraction tube with top T 40/50 joint, 250-mL flask and condenser, Fisher Scientific, Cat. No. 09-556B (see *Note*)

Tubing, tygon, 7.9-mm ID, 15-m long, Fisher Scientific, Cat. No. 14-169-1M

Vials with caps, 22.2-mL Fisher Scientific, Cat. No. 03-338-H

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. References to water mean deionized or distilled.

Calcium chloride, anhydrous, dessicant, 4-20 mesh, Fisher Scientific, Cat. No. C614

Methylene chloride, 99.8% minimum purity, Fisher Scientific, Cat. No. D37

Nitrogen, 99.99% minimum purity

Pencil, soft lead (F or 2)

Thimbles, Whatman, pure cellulose, 33- x 80-mm, Fisher Scientific, Cat. No. 09-656B

Water, deionized or distilled

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of Soxhlet extraction, and the equipment being used. Additional information on the technique of Soxhlet extraction, including a diagram of the apparatus, can be found in ASTM Method G120, "Determination of Soluble Residual Contamination by Soxhlet Extraction."

Qualitative Extraction Preceding Carbon Determination

1. Grind the catalyst according to UOP Method 918, "Collection and Grinding of Adsorbents and Catalysts for Analysis," or with a mortar and pestle to a fine mesh size.
2. Place approximately 5 g of catalyst into the extraction thimble. Close the thimble by folding over the top 1.5 cm and pressing it shut with the forceps. Use a pencil to identify the contents of the thimble. Do not use an ink pen. Place the thimble in the extraction apparatus.
 - Less than 5 grams may be used if a limited supply of sample is available and the amount is sufficient for subsequent analyses.
3. Transfer 150 mL of methylene chloride with a graduated cylinder into the solvent flask.
4. Lubricate the ground glass joints by marking them liberally with a soft lead pencil.
5. Assemble the apparatus in a fume hood according to manufacturer's specification and turn on the cooling water to the condensers.
6. Turn on and increase heat to obtain a gentle reflux of the solvent.
7. Continue to reflux for a minimum of four hours, or until the solvent in the thimble is visibly clear when nearly full.
8. When the extraction is complete, turn off the heat and allow the apparatus to cool for 30 minutes.
9. Turn off the water to the condensers.
10. Disconnect the condenser from the thimble chamber and, with the forceps, lift the thimble, set it in the opening of the thimble chamber, and allow it to drain for approximately 15 minutes to remove excess solvent.
11. Discard the solvent, unless it is required for organic type identification.
12. Place the thimble in a clean beaker and dry in the nitrogen-purged, vented oven at 105°C for a minimum of 4 hours.
 - The thimble may be left in the oven at 105°C overnight.
13. Remove the beaker with the thimble from oven, place in a desiccator, and allow to cool.
14. Transfer the sample to a vial and cap immediately.

Quantitative Extraction to Determine Hydrocarbon Content

1. Place a new extraction thimble in a 250 mL beaker and dry it for 30 ± 1 minutes in a nitrogen-purged, vented oven at 105°C.
2. Grind the catalyst according to UOP Method 918, "Collection and Grinding of Adsorbents and Catalysts for Analysis," or with a mortar and pestle to a fine mesh size.
3. Remove the beaker and thimble from the oven, place in a desiccator, and allow to cool for 15 ± 1 minutes.
4. Weigh the beaker and thimble to the nearest 0.0001 g to obtain a tare weight.