



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.

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).

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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.

5. Transfer a sufficient quantity of the ground catalyst to fill the thimble approximately 2/3 full.
6. Weigh and record the gross weight of the sample, thimble and beaker.
7. Follow the extraction procedure as described under *Qualitative Extraction Preceding Carbon Determination*, Steps 3 through 11.
8. Put the thimble in the tared beaker originally used and place in the drying oven at 105°C for four hours.
9. Remove the beaker with the thimble from oven, place in a desiccator, and allow to cool for 15 ± 1 minutes.
10. Weigh the beaker containing the thimble and extracted sample to the nearest 0.1 mg. Record the weight.

Extracts for Other Analysis

If subsequent analysis of the extracted material is required, the hydrocarbon can be provided as a solution in the extraction solvent or as a quantitative, non-volatile residue.

When a solution is required:

1. Quantitatively transfer the solvent solution resulting from either of the preceding sections to a 250-mL volumetric flask.
2. Rinse the apparatus, collecting the rinsings in the volumetric flask and dilute to the mark with additional solvent.

When a quantitative non-volatile residue is required:

1. Extract the sample as described under *Quantitative Extraction to Determine Hydrocarbon Content*.
2. Transfer all of the solvent from the extraction apparatus to a tared beaker.
3. In a hood, carefully evaporate to 5-10 mL on a hot plate at a low temperature.
4. Place the beaker in the nitrogen-purged, vented oven at 105°C for one hour.
5. Remove the beaker with the thimble from oven, place in a desiccator, and allow to cool.
6. Weigh the beaker and residue to the nearest 0.1 mg.

Calculations

If a quantitative extraction to determine hydrocarbon content is performed, use the following calculations to determine extractable hydrocarbons and/or non-volatile extracted materials.

Calculate the mass-% extractable hydrocarbon content of the catalyst sample using Equation 1. Any water present in the catalyst sample that is removed during the four-hour drying procedure will be included in the extractable hydrocarbons value. Report to the nearest 0.1 mass-%.

$$\text{Extractable hydrocarbons, mass-\%} = \frac{100(A - B)}{C} \quad (1)$$

where:

- A** = mass of beaker, thimble and sample before extraction, g
- B** = mass of beaker, thimble and sample after extraction and oven drying, g
- C** = mass of sample before extraction, g
- 100** = factor to convert from g/g to mass-%

Calculate the mass-% non-volatile extracted materials after oven drying at 105°C using Equation 2. Report to the nearest 0.1 mass-%.

$$\text{Non-volatile extracted materials, mass-\%} = \frac{100(D - E)}{C} \quad (2)$$

where:

C = mass of sample before extraction, g
D = mass of beaker and extracted material, g
E = mass of empty beaker, g
100 = factor to convert from g/g to mass-%

Note

Several vendors offer complete ‘automated systems’ that are satisfactory alternatives. The use of one such system is described in the *Appendix*.

Precision

Repeatability and Reproducibility

Because this method is primarily used for the qualitative removal of hydrocarbons from catalysts prior to carbon determination, the repeatability and reproducibility of the test are not calculated.

Time for Analysis

Table 1 shows the labor requirements and elapsed times for the various extraction procedures.

Table 1
Times for Analysis

	Labor Requirement, <u>Hours</u>	Elapsed Time, <u>Hours</u>
Qualitative extraction preceding carbon determination	0.2	10
Quantitative extraction to determine hydrocarbon content	1.5	12
Extracts for other analysis	0.4	5

Suggested Suppliers

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785, USA (412-490-8300)
www.fishersci.com

Matheson Tri-Gas, 166 Keystone Dr., Montgomeryville, PA 18936, USA (215-641-2700)
www.mathesontrigas.com

Appendix

Soxhlet Extraction Using an Automated System

Scope

This procedure is for separating hydrocarbons from catalyst samples by extraction with a volatile solvent using an automated extraction system. This extraction is used to obtain a hydrocarbon and water free catalyst sample for carbon determination by UOP Method 703. It can also be used to determine the non-volatile hydrocarbon content of the sample, or to provide an extract for subsequent organic types analysis. Up to four samples can be extracted simultaneously in the four-position extraction system specified.

Outline of Method

A ground sample is placed in an extraction thimble and Soxhlet extracted with methylene chloride for two hours. 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.

Apparatus

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

The following items are required in addition to items in the body of the method.

Extraction system, Model B-811, Buchi

Glass collars, for 27mm O.D. extraction thimbles, Buchi, Cat. No. 037277

Solvent beakers, Buchi, Cat. No. 037276.

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.

The following item is required in addition to items in the body of the method.

Thimbles, Whatman, pure cellulose, 25-mm I.D. x 100-mm, Fisher Scientific, Cat. No. 09-656L

Procedure

The Buchi B-811 extraction system is configured for the “Soxhlet Warm” extraction type. This reduces the required extraction time from 4 hours to 2 hours by heating the extraction chamber. Set the upper and lower heat settings to those recommended by the manufacturer for the solvent used. See Table A1 for an example.

Table A1
Recommended Settings For Buchi B-811 Extraction System

Extraction type	Soxhlet Warm
Solvent	methylene chloride
Lower heater temperature	9
Upper heater temperature	2
Extraction time	2 hours
Rinse time	10 minutes
Dry time	0
Inert gas purge	optional

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. Use a pencil to identify the contents of the thimble. Do not use an ink pen.
 - Less than 5 grams may be used if a limited supply of sample is available and the amount is sufficient for subsequent analyses.
3. Insert the extraction thimble into the glass collar such that all but 2-3 mm of the thimble extends from the bottom of the collar. The notches must be at the top.
4. Mount the collar to the sample holder at the end of the condensation tube.
5. Repeat steps 2 - 4 for all remaining samples, then lower the condensers and turn clockwise to seal the system.
6. Transfer 100 mL of methylene chloride with a graduated cylinder into each solvent beaker.
7. Lift the solvent beakers into the holder ring and turn to lock in place.
8. Turn on water supply, if not already on.
9. Lift the lower heating elements up to the solvent beakers. They will lock in place when fully raised.
10. The position of the optical sensor should be adjusted to be approximately 1 cm above the top of the sample. This position can be marked on the guide rod with permanent marker.
11. Start the program to begin the extraction. The samples will extract for two hours, rinse for 10 minutes, and then lift up within the extraction chamber to drain any remaining solvent.
12. When the extraction is complete, lower the heating elements and allow the thimbles to drain for 15 minutes to remove excess solvent.
13. Rotate the condensers counter-clockwise to unlock. The condensers will lift to the upper position automatically.
14. Remove the thimbles from the collars and place the thimbles in a clean beaker.
15. Raise the heating elements to the locked position. Press the SOLVENT button on the control panel. This will drain any remaining solvent back to the solvent beaker.
16. Discard the solvent, unless it is required for organic type identification.
17. Dry the extraction thimbles in the nitrogen-purged, vented oven at 105°C for 4 hours.

- For self-heating samples, place the thimbles individually into small beakers, and allow the sample to cool to room temperature in the nitrogen-purged, vented oven before proceeding.
18. Remove from oven, place in a desiccator, and allow to cool.
 19. 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 with the thimble from 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.
5. Transfer a sufficient quantity of the ground catalyst to fill the thimble approximately 1/2 full.
6. Weigh and record the gross weight of the sample, thimble and beaker.
7. Follow the extraction procedure as described under *Qualitative Extraction Preceding Carbon Determination*, Steps 3 through 13, and Steps 15 and 16.
8. Put the thimble in the tared beaker originally used and place in the nitrogen-purged drying oven at 105°C for four hours.
 - For self-heating samples, place the thimbles individually into small beakers, and allow the sample to cool to room temperature in the nitrogen-purged, vented oven before proceeding.
9. Remove the beaker with the thimble from oven, place in a desiccator, and allow to cool for 15 ± 1 minutes.
10. Weigh the beaker containing the thimble and extracted sample to the nearest 0.1 mg. Record the weight.

Extracts for Other Analysis

If subsequent analysis of the extracted material is required, the hydrocarbon can be provided as a solution in the extraction solvent or as a quantitative non-volatile residue.

When a solution is required:

1. Quantitatively transfer the solvent solution resulting from either of the preceding sections to a 250-mL volumetric flask.
2. Rinse the apparatus, collecting the rinsings in the volumetric flask and dilute to the mark with additional solvent.

When a quantitative non-volatile residue is required:

1. Tare a clean solvent beaker for each sample.
2. Use a modified version of the extraction program which contains a drying step (Program Step 3). Fifteen minutes on heat level 7 is sufficient for 100 mL of methylene chloride. This alternative extraction method should be saved with a different program number.

3. If needed, the inert gas purge of the B-811 should be started before beginning the extraction.
4. Extract the sample as described under *Quantitative Extraction to Determine Hydrocarbon Content*, Steps 1 through 10.
5. Remove the solvent beaker from the extraction unit and place the beaker in the nitrogen-purged, vented oven at 105°C for one hour. Repeat for all samples extracted in this set.
6. Place an empty solvent beaker under each extracted sample position. Raise the heating element and press the SOLVENT button to drain the solvent back to the solvent beaker. Discard the used solvent.
7. Remove the beaker from the oven, place in a desiccator, and allow it to cool.
8. Weigh the beaker and residue to the nearest 0.1 mg.

All calculations are the same as in *Calculations* in the body of the method.

Time for Analysis

Table A2 shows the labor requirements and elapsed times for the various extraction procedures.

Table A2
Times for Analyses

	Labor Requirement, <u>Hours</u>	Elapsed Time, <u>Hours</u>
Qualitative extraction preceding carbon determination	0.2	7
Quantitative extraction to determine hydrocarbon content	0.5	9
Extracts for other analysis	0.3	3

Suggested Supplier

BÜCHI Corporation, 19 Lukens Drive, Suite 400, New Castle, DE 19720, USA (302-652-3000),
www.myluchi.com