

SRI LANKA STANDARD 666:1984
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**SPECIFICATION FOR
DISSOLVED ACETYLENE**

SRI LANKA STANDARDS INSTITUTION

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SLS 666:1984

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SPECIFICATION FOR DISSOLVED ACETYLENE

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1984-10-31 after the draft, finalized by the Drafting Committee on Industrial and Medicinal Gases, had been approved by the Chemicals Divisional Committee.

All standard values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification, the assistance obtained from the publications of the Indian Standards Institution and the Federal Supply Service of the General Services Administration of United States of America is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements and methods of sampling and test for industrial acetylene gas dissolved in acetone for use in industry.

2 REFERENCES

- BS 349 Identification of the contents of industrial gas containers
- CS 102 Presentation of numerical values
- SIS 428 Random sampling methods

3 DEFINITIONS

For the purpose of this specification the following definitions shall apply:

3.1 tare A: The mass of the vessel consisting of the cylinder, a valve, safety devices if any, and valve protection devices permanently fixed directly to the cylinder together with the porous mass and acetone for the storage of dissolved acetylene.

3.2 tare S: The mass of the vessel consisting of the cylinder, a valve, safety devices if any, and valve protection devices permanently fixed directly to the cylinder together with the porous mass, acetone for the storage of dissolved acetylene and the acetylene required to saturate the solvent at atmospheric pressure and at a temperature of 15 °C.

4 REQUIREMENTS

The gas shall consist essentially of acetylene, C_2H_2 , dissolved in acetone in cylinders containing a porous mass, complying with the requirements specified in Table 1.

TABLE 1 - Requirements for acetylene

Characteristic	Requirement	Method of test (Ref. to Appendix)
Acetylene content (as C_2H_2), per cent (V/V), minimum*	98.0	B
Phosphorus (as PH_3) per cent (V/V), maximum	0.05	C** and D
Sulfur (as H_2S), per cent (V/V), maximum	0.05	C** and D
Ammonium (as NH_3), per cent (V/V), maximum	0.05	E

* This requirement is based on the solvent-free material.

** Appendix C is a qualitative determination. In case of dispute, the acetylene shall be quantitatively analysed by the method in Appendix D.

5 PACKAGING

- 5.1 Acetylene shall be supplied in steel cylinders of a suitable design and construction as agreed to between the purchaser and the supplier.
- 5.2 The contents of such cylinders shall meet the filling limitations authorized for the cylinder.
- 5.3 The cylinders shall be free from dents and corrosion and shall be examined annually.

6 MARKING

- 6.1 Cylinders shall be painted with the maroon colour as specified in BS 349.
- 6.2 Each cylinder shall be stamped or marked in a permanent nature on the shoulder or on a reinforced part of the cylinder or on the collar or neck with the following marks.
- a) Gas identification *ACETYLENE* and the chemical symbol C_2H_2 ;
 - b) Name and address of the manufacturer;
 - c) Serial number of the cylinder;
 - d) Tare, in kilograms, with suffix A or S as defined in 3;
 - e) Maximum mass, in kilograms, of acetylene to be charged into the cylinder;
 - i) inclusive of saturation mass, in kilograms of acetylene in the case of tare marked A.
 - ii) not inclusive of saturation mass, in kilograms, of acetylene in the case of tare marked S; and
 - f) Maximum pressure, in kilopascals at 27 ± 2 °C.

6.3 The cylinders may also be marked with the Certification Mark of the Sri Lanka Standards Institution illustrated below on permission being granted for such marking by the Sri Lanka Standards Institution.



NOTE - The use of the Sri Lanka Standards Institution Certification Mark (SLS Mark) is governed by the provisions of the Sri Lanka

Standards Institution Act and the regulations framed thereunder. The SLS mark on products covered by a Sri Lanka Standard is an assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control, which is devised and supervised by the Institution and operated by the producer. SLS marked products are also continuously checked by the Institution for conformity to that standard as a further safeguard. Details of conditions under which a permit for the use of the Certification Mark may be granted to manufacturers or processors may be obtained from the Sri Lanka Standards Institution.

7 SAMPLING

Unless otherwise agreed to between the buyer and the seller, the procedure for sampling shall be as given in Appendix A.

8 METHODS OF TEST

8.1 The material shall be tested by the appropriate methods prescribed in Table 1.

8.2 Unless otherwise specified, distilled water and analytical reagent grade chemicals shall be used throughout the tests.

9 CONFORMITY TO STANDARD

A lot shall be declared as conforming to the requirements of this specification if all the samples tested as in A.5 satisfy the relevant requirements.

APPENDIX A

SAMPLING

A.1 LOT

All cylinders containing acetylene manufactured at one time using one mix of raw materials shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, storing, and handling the samples the following precautions and directions shall be observed.

A.2.1 Because of hazards involved with dissolved acetylene it should be sampled by, or sampled under the supervision of, persons familiar with the necessary safety precautions.

A.2.2 Metal sample containers of a type which insures maximum safety and which are corrosion-resistant to product being sampled shall be used to collect samples. The container may be of the one valve or the two valve type. The size of the container depends upon the amount of sample required for the laboratory tests that are to be made.

A.2.3 Samples shall be kept in a cool location soon after they are drawn and shall be kept there until all the tests have been completed.

A.2.4 The valve of the sample container shall be protected either by packing the container in a crate, in an appropriate manner or by using a protective cap so that accidental unseating of the valve is avoided.

A.2.5 Each sample container after filling shall be marked with necessary details of sampling.

A.3 SCALE OF SAMPLING

A.3.1 Samples from each lot shall be treated separately for ascertaining conformity of the product to the requirements of this specification.

A.3.2 The number of cylinders to be selected for taking samples shall be in accordance with Table 2.

A.3.3 The cylinders shall be selected at random. In order to ensure randomness of selection random number tables as given in SLS 428 shall be used.

TABLE 2 - Scale of sampling

Number of cylinders in the lot	Number of cylinders to be selected
Upto 05	02
6 to 15	03
16 to 25	04
26 to 50	05
51 to 100	06
101 and above	08

A.4 METHOD OF SAMPLING

Samples of dissolved acetylene shall be taken from a cylinder containing the gas direct to the sample container. A regulator shall always be fitted to the cylinder valve and the down stream pressure set at not more than 30 kPa. The valve regulator, connecting lines and sample containers shall be carefully purged before taking samples.

A.5 NUMBER OF TESTS

Each sample selected as in A.3.2 shall be subjected to all the requirements of this specification.

APPENDIX B

DETERMINATION OF ACETYLENE CONTENT

B.1 PRINCIPLE

A measured volume of sample is treated with fuming sulfuric acid which absorbs the acetylene gas. The volume of any residual gas is then measured in a burette.

B.2 APPARATUS

The apparatus shall be of the type designed for determination of the percentage of gas by volume from the shrinkage in volume because of the absorption of the gas by the reagents used, and the apparatus shall measure with an error of not more than ± 0.1 per cent.

The apparatus shall consist of a burette having a capacity of 100 ml, with graduated stems at the upper and lower ends and an ungraduated bulb in the middle. The graduated parts shall each hold about 10 per cent of the capacity of the burette. A water jacket enclosing the burette shall be provided. The burette shall be connected to absorption pipettes by means of a flushing manifold. The pipettes shall be of the bubbler type, made entirely of glass. Flexible rubber bags of such quality as will not be appreciably affected by sulfur trioxide fumes, or additional pipettes containing saturated sodium chloride solution or other suitable liquid, shall be attached to the rear compartment of the pipettes. A vertical support rod for the liquid levelling bottle shall be provided. A bottom entry, top discharge prefilter tube containing between 20 g and 30 g of steam activated wood charcoal shall be inserted between the dissolved acetylene supply and the apparatus. The steam activated wood charcoal shall be situated between two glass fibre strands retaining plugs. The purpose is to remove the solvent carry-over from the gas sample.

B.3 GAS-DISPLACEMENT LIQUID

The gas-displacement liquid shall be mercury, with about 0.1 ml of an aqueous solution of approximately 2 per cent of sulfuric acid on the surface of the mercury in the burette for the purpose of clearing up precipitation on the interior of the burette tube.

B.4 REAGENT

The reagent used in the first pipette shall be fuming sulfuric acid containing 20 per cent of free sulfur trioxide. The reagent used in the second pipette shall be sulfuric acid (A.R. grade $\rho = 1.70$) made by diluting 373 ml of concentrated sulfuric acid ($\rho = 1.84$) to 500 ml with distilled water.

B.5 PROCEDURE

Flush out the manifold with nitrogen or dry air. Purge the burette with the sample by drawing in and discarding several samples, being careful not to displace the nitrogen above the absorption pipette. Draw a measured volume of 100-ml sample into the burette by means of a levelling bottle. Observe and record the temperature at this time. Pass the sample into the first absorption pipette containing the fuming sulfuric acid and then back into the burette. Report this step until no further change in volume occurs as indicated by successive measurements. Store the gas in the first pipette without disturbing the gas in the pipette or in the manifold, fill the burette with mercury and draw in 5 ml of dry air or nitrogen. Return the residual gas sample stored in the first pipette, to the burette and mix with dry air. Pass this gas mixture again several times between the first absorption pipette and the burette until two successive volume readings are within 0.1 ml of each other. Pass the gas into the second pipette containing the dilute sulfuric acid ($\rho = 1.70$) and then back into the burette. Repeat this step until no further change in volume occurs as indicated by two successive volume readings lying within 0.1 ml of each other. Record the temperature, if the difference between the first and second temperature readings is greater than 2°C , repeat the test.

B.6 CALCULATION

Acetylene content per cent (V/V) = $105 - V$

where,

V = final volume reading in millilitres.

APPENDIX C

QUALITATIVE DETERMINATION OF PHOSPHORUS AND SULFUR

C.1 REAGENT

C.1.1 *Silver nitrate*, 10 per cent (m/V) solution.

C.2 PROCEDURE

Hold a piece of white filter paper saturated in silver nitrate solution in a flow of the gas from the cylinder for at least 30 seconds. If the paper remains white or becomes a light yellow, the acetylene is acceptable. If the paper turns dark brown or black, the acetylene shall have failed the test. Do not allow the paper to dry before the test.

NOTE - Used silver nitrate test papers should be immediately saturated with water and disposed of as waste paper.

APPENDIX D

QUANTITATIVE DETERMINATION OF PHOSPHORUS AND SULFUR

D.1 PRINCIPLE

A measured volume of sample is passed through sodium hypochlorite solution. Phosphorus and sulfur compounds are trapped in the solution and subsequently converted into ortho-phosphoric acid and sulfuric acid respectively. Phosphorous and sulfur are thereafter determined by volumetric and gravimetric methods respectively.

D.2 REAGENTS

D.2.1 *Sodium hypochlorite, solution*, two to three per cent, make a 1 : 1 dilution with a 4 to 6 per cent solution of sodium hypochlorite commercially available.

D.2.2 *Molybdate reagent*, make the molybdate reagent by either method as follows:

D.2.2.1 Method A

D.2.2.1.1 Solution 1

Mix 118 g of 85 per cent molybdic acid with 400 ml of distilled water, add 80 ml concentrated ammonium hydroxide, and filter when solution is complete.

D.2.2.1.2 Solution 2

Mix 400 ml of concentrated nitric acid with 600 ml of distilled water. Vigorously agitate Solution 2 by means of a current of air and add Solution 1 very slowly through a tube dipping under the surface of the agitated solution. When all of Solution 1 has been added, continue the current of air for 1 to 2 hours. Let stand, filter if necessary, and store in a glass-stoppered bottle.

D.2.2.2 Method B

D.2.2.2.1 Solution 1

Dissolve 100 g of ammonium molybdate (A.R. grade) in 400 ml of distilled water and 80 ml of concentrated ammonium hydroxide. Filter if necessary.

D.2.2.2.2 Solution 2

Mix 400 ml of 16 N nitric acid with 600 ml of water. For use, mix the necessary amount of Solution 1 with twice its volume of Solution 2, adding Solution 1 to Solution 2 gradually with vigorous stirring.

D.3 PREPARATION OF SAMPLE

Approximately 30 litres of acetylene gas from the sample cylinder shall be passed at a rate of one litre per minute through two Fisher-Milligan Gas Washer bottles connected in series and each containing 200 ml of the 2 to 3 per cent sodium hypochlorite solution. The effluent gas shall be vented to outdoor spaces to avoid explosive atmospheres. Measure the volume of gas by means of a wet-test meter after the gas has passed through the absorption bottles. At the conclusion of the run, shut off the flow of gas and record the volume of gas used; next, record the barometric pressure and the temperature. Remove plastic caps from the Fisher-Milligan bottles and rinse down with a few millilitres of distilled water, allowing the rinsings to fall into the bottle. Transfer the solutions from both bottles into the same 500-ml volumetric flask. Rinse each bottle and glass with three 10-ml portions of distilled water, transfer rinsings to volumetric flask, and make up to volume with distilled water. Mix the contents thoroughly and pipette 250 ml into a 400-ml beaker together with rinsings from the flask and pipette.

D.4 PROCEDURE

D.4.1 Analysis for phosphine

To the first beaker of solution obtained as specified in D.3 above, add 30 ml of concentrated nitric acid and boil it down to 100 ml to 125 ml. Transfer the solution to a 300-ml glass-stoppered erlenmeyer

flask, cool, and cautiously add 10 ml of concentrated ammonium hydroxide. Adjust the temperature to approximately 40 °C and add 100 ml of the molybdate reagent prepared in accordance with D.2.2 above. Let the solution settle for 10 to 30 minutes. Filter it on a small Gooch crucible, and wash the flask and the crucible with a 1 per cent potassium-nitrate solution until they are acid-free as indicated by pH indicator paper.

Transfer the mat with the precipitate from the crucible into the flask and add a known volume of standard sodium hydroxide solution until the precipitate is completely dissolved, as indicated by the absence of yellow colour. Add a few drops of phenolphthalein indicator solution and then back titrate with standard hydrochloric acid solution until the indicator colour disappears. Repeat this procedure, using sodium hypochlorite solution and reagents only, to obtain a blank determination.

D.4.1.1 Calculation

$$\text{Phosphorus (as PH}_3\text{), per cent (V/V)} = \frac{[(N_1c_1 - N_2c_2) - B] \times (273+T) \times 0.072}{P \times V}$$

where,

- B = value obtained in blank determination;
- T = temperature, in degrees Celsius, of gas;
- V = measured volume, in litres, of acetylene;
- P = barometric pressure, in kilopascals;
- V_1 = volume, in millilitres, of standard sodium hydroxide;
- V_2 = volume, in millilitres, of standard hydrochloric acid;
- c_1 = concentration of the standard sodium hydroxide solution in mol/l; and
- c_2 = concentration of the standard hydrochloric acid solution in mol/l.

NOTE - The above calculation is derived as follows:

$$1 \text{ mol NaOH} = 1/23 \text{ mol PH}_3 = \frac{22.412}{23} \text{ litres PH}_3 \text{ at NTP}$$

$$1 \text{ millimol NaOH} = 0.9744 \times 10^{-3} \text{ litres PH}_3$$

$$\frac{[(\text{ml NaOH} \times N_1 - \text{ml HCl} \times N_2) - B] \times 0.9744 \times 10^{-3} \times (273 + T) \times 101.3 \times 2^* \times 100}{V \times P \times 273}$$

$$= \frac{[(\text{ml NaOH} \times N_1 - \text{ml HCl} \times N_2) - B] \times (273 + T) \times 0.072}{V \times P} = \% \text{ PH}_3 \text{ (by vol.)}$$

*NOTE - The factor "2" was introduced in the above calculation because the aliquot used represents one half of the original sample.

D.4.2 Analysis for hydrogen sulfide

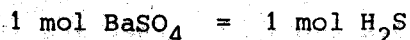
To the second beaker of solution obtained as specified in D.3 above, add 30 ml of concentrated hydrochloric acid and boil down to 100 ml to 125 ml. Then carefully neutralize with ammonium hydroxide to the methyl orange end point and add one ml of concentrated hydrochloric acid. Heat to boiling and add 10 ml of 10 per cent barium chloride solution while stirring vigorously. Let it stand at the side of a steam bath for 30 minutes. Filter wash, and ignite to constant mass: record the mass of the remaining barium sulfate in grams repeat this procedure using hypochlorite solution and reagents only to obtain a blank determination. Record mass of barium sulfate in grams.

D.4.2.1 Calculation

Sulfur (as H₂S) per cent (V/V) =

$$\frac{(m_1 - m_0) \times (273 + T) \times 7.13}{P \times V}$$

where,

 m_1 = mass in grams, of barium sulfate; m_0 = mass, in grams, of barium sulfate in blank determination; P = barometric pressure, in kilopascals; V = measured volume, in litres, of acetylene; and T = temperature, in degrees Celsius, of gas.*NOTE - The above calculation is derived as follows:*

$$1 \text{ mol BaSO}_4 = \frac{1 \text{ mol H}_2\text{S}}{233.42} = \frac{22.412}{233.42} \text{ litres H}_2\text{S at NTP} = 0.09602 \text{ litres H}_2\text{S}$$

$$= \frac{(\text{mass BaSO}_4 - \text{mass blank}) \times (273 + T) \times 7.13}{V \times P} = \% \text{ H}_2\text{S (by vol.)}$$

APPENDIX E

DETERMINATION OF AMMONIUM COMPOUNDS

E.1 PRINCIPLE

A measured volume of sample is passed through a known volume of standard sulfuric acid. Any ammonia gas present reacts with an equivalent amount of the standard acid and the excess acid is determined by standard sodium hydroxide.

E.2 APPARATUS

E.2.1 *Gas meter*, accurate to one per cent.

E.2.2 *Two gas scrubbers*, as shown in Fig. 1.

E.3 REAGENTS

E.3.1 *Sulfuric acid*, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0.05$

E.3.2 *Sodium hydroxide*, standard volumetric solution, $c(\text{NaOH}) = 0.1$

E.3.3 *Methyl orange indicator*. Dissolve 0.1 g of methyl red in 100 ml of 95 per cent (V/V) ethanol.

E.4 PROCEDURE

Transfer 20 ml of standard sulfuric acid into each of the two gas scrubbers. Add 2 drops of methyl orange indicator to each. Assemble the scrubbers as shown in Fig. 1. Connect the inlet to the gas sample reservoir. Record the meter reading, temperature, and barometric pressure. Pass the gas to be tested through the gas scrubbers at a rate of 10 litres/hour for 4 hours to 5 hours. Cut off the gas and again record the meter reading, temperature and barometric pressure. Transfer the solution from both the gas scrubbers to a 250-ml beaker and titrate the excess of acid with standard sodium hydroxide solution. Carry out a blank titration with the same quantity of standard sulfuric acid, (that is: 40 ml) without passing the gas through it.

E.5 CALCULATION

Convert the volume of acetylene gas passed through the gas scrubbers to that at NTP assuming the temperature and pressure to be the mean of the two readings taken.

$$\text{Ammonium compounds (as NH}_3\text{), per cent (V/V)} = \frac{2.241 (V_1 - V_2) c}{V}$$

where,

V_1 = volume, in millilitres, of standard sodium hydroxide solution used in the blank;

V_2 = volume, in millilitres, of standard sodium hydroxide solution used in the experiment with the gas;

c = concentration of the sodium hydroxide solution (E.3.2) in mol/l; and

V = volume, in litres, at NTP of the gas taken for the test.

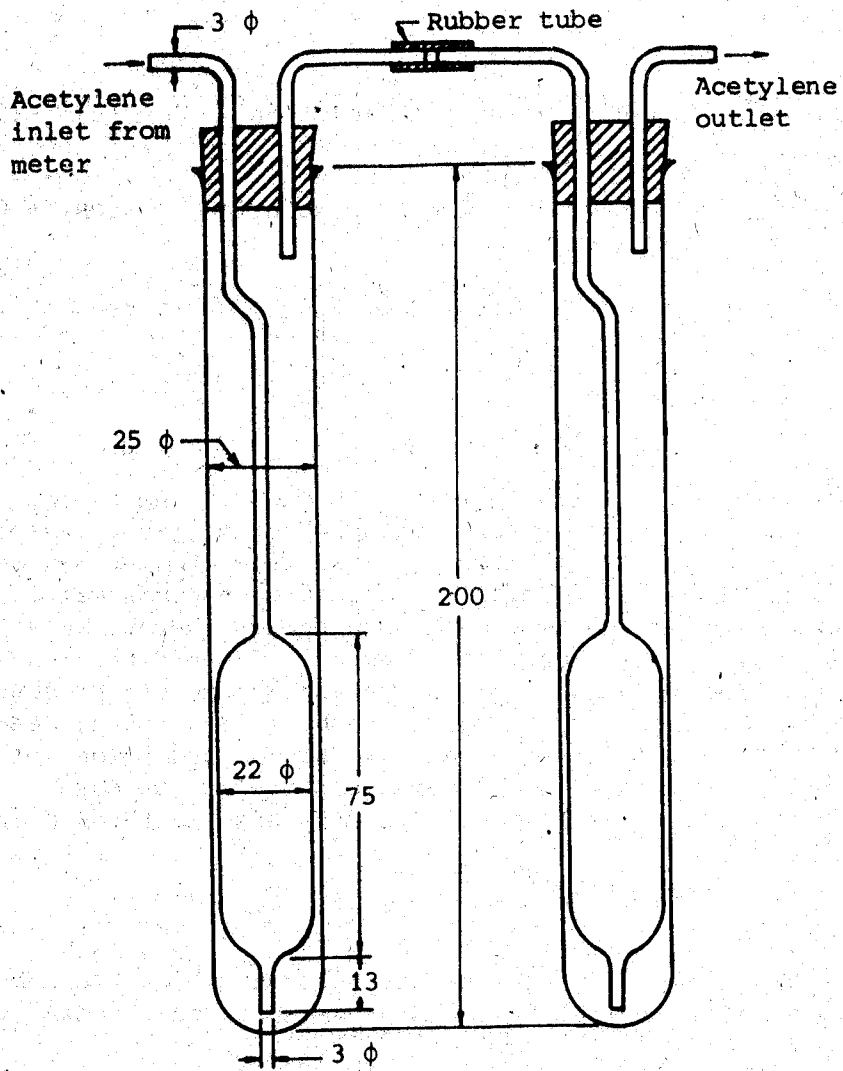


FIGURE 3 - Details of gas scrubbers

SRI LANKA STANDARDS INSTITUTION

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