

DEWAXING OF PETROLEUM PRODUCTS

UOP Method 847-86

SCOPE

This is a semi-quantitative method for reducing the wax content of wax-containing petroleum charge stocks or products having initial boiling points greater than 200 C. It is primarily intended as a procedure to prepare small quantities of dewaxed oil for subsequent analyses. The wax content of the sample can be quantitatively determined by UOP Method 46.

OUTLINE OF METHOD

The sample is dissolved in dichloromethane (methylene chloride) and the resulting solution is cooled to -28 C to precipitate wax. Other solvents or temperatures may be used as specified by the ultimate use of the dewaxed oil being investigated. The precipitated wax is removed by filtration using vacuum. The solvent is removed from the dewaxed oil by distillation and evaporation. A material balance is calculated and reported.

APPARATUS

Balance, readability 0.01-g

Bath, water, stainless steel, 254-mm diameter, Sargent-Welch Scientific, Cat. No. S-84215-C, or equivalent

Beakers, 1500- and 2000-mL

Clamp, utility, V-jaw, Sargent-Welch Scientific, Cat. No. S-19150, or equivalent

Condenser, Liebig, 400-mm jacket, Sargent-Welch Scientific, Cat. No. S-22645-D, or equivalent

Dewaxing apparatus (Fig. 1), consisting of:

Bath, cooling, stainless steel, 280-mm OD by 165-mm deep, covered with 25 mm of foam insulation, 44-mm diameter hole in the center of the bottom

Filter paper, Whatman Grade AH, glass fiber, No. 934AH, 9-cm diameter, Sargent-Welch Scientific, Cat. No. S-33340-G, or equivalent

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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Flask, flat-bottom, distillation, with side tube, 5000-mL

Flask, polyethylene, foam insulated, 2000-mL Sargent-Welch Scientific, Cat. No. S-34755-B, or equivalent

Funnel, Buchner, fritted glass, medium porosity, borosilicate, 600-mL, Sargent-Welch Scientific, Cat. No. S-35591-H, or equivalent

Gauze, wire, iron, 10 x 10-cm, Sargent-Welch Scientific, Cat. No. S-85325-A, or equivalent

Ring stands, 15 x 28-cm base, 91-cm support rod, Sargent-Welch Scientific, Cat. No. S-78306-D, or equivalent (three required)

Stoppers, rubber, one-holed to accommodate funnel stem, Nos. 9 and 11, Sargent-Welch Scientific, Cat. Nos. S-73315-N and R, or equivalent

Support rings, extension, 152-mm ID, 178-mm stem, VWR Scientific, Cat. No. 60135-100, or equivalent

Support shelves, with screw clamp, 127-mm diameter, VWR Scientific, Cat. No. 60125-004, or equivalent

Thermometer, ASTM 6C, Sargent-Welch Scientific, Cat. No. S-80585, or equivalent

Thermometer clamp and clamp holder, Fisher Scientific, Cat. Nos. 05-809-10 and 05-754, respectively, or equivalent

Trap, dry ice, 4-mm wall thickness, 45-mm OD, 284-mm length, (Fig. 2), UOP Inc., or equivalent

Tubing, rubber, vacuum, 6-mm OD

Hot plate, with stirrer and stirring bar, Sargent-Welch Scientific, Cat. No. S-41059-25, or equivalent

Pump, vacuum, heavy-duty, Sargent-Welch Scientific, Cat. No. S-71381, or equivalent

Regulator, nitrogen, two-stage, with flowmeter, Matheson 8-BF, CGA No. 580, or equivalent

Stopper, rubber, size 11, Sargent-Welch Scientific, Cat. No. S-73445-Q, or equivalent

Stopper, rubber, one-hole, size 11, Sargent-Welch Scientific, Cat. No. S-73315-R, or equivalent

Tubing, glass, 60-cm, 6-mm OD, Standard wall

REAGENTS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available unless otherwise specified.

Carbon dioxide, solid

Dichloromethane (methylene chloride), 95% minimum purity, Sargent-Welch Scientific, Cat. No. SC 13551, or equivalent (CAUTION: Avoid prolonged or repeated breathing of vapor. Use only with adequate ventilation.)

Nitrogen, 99.9% minimum purity, extra dry

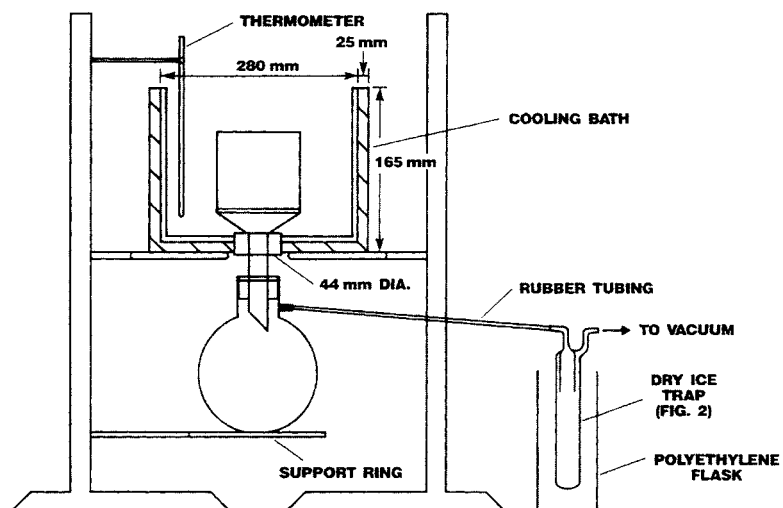


Figure 1

Dewaxing Apparatus

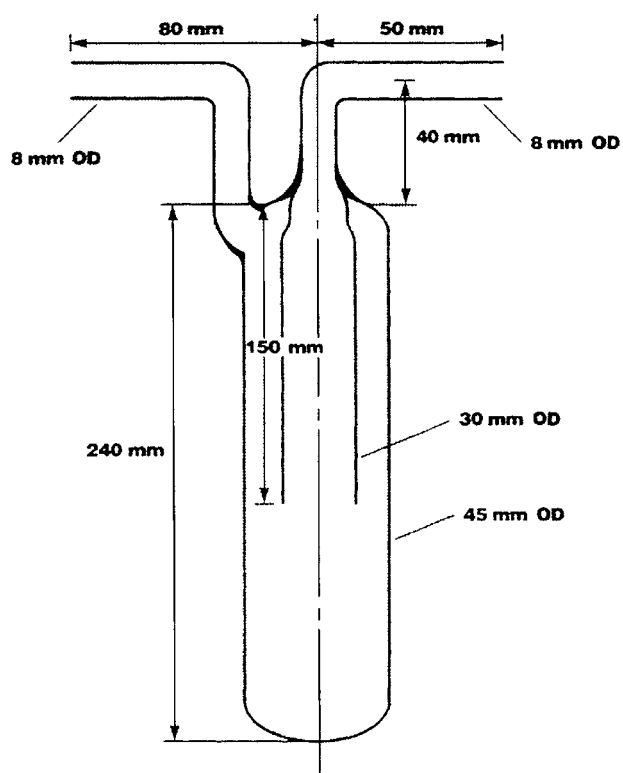


Figure 2

Dry Ice Trap

PROCEDURE

Weigh 100-150 g of sample to the nearest 0.01 g into the appropriate size beaker. Record the mass of sample taken. The amount of sample to be dewaxed depends on the quantity of dewaxed oil that is required for subsequent analysis. Typically, the wax content of wax-containing oils is approximately 15%.

Dissolve the sample by adding dichloromethane solvent to the beaker in the proportion of one-part sample to 10-parts solvent by volume. Cool the solution to -28 C by whatever means are available or practical. The solution should be cooled gradually over a period of approximately two hours to avoid localized cooling that will cause the wax to adhere to the sides of the beaker.

While the sample solution is cooling, place a piece of filter paper on the fritted glass at the bottom of the Buchner funnel. Place the dry ice trap (Fig. 2) in the polyethylene flask containing a dry ice and dichloromethane mixture. Weigh a 5-L distilling flask containing a stirring bar to the nearest 0.01 g. Assemble the dewaxing apparatus as shown in Fig. 1. Fill the stainless steel cooling bath with dichloromethane to within one centimeter of the top of the Buchner funnel. Slowly add pieces of dry ice of sufficient size to avoid extreme agitation of the dichloromethane. Continue adding dry ice until the operating temperature of -28 C is reached. Periodically add smaller pieces of dry ice to the bath to maintain this temperature.

Using vacuum, filter the sample solution. Throughout the filtration operation, maintain the beaker of sample solution at -28 C, stir the sample solution occasionally, and do not allow the wax filter cake to dry. Rinse the wax filter cake with additional dichloromethane that has been cooled to -28 C until the filtrate is colorless.

Turn off the vacuum and remove the flask from the dewaxing apparatus. Place a solid rubber stopper in the neck of the flask and attach the condenser to the flask at the side arm with rubber tubing. Place the flask on the water bath and support the condenser with a ringstand. Heat the water bath to approximately 100 C and cool the condenser with running tap water. Collect the distilled solvent and retain for reuse. When solvent ceases to drip from the condenser, remove the condenser and transfer the flask to a hot plate. Connect the distilling flask side arm to the dry ice trap and vacuum pump used for the dewaxing. Using a one-hole stopper, attach a nitrogen line to the flask. Turn on the hot plate and stirrer. Set the heater control to approximately 100 C. Heat and stir the sample under vacuum with a nitrogen flow of several milliliters per minute for a minimum of 5 hours to remove the remaining solvent from the dewaxed oil.

Transfer the wax cake from the funnel to a tared beaker and remove the filter paper. Evaporate the solvent from the wax by placing the beaker on a water bath maintained at approximately 100 C and directing a stream of nitrogen into the beaker. When the wax stops boiling and no more solvent appears to be coming off, place the beaker on the hot plate set at approximately 100 C for 3 hours to remove the remaining solvent. Place the wire gauze between the beaker and hot plate to prevent the wax from spattering.

Weigh the beaker after it has cooled and record the mass of the wax. Weigh the distilling flask and record the mass of the dewaxed oil.

CALCULATIONS

Calculate the mass-% of wax using the following equation:

$$W = \frac{100 M}{C}$$

where:

C = mass of sample charged, g

M = mass of wax, g

W = wax, mass-%

100 = percentage constant

Calculate the mass-% of oil using the following equation:

$$R = \frac{100 D}{C}$$

where:

C = previously defined

D = mass of dewaxed oil, g

R = dewaxed oil, mass-%

100 = percentage constant

Calculate the mass-% of loss using the following equation:

$$L = 100 - (W + R)$$

where:

L = loss, mass-%

R, W and 100 = previously defined

A loss of more than 2% is considered unacceptable and the procedure must be repeated with a fresh sample. A gain in mass is an indication of incomplete solvent removal.

PRECISION

An estimated standard deviation is not reported because insufficient data are available at present to permit this calculation with at least 4 degrees of freedom.

TIME FOR ANALYSES

The elapsed time for one analysis is 16 hours. The labor requirement is 8 hours.

REFERENCES

BP Method 237/55 "Wax Content of Petroleum Products," British Petroleum Company Ltd., Chertsey Rd., Sunbury-on-Thames, Middlesex, 1W16 7LN, United Kingdom
UOP Method 46

SUGGESTED SUPPLIERS

Fisher Scientific, 1600 W. Glenlake Ave., Itasca, IL 60143
Matheson, P.O. Box 96, Joliet, IL 60434
Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
VWR Scientific, P.O. Box 66929, O'Hare AMF, Chicago, IL 60