

FRACTIONATION OF PETROLEUM DISTILLATES

UOP Method 79-87

SCOPE

This method describes equipment and procedures used for laboratory fractionation of petroleum distillates and products. Procedures are given for quantitatively removing non-condensable and condensable gases, as well as obtaining liquid fractions boiling within the range of 10 to 400 C. For the fractionation of crude oil and the preparation of distillation curves from true boiling point (TBP) data, see ASTM D 2892.

OUTLINE OF METHOD

A known mass of a petroleum distillate or product is fractionated in a high efficiency laboratory column. Procedures are given for the quantitative separation of normally gaseous hydrocarbons, such as C₃ and/or C₄ hydrocarbons from C₅ and heavier fractions, and for the precision fractionation at atmospheric and reduced pressures of normally liquid hydrocarbons. Procedures for the collection of gas/liquid samples for sulfur determinations are also included.

APPARATUS

Balance, top-loading, 0 - 30,000 g, readability 0.1-g, Sartorius, Model 3808 MP8, Sargent-Welch Scientific, Cat. No. S-2714-21A, or equivalent

Barometer, mercurial, Sargent-Welch Scientific, Cat. No. S-4565, or equivalent

Cylinder, graduated, 2000-mL

Cylinders, type 304 stainless steel, high pressure, 12,400 kPa (1800 psig) maximum working pressure, 75- and 1000-mL capacity, Whitey Co., Cat. Nos. 304L-HDF4-75 and 304L-HDF4-1000, respectively, or equivalent. Equip each cylinder with 2 valves, Whitey Co., Cat. No. 16DKM4-F4, or equivalent, and a hose fitting, Matheson, Cat. No. 121M, or equivalent.

Density meter, and support apparatus, as specified in ASTM D 4052 (optional)

Flasks, Dewar, 350- and 1000-mL capacities, Sargent-Welch Scientific, Cat. No. S-34707-AA and B, or equivalent

Fractionating system (Figs. 1 - 4), Reliance Glass Works Inc., or equivalent, consisting of the following:

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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Flasks, distillation, round bottom, ranging in capacity from 200- to 5000-mL, equipped with a stopcock in the sidearm, a thermowell, and a 50/30 female spherical glass joint

Fractionating column, Podbielniak HYPER-CAL, 30-mm ID x 1.2 m, Cat. No. 3438, containing a one-meter section of HELI-PAK column packing, Cat. No. 3008. The use of O-ring joints is recommended wherever possible to avoid contamination from the lubricating grease. The overall height of the assembled system is 2.2 m.

Gauge, McLeod, 0 - 15-mm. A pressure transducer, including power supply and digital readout, may be substituted.

Heating mantles, "Glas-Col" mantles, in sizes corresponding to those of the distillation flasks

Light overhead condenser (Fig. 2). This is an external condenser which is filled with dry ice when refluxing propane and butane prior to debutanization of a gasoline.

Manometers, open-arm type

Manostat

Potentiometer, 0 to 500 C range

Receivers, jacketed and unjacketed, calibrated for accurate measurement of fraction volumes

Refrigeration unit and pump, capable of circulating an ethylene glycol-water mixture through the reflux condensers at approximately -20 C (-4 F)

Surge tank

Thermocouples, calibrated, iron-constantan wire

Time switch

Vacuum pump

Gas condensate traps, 100- and 300-mL capacity, UOP Inc., or equivalent

Gas sampling bottle, 2 required, 8-L, aspirator type (Fig. 5), UOP Inc., or equivalent

Hydrometers, petroleum, and support apparatus, as specified in ASTM D 1298

Regulator, nitrogen, two-stage, high purity, Matheson, Model 3104-580, or equivalent

Tubing, latex, translucent, heavy wall, 6.4-mm ID, 2.4-mm wall, Sargent-Welch Scientific, Cat. No. S-73571-D, or equivalent

REAGENTS AND MATERIALS

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. References to water mean deionized or distilled water.

Acetone, 98% minimum purity

Cleaning solution, Micro, International Products Corporation, or equivalent

Dry ice

Ethylene glycol, 98% minimum purity

Hydrochloric acid, concentrated

Mercury, triple distilled

Methyl orange, powder, Sargent-Welch Scientific, Cat. No. EKC-432, or equivalent

Nitrogen, extra dry, 99.9% minimum purity

Sodium chloride, crystal, Sargent-Welch Scientific, Cat. No. SC-14767, or equivalent

Sodium chloride solution, saturated and acidified. To prepare, place approximately 860 g of sodium chloride in an 8-L gas sampling bottle. Add water to almost fill the bottle and shake or stir to dissolve the sodium chloride. Add approximately 0.2 g of methyl orange, 5 drops of concentrated hydrochloric acid and shake or stir to mix.

Stopcock grease, Dow-Corning High Vacuum Silicone Grease, Sargent-Welch Scientific, Cat. No. S-77324-01, or equivalent

Toluene, 99.9% minimum purity

PREPARATION OF APPARATUS

Assemble the apparatus appropriate to the fractionation desired, as shown in Figs. 1 - 4.

After assembly, clean the column with suitable solvents and dry with a stream of nitrogen. Lubricate all stopcocks and ground-glass joints not equipped with O-rings with the specified grease.

To check the apparatus for leaks, attach a flask to the spherical joint at the bottom of the column. Close the system and attach a vacuum line to the flask at the stopcock. Evacuate the system to approximately 2 - 3 mm Hg (0.27 - 0.40 kPa) absolute. Close the stopcock and detach the vacuum line. If the reading on Manometer B remains constant over a period of 20 to 25 minutes, the apparatus may be considered leak-proof.

Determine the accuracy of the thermocouple and potentiometer according to Paragraph A6 of ASTM D 2892. Examine the flask and the condensate trap for cracks in the glass. Rinse with acetone and dry before use. Clean glassware with cleaning solution whenever necessary.

PROCEDURE

Because this method covers materials with such diverse boiling ranges, not all sections of the procedure may be applicable to any one particular sample. Each section contains directions for assembly and shut-down so that it can be performed independently if required. Determine which sections are applicable to the sample to be fractionated. Follow the directions for assembly in the section where the fractionation begins, and the directions for shut-down in the section where the fractionation is concluded.

Part I - Fractionation of Unstabilized Samples

Included in this category are fractionations at atmospheric pressure of unstabilized hydrocarbon samples for removal of C₄ and lighter components. The composition of these fractions may subsequently be determined by mass spectrometry, gas-liquid chromatography or an equivalent method. Unstabilized hydrocarbon samples have a Reid vapor pressure of 15 psig (103 kPa) or greater as determined by ASTM D 323, and are by necessity contained in stainless steel sample cylinders under pressure.

A. Collection of Non-Condensable Gases

Assemble the apparatus as shown in Fig. 1 and follow the procedure as specified under *PREPARATION OF APPARATUS*. Weigh and record the mass of a clean flask to the nearest 0.1 g. Attach the flask to the spherical joint at the bottom of the column. The flask should be of proper size for the volume of sample to be charged (not over 80% of the capacity of the flask), making due allowance for thermal expansion. The stopcock on the side arm of the flask is used for sampling directly into an evacuated system.

Weigh and record the mass of a clean gas condensate trap to the nearest 0.1 g. Pre-cool the trap by immersion in a Dewar flask filled with a dry ice-acetone mixture. With a short piece of latex tubing, attach the gas vent line to the coil end of the trap immersed in the cooling mixture. Pre-cool the ethylene glycol solution and start circulation of the solution through the reflux condenser only.

Fill an 8-L gas sampling bottle (Fig. 5) with sodium chloride solution. Prepare fresh sodium chloride solution when the methyl orange indicator starts to fade from red-orange to yellow. Using latex tubing, attach stopcock B of the gas sampling bottle to the exit end of the gas condensate trap. Close stopcocks A and B. Open stopcock D on the gas line vent. Evacuate the entire system to approximately 2 - 3 mm Hg (0.27 - 0.40 kPa) absolute. Check the system for leaks.

Set up a second bottle containing a known volume, typically 1000 mL, of sodium chloride solution. Attach a piece of latex tubing to stopcock E. Open stopcock E and carefully fill the tubing with solution. Attach the tubing to stopcock C of the gas collecting bottle.

Weigh the sample cylinder and record the mass to the nearest 0.1 g. Cool the cylinder in a refrigerator or in a container filled with ice. Surround the distillation flask with dry ice and allow it to cool for 10 minutes. With the cylinder in a vertical position, connect the bottom valve of the cylinder to the stopcock on the flask with a short piece of latex tubing. Open the stopcock. Carefully open the valve on the cylinder and adjust the flow so that the sample will enter the flask slowly. When the column reaches a pressure of 50 mm (6.7 kPa) above atmospheric, as indicated by Manometer B, open stopcock C at the bottom of the gas sampling bottle, and carefully open stopcock B between the trap and the bottle. Begin collecting the non-condensable gases by displacing the sodium chloride solution. Continually adjust the flow of the sample into the flask and the non-condensable gas into the collecting bottle so that a positive pressure is maintained in the system. Continue charging until the entire contents of the cylinder are charged to the flask as evidenced by a loss of positive pressure. Close stopcock B. Close the valve on the cylinder.

Replace the dry ice surrounding the flask with the mantle heater. Allow the flask to warm to room temperature and turn on the mantle heater at the lowest setting. Slowly increase the rate of heat input until the sample begins to boil. Carefully open stopcock B when a positive pressure is re-established. Maintain a positive pressure in the system by opening and closing stopcock B as the pressure indicates until the pressure remains negative with stopcock B closed. At this point, all of the non-condensable gases have been

separated from the sample and the remainder of the gas will condense in the trap. Close the stopcock on the distillation flask. Disconnect the gas bottle from the trap, leaving the trap connected to the column but vented to the atmosphere. Weigh the sample cylinder and record the mass to the nearest 0.1 g. Calculate the mass of the charge.

In order to adjust the pressure in the gas collecting bottle to atmospheric, raise or lower the second bottle until the levels of the sodium chloride solution in the two bottles are equal. Close stopcocks C and E. Measure the height, in mm, of the solution left in the gas collecting bottle above the discharge point. Disconnect the second bottle and measure the volume of solution displaced. From this volume subtract the volume of solution that was originally in the bottle. Record this value as the uncorrected gas volume. Record the room temperature and the barometric pressure. Determine the mol-percent of each component present in the non-condensable gas fraction by mass spectrometry, gas-liquid chromatography or equivalent method.

If the volume of non-condensable gases exceeds the capacity of the gas-collecting bottle, the distillation will have to be interrupted to change bottles. Close the valve on the cylinder. Return the distillation flask to the dry ice bath, and follow the procedure in the preceding paragraph. Replace or refill the bottle, after taking a sample for analysis, and restart the distillation.

B. Collection of Condensable Gases (Debutanization)

Assemble the apparatus as shown in Fig. 2. Before the sample is charged add dry ice to the light overhead condenser. This will help to establish a good condensable gas reflux in the column. It will also provide a better separation between the C₄ and C₅ components and minimize the amount of C₅ and heavier components distilled into the condensate trap. Once a good reflux is established in the column, as evidenced by a steady drip from the reflux condenser (3 - 4 drops per second), allow the dry ice in the light overhead condenser to vaporize and do not add any more.

Follow the procedure in *Part I Section A* to remove the non-condensable gases. Warm the sample as previously described. When all the non-condensable gases have been removed, and a good reflux established, turn on the time switch set to 30:1 (30 seconds off: 1 second on). The cold liquid reflux will pass into the warm receiver and vaporize. The gas vapors will be drawn into the gas condensate trap and be collected. Continue the distillation until the vapor temperature reaches 10 C (50 F). Place the column on total reflux for 10 minutes by shutting off the time switch. If the vapor temperature drops more than 3 degrees, resume the distillation until it increases to 10 C. Continue alternating between distillation and total reflux until the vapor temperature stabilizes at 10 C for a minimum of 10 minutes. After stabilization is attained turn the time switch on again and distill to 15 C (59 F). Shut off the time switch. Cool the distillation flask to 0 C (32 F) with ice. Disconnect the gas condensate trap from the apparatus. Remove the trap from the Dewar and wipe it free of acetone and dry ice. Quickly weigh the trap and record the mass of the gas condensate fraction. Replace the trap containing the gas condensate fraction in the Dewar flask, add sufficient dry ice-acetone mixture to the Dewar flask to fill it and label the trap with the sample identification. Analyze the gas condensate, employing mass spectrometry, gas-liquid chromatography or an equivalent method.

Weigh and record the mass of the debutanized bottoms. Determine the relative density of the bottoms fraction by ASTM D 1298. Store the debutanized bottoms in a labeled bottle in a refrigerator for further analysis.

Alternatively, follow the procedure for the fractionation and collection of the gas condensate, pentanes and subsequent fractions, if desired, as described for stabilized hydrocarbon samples (*Part II*).

C. Preparation of Samples for Sulfur Analyses

Total sulfur content may be determined on the gas condensate fraction by UOP Method 727. The sample used for this purpose must be transferred from the gas condensate trap by the following procedure.

Select a clean, dry, 75-mL stainless steel cylinder that has been “pickled” according to UOP Method 516. Evacuate the cylinder and weigh it to the nearest 0.1 g. Pack the cylinder in dry ice to cool it. Connect a length of 1/8-inch standard wall stainless steel tubing to the valve. Insert the tubing into the cold condensate, open the cylinder valve and draw 25 - 35 g of the gas condensate into the cylinder. Care must be taken so that the cylinder does not become liquid-full, since a considerable expansion in liquid volume will occur when the condensate is warmed from - 78 C to ambient temperature. Weigh the cold cylinder immediately to the nearest 1.0 g to verify that the quantity of sample in the cylinder does not exceed 35 g. Bleed off a small amount of liquid phase if necessary. Let the cylinder and condensate warm to room temperature and weigh to the nearest 0.1 g to determine the exact mass of condensate in the cylinder. Analyze the sample for total sulfur as soon as possible.

When a hydrocarbon sample contains measurable amounts of hydrogen sulfide and/or mercaptan sulfur, it may be necessary to collect them for analysis. Connect aqueous potassium hydroxide scrubbers, such as described in UOP Method 212, to the system between the column and the gas condensate trap. Bubble the gas through the scrubbers to the vapor temperature of 10 C (50 F), or until there is an indication that the system is approaching sub-atmospheric pressure. Remove the scrubbers immediately, as the caustic solution may otherwise be drawn into the fractionation system. The hydrogen sulfide and mercaptan sulfur in the sample are retained in the potassium hydroxide solution during the scrubbing period. Obtain an analysis of the potassium hydroxide solution as described in UOP Method 212.

Part II - Fractionation of Stabilized Samples (Depentanization)

Included in this category are fractionations at atmospheric pressure of gasoline samples with vapor pressures of less than 15 psig (103 kPa), as determined by ASTM D 323, for removal of C₅ and lighter components. The composition of these light components may be determined by mass spectrometry, gas-liquid chromatography or an equivalent method.

Assemble the apparatus as shown in Fig. 2 and follow the procedure as specified in *PREPARATION OF APPARATUS*. Prepare and attach a clean flask and gas condensate trap, as previously described.

Chill the sample to 0 C (32 F) and determine the relative density according to ASTM D 1298. Weigh and record the mass of the sample and its container. Using a short section of latex tubing, connect the stopcock on the flask to a piece of cold glass tubing placed in the chilled sample. By means of the vacuum in the system draw sufficient sample into the flask for the fractionation. Reweigh the sample container and record the mass of the sample charged.

Open stopcock D to the gas condensate trap to allow collection of any condensable gases present. Turn on the current to the flask heater. Regulate the heat input to the flask to establish and maintain a reflux return rate of approximately 3 to 4 drops per second, observed at the dripper at the bottom of the take-off valve. Set the time switch at 30:1.

Condensable gases distilled from the sample while establishing the proper reflux will be collected in the gas condensate trap. To provide better separation between the C₄ and C₅ components and minimize the amount of C₅ and heavier components distilled into the gas condensate trap, follow the procedure specified in *Part I, Section B* for debutanization. When the vapor temperature reaches 15 C, start the cooling solution through the receiver jacket. This temperature is the initial vapor temperature. Record the temperature of the liquid in the distillation flask.

Continue the distillation until the vapor temperature reaches 40 C (100 F). Place the column on total reflux for 10 minutes by shutting off the time switch. If the vapor temperature drops more than 3 degrees, resume the distillation until it increases to 40 C. Continue alternating between distillation and total reflux until the vapor temperature stabilizes at 40 C for a minimum of 10 minutes. After stabilization is attained, turn the time switch on again and distill to 46 C (115 F). Shut off the time switch. Record the mass of the material in the receiver and transfer it to a chilled bottle. Determine the relative density by ASTM D 1298. Label the sample as "C₅ Fraction" and store in a refrigerator.

Disconnect the gas condensate trap from the apparatus. Remove the trap from the Dewar and wipe it free of acetone and dry ice. Weigh the trap and record the mass of the gas condensate fraction. Replace the trap containing the gas condensate fraction in the Dewar flask, add sufficient dry ice-acetone mixture to the jar to fill it, and label the trap with the sample identification. Analyze the gas condensate, employing mass spectrometry, gas-liquid chromatography or an equivalent method. Alternatively, the trap contents may be blended back into the C₅ fraction.

If no further fractionation of the C₆ and heavier residue remaining in the distillation flask is required, turn off the heat and remove the mantle heater. Allow the flask containing the C₆ and heavier residue to cool to 15.6 C (60 F) and remove it from the column. Wipe the ground-glass joint free of grease, if necessary. Weigh and record the mass of the C₆ and heavier residue, and determine the relative density by ASTM D 4052 or D 1298. Transfer it to a container of suitable size, mark with proper identification and store for further analysis.

Part III - Fractionation of Depentanized Samples

Assemble the apparatus as shown in Fig. 3 and follow the procedure as specified under *PREPARATION OF APPARATUS*. Attach a clean flask of appropriate size to the spherical joint at the bottom of the column. The size of the charge depends upon the yield and analyses desired of any specific fraction and the amount of sample available.

Attach the coil end of a tared glass condensate trap to the gas vent line. Immerse the trap in a Dewar flask filled with a dry ice-acetone mixture. Start the circulation of cold water through the reflux condenser. Evacuate the system. The pressure drop through the column is measured with Manometer A.

Obtain and record the relative density of the sample and the mass of the sample and container. Charge the appropriate amount of sample in the same manner described for stabilized samples in *Part II*. Weigh the sample container and calculate the mass and volume of sample charged.

Establish the proper reflux as prescribed in *Part II*. Turn on the time switch to begin the fractionation and record the vapor temperature and the liquid temperature. Collect the necessary fractions for the required inspections. Record the vapor temperature, the liquid temperature, mass and the relative density for each fraction.

Continue the distillation to minimum bottoms, 5 mL per 1000 mL flask size remaining in the bottom of the flask, or the desired vapor temperature, whichever is reached first. If the sample begins to show signs of cracking, as evidenced by the evolution of fumes or erratic temperature readings, discontinue the distillation and proceed according to *Part IV*.

To discontinue a distillation, shut off the heat and time switch. Allow the column to cool and drain. Record the mass and volume of the last cut after drainage is completed. Remove the flask from the column and weigh. Record the mass of the flask residue and, if there is sufficient material, the relative density. Pour the residue into a storage container and label for identification. Remove and weigh the trap to obtain the weight of any condensate collected.

Part IV - Vacuum Fractionation of High-Boiling Samples

Included in this category are fractionations of samples with initial boiling points above 180 C (350 F) and materials that are unstable at the temperatures attained in an atmospheric distillation and, therefore, must be distilled under vacuum.

Assemble the apparatus as shown in Fig. 4 and follow the procedure specified under *PREPARATION OF APPARATUS*. Charge the sample in the same manner as in *Part II* unless it is viscous. In the latter case, heat it sufficiently to permit transfer directly into a tared flask. Weigh the charge and the flask and attach it to the column. Be sure that a vapor-tight seal is made at the column joint.

Start the flow of water through the reflux condenser. Insert the vapor trap located between Manometer B and the fractionating system into a Dewar flask containing a dry ice-acetone mixture. Attach the straight end of the gas condensate trap, which is submerged in a dry ice-acetone mixture, to the gas vent line. Insert a clean manostat into the system between the surge tank and the vacuum pump. Turn on the vacuum pump. A pressure of 6 to 8 mm (0.80 to 1.1 kPa) is typically used for higher boiling distillates.

Turn on the mantle heater, making adjustments to give the proper reflux rate specified in *Part II*. Collect the necessary fractions for the required inspections. Record the pressure and the vapor temperature converted to 760 mm Hg, in addition to the items specified in *Part III*. Read the distillation pressure above 15 mm by Manometer B, and pressures below 15 mm by the McLeod gauge. Refer to Paragraph A9 of ASTM D 2892 for the equations to convert observed temperature readings to atmospheric equivalent temperature (AET). To change distillate receivers, close off the full receiver from the system and direct the distillate flow into the empty one. Vent the full receiver to atmospheric pressure before disconnecting it. Connect a clean receiver to the distillate changer and evacuate it using a separate vacuum line.

Continue the fractionation until the required overhead temperature is reached or until the sample shows signs of cracking, i.e., the pressure cannot be maintained due to the evolution of fumes. It may be possible to continue the fractionation at a lower pressure. Place the column on total reflux, decrease the heat, readjust the manostat and allow the pump to increase the vacuum. When the desired pressure is attained, increase the heat and re-establish a steady reflux. Resume the fractionation. At the first indication of a pressure increase or a sudden decrease in temperature, discontinue the distillation. Decomposition of the oil will preclude further fractionation under these conditions.

To discontinue a fractionation, shut off the heater switch and remove the heating mantle. Allow the distillation flask to cool while under vacuum for one hour. Disconnect the vacuum line from the gas vent line and replace with a nitrogen line. Using nitrogen, slowly bring the system back to atmospheric pressure. Turn off the nitrogen but leave the line connected. Allow the system to cool for an additional four hours or

until the liquid temperature reaches approximately 50 C. Disconnect the flask. Determine the mass and relative density of the residue. Remove the trap and obtain the mass of the condensate.

Recovery of Static Hold-up

Static hold-up or “wettage” is the quantity of material retained in the column after a distillation is completed. The quantity retained is characteristic of the packing and dependent upon the composition of the material that was refluxing in the column at the time of the final cut. In general, for atmospheric distillations, the amount of hold-up is minimal and may be ignored. For vacuum distillations involving heavy material, however, the amount of hold-up may be substantial and it may be necessary to recover it.

After the column has drained and the flask removed, attach a distillation flask containing 600 to 800 mL of a suitable solvent, typically toluene. Apply heat to the flask and allow the solvent to reflux for 45 minutes to an hour. Turn off the heat and allow the system to cool and drain for one hour. Quantitatively transfer the solution to a tared beaker. In a hood or well-ventilated area, remove the solvent by evaporation on a steam bath. Use a nitrogen purge to aid the evaporation. When all of the solvent appears to have been removed, cool and weigh the beaker. Return the beaker to the steam bath for one hour. Alternate heating the beaker with cooling and weighing until the mass of the beaker remains constant. This indicates that all of the solvent has been removed. Calculate the mass of the hold-up. This material may be treated as a separate small cut, or it may be blended into the residue before further analyses are performed.

CALCULATIONS

Non-Condensable Gases

Calculate the volume of gas collected in the gas sampling bottle as follows:

$$G = \frac{273 V (P_1 - P_2 - P_3)}{760 (273 + T)} \quad (1)$$

where:

G = gas volume corrected to 0 C (32 F) and 760 mm Hg, mL

P₁ = barometric pressure, mm of Hg

P₂ = pressure correction for the vapor tension of the saturated sodium chloride confining solution obtained from Fig. 6, mm of Hg

P₃ = pressure correction for the height of saturated sodium chloride solution in the gas bottle, mm of Hg (Eq. 2)

T = temperature at which the gas was collected, C

V = volume of displaced sodium chloride solution, mL

273 = conversion factor to degrees Kelvin

760 = standard atmospheric pressure, mm of Hg

$$P_3 = \frac{1.20 H}{13.5} \quad (2)$$

where:

H = height of the sodium chloride solution above the discharge point, mm

1.20 = relative density of the solution, g/mL

13.5 = relative density of mercury, g/mL

Calculate the gas volume of each of the gaseous components as follows:

$$C = \frac{GM}{100} \quad (3)$$

where:

C = gas volume of a given component, mL

G = previously defined, mL

M = concentration of the given component in the gas collected, mol-%

100 = percentage constant

Calculate the number of moles of each of the gaseous components as follows:

$$N = \frac{C}{22,400} \quad (4)$$

where:

C = previously defined

N = number of moles

22,400 = ideal gas law constant, mL/mole at 0 C and 760 mm Hg

Calculate the mass of each of the gaseous components as follows:

$$W = DN \quad (5)$$

where:

D = molecular weight, g/mole (See Table)

N = previously defined

W = mass of given component, g

Gas Condensate

Calculate the mass of each of the components in the gas condensate as follows:

$$W_x = \frac{E(D_x M_x)}{\sum D_x M_x} \quad (6)$$

where:

E = mass of contents of gas condensate trap, g

D_x = molecular weight of given component, g/mole (See Table)

M_x = concentration of given component, mol-%

W_x = mass of given component, g

Liquid Fractions

The mass of the liquid fractions is measured directly.

Material Balance

Convert mass to volume at 15.6 C (60 F) as follows:

$$S = \frac{W}{U} \quad (7)$$

where:

S = volume, mL

U = relative density, g/mL (See Table)

W = mass, g

Calculate mass-percent of each distillate fraction and the residue, to the nearest 0.1 mass-% as follows:

$$\text{Distillate fraction or residue, mass - \%} = \frac{100 W}{Z} \quad (8)$$

where:

W = previously defined

Z = mass of total charge, g

100 = percentage constant

Calculate liquid-volume percent of each distillate fraction and the residue to the nearest 0.1 LV-% as follows:

$$\text{Distillate fraction or residue, LV - \%} = \frac{100 S}{Y} \quad (9)$$

where:

S = previously defined

Y = volume of total charge, mL

100 = percentage constant

Calculate percent-loss as follows:

$$L = 100 - X \quad (10)$$

where:

L = loss, mass-%

X = the sum of the mass-% of all fractions and residue

100 = percentage constant

If the percent-loss exceeds 0.5%, follow the procedure for recovery of hold-up if it has not already been done. If the loss still exceeds 0.5%, the distillation must be discarded.

PRECISION

Insufficient data are available to calculate an estimated standard deviation with at least 4 degrees of freedom for the many situations to which this method is applicable.

TIME FOR ANALYSIS

Depending upon the volume of sample distilled, as well as the type of sample, the elapsed time per test may vary from 4 to 90 hours, and the labor requirement, 4 to 20 hours.

REFERENCES

ASTM Standard Test Methods D 323, D 1298, D 2892, D 4052, www.astm.org
UOP Methods 212, 516 and 727

SUPPLIERS

International Products Corporation, P. O. Box 118, Trenton, NJ 08601-0118
Matheson, P.O. Box 96, Joliet, IL 60434
Reliance Glass Works, Inc., P. O. Box 825, Bensenville, IL 60106
Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
UOP Inc., Monirex Div., P. O. Box 5017, Des Plaines, IL 60017
Whitey Co., 318 Bishop Rd., Highland Heights, OH 44143

Table		
Hydrocarbon	Molecular Weight, g/mole	Relative Density, g/mL @ 15.6 C
Methane	16.042	0.262
Ethane	30.068	0.368
Ethylene	28.052	0.371
Propane	44.094	0.5077
Propylene	42.078	0.5218
Isobutane	58.120	0.5631
<i>n</i> -Butane	58.120	0.5844
Isobutylene	56.104	0.6002
1-Butene	56.104	0.6011
<i>cis</i> -2-Butene	56.104	0.6272
<i>trans</i> -2-Butene	56.104	0.6100
Isopentane	72.046	0.6248
<i>n</i> -Pentane	72.106	0.6312
1-Pentene	70.130	0.6457
<i>cis</i> -2-Pentene	70.130	0.6608
<i>trans</i> -2-Pentene	70.130	0.6533
3-Methyl-1-Butene	70.130	0.6325
2-Methyl-2-Butene	70.130	0.6676

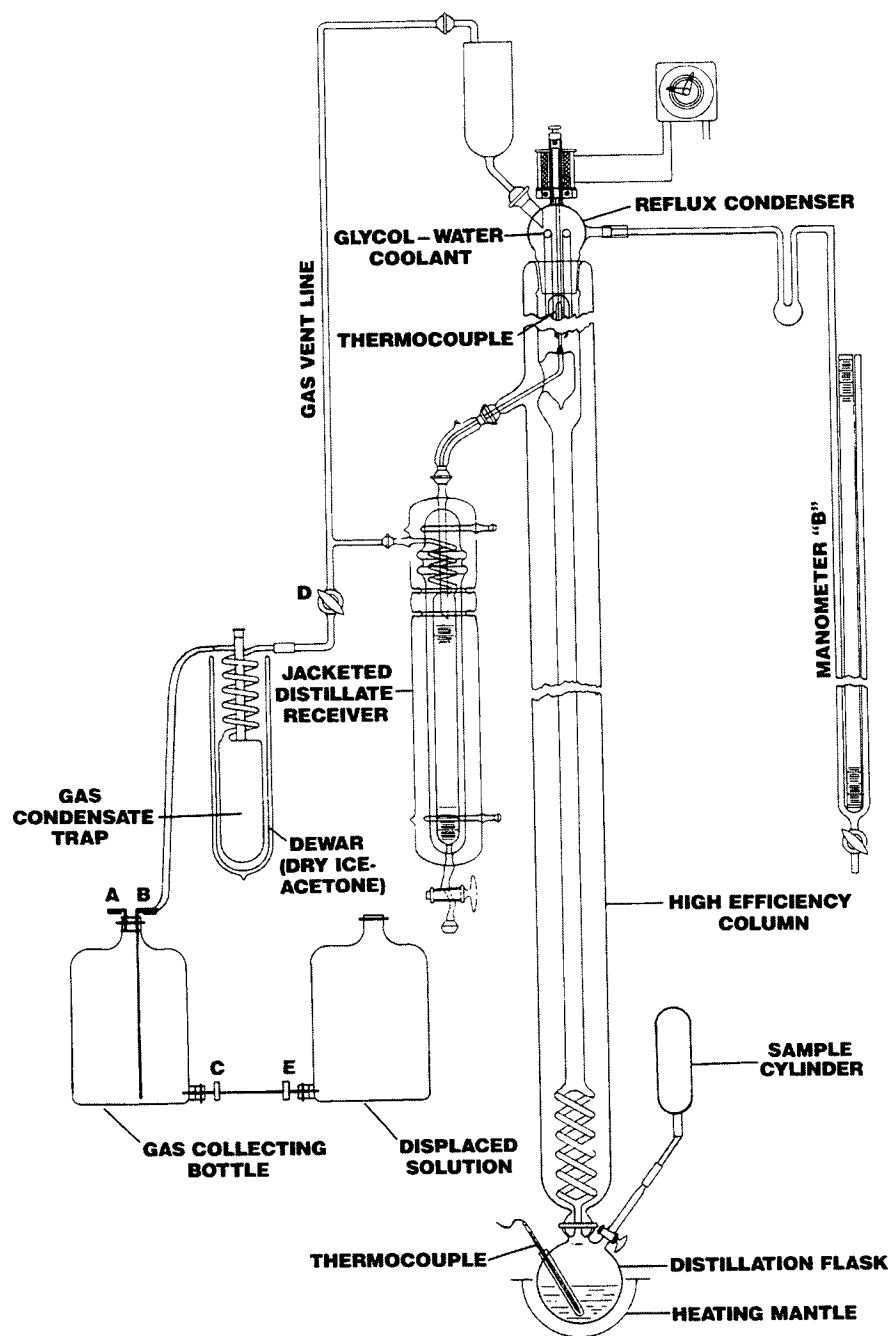


Figure 1
 Fractionation Equipment
 for Non-Condensable Gases

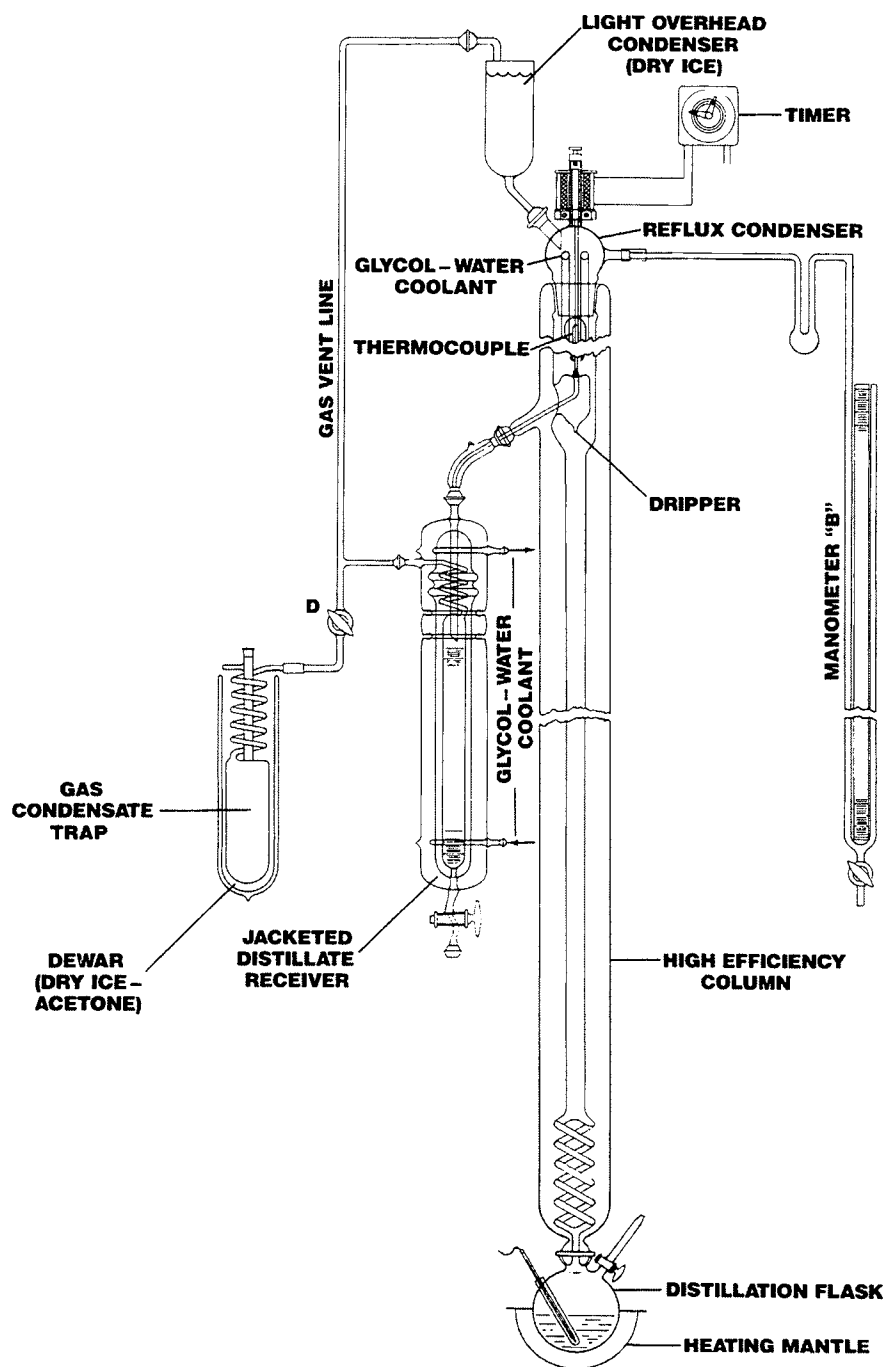


Figure 2

Fractionation Equipment
for Debutanization and Depentanization

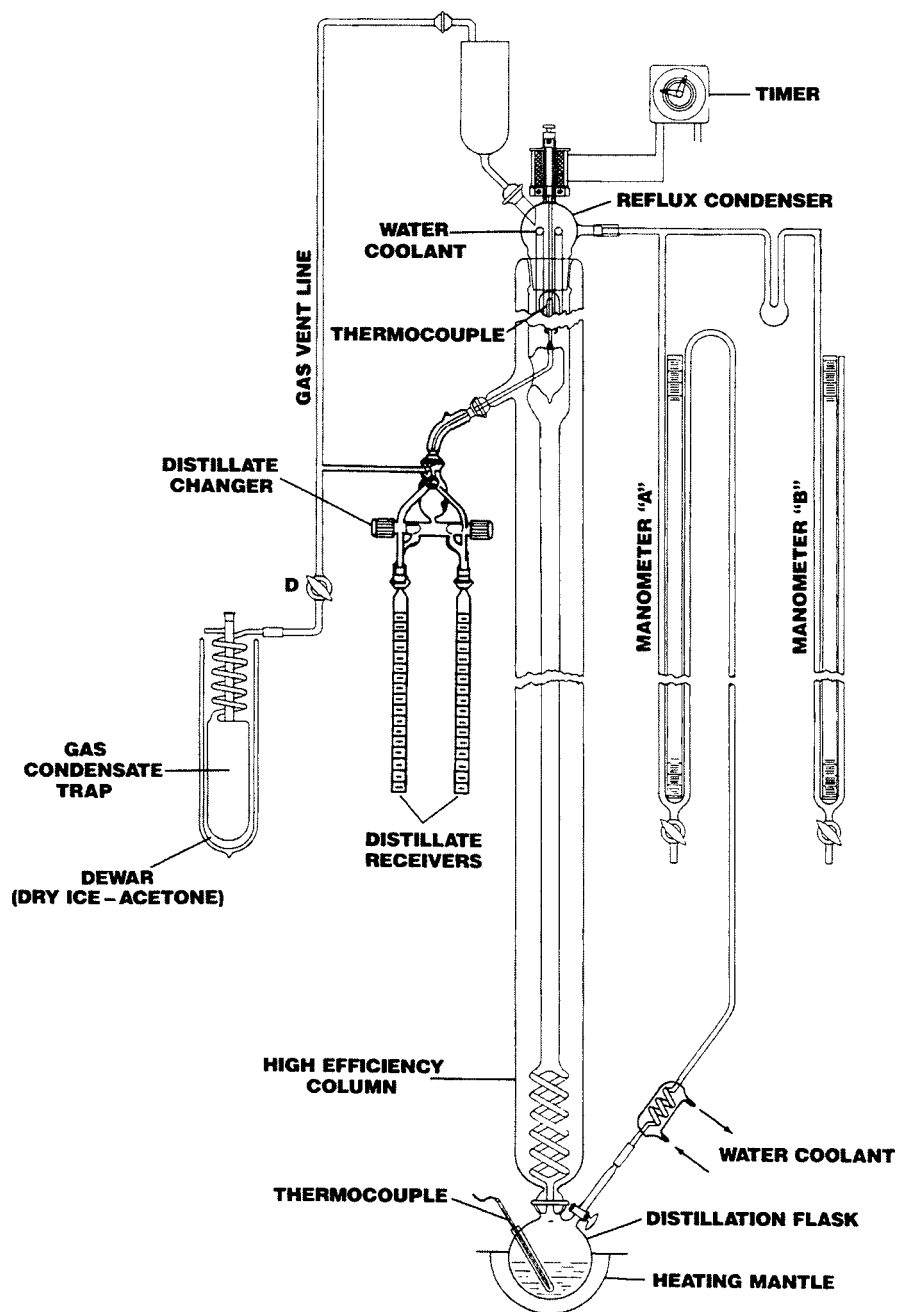


Figure 3

Atmospheric Fractionation Equipment

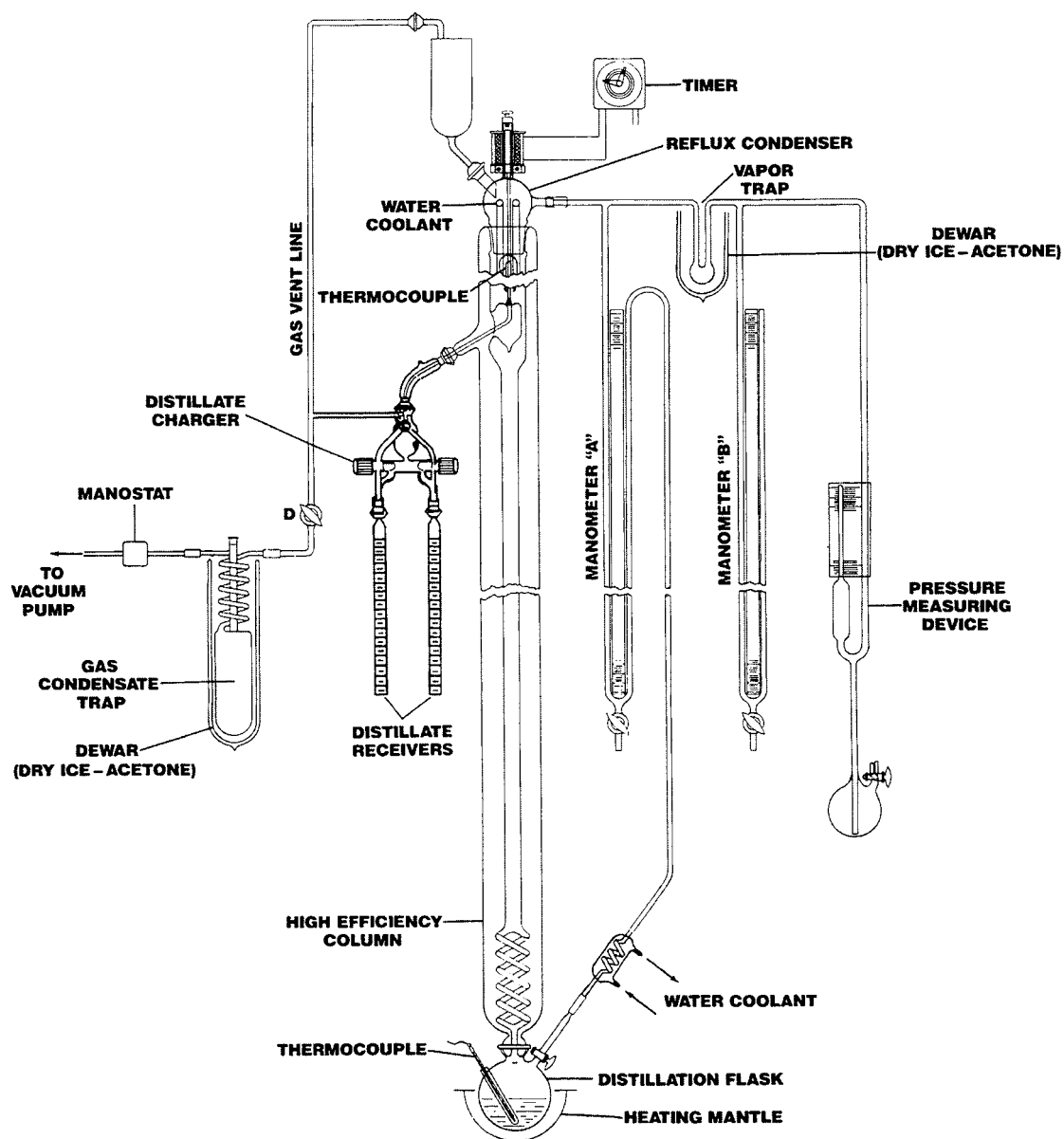


Figure 4

Vacuum Fractionation Equipment

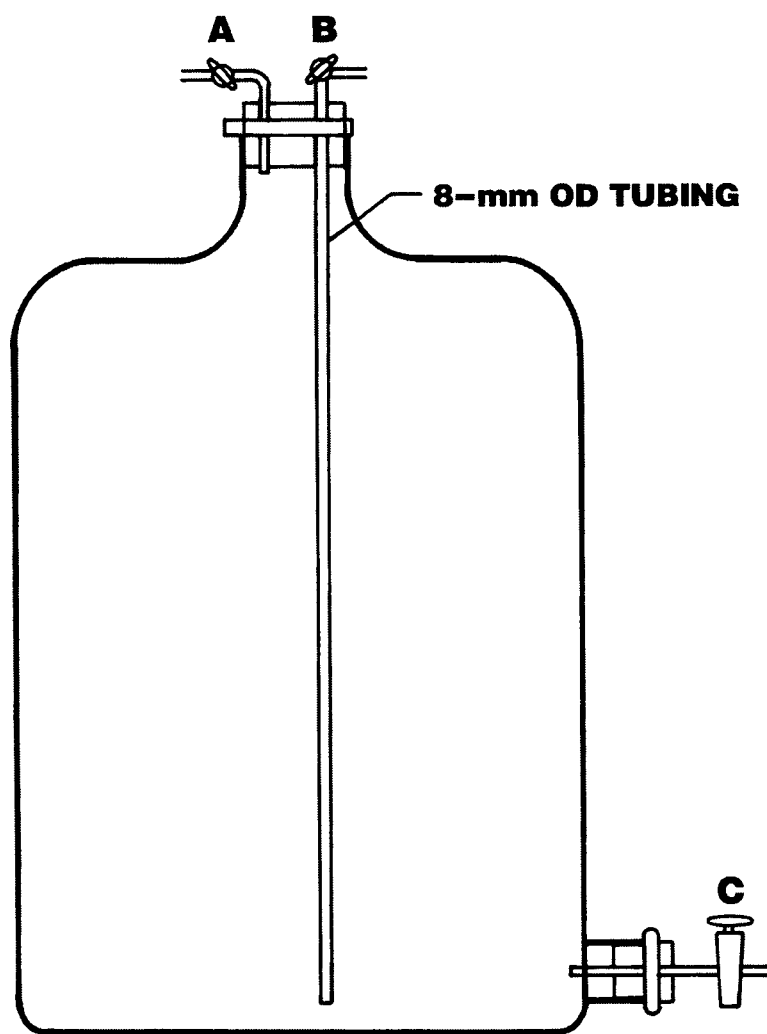


Figure 5
Gas Sampling Bottle

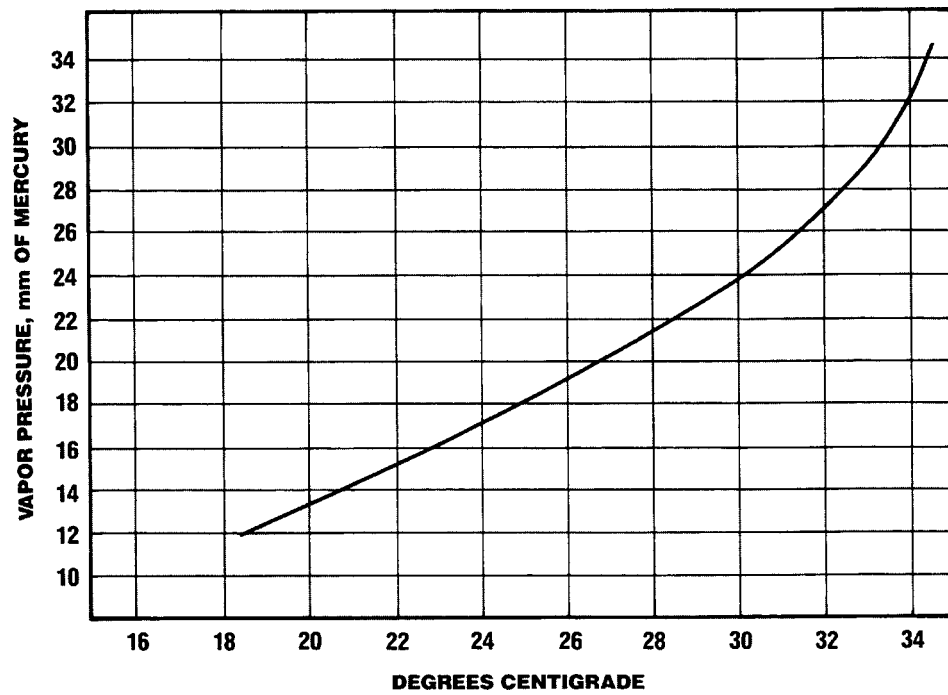


Figure 6

Vapor Pressure — Temperature Curve
For Saturated Sodium Chloride Solution