

# SAMPLING AND HANDLING OF GASOLINES, DISTILLATE FUELS AND C<sub>3</sub>-C<sub>4</sub> FRACTIONS

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## UOP Method 516-00

### SCOPE

This method is for the preparation and cleaning of sample cylinders, and for sampling techniques necessary to obtain an air-free sample of a liquid hydrocarbon. The procedures are applicable to liquefied natural gases, high vapor pressure gasolines, various liquefied petroleum gases, air-unstable gasolines or other liquid hydrocarbon products that require rigorous exclusion of air. Sampling for the analysis of sulfur or other heteroatom compounds in C<sub>3</sub>-C<sub>4</sub> fractions requires a special cylinder or a special cylinder preparation. An alternative to the sampling system and procedure is available as “Closed Sampling System”, UOP, PIC. If the sample is to be transported offsite, see *NOTE 1*.

### OUTLINE OF METHOD

Cylinder cleaning and sampling techniques are specified to obtain a representative, air-free sample in a suitable container, e.g., a double-valved, stainless steel cylinder. For trace sulfur or other heteroatom compound analysis, a Silcosteel<sup>®</sup> coated cylinder is preferred. When coated cylinders are unavailable or impractical, a conventional cylinder is treated (pickled) to make it more stable to sulfur compounds. The clean, leak-free cylinder is mounted vertically near the sampling point and connected to the plant sample line by steel tubing or pipe. The connecting lines are flushed with the sample. After line flushing, the sample is allowed to flow upward through the cylinder. Several volumes of sample are discarded before closing the valves of the cylinder. The cylinders that are used for collection are constructed to provide safe outage.

### APPARATUS

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used, unless stated otherwise.

*Cylinder*, C<sub>3</sub>-C<sub>4</sub> fractions, type 304 stainless steel, high pressure, 12,400 kPa, gauge (1800 psig) maximum working pressure, one-liter capacity, Whitey, Cat. No. 304L-HDF4-1000, or equivalent. Equip the cylinder with outage tube, Whitey, Cat. No. 304L-DTM4-F4-038, two stainless steel valves, Whitey, Cat. No. SS-16DKM4-F4, and two plugs, Whitey, Cat. No. SS-4-P, or equivalent. Smaller cylinders, such as 500-mL, may be used when large quantities of sample are not required.

**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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*Cylinder*, C<sub>3</sub>-C<sub>4</sub> fractions, Silcosteel coated, for trace sulfur and other applications which require a more inert surface than stainless steel, 1000 mL, Restek, Cat. No. 24275. Equip the cylinder with two Silcosteel coated valves, Restek, Cat. No. 24278, an outage tube, Whitey, Cat. No. 304L-DTM4-F4-038, and two caps, Whitey, Cat. No. SS-4-CP. Ship the outage tube to Restek for Silcosteel coating, Restek, Cat. No. 54852. Caustic materials and strong acids will damage the Silcosteel surface. Samples and cleaning procedures must be caustic and acid free.

*Cylinder*, gasoline and distillate fuels, type 304 stainless steel, 690 kPa, gauge (100 psig) maximum working pressure, Code No. 2, 28-L (7-gal) capacity, with dip tubes, equipped with 2 stainless steel valves. The cylinder without valves is available from Alloy Products, or equivalent. Valves are available from Whitey, Cat. No. SS-1KF4, or equivalent. The cylinder, as received, will be equipped with a long dip tube; the short dip tube, made from 1/4-inch OD stainless steel, must be fabricated and welded to the valve coupling extending into the cylinder approximately 14 cm. Air-free samples of gasoline or distillate fuel oils to be used for treating, oxidation or corrosion studies are typically taken in this cylinder. **CAUTION:** This cylinder cannot be used for samples having a vapor pressure above 275 kPa, gauge (40 psig) at 21°C (70°F).

*Cylinder Sampling Assembly*, see Fig. 3, required for sampling gasolines or distillates

*Fittings*, pressure-rated tubing or pipe made of stainless steel for use as transfer and manifold lines with appropriate valves, tees and unions or couplings of these materials. **CAUTION.** Do not use brass, aluminum or copper pipe, tubing, valves, unions, ferrules, or couplings.

*Grounding cable*, heavy gauge wire or cable connected to a water pipe or to a rod driven into the ground, according to local codes, having a suitable spring clamp to attach it to the metal cylinder; necessary to prevent explosion due to static electricity.

*Oven*, gravity convection, capable of maintaining 150°C, Fisher Scientific, Cat. No. 13-247-650G

*Pail*, polyethylene, 13-L capacity, Fisher Scientific, Cat. No. 03-687-10

*Regulator*, nitrogen, delivery pressure 345-4480 kPa, gauge (50-650 psig), Matheson, Model No. 2-580, or equivalent

*Regulator*, nitrogen, two-stage, delivery pressure 28-690 kPa, gauge (4-100 psig), Matheson, Model No. 3104-580, or equivalent

*Thermometer*, Fisher Scientific, Cat. No. 15-077-2B

*Vacuum pump*, of sufficient capacity to evacuate the cylinder to the extent that the cleaning solution may be drawn into the cylinder

## REAGENTS

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

*Acetone*, 99.5% minimum purity, Fisher Scientific, Cat. No. A949SK-4

*Ferric chloride*, Fisher Scientific, Cat. No. I89-500

*Hydrochloric acid*, 1:1, Fisher Scientific, Cat. No. LC15130-3

*Isooctane*, 99.5%, low residue, Fisher Scientific, Cat. No. O-301-4

*Leak detector*, solution, Leak-Check, Alltech Associates, Cat. No. 2025, or equivalent

*Methanol*, 99.8% minimum purity, Fisher Scientific, Cat. No. A408SK-4

*Methylene chloride*, 99.9%, low residue, Fisher Scientific, Cat. No. D151-4

*Nitrogen*, 99.99% minimum purity

*Pentane*, 99%, low residue, Fisher Scientific, Cat. No. P399SK-4

*Pickling solution*. Prepare by dissolving  $10 \pm 0.1$  g of ferric chloride in one liter of 1:1 hydrochloric acid.

*Steam*, local supply

*TAM(toluene-acetone-methanol) solution*. To prepare, mix equal portions of toluene, acetone and methanol.

*TI(toluene-isooctane) solution*. To prepare, mix equal portions of toluene and isooctane.

*Toluene*, 99.8% minimum purity, Fisher Scientific, Cat. No. T290SK-4

*Water*, deionized or distilled

*Water*, tap, hot

## PREPARATION OF APPARATUS

### Cleaning and Drying Cylinders

Proper preparation and cleaning of sample cylinders is of paramount importance. In no case may an improperly cleaned cylinder be used as a sample container. Clean and prepare cylinders as described.

### Cylinders for C<sub>3</sub>-C<sub>4</sub> Fractions

#### A. Silcosteel Coated Stainless Steel, or Conventional Stainless Steel if No Sulfur or Other Heteroatom Analysis Is Required

1. Completely empty the cylinder of any residual material in a well ventilated area or fume hood.
2. If the cylinder contained aqueous material, partially fill the cylinder with deionized or distilled water, agitate and drain. Follow the same procedure with methanol.
  - Dispose of all used cleaning solutions in an environmentally safe manner.
3. Partially fill the cylinder (about 1/4) with TI solution, agitate and drain. Follow the same procedure sequentially with pentane, methylene chloride and methanol.