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Methods of testing petroleum and associated products

Test for ethyl mercaptan odorant in propane, field method

Developed by the Canadian General Standards Board

Canada

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Canadian General Standards Board

Committee on Fuel Test Methods

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Preface

This National Standard of Canada has been reaffirmed by the CGSB Committee on Fuel Test Methods.

Changes since the previous edition

- ICS Code changed from ICS 71.040.40, Chemical analysis Including analysis of gases and surface chemical analysis to ICS 75.160.30, Gaseous fuels Including liquefied petroleum gases.

The following definitions apply in understanding how to implement this National Standard of Canada:

- "shall" indicates a **requirement**;
- "should" indicates a **recommendation**;
- "may" is used to indicate that something is **permitted**;
- "can" is used to indicate that something is **possible**, for example, that an organization is able to do something.

Notes accompanying clauses do not include requirements or alternative requirements. The purpose of a note accompanying a clause is to separate explanatory or informative material from the text. Annexes are designated normative (mandatory) or informative (non-mandatory) to define their application.

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Methods of testing petroleum and associated products

Test for ethyl mercaptan odorant in propane, field method

1 Scope

This method is intended for the semi-quantitative determination of ethyl mercaptan odorant (within the range of 5 to 48 mg/L) in liquid propane in order to confirm that recently stenched propane is odorized to the required level. Two procedures are given which are intended for field application and may be suitable for use by non-laboratory personnel after suitable training. A gas chromatographic method should be used for more precise, quantitative determination of ethyl mercaptan in propane.

Other low molecular weight mercaptans, such as methyl mercaptan, can interfere by giving a positive result.

The primary stain tube described in this method is suitable for Grade 1 propane but is not suitable for Grade 2 (commercial) propane, which normally contains more than 5% propylene by volume.

The testing and evaluation of a product against this method require the use of materials and/or equipment that could be hazardous. This document does not purport to address all the safety aspects associated with its use. Anyone using this method has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any applicable regulatory requirements prior to its use.

2 Normative references

The following normative documents contain provisions that, through reference in this text, constitute provisions of this method. The referenced documents may be obtained from the sources noted below.

Note: The contact information provided below was valid at the date of publication of this method.

An undated reference is to the latest edition or revision of the reference or document in question, unless otherwise specified by the authority applying this method. A dated reference is to the specified revision or edition of the reference or document in question.

2.1 Canadian General Standards Board (CGSB)

CAN/CGSB-3.14 — *Propane for fuel purposes*

2.1.1 Contact information

The above may be obtained from the Canadian General Standards Board. Telephone: 1-800-665-2472. Email: ncr.cgsb-ongc@tpsgc-pwgsc.gc.ca. Web site: https://www.tpsgc-pwgsc.gc.ca/ongc-cgsb/index-eng.html.

2.2 CSA Group

CAN/CSA-B149.2 — *Propane storage and handling code*

2.2.1 Contact information

The above may be obtained from the CSA Group. Telephone: 416-747-4044 or 1-800-463-6727. Web site: https://www.csagroup.org.

2.3 ASTM International

D1265 — *Standard Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method*

D5305 — *Standard Test Method for Determination of Ethyl Mercaptan in LP-Gas Vapor*

E29 — *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*

2.3.1 Contact information

The above may be obtained from ASTM International. Telephone: 1-877-909-2786, Web site: https://www.astm.org. They can also be obtained from Standards Store by Accuris. Telephone: 1-800-447-2273. Web site: https://store.accuristech.com/.

3 Summary of test method

A representative sample of liquid propane is completely vaporized by one of two procedures (a flowing stream of gaseous propane or evaporation of a sample in a plastic bag) and then tested for odorant concentration by use of a hand-held tester that utilizes a detector tube (stain tube) specific for ethyl mercaptan.

4 Significance and use

Propane, a colourless, odourless gas, is intentionally treated with an odorant, ethyl mercaptan for detecting propane leaks. However, the concentration of ethyl mercaptan in propane may decrease (fade) over time because of adsorption on metal surfaces and oxidation, leading to odorant depletion. Therefore, a field test is required to confirm the concentration of odorant in recently stenched propane and to allow determination of ethyl mercaptan in propane in the distribution system.

5 Safety precautions

5.1 Note the extreme flammability of propane and ensure that this test is performed in a well-ventilated area, free of ignition sources.

5.2 Liquid propane evaporates rapidly and can readily freeze flesh. Avoid skin contact.

6 Apparatus and materials

6.1 Gastec precision gas sampling pump: GV-100 or GV-100S or equivalent, incorporating a “test complete” indicator.

6.2 Gastec ethyl mercaptan detector tube: No. 72 calibrated over the range of 0.5 to 120 mL/m^3, or equivalent.

6.3 Sample container: (i.e. pressure cylinder), suitable for collecting and holding liquid propane, and conditioned by prior exposure to propane odorized with expected levels of ethyl mercaptan to minimize adsorption of ethyl mercaptan from the sample. A sample cylinder of at least 500 mL capacity is required for Procedure A. The following sample cylinders (with outage tube and resettable pressure-relief safety valve with monel spring) have been found to be suitable:

6.3.1 Silcosteel-treated, Sulfinert-treated[1] or Teflon-lined stainless steel pressure cylinders.

6.3.2 316 stainless steel pressure cylinders dedicated to stenched propane service.

6.3.2.1 Initial commissioning of new or cleaned/reconditioned cylinders should include charging the cylinder with stenched propane, storing for at least 24 h, and repeating this charging/storing process at least five times.

6.3.2.2 Sample cylinders for stenched propane service should not routinely be left open to the atmosphere or cleaned (e.g. steam cleaning). A cylinder that has been exposed to oxygen or water (vapour) shall be recommissioned as described in 6.3.2.

[1] One of the suppliers of Silcosteel and Sulfinert-treated stainless steel pressure cylinders is Restek Corporation. Telephone: 1-800-356-1688, ext. 3 or 1-814-353-1300, ext. 3. Alternatively, pressure cylinders can be provided to Restek Corporation to receive the Silcosteel or Sulfinert treatment.

6.4 Vaporization device: depending on the particular sample preparation method chosen.

6.4.1 Heating bath (40 to 50 °C — very hot tap water) with 3 m of coiled 3 mm stainless steel tubing (see 11.2), fittings for sample cylinder, a flow meter (optional) and control valve (Figure 1) or;

6.4.2 Polyethylene freezer/food storage bags, nominally 20 to 35 cm per side (large size), equipped with a resealable closure (Ziploc® or “zipper” type) capable of holding a gas-tight seal after the bag is filled while the contents are warmed to near room temperature in preparation for the stain tube determination.

6.4.2.1 Determine the “standard volume” of liquid propane to be put into each polyethylene bag by pouring 15 mL of water into one bag, suspending the bag freely from a top corner so that the water fills a lower corner of the bag. Mark a line diagonally across the corner of the bag to represent the 15 mL volume. Mark each plastic bag with a line at this determined location (but do not use a bag that has contained water for an actual ethyl mercaptan test).

7 Procedure

Caution: This test method requires the release of combustible propane liquid and vapour. The user is warned to take proper safety precautions to prevent freezing of flesh and ignition. This procedure should never be performed in an enclosed building without adequate fume exhaust or near a source of ignition. An experienced tester should train personnel who have not performed this test.

7.1 Either collect a representative sample of liquid propane in an appropriate sample container, by ASTM D1265, taking care to leave a vapour space in the cylinder (i.e. minimum 20%) to allow for expansion of the liquid propane, or arrange for a fine flow of liquid propane at the sample point for testing on site.

Caution: A fully charged cylinder, without “outage” (i.e. minimum 20% vapour space), can explode when allowed to warm up.

7.2 Proceed with the determination of ethyl mercaptan odorant by Procedure A or Procedure B. Both procedures achieve total vaporization of a portion of liquid propane and allow measurement of ethyl mercaptan in the vapour phase (see 11.1).

7.3 Procedure A — Flowing propane gas stream analysis

7.3.1 Assemble the apparatus as shown in Figure 1, in a fume hood or other well-ventilated place, at an ambient temperature of 15 to 30 °C. Ensure that the sample cylinder is mounted vertically.

7.3.2 Open the lower valve on the sample cylinder.

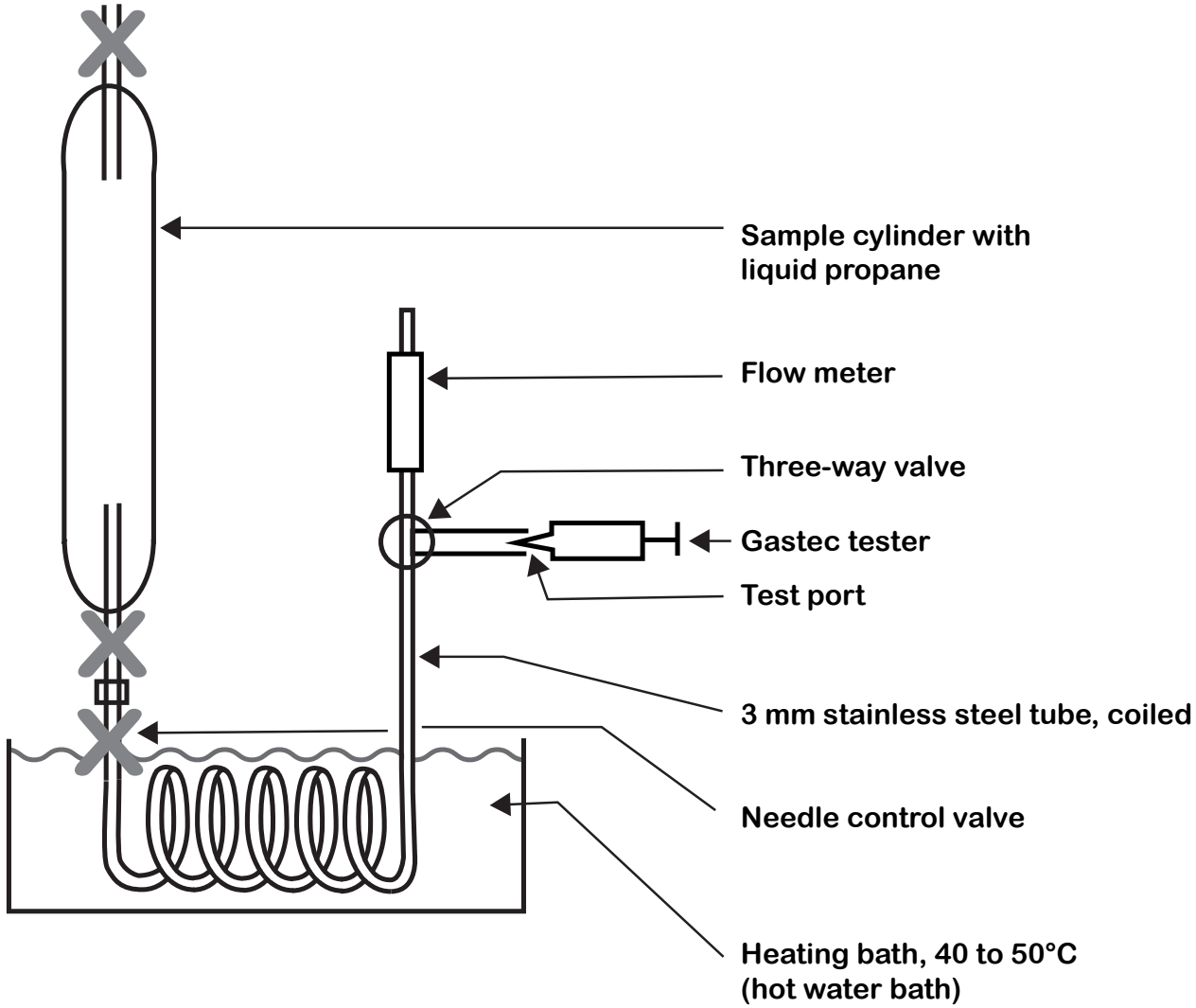
7.3.3 Carefully open the needle control valve to allow a gas stream of approximately 500 mL/min to flow through the coiled tube, three-way valve and optional flow meter.

7.3.4 Purge the tubing for 2 min until the flow of propane gas has stabilized. Readjust the control valve to approximately 500 mL/min.

7.3.5 Turn the three-way valve to cause the propane vapour to exit through the open test port.

7.3.6 Immediately before each series of measurements, test the pump for leakage according to the manufacturer's instruction: insert an unopened tube, align the guide marks, pull a full stroke until handle locks, wait for one minute, and carefully let the handle return. The pump handle should return to within 6 mm of the fully closed position. If it does not, replace worn parts or re-lubricate, and retest.

Figure 1 — Apparatus for procedure A — Flowing propane gas stream analysis



7.3.7 Prepare the detector (stain) tube as specified by the manufacturer (i.e. break both ends off the tube by inserting them in the tube tip breaker of the pump). Then insert the tube into the rubber inlet of the pump with the arrow on the tube pointing towards the pump.

7.3.8 Make certain the pump handle is all the way in. Align the marks on the shaft and housing of the pump.

7.3.9 Place the open tip of the detector tube inside the open test port, in the flowing stream of propane gas, and draw back the handle all the way out for one full stroke until it locks at the 100 mL mark. Keep the tip of the detector tube in the port for 2 min or until the indicating device shows that sampling is completed, to allow the correct volume of gas to pass through the tube.

7.3.10 Immediately (within 30 s) read the coloured (yellow) portion of the tube. Low-level readings (below 10 mL/m^3) may not be very reliable by this procedure but are indicative of a low odorant level.

7.3.11 If the Gastec detector tube No. 72 turns grey, it indicates the presence of propylene. Discontinue the test if the greying of the Gastec tube is such that the first faint indication of yellow cannot be detected.

Note 1: For propane containing less than 1% propylene by volume, the Gastec detector tube No. 72 remains white, and a yellow stain is produced when ethyl mercaptan is detected. The presence of propylene in propane can interfere with the Gastec detector tube because of a competing reaction, which generates a grey colour throughout the tube. Gastec tube No. 72 is

suitable for use with Grade 1 propane (“HD-5,” the quality of retail propane generally sold in Canada), which contains less than 5% propylene by volume. It is not suitable for Grade 2 propane, which normally contains more than 5% propylene by volume.

Note 2: Other detector tubes for the detection of mercaptans, which use different chemistries from Gastec tube No. 72, may not be affected by the presence of propylene. The use of a different detector tube or sampling pump constitutes a modification of this method and requires that suitable procedures for calibration and the calculation of results be established to ensure equivalency.

Note 3: The Gastec ethyl mercaptan detector tube will give an erroneously low result if the temperature of the tube is much below 20 °C. The tubes should be kept in a warm place (20 to 30 °C) before use in cool weather.

7.3.12 If the reading is below 10 mL/m^3, repeat the test once (using the same tube) as follows. Turn the handle 1/4 turn in either direction, and return it to the starting point. Repeat the steps described in 7.3.8 to 7.3.11. Divide the reading of a double stroke test by two.

7.3.13 Close the sample cylinder and control valve and ensure the testing equipment and area are cleared of propane vapours.

7.4 Procedure B — Analysis of propane in a plastic bag (“BAGGIE” method)

7.4.1 Perform this test in a well-ventilated area, at an ambient temperature of 15 to 30 °C.

7.4.2 Immediately before each series of measurements, test the pump for leakage as in 7.3.6.

7.4.3 Clamp the sample cylinder in a vertical position. Attach a short tube to the lower valve (e.g. 2 cm of 5 mm tubing).

7.4.4 Prepare the detector (stain) tube as specified by the manufacturer (i.e. break both ends off the tube by inserting them in the tube tip breaker of the pump). Then insert the tube into the rubber inlet of the pump with the arrow on the tube pointing towards the pump.

7.4.5 Make certain the pump handle is all the way in. Align the marks on the shaft and housing of the pump.

7.4.6 Take a new plastic bag (with a diagonal line across one corner to indicate a 15 mL volume) and open a short length (e.g. 2 to 3 cm) of the bag, minimizing the volume of the bag that is open to the air.

7.4.7 Slightly open the lower valve of the sample cylinder (or sampling valve on a bulk tank) and allow a slow stream of liquid propane to flow out before placing the bag for sample collection under the stream.

Note: Ensure there are no ignition sources in the area of the test. Bond and ground the sample cylinder. Avoid contact of stenched propane with flesh or clothing. Wear gloves for the steps described in 7.4.7 to 7.4.9.

7.4.8 Immediately collect about 15 mL of liquid propane (fill to the diagonal line) in the plastic bag; seal the bag and close the valve on the sample cylinder or bulk tank.

7.4.9 Shake or tumble the bag gently to aid evaporation of the liquid propane and to mix the gases thoroughly.

7.4.10 As soon as the last drop of propane evaporates, proceed with the detector (stain) tube test by one of the following procedures.

Note: If the bag becomes overinflated (drum tight), stop the test and restart it using a new bag. If the pressure inside the bag causes the seal to break, restart the test using a new bag.

7.4.10.1 Puncture the plastic bag by piercing it with the detector (stain) tube, and proceed with the test by drawing back the handle of the pump. (It may be necessary to press down on one part of the bag to give a firmly inflated bag to pierce.)

7.4.10.2 Alternatively, open a small corner of the bag (e.g. 1 to 2 cm length), insert the ethyl mercaptan detector tube all the way and draw back the handle of the pump until it locks. Reseal the bag around the tube as much as possible. Keep the tube in place for 2 min, or until the indicating device shows that sampling is complete, to allow the correct volume of gas to pass through the detector tube.

7.4.10.3 Immediately (within 30 s) read the coloured (yellow) section of the tube. Low-level results (below 10 mL/m^3) may not be reliable by this procedure but are indicative of a low odorant level.

Note: Leave the tube in the bag while taking the reading in case a second pump stroke is required (see 7.4.10.5).

7.4.10.4 If the Gastec detector tube No. 72 turns grey, it indicates the presence of propylene. Discontinue the test if the greying of the Gastec tube is such that the first faint indication of yellow cannot be detected.

Note 1: For propane containing less than 1% propylene by volume, the Gastec detector tube No. 72 remains white, and a yellow stain is produced when ethyl mercaptan is detected. The presence of propylene in propane can interfere with the Gastec detector tube because of a competing reaction, which generates a grey colour throughout the tube. Gastec tube No. 72 is suitable for use with Grade 1 propane ("HD-5," the quality of retail propane generally sold in Canada), which contains less than 5% propylene by volume. It is not suitable for Grade 2 propane, which normally contains more than 5% propylene by volume.

Note 2: Other detector tubes for the detection of mercaptans, which use different chemistries from Gastec tube No. 72, may not be affected by the presence of propylene. The use of a different detector tube or sampling pump constitutes a modification of this method and requires that suitable procedures for calibration and the calculation of results be established to ensure equivalency.

Note 3: The Gastec ethyl mercaptan detector tube will give an erroneously low result if the temperature of the tube is much below 20 °C. The tubes should be kept in a warm place (20 to 30 °C) before use in cool weather.

7.4.10.5 If the reading is below 10 mL/m^3, repeat the test once (using the same tube) as follows. Turn the handle 1/4 turn in either direction, and return it to the starting point. Repeat the steps described in 7.4.10 to 7.4.10.4. Divide the reading of a double stroke test by two.

7.4.10.6 Destroy the sample bag by literally tearing it apart, and ensure that the test area is cleared of propane vapours. (A bag of propane vapour is a serious explosive hazard.)

8 Calculation

8.1 Using Table 1, convert the detector (stain) tube reading in millilitres of ethyl mercaptan per cubic metre of propane vapour (column 1, mL/m^3) to milligrams of ethyl mercaptan per litre of liquid propane (column 2, mg/L).

8.2 Replicate results may be averaged. Round off the result to the nearest whole number by the rounding-off method in ASTM E29.

9 Report

Report the ethyl mercaptan content in liquid propane in mg/L to the nearest whole number and the procedure used (A or B).

10 Precision

The precision of this test method has not been determined by a formal round-robin study. Informal testing indicates a repeatability of ± 15%.

11 Notes

11.1 Ethyl mercaptan is very soluble in propane but has a lower vapour pressure. Therefore, the equilibrium concentration of ethyl mercaptan in the vapour phase of a cylinder is much lower (by a factor of 0.1 to 0.2) than the concentration of ethyl mercaptan in the liquid propane (depending on the temperature and amount of propane in the cylinder). Thus if a sample of propane were taken from the vapour space of a cylinder, the amount of ethyl mercaptan present would generally be only a fraction of that measured in a sample of liquid propane. Since this method has a lower detection limit of about 5 mg of ethyl mercaptan per litre of propane, it is better to analyze a liquid sample (which is totally vaporized by this method) and thus expect to get a result in the range of 15 to 25 mg/L.

11.2 Regarding 6.4 a), 7.3.3 and 7.3.4, apparently the average flow rate of gas into the Gastec detector is 200 mL/min. However, the peak flow rate at the beginning of a stroke is much higher than 200 mL/min. Therefore, a flow rate of 500 mL/min for propane vapour at the test port is suggested. Note that ASTM D5305, designed to measure ethyl mercaptan in vapour phase propane which is assumed to be in equilibrium with the ethyl mercaptan concentration in the liquid phase, uses a gas reservoir as the point of testing with the stain tube device.

11.3 Annex A is a proposed data sheet for recording ethyl mercaptan test results.

Table 1[2] — Equivalent concentrations of ethyl mercaptan in liquid propane

Column 1	Column 2	Other concentration units		
Stain tube reading	Reporting units			
mL/m^3 or ppm v/v — Vapour phase	mg/L	lb/10,000 USG	mL/m^3 or ppm v/v — Liquid phase	g/1000 kg or ppm m/m
0	0	0	0	0
1.7	1.2	0.1	1.4	2.3
3.3	2.4	0.2	2.8	4.7
5	3.6	0.3	4.3	7
6.7	4.8	0.4	5.7	9.4
8.3	6	0.5	7.1	11.7
10	7.2	0.6	8.5	14.1
11.7	8.4	0.7	9.9	16.4
13.3	9.6	0.8	11.3	18.8
15	10.8	0.9	12.8	21.1
16.7	12.0[a]	1	14.2	23.5
18.3	13.2	1.1	15.6	25.9
20	14.4	1.2	17	28.2
21.7	15.6	1.3	18.4	30.5
23.3	16.8	1.4	19.9	32.9
25	18	1.5	21.3	35.2
26.7	19.2	1.6	22.7	37.6
28.3	20.4	1.7	24.1	39.9
30	21.6	1.8	25.5	42.3
31.7	22.8	1.9	26.9	44.6
33.3	24	2	28.4	47
35	25.2	2.1	29.8	49.3
36.7	26.4	2.2	31.2	51.7
38.4	27.6	2.3	32.6	54
40	28.8	2.4	34	56.4
41.7	30	2.5	35.5	58.7
43.4	31.2	2.6	36.9	61.1
45	32.4	2.7	38.3	63.4
46.7	33.6	2.8	39.7	65.8
48.4	34.8	2.9	41.1	68.1
50	35.9	3	42.5	70.5
51.7	37.1	3.1	44	72.8
53.4	38.3	3.2	45.4	75.2
55	39.5	3.3	46.8	77.5
56.7	40.7	3.4	48.2	79.9
58.4	41.9	3.5	49.6	82.2
60	43.1	3.6	51.1	84.6
61.7	44.3	3.7	52.5	86.9
63.4	45.5	3.8	53.9	89.3

[2] Use of this table of conversions of ethyl mercaptan concentration is restricted to situations where the vapour phase is representative of the liquid phase, as achieved by totally evaporating a flowing stream of liquid propane as described in Procedure A or totally evaporating a portion of liquid propane in a plastic bag as described in Procedure B ("BAGGIE Method"). The table of conversions is **not** applicable to conversion of ethyl mercaptan concentration in vapour phase propane which is in equilibrium with liquid propane, such as the vapour phase in the top of a storage tank or cylinder, which is not representative of the composition of the liquid phase.

Column 1	Column 2	Other concentration units		
Stain tube reading	Reporting units			
mL/m^3 or ppm v/v — Vapour phase	mg/L	lb/10,000 USG	mL/m^3 or ppm v/v — Liquid phase	g/1000 kg or ppm m/m
65	46.7	3.9	55.3	91.6
66.7	47.9	4	56.7	94
[a] The historical requirement for ethyl mercaptan in propane has been 1.0 lb/10,000 U.S. gallons, which is 12.0 mg/L of ethyl mercaptan in liquid propane.				

Annex A

(informative)

Propane odorant stain tube test/Data sheet

A-0000001

Location: _____ Date: _____

Test conducted by: _____ Time: _____

Container data:

Owner: _____

Location: _____

Capacity: _____ lb. _____ litres _____ gal. (imp.) _____ gal. (U.S.)

Name of manufacturer: _____

Year of manufacturing: _____ Serial no.: _____

Tank specification: _____ ASME _____ CTC _____

Year requalified: _____ Type of services: _____ Liquid _____ Vapour

Date of container installation at present site? _____

New when installed? Y/N _____ Refilling schedule _____ On call _____ Scheduled

Was container relocated from previous service? _____ Yes _____ No

Container throughput during last twelve months (est.): _____ (lb./L/gal.)

Liquid level in container when tested (e.g. 20% or 10 lb.) _____

Temperature of product in container when tested (°F or °C) _____

_____

Weather data: Temperature: _____ (°F or °C)

(X appropriate)

_____ Calm _____ Windy _____ Sunny _____ or Partly cloudy _____ Cloudy

Precipitation at time of test: _____ Dry _____ Rain _____ Snow

Odorant data:

Odorant in use: _____ Ethyl mercaptan _____ Thiophane _____ Not known

Odorized by: _____ Producer _____ Processor _____ Distributor _____ Not known

Manufacturer of test pump: _____

Manufacturer of stain tube used: _____

Tube no.: _____ Lot no.: _____ Date of expiry: _____

Test result: _____ mL/m^3 or ppm v/v — Vapour phase _____ mg/L

Was container returned to service? Yes _____ No _____