

COMBINED NITROGEN IN LPG BY CHEMILUMINESCENCE

UOP Method 936-95

SCOPE

This method is for determining combined nitrogen in liquefied petroleum gas (LPG). The method determines all nitrogen compounds that are extractable by aqueous acid, including ammonia, amines, nitriles and amides. The method is particularly useful for LPG samples containing nitrogen species, such as ammonia and amines, which can readily adsorb on stainless steel surfaces in contact with the LPG. The procedure yields quantitative results in the range of approximately 0.1 mass-ppm to approximately 3 mass-%.

OUTLINE OF METHOD

Nitrogen compounds are quantitatively extracted from an LPG sample using a solution of 2-N acetic acid in water. A measured mass of LPG is collected in a cleaned, stainless steel, sample cylinder. A known amount of acid solution is added to a second cylinder, the two vessels are connected and the acid solution is transferred under helium pressure into the LPG sample cylinder. The nitrogen components are extracted into the acid solution by mixing the liquids for several minutes. The LPG and acid extract are allowed to separate. The acid extract is withdrawn and analyzed for total nitrogen by chemiluminescence, utilizing ASTM Method D 4629 with the optional boat inlet system.

APPARATUS

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

Adapter, male, 2 required, for charging acetic acid cylinder, available from Dearborn Valve and Fitting, Swagelok, Cat. No. SS-4-TA-1-4 (see also *Connector*)

Balance, readability 0.1-mg

Balance, readability 0.1-g, capacity 4000-g

Beaker, polypropylene, 100-mL, Fisher Scientific, Cat. No. 02-591-17B

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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UOP Methods are available through ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959, United States. The Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Calculator, capable of least squares linear regression calculation, required only if the nitrogen analyzer does not permit direct calibration and direct reading of the nitrogen in the extract

Clamps, for ring stand and sample cylinders, several required, with holders and cast iron ring, Fisher Scientific, Cat. Nos. 05-769-8, 05-754 and 14-050C, respectively

Connector, stainless steel, used to connect the 300-mL and the 1000-mL cylinders vertically together and consisting of a male connector, Swagelok, Cat. No. SS-400-1-4 joined together with one male adapter, Swagelok, Cat. No. SS-4-TA-1-4. All parts are available from Dearborn Valve and Fitting.

Cylinders, for sample collection and analysis, type 304 stainless steel, high pressure, 12,400 kPa gauge (1800 psi) maximum working pressure, 1000- and 300-mL capacity, Whitey, Cat. Nos. SS-304L-HDF4-1000 and -300, respectively. Equip the cylinders with two stainless steel valves each, Whitey, Cat. No. SS-16DKM4-F4, and two pipe-plugs each, Cajon, quarter-inch P-male-NPT, Swagelok, Cat. No. SS-4-P. All parts are available from Dearborn Valve and Fitting.

Cylinders, graduated, borosilicate glass, 50-, 100- and 250-mL, Fisher Scientific, Cat. Nos. 08-556C, D and E, respectively

Extractor (optional), special construction, see *APPENDIX*

Flasks, volumetric, 100-, 1000- and 2000-mL, with caps, Fisher Scientific, Cat. Nos. 10-210-5C, -5G and -5H, respectively

Gauge, pressure, 0-20, 700 kPa gauge (0-3000 psig), 2-inch diameter, 0.25-inch MNPT, used for testing the pressure of an unknown LPG sample gas cylinder, Matheson Gas Products, Model 63-2233

Jar-Mill (optional), 20-300 rpm, Model 755 RMV, Fisher Scientific, Cat. No. 08-381-1. Required only if optional extractor is used.

Nitrogen analyzer, total nitrogen by chemiluminescence, with boat control system for aqueous sample, Mitsubishi Instruments, TN-10, available from Cosa Scientific

Oven, drying, capable of maintaining 105°C

Pipet, Mohr, 1-, 5- and 25-mL, Fisher Scientific, Cat. Nos. 13-665-F, -K and -N, respectively

Quartz beads, -10 to + 60 mesh, Pyromatics, Cat. No. 7323

Regulators, helium, two-stage, Matheson Gas Products, Models 3104C-580 (used for the nitrogen analyzer) and 3030-580 (used for acetic acid transfer between cylinders)

Regulator, oxygen, two-stage, high purity, Matheson Gas Products, Model 3813-CGA-540, cleaned for oxygen service by the supplier, used with Nitrogen Analyzer

Ring stand, with rectangular base, 170- x 280-mm base with 910-mm rod, Fisher Scientific, Cat. No. 14-670D

Sample vials, for extracted sample, Kimble Scientific Products, Cat. No. 60957-1, with Quorpack black phenolic caps and polyseal liners, Cat. No. 02-883-5G, available from Fisher Scientific

Syringe, 50- μ L, Hamilton, Model 705N, Fisher Scientific Products, Cat. No. 14-824-5

Tubing, polyethylene, "POLYFLOW", for charging acetic acid cylinder, 6.4-mm (0.25-inch) ID, Lesman Instruments, Cat. No. 44-P-N

Tubing, Tygon, R3603, for charging acetic acid cylinder 6.4-mm (0.25-inch) ID, Fisher Scientific, Cat. No. 14-169-3C

Tubing, vacuum, 1/4-inch ID (6.4-mm), Fisher Scientific, Cat. No. 14-173C

Vacuum pump, aspirator, Nalgene, Fisher Scientific, Cat. No. 09-960-2

Valve, check, 20,700 kPa gauge (3000 psig), Matheson Gas Products, Model 400V

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.

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

Acetic acid, approximately 99.7%, Fisher Certified, Fisher Scientific, Cat. No. A38-212

Acetic acid, approximately 2-N. Prepare two liters by slowly adding 232 mL of concentrated acetic acid from a graduated cylinder to approximately 1500 mL of water in a 2000-mL volumetric flask. After the heat of dilution dissipates, dilute to volume with water and mix thoroughly by capping the flask and inverting at least 10 times.

Acetone, Fisher Certified, Fisher Scientific, Cat. No. A18-4

Helium, Ultra High Purity, 99.999%, Matheson Gas Products

Isooctane, Certified, Fisher Scientific, Cat. No. 0299-4

Isooctane-acetone, 9 to 1, Measure 900 mL of isooctane into a 1000-mL volumetric flask and fill with acetone to the mark. Cap and mix thoroughly by inverting at least ten times.

Methanol, Fisher Certified, ACS, Fisher Scientific, Cat. No. A412-4

Methanol-acetone, 1 to 1. Measure 500 mL of acetone with a graduated cylinder into a 1000-mL volumetric flask and fill to the mark with methanol. Cap the flask and mix by inverting at least ten times.

Oxygen, 99.98%, Ultra-High Purity, Matheson Gas Products

pH paper, CF strips, 0 to 14 pH units, Fisher Scientific, Cat. No. 09-876-17

Pyridine, 99.9%, Aldrich Chemical, Cat. No. 27,040-7

Sulfuric acid, reagent grade, ACS, 95.5%, Fisher Scientific, Cat. No. A300-212

Sulfuric acid, 10%. Prepare one liter by slowly adding 100 mL of concentrated sulfuric acid from a graduated cylinder to approximately 800 mL of water in a 1000-mL volumetric flask while cooling in ice water. After the heat of dilution dissipates, dilute to volume with water. Mix thoroughly by capping the flask and inverting at least 10 times.

Teflon-tape, PTFE, roll, 12.5-mm (0.5-inch), Fisher Scientific, Cat. No. 14-831-300A

PROCEDURE

Cylinder Preparation

1. Clean the interior surfaces of the 1000-mL LPG sample cylinder and valves as follows: Rinse the cylinder with successive 100-mL portions of the isooctane-acetone mixture until the rinse is colorless and follow with 100-mL rinses of the methanol-acetone mixture until the rinse is colorless. Rinse with one 100-mL portion of the 10% sulfuric acid solution. Rinse with water until the rinse is neutral to the pH paper. Appropriately discard the washes.
2. Remove the water by rinsing the 1000-mL cylinder and valves with two successive 100-mL portions of the methanol-acetone and discard the rinses.
 - Rinsing with the solvents, in the order given, removes adsorbed nitrogen compounds. Any convenient procedure that assures contact of the wetted valve and cylinder surfaces with solvents may be used. One workable procedure is outlined in Note 1.
3. Flush the cylinder with helium to remove the remaining solvents and then dry in the drying oven for at least one hour at 105°C.
 - Nitrogen may be substituted for the more expensive helium in this step.
4. Allow the cylinder to cool to room temperature. Tare the cylinder and record the tare mass to the nearest 0.1 g.
 - The weight of the cylinder should not include any detachable parts, such as end-caps, tags, etc. to assure that an accurate mass is recorded.

Sample Collection

1. Collect sufficient LPG in the 1000-mL sample cylinder to fill it approximately half full with liquid, according to UOP Method 516.
 - In no case should a cylinder be filled completely with liquid because the hydrostatic pressure in the cylinder will exceed the rated pressure of the cylinder, if the liquid expands after filling.
 - The mass of the LPG will be approximately 250 g, but should never exceed 300 g when the cylinder is half full.

- The LPG sample size should be adjusted at the source, based on the nitrogen concentration. A sample size of 500 mL was selected to analyze samples containing 300 mass-ppm N or less. If the nitrogen content is greater than 300 mass-ppm, the LPG sample size must be adjusted as shown in Table 1. Higher nitrogen concentrations also require the use of more extractant, as described later in the method.
- The ratio of extractant to LPG must never be less than the values shown in Table 1. If larger volumes of extractant are injected accidentally, the analysis is still accurate.

Table 1
Quantities of LPG and Extractant to Use
for Various Nitrogen Concentrations

<u>Mass LPG, g</u>	<u>N, mass-ppm</u>	<u>Extractant, g</u>
250	0.1 to 300	50 g
50	300 to 3,000	100 g
25	3,000 to 30,000	250 g

2. Measure and record the pressure in the half-filled 1000-mL cylinder using the pressure gauge (0-20,700 kPa) and indicate the pressure on a tag to be attached to the cylinder.
 - The pressure of the sample cylinder should not exceed 500 psi. If the pressure is greater than 500 psi, the sample is likely not pure LPG and should not be analyzed. Consult with the plant engineer or originator of the sample.
3. Reweigh the cylinder and record the LPG sample mass, to the nearest 0.1 g, on a tag to be attached to the cylinder as the final cylinder mass minus the tare mass from *Cylinder Preparation*, Step 4.

Extraction

1. Clean the 300-mL cylinder in the same manner as directed for the 1000-mL cylinder under *Cylinder Preparation*, Steps 1 through 4.
2. Mount the 300-mL cylinder vertically on the ring stand with the clamps and attach a vacuum hose to a male adapter at the open valve at the top of the cylinder, but keep the bottom valve closed. Apply vacuum to the cylinder at the open top valve.
3. Note the appropriate volume of 2-N acetic acid solution to be added to the 300-mL cylinder based on the nitrogen content of the LPG and Table 1 and measure the acid into an appropriately sized graduated cylinder.
4. Attach a piece of Tygon and polyethylene “POLYFLOW” tubing to the male adapter at the bottom valve, so that the polyethylene tubing is submerged at the bottom of the graduated cylinder containing the appropriate volume of acetic acid and slowly open the bottom valve to withdraw the acetic acid with vacuum into the 300-mL cylinder. Next, close first the lower, then the upper valve and disconnect the vacuum hose.
 - Take care to control the rate of withdrawal of the acetic acid into the evacuated 300-mL cylinder so that none of the acid is aspirated out of the opened top valve.

5. Assemble the cylinders attached to the helium tank as shown in the Figure and pressurize the 300-mL cylinder with helium, until the pressure in the 300-mL cylinder exceeds the pressure in the 1000-mL cylinder by approximately 340 kPa gauge (50 psig).
 - The pressure in the LPG sample cylinder was recorded in *Sample Collection*, Step 2. If no record is present, the pressure must be measured by attaching the cylinder to the ring stand in a vertical position, attaching a gauge to the top of the cylinder, and measuring the pressure. For this method, the pressure should not exceed 500 psi, but in no case should the pressure in the cylinder exceed the safe operating pressure of the vessel, typically 12,400 kPa (1800 psig).
 - The two cylinders are attached vertically to a ring stand, positioned in a hood, using the clamps. The bottom valve of the top cylinder is attached to the top valve of the lower LPG sample cylinder with the straight stainless steel connector. Any substitute connector tube should be no more than 51 mm (2 inches) in length and the shortest possible section of straight, stainless steel tubing and fittings should be used to minimize hold up of the acetic acid. Ensure that all fittings are leak free at the pressure in the 300-mL cylinder. The cylinder assembly is shown in the Figure, without indicating the location of the ring stand and clamps.
6. Open the bottom valve of the top cylinder slowly.
7. Open the top valve of the 1000-mL LPG cylinder.
 - The sound of the 2-N acetic acid solution being forced into the LPG sample should be audible.
8. Close the top valve of the LPG sample cylinder and then close the helium cylinder valves.
 - Development of heat of the sample cylinder is not expected. If, however, the cylinder should warm up due to caustic or other impurities in the sample, immediately stop the analysis and consult with the plant engineers or originators of the sample.
9. Disconnect the helium tubing connection at the top valve of the 300-mL cylinder slowly to relieve pressure. Disconnect the 300-mL cylinder from the LPG cylinder. Remove the LPG cylinder from the ring stand and reweigh the LPG cylinder to determine the mass of the acid added (± 0.1 g).
 - The weight of the cylinder should not include any detachable parts, such as end-caps, tags etc., to assure that an accurate mass is recorded.
 - The 1000-mL LPG cylinder is plugged at both ends after weighing, using the stainless steel plugs.
10. Mix the LPG and acid mixture thoroughly by inverting and rotating the cylinder for at least 30 minutes. Use a rocking, rotating motion to thoroughly extract the nitrogen compounds or use a mechanical extractor (See Note 2).
 - Nitrogen species can adsorb on the walls of the cylinder, therefore the acetic acid solution must contact the entire interior surface.
11. Reattach the 1000-mL LPG sample cylinder to the ring stand in a vertical position, and allow the contents to separate and then remove the end caps.
 - Separation can occur in 10 minutes. However, the individual LPG will affect the length of time required for separation and some experimentation may be needed to establish a suitable setting time. Longer periods are required for LPG containing ethers, alcohols or other non-paraffinic impurities.
12. Drain the acid extract from the LPG sample cylinder by partially opening the bottom valve of the 1000-mL cylinder into a clean, dry, appropriately sized volumetric flask placed under the valve.
 - The sample pressure will force the separated acid extract out of the cylinder. If the valve is opened too far, the acid solution may spatter and appropriate safety shield, goggles, etc. must be used for personal protection. Usually the acetic acid drains as a milky acid-gas emulsion, which separates easily in a few minutes.

13. Decant a portion of the acid extract from the volumetric flask into a 15-mL sample vial and analyze the acid extract on the chemiluminescence nitrogen analyzer as directed in ASTM Method D 4629 and according to the following procedure.

Instrument Set-Up

The following steps are specific to the Mitsubishi TN-10 Chemiluminescence Analyzer. If another instrument is used, follow that manufacturer's recommended procedure.

1. Install the nitrogen analyzer with the optional boat inlet system as recommended by the instrument manufacturer.
2. Set the parameters of the instrument to analyze aqueous acid solutions, as prescribed by the instrument manufacturer.
 - The recommended temperatures for the tube-furnace of the Mitsubishi TN-10 are 850°C at the inlet and 950°C for the catalyst.
3. Replace the septa.
 - The septa should be replaced after about every 20 injections.
4. Set the main oxygen flow to 300 mL/min, helium flow to 0.5 L/min, and the secondary oxygen flow to 0.5 L/min.
5. Turn on the vacuum pump for the Mitsubishi TN-10 Analyzer.
6. Turn on the ozonizer.
 - To prevent damage of the quartz cell, do not turn on the ozonizer until the vacuum pump is turned on and the gas flows are set.
7. Fill the sample boat approximately half full with quartz beads.
8. Prepare the boat by manually pushing it into the furnace and allowing it to remain there for 5 minutes.
9. Pull the boat manually back into the cooling chamber and allow it to cool for 5 minutes.

Calibration

1. Prepare a standard stock solution containing approximately 500 mass-ppm of nitrogen by weighing 0.285 g, to the nearest 0.001 g, of pyridine into a 100-mL volumetric flask containing approximately 90 mL of 2-*N* acetic acid. Bring the solution to volume with more 2-*N* acetic acid and mix thoroughly.
 - The density of the stock solution is 1.0 g/mL and it is stable for at least eight weeks.
 - Calculate the exact concentration of nitrogen and mark it on the flask.

2. Prepare a series of three working standards by serial dilution of the stock solution to span the concentration range of nitrogen expected in the sample(s).
 - The working standards are prepared using a Mohr pipet, volumetric flasks and additional 2 N acetic acid.
 - The range of the working standards should be minimized to avoid non-linearity in instrument response. The three standards should bracket the unknown and the total range for the standards should not exceed 3-4 times the lowest standard.
3. Set the instrument to the calibration mode.
4. Use the working standards to generate a calibration curve directly in the instrument, according to the instrument manufacturer's instructions.
 - A few instruments do not permit direct calibration. Then, *Calibration* must be performed by recording the individual instrument responses of the calibration standards and proceeding as indicated in *CALCULATIONS* before proceeding with Step 5.
 - The average of at least three analyses of each standard must be used for each calibration point.
 - The correlation coefficient should be 0.9995 or higher.
5. Check the calibration curve by analyzing one of the standards as a sample. This standard check should be performed after every 5 samples.
 - The standard concentration should check within 3% relative, otherwise the *Calibration* must be repeated beginning with Step 1.

Sample Analysis

1. Set the instrument to the analyze mode.
2. Enter "1", as the density of the acetic acid sample.
3. Enter the volume of acetic acid that is to be injected.
 - Approximately 30 μ L of sample (or standard) is recommended for an injection volume.
4. Enter the dilution volume and the original sample extract volume diluted, if dilution of the acetic acid extract is necessary.
 - If the nitrogen concentration of the extract exceeds the concentration of the highest standard, the extract must be serially diluted, using Mohr pipet, volumetric flasks and 2-N acetic acid until the standards span the concentration of the diluted extract.
5. Inject the sample and read the nitrogen content of the extract directly, according to the instrument manufacturer's instructions.
 - Some instruments do not permit direct reading of the nitrogen in the acetic acid extract. Then, the instrument response of the sample must be recorded and the nitrogen content of the extract determined as indicated in *CALCULATIONS*.
 - 5-6 samples may be injected in triplicate (maximum 20 injections) before the septum should be replaced as otherwise gas leaks will cause non-reproducibility.

CALCULATIONS

Verify that the acetic acid sample concentration is spanned by the calibration standards. Most instruments permit direct calibration as described in *Calibration* and report the nitrogen concentration directly in mass-ppm as described in Step 5 of *Sample Analysis*. If an instrument is used that does not have these capabilities, the *Calibration* must be performed using the individual instrument responses for the calibration

standards. Then, the best straight line calibration must be calculated, using a calculator or computer. Similarly, the sample response must be read from the instrument, and the concentration of the extract must be calculated manually with the calculator using the calibration curve, the instrument response for the sample, and any dilution factor that was used.

Calculate the concentration of the combined nitrogen in the LPG sample in mass-ppm, as follows, rounding the result to two significant figures at or above one mass-ppm, and to one significant figure below one mass-ppm. Concentrations at or above 10,000 mass-ppm can be converted to mass-% by dividing the mass-ppm value by 10,000.

$$N = \frac{SM}{L}$$

where:

L = mass of the LPG, g

M = mass of the acetic acid extract added to the LPG cylinder, g

N = concentration of the combined nitrogen in the LPG, mass-ppm

S = combined nitrogen concentration of the acetic acid extract, as read from the instrument, mass-ppm

NOTES

1. Cleaning of the interior, wetted surfaces of the cylinders and attached valves with the cleaning solvents can be performed as follows:
 - a. Mount the cylinder vertically on the ring stand in the hood and open both valves.
 - b. Evacuate the cylinder, by pressing a vacuum hose against the top cylinder valve by hand to create some vacuum in the cylinder.
 - c. Withdraw the solvent into the cylinder by submersing the bottom valve in the solvent held in the 100-mL polypropylene beaker. Close first the bottom, then the top cylinder valve.
 - d. Remove the cylinder from the ring stand and rotate and invert the cylinder manually for about two minutes.
 - e. Reattach the cylinder to the ring stand and place the polypropylene beaker under the lower valve. Open both valves to drain the solvent from the cylinder and observe the color. Then discard the rinse.
 - f. Repeat the rinse with the solvent until the rinse is colorless. Then repeat Steps a through f, alternating the valves that are cleaned, until all the solvent required has been used to clean the cylinder and valves.

2. The extraction or cleaning procedure can be automated by using any commercial mixer that inverts the cylinders regularly as described. A home-made device for the extraction can be constructed as described in the *APPENDIX*.

PRECISION

Repeatability

Based on two tests performed by each of two analysts on each of two different days (8 tests), the within-laboratory estimated standard deviation (esd) was calculated to be 0.08 mass-ppm nitrogen at the 5.9 mass-ppm level. Two tests performed in one laboratory by different analysts on different days should not differ by more than 0.3 mass-ppm (95% probability) at the 5.9 mass-ppm level.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

TIME FOR ANALYSIS

The labor required for the analysis is 2 hours and the elapsed time is also 2 hours.

REFERENCES

ASTM Method D 4629, www.astm.org

UOP Method 516, "Sampling of Gasolines, Distillate Fuels and C₂-C₄ Fractions"

SUGGESTED SUPPLIERS

Aldrich Chemical Co., 1001 W. St. Paul Ave., Milwaukee, WI 53233 (414-273-3850)

Cosa Scientific, 55 Oak St., Norwood, NJ 07648 (201-767-6600)

Dearborn Valve and Fitting, 1540 Old Rand Rd., Wauconda, IL 60048 (708-526-6900)

Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219-4785 (412-562-8300)

Lesman Instruments, 215 Wrightwood Ave., Elmhurst, IL 60126 (708-834-2200)

Matheson Gas Products, 932 Paterson Plank Rd., P.O. Box 85, East Rutherford, NJ 07073 (201-933-2400)

Pyromatics Inc., P.O. Box 764, 3985 Ben-Hur Ave., Willoughby, OH 44094 (216-951-3985)

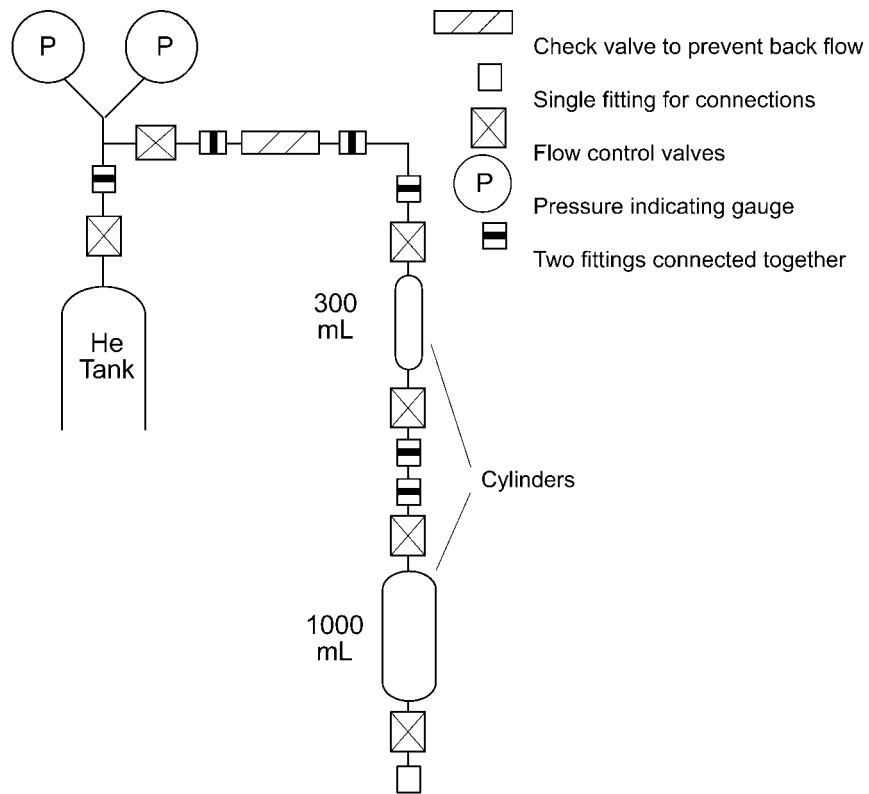


Figure
Cylinder
Assembly

APPENDIX

MATERIALS

Extractor, (optional), special construction, consisting of the following:

Hex nuts, plain steel, grade 8, McMaster-Carr, Cat. No. 90499A033, 16 required

Polycarbonate sheet, “Hyzod”, general purpose, ½-inch thickness, Piper Plastics, Cat. No. GP-9100

Rod, alloy steel, B-7, coarse thread, ½-inch-13, approximately 4 m (158 inches) required, cut to length, McMaster-Carr, Cat. No. 98957A636

Rubber-Mat, V-rib, corrugated, heavy duty, 0.25 inch thickness, McMaster-Carr, Cat. No. 6973T12

Washers, flat steel, 24 needed, McMaster-Carr, Cat. No. 90108A033

Wing-nuts, iron, diameter ½-inch-13, 8 needed, McMaster-Carr, Cat. No. 90877A033

PROCEDURE

Any machine or carpenter shop familiar in the arts can fabricate the optional extractor as follows:

1. Trace two 500-mm (20-inch diameter) circular patterns on a rigid material, such as heavy polycarbonate sheet.
2. Cut two circular wheels from the polycarbonate in Step 1 and sand or polish the edges until smooth.
3. Drill 16-mm holes into the wheels for placement of the axles and clamps in the positions shown in Fig. A-1.
4. Glue a piece of rubber-mat (80 x 200 mm) into the center of each wheel between the clamp holes (Figures A-1 and A-3). These mats will serve as a cushion for the stainless steel LPG cylinder(s).
5. Connect the wheels as shown in Figs. A-2 and A-3, using the threaded rods, washers and nuts as indicated.
6. Heat treat and bend a length of threaded rod with an acetylene-oxygen torch into four equal size U-shaped clamps using an empty stainless steel cylinder as a form. Cut off and grind smooth the ends of each U-shaped clamp at a height of 160 mm. Insert two clamps for each cylinder into the appropriate holes in the center of each wheel and secure the clamp in place with the washers and wingnuts as shown in Fig. A-3 and A-4.
7. Attach the cylinder(s) to the extractor using the clamps as shown in Fig. A-4.
 - Two extractions can be performed simultaneously, as indicated in Fig. A-2.

8. Place the extractor onto the jar mill and turn on the mill.
9. Proceed with *Extraction*, Step 10, in the body of the method. Operate the mixer for 30 minutes per extraction.

SUGGESTED SUPPLIERS

McMaster-Carr Supply Co., 600 County Line Rd., Elmhurst, IL 60126-2081 (708-833-0300)
Piper Plastics Inc., 1921 Industrial Dr., Libertyville, IL 60048 (708-367-0110)

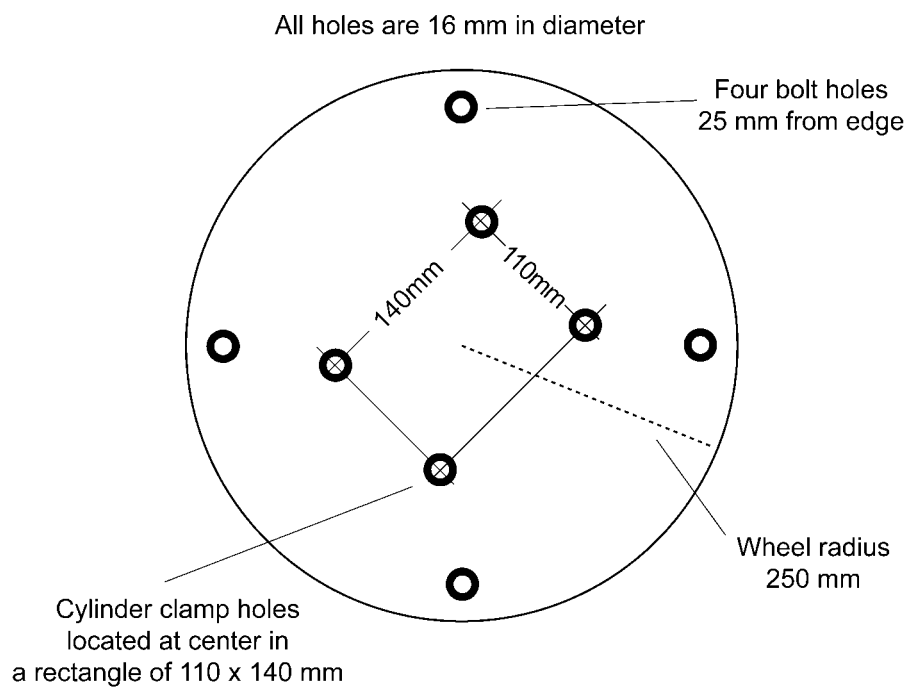


Figure A-1
Extractor Wheel (2 Required)
Showing Dimensions and Holes

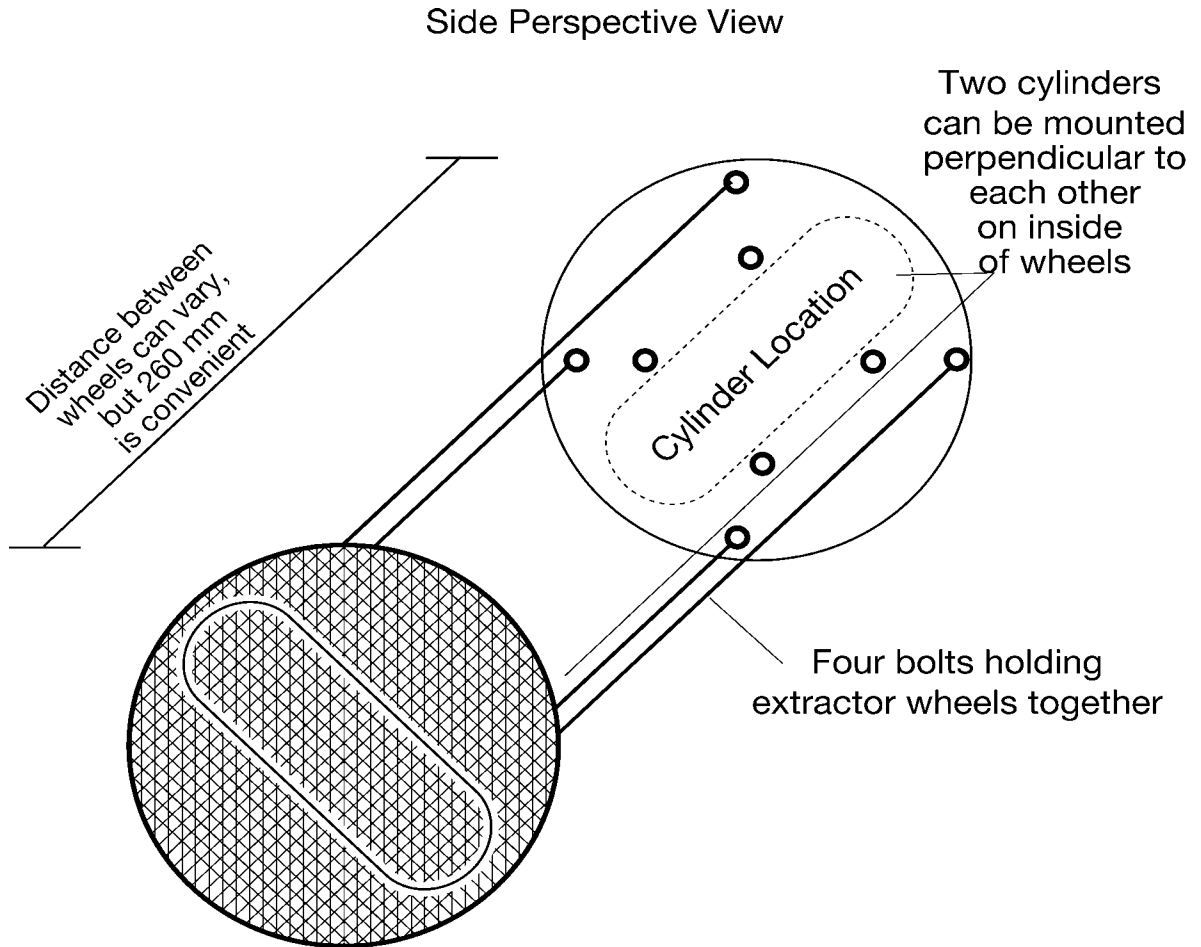


Figure A-2
Assembled Extractor Wheels, Showing Bolts
and Cylinder Locations

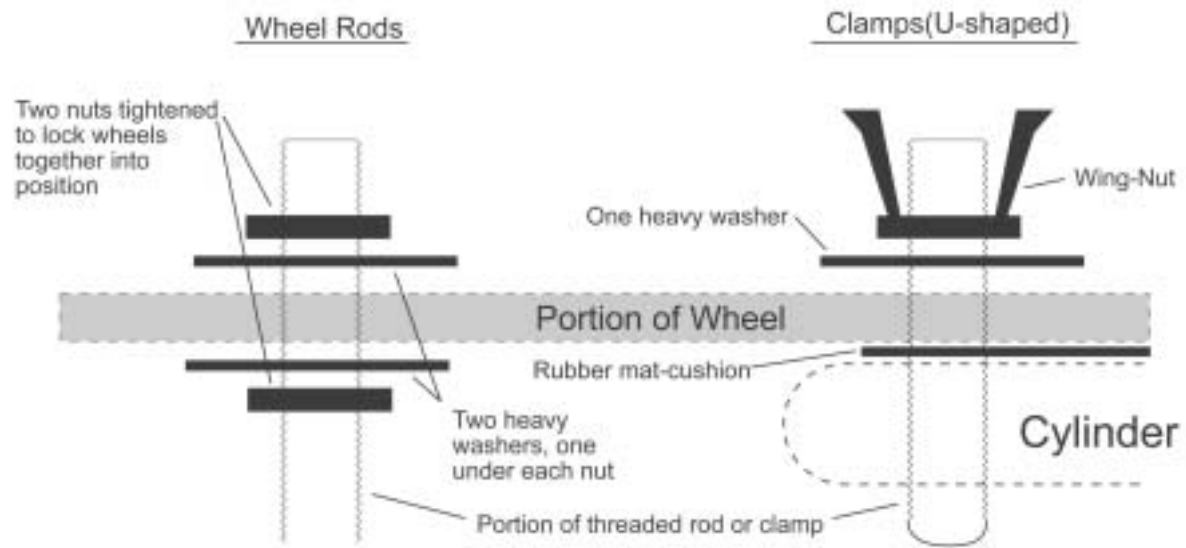


Figure A-3
Detail Showing Nuts and Washers on
Extractor-Wheel Bolts or Clamps, Side View

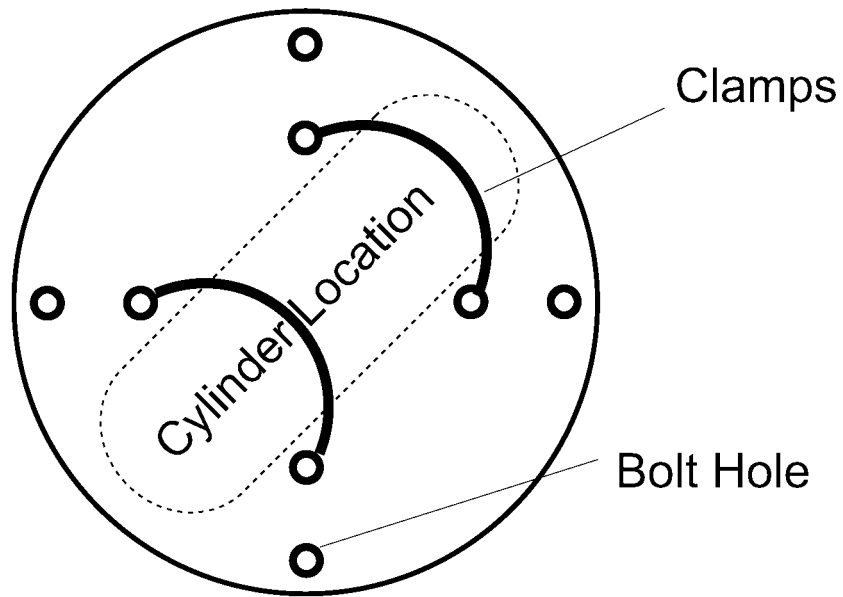


Figure A-4
Cylinder Clamped to Extractor Wheel,
Not to Scale