### DRAFT TANZANIA STANDARD

Gaseous Carbon dioxide – technical grade – Specification

TANZANIA BUREAUOFSTANDARDS

2<sup>nd</sup> edition

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Tanzania Bureau of Standards (TBS) is a statutory national standards body for Tanzania established under the Standards Act No. 3 of 1975 and replace by the Standards Act No. 2 of 2009.

This standard was prepared under the supervision of Chemicals Divisional Standards Committee which consists of consists of representatives from the following organizations;

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#### FOREWORD

This Tanzania Standard was prepared by the Gases Products Technical Committee CDC 9 under the supervision of Chemical Standards Division Committee and it is in accordance with the procedures of the bureau.

In the preparation of this standard assistance has been derived from:

IS 307:1996 Carbon dioxide - Specification, published by the Bureau of Indian Standards,

Carbon dioxide, prepared at the  $49^{th}$  JECFA (1996), published by FAO in FNP 52 Add 5 (1997),

BS 4105:1967, Specification for liquid carbon dioxide Industrial, published by the British Standards Institute.

This second edition cancels and replaces the second edition (TZS 216:2017), which has been technically revised

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated expressing the result(s) of a test or analysis shall, be rounded off in accordance with TZS 4 (see clause 2). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Replecience

# 1. SCOPE

This Draft Tanzania Standard prescribes requirements, sampling, and test methods for gaseous carbon dioxide of technical grades.

Gaseous Carbon dioxide (technical grade) is intended for fire extinguishers, refrigeration and general purposes.

It doesn't intend to be used as a food preservative and medical purpose

### 2. Normative references

The following normative references are indispensable for the application of this standard. For dated references, only the edition citied applies. For undated references, the latest edition of the normative references (including amendments) applies

TZS 59, Water – Distilled quality – Specification

TZS 97, Specification for identification of contents of industrial gas cylinders

TZS 4, Rounding off numerical values

### 3. Requirements

### 3.1 General requirements

i.In gaseous form carbon dioxide shall be odourless, colourless, non-flammable, and slightly acidic.

ii.A gaseous carbon dioxide of technical grade when passed through a solution of barium hydroxide, a white precipitate will be formed which dissolves with effervescence with dilute acetic acid.

### 3.2 Specific requirements

When tested according to respective method, gaseous carbon dioxide shall comply with the specific requirements in Table 1.

S/ No.	Characteristic	Requirement	Test method
(i)	Carbon dioxide, percent by volume, min.	99.0	Annex A
(ii)	Carbon monoxide, mg/Kg, max.	10	Annex B
(iii)	Acid and Sulphur dioxide	To pass test	Annex C
(iv)	Phosphine and hydrogen sulphide	To pass test	Annex D
(v)	Moisture, percent by volume, max.	0.1	Annex E
(vi)	Arsenic, mg/Nm <sup>3</sup> , max.	0.5	Annex F
(vii)	Oil, mg/kg(m/m), max.	5	Annex I

### Table 1– Requirements for gaseous carbon dioxide – technical grade

### 4 Sampling

### 4.1 Scale of sampling

4.1.1 In any consignment all cylinders charged during one work shift from one charging manifold shall be grouped together to constitute a lot.

4.1.2 Samples shall be tested for each lot for ascertaining- the conformity of the material to the requirements of this specification.

### 4.2 Method of sampling

4.2.1 The number of cylinders to be selected from each lot shall be in accordance with Table 2.

Lot size "N"	Sample size "n"	
Up to 50	5	
51 – 100	8	
101 – 150	15	
151 – 300	20	
300 and above	25	

### Table 2– Numberof cylinders to be selected

4.2.2 The cylinders shall be selected at random and to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier, shall be used. In case such a table is not available, the following procedure is recommended.

Starting from any cylinder in the lot, count them as 1, 2, 3, up to "r" and so on, where "r" is the integral part of "N" (N being the number of cylinders in the lot and "n" the number to be selected as sample. Every' *r*th cylinder thus counted, shall be withdrawn to constitute a sample, till the required number of cylinders is obtained.

### 5 Number of tests

5.1 From ea.ch of the cylinders selected according to 4.2.2 two separate samples of each gas shall be drawn.

5.2 All the samples of the gas shall be tested individually for all the requirements given in 3.1, 3.2 and Table 1.

### 6 Criteria for conformity

A lot shall be declared as conforming to the requirements of this specification if all the individual test results satisfy the relevant requirements given in clause 3.

### 7 Quality of reagents

Analytical grade reagents and distilled water that comply with TZS 59 (See Clause 2) or deionized water of equivalent purity shall be used for the appropriate tests.

### 8 Packaging and marking

### 8.1 Packaging

Carbon dioxide of technical grade can be packed in pressurized cylinder or low pressure bulk liquid system, or in solid blocks or in any suitable form.

# 8.2 Marking

The marking and labelling shall be in accordance with TZS 97.

### Annex A

#### (normative)

#### Analysis of carbon dioxide

### A.1 Apparatus

The apparatus shall consist of the following parts assembled as shown in Figure 1.

**A.1.1** Pipette (D) – Semet Solvay modification of hempel pipette for the absorption of carbon dioxide.

**A.1.2** Burette (A)–of100 ml capacity with a graduated tube (at the top) of capacity 1.0 ml graduated 0.05 ml beginning at the three way stopcock *C*. One branch of stopcock *C* communicates with the pipette *D* while the other branch with the sample to be analysed. The burette is enclosed in jacket filled with water.

**A.1.3** Levelling Bottle (B) – filled with acidulated water, saturated with the gas to be analysed and coloured red with methyl orange, to facilitate reading.



Figure 1 - Assembly of apparatus for determination of carbon dioxide

### A.2 Reagents

A.2.1 Acidulated water – containing 0.5% by mass of sulphuric acid.

**A.2.2** *Potassium hydroxide solution* – Dissolve 360 g of potassium hydroxide in water and dilute to one litre.

#### A.3 Procedure

**A.3.1** Preparation of the apparatus – Make sure that the temperature of water in the jacket and the levelling bottle *B* is about the same as the room temperature. Grease the stopcock *C* of the burette *A*, and test theburette and connections for gas tightness. To do this, connect *B* to the burette *A* with *A* rubber tubing and draw in 100 ml of air closing the stopcock *C* and reading the volume. Raise the levelling bottle *B* to the height of *C* and keep it in the position for 3 minutes. Large leaks will be observed by the meniscus gradually rising while smaller leaks will be disclosed by again reading the volume of air at the end of 3 minutes.

**A.3.2**Turn the stopcock *C* so that the burette *A* communicates with the atmosphere through the left hand capillary and raise the levelling bottle *B* to force the air out of the burette *A*. When the water reaches the top of the capillary, close *C* by turning the stopcock through 90 degrees and then connect the left hand capillary with the vessel containing the sample of gas to be analysed. Lower *B*, open the connection to the sample reservoir and then open *C* drawing in about 25 ml of this gas. Close *C* while the gas is still entering. Then turn the stopcock *C* to connect the burette *A* with theright hand capillary. Drive out and discard the gas contained in the burette *A* as it usually contains some air, When wateremerges from the top of the capillary, turn *C* to connect again with the gas sample reservoir, lower *B* and draw into the burette *A* slightly more than 100 ml of gas. Close *C* and disconnect it from the gas sample reservoir. Allow the burette to drain for 30 s. Then raise *B* untilthe meniscus is at 100 ml mark. Pinch the rubber tubing with one hand and with the other open *C* momentarily to the air through the right hand capillary tube and allow the excess gas to pass out bringing the gas in the burette *A* to atmospheric pressure. Immediately close *C* and bring the water in *B* and in burette *A* exactly to the same level. Read and record the volume of gas. Thevolume of gas should be 100 ml in the burette.

**A.3.3** Place the pipette D in the stand E and adjust height. Fill the pipette D by pouring potassium hydroxide solution through a funnel inserted in the open stem of the bulb J so that the meniscus of the potassium hydroxide solution is near the lower edge of the stopcock G. Before connecting the pipette D to the right hand capillary of the burette A put a little acidulated water in cup F of the pipette D and by turning the three way stopcock G allow acidulated water to fill the capillary tube completely, and then close stopcock G. Connect the pipette D with the burette A with a rubber tube, squeezing the rubber tube between the thumb and forefinger and slipping it over the capillary of the burette without introducing air. Raise B sufficiently to put the gas in the burette under slight pressure. Open C to connect to the right hand capillary and then cautiously turn G off the pipette to allow the gas to drive the water in the capillary back into cup F. When the gas reaches stop cock G turn it to communicate with pipette bulb H and pass gas into the pipette D by raising B until the water reaches the top of the burette A. Allow it to remain for 5 to 7 seconds and then lower B and return all the gas to the burette A. Repeat this process 3 to 4 times. Lower B and draw the gas back into the burette until the potassium hydroxide solution reaches its initial mark on the capillary below G. Turn G to connect with F and draw acidulated water from the cup through the capillary to G and close C. Allow the burette to drain for 30 s and the reading of the burette. Repeat this operation by again transferring the gas to the pipette to ensure complete absorption. Two consecutive readings should agree within 0.05 ml.

### A.4 Calculation

Carbon dioxide, percentage by volume = 
$$\frac{V_2 - V_1}{V_2} \times 100\%$$

Where:

- $V_2$  = Volume of the gas sample taken for the test, in mI and,
- $V_1$  = Volume of the gas after absorption, in ml.

### Annex B

# (normative)

# Determination of carbon monoxide

### B.1 Method

A known volume of the gas is passed through hot iodine pentoxide. Any carbon monoxide present is thus oxidized to carbon dioxide which is estimated by absorption in baryta solution.

### **B. 2 Apparatus**

The apparatus shall consist of the following components assembled as shown in Figure 2.

**B.2.1** *Mercury filled by*-pass safety valves (A)

**B.2.2** Gas scrubbers (B) – Containing 40% potassium hydroxide solution

**B.2.3***Gas bubblers (C)* – containing saturated solution of chromic acid in concentrated sulphuric acid.

B.2.4 U-tube - one limb (D) - Containing magnesium perchlorate.

- **B.2.5** *U-tube* (*F*) Containing iodine pentoxide
- B.2.6 Thermometer (H) reading up to 200°C, graduated to 0.5°C

**B.2.7** Absorber (J) – Containing potassium iodine solution

**B.2.8** Carbon dioxide absorbers (K) – Two each containing baryta solution and fitted with burette containing standard hydrochloric acid.



Figure 2 - Assembly of iodine pentoxide apparatus for determination of carbon monoxide

### **B.3 Reagents**

### B.3.1 Standard hydrochloric acid - Approximately 0.02 mol/L.

**B.3.2** Baryta solution – Dissolve about 4 g of barium hydroxide in 200 ml of water in the large flask. Close the flask and shake until the crystals have completely disappeared and a light, insoluble powder of barium carbonate remains. Allow the solution to stand for two days, until the barium carbonate has completely settled; siphon it into a bottle through which current of air (free from carbon dioxide) has been passed for 2 hours previously. Connect this bottle with soda-lime tube and with burette as shown in Figure 3.



Figure 3 - Bottle for storing baryta solution

B.3.3 lodine pentoxide

B.3.4 Potassium iodide solution - Dissolve 5g of potassium iodide crystals in 100 ml of water

B.3.5 Phenolphthalein indicator - Dissolve 0.50g of phenolphthalein in 100 ml of rectified spirit

### **B.4 Procedure**

**B.4.1** Take 25 ml of baryta solution in each carbon dioxide absorber K and add water to about half its height. Maintain the temperature of iodine pentoxide bath at 150°c. Flush apparatus with about 5 litres of nitrogen. Pass the carbon dioxide mixed with an equal volume of pure nitrogen at the rate of about 5 litres per hour. Measure the rate of flow of the gas by suitable device and record the temperature and atmospheric pressure at regular interval. Pass 10 litres of carbon dioxide, keeping the temperature and pressure of the gas constant. Titrate individually the baryta solution in both absorbers with standard hydrochloric acid using phenolphthalein as indicator. Find out the total volume of standard hydrochloric acid used in the two titrations.

B.4.2 Cary out blank titration with an equal volume of baryta solution used for absorption

### **B.5 Calculation**

Carbon monoxide, ppm by volume = 
$$\frac{3.098 (V_1 - V_2)(273 + t) M}{P}$$

Where

- V1= Volume in ml of standard hydrochloric acid used for the blank titration baryta solution in B.3.2
- V<sub>2</sub> = Volume in mI of standard hydrochloric acid used for titration of baryta solution in B.3.1
- T = Average temperature in degrees centigrade
- M =Molarity of the standard hydrochloric acid and
- P = Average pressure in millimeters of mercury

### Annex C

#### (Normative)

#### Test for acids and sulphur dioxide

#### C.1 Apparatus

C.1.1 Nessler tubes – 50 ml capacity.

#### C.2 Reagents

C.2.1 Methyl orange indicator solution – Dissolve 0.01 g of methyl orange in 100 ml of water.

C.2.2 Standard hydrochloric acid – 0.01 mol/l.

#### C.3 Procedure

**C.3.1** Take 50 ml of carbon dioxide free cooled water in a vessel of such diameter that a hydrostatic column of 12 to 14 cm is produced. Pass carbon dioxide equivalent to 1000 ml at normal temperature and pressure, through the water using a delivery tube having an orifice about 1 mm in diameter and extending to within 2 mm of the bottom of the vessel. Regulate the flow of gas so that about 15 min are taken for passing the gas. After passing the gas, transfer the liquid to a Nessler tube and add 0.1 ml of methyl orange, indicator solution. Carry out a control test in another Nessler tube, adding 50 ml of carbon dioxide free water, 1 ml of standard hydrochloric acid and 0.1 ml of methyl orange indicator.

**C.3.2** The material shall be taken to have passed the test if the intensity of red colour of the test solution is not greater than that in the control test.

### Annex D

### (Normative)

#### Test for phosphine and hydrogen sulphide

#### **D.1 Reagents**

**D.1.1** Silver nitrate solution – 10%.

**D.1.2** Ammonium hydroxide – 1:1 (v:v).

#### D.3 Procedure

**D.3.1** Pass carbon dioxide equivalent to 1 000 ml at normal temperature and pressure through a mixture of 25 ml of silver nitrate solution, 5 ml ammonium hydroxide and 20 ml of water.

**D.3.2** The material shall be taken to have passed the test if no turbidity is produced and the solution is not darker than an equal quantity of test solution through which no gas has been passed.

### Annex E

#### (normative)

### **Determination of moisture**

### E.1 Absorption method

### E.1.1 Apparatus

The apparatus consists of the following parts assembled as shown in Figure 4.

E.5.1.1 Gas meter, accurate to 1 percent.

**E.5.1.2** Absorption Train, three U tubes containing phosphorous pentoxide connected in a series to the gas meter. The one near to the gas meter will serve as a guard to prevent moisture from backing into the first tube.



Figure 4 - Assembly of apparatus for determination of moisture

### E.5.2 Procedure

Connect the 3 tubes in series to the inlet of the gas meter and connect the free end of u –tubesto the gas Sample. Allow the gas to flow through the train for a while to saturate the water in the gas meter with the gas as well as replacing the air in the first two U-tubes. Stop the gas flow, close the stopcocks of the u-tubes and weigh the first two U-tubes. Connect again, record the meter reading. Pass the gas through the train at the rate of 10 litres per hour for 4 to 5 h. Close the gas supply end record the meter reading. Remove the first two U-tubes and weigh.

### E.5.3 Calculation

Convert the volume of the gas taken for test to normal temperature and pressure. From the increase in weight, calculate the volume of moisture present on the basis that 1 g of water is equivalent to 1.244 litres of water vapour at normal temperature and pressure and express the results as percentage by volume.

# Annex F

# (Normative)

### Test for arsenic

# F.1 Apparatus

The apparatus shall consist of the following parts assembled as shown in Figure 5.

**F.1.1** *Evolution flask* – capacity 100 to 125 ml and scrubber with glass wool attached to the reaction flask by ground join.

**F.1.2** Arsenic Absorber – with a calibrated mark at 4 ml. This is connected to the scrubber by means of ball and socket joint through the side capillary tube of the absorber.

**F.1.3** Spectrophotometer – with 10 mm cell.

**F.1.4** *Dreschel Bottle* – with fitted tube.

F.1.5 Gas meter, wet type.



# Figure 5 - Assembly of apparatus for testing of Arsenic

F.2 Reagents – only arsenic free reagents should be used.

F.2.1 Lead acetate solution - Dissolve 10g of lead acetate in 100 ml of water.

**F.2.2** Silver Diethyldithiocarbamate-Pyridine Solution – Dissolve 1.0 g of pure drydiethyldithiocarbamate in 200 ml of pyridine, store in amber coloured bottle.

F.2.3 Potassium Iodine Solution – Dissolve 15 g of potassium iodide in 100 ml of water.

**F.2.4** Standard Arsenic Solution – Dissolve 1.320 g of arsenious oxide in minimum volume of 1 M sodium hydroxide solution, acidify with dilute hydrochloric acid and make up to one litre in a volumetric flask. One millilitre contains 1 mg of arsenic. Dilute to get a solution containing 1  $\mu$ g per ml.

#### F.2.5 Concentrated Hydrochloric Acid

**F.2.6** *Stannous Chloride Solution* – Dissolve 40 g of stannous chloride (SnCl<sub>2</sub>.2H<sub>2</sub>O) in 100 ml of concentrated hydrochloric acid.

**F.2.7** Zinc Powder – 500 to 850 µ particle size.

F.2.8 Potassium Hydroxide Solution – 0.1 M.

NOTE: All glassware used should be thoroughly cleaned with either hot concentrated sulphuric acid or boiled nitric acid, followed by rinsing with distilled water, and then with acetone.

### F.3 Procedure

**F.3.1** Impregnate the glass wool (purified) in the scrubber with lead acetate solution. Charge the absorption tube with 4.00 ml of silver diethyldithiocarbamate reagent.

**F.3.2** Prepare a calibration curve by pipetting suitable aliquots of the dilute standard arsenic solution into a series of clean evolution flasks. Cover the range 0 to 10  $\mu$ g of arsenic. To each add 5 ml of concentrated, 2.0 ml of 15% potassium iodide solution and 8 drops of stannous chloride solution. Swirl the contents of the flasks and allow them to stand for 15 minutes to ensure complete reduction. Add 5.0 g of zinc and insert hydrogen sulphide scrubber immediately the evolution of arsine is 99 percent complete in 30 minutes and virtually complete in 40 minutes. If necessary, dilute the liquid in the arsine absorber with pure pyridine to the 4 ml mark and pass gentle stream of air through the absorber to mix the solution. Transfer the absorbing solution to a 10 mm cell and measure the transmittance at 540 m $\mu$  in the spectrophotometer. Repeat the procedure with the remaining flasks. Plot the transmittance of each aliquot (less that of the blank) against arsenic content in  $\mu$ g.

**F.3.3** Pass 25 to 50 litres of carbon dioxide through 25 ml of 0.1 M potassium hydroxide solution in 100 ml dreschel bottle at the rate of 10 litres per hour. The gas is measured by the gas meter placed after the bubbler. The water in the gas meter is saturated with the gas prior to the experiment.

**F.3.4** Take an aliquot from the treated potassium hydroxide in F.3.3 to give about 5.0  $\mu$ g of arsenic and follow the same procedure as in F.3.2. From the transmittance obtained at 540 m $\mu$  evaluate the arsenic content of the sample by reference to the calibration graph.

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#### Annex G

#### (normative)

#### Determination of non – volatile hydrocarbons

**G.1** Pass a sample of liquid carbon dioxide from storage container or sample cylinder through a commercial carbon dioxide snow horn directly into an open, clean container. Weigh 500 g of this sample into a clean beaker. Allow carbon dioxide solid to sublime completely, with a watch-glass placed over the beaker to prevent ambient contamination. Wash the beaker with a residue-free solvent, and transfer the solvent from the beaker to a clean, tared watch-glass or petri dish with two additional rinses of the beaker with the solvent. Allow the solvent evaporate, suing heating to 104°Cuntil the watch-glass or petri dish is at a constant weight. Determine the weight of residue by difference. The weight of the residue does not exceed 5 mg.

#### Annex H

#### (normative)

#### Determination of volatile hydrocarbons

#### H.1Standards Preparation

Flusha 500 ml glass, septum-equipped sampling bulb with helium, and inject into the bulb a 5.00 ml sample of methane. Allow the bulb to stand for 15 minutes to permit the gases to mix, and then inject 2.50 ml of the mixture into a second 500 ml sampling bulb, also flushed with helium, and allow this tube to stand for 15 minutes to permit the gases to mix. This mixture is a nominal 50 ppm v/v standard. Determine the exact concentration from the exact volumes of the gas sampling bulbs. To determine these volumes, weigh the empty tubes, fill them with water, and reweigh. From the weight of the water and its temperature, calculate the volume of the tube.

#### H.2 Chromatographic system

The gas chromatograph is equipped with flame ionization detector and a 1.8-m x 3-mm o.d. metal column packed with 80- to 100-mesh Hayesep Q (or equivalent). The carrier gas is helium at flow rate 30ml/min. the injector temperature and the detector temperatures both are maintained at 230°C. The column temperature is programmed according to the following steps: it is held at 70°C for 1 min, then increased to 200°C at a rate 20°C/min, and then held at 200°C for 10 minutes. The parameters for the detector ae sensitivity range: 10<sup>-12</sup> A/mV; attenuation: 32. The concentration of volatile hydrocarbons is reported in methane equivalents. The various gas chromatograph responses, excluding the carbon dioxide response, are summed to yield the total volatile hydrocarbon concentration. The composition of hydrocarbons present will vary from sample to sample. Typical retention times are methane: 0.4 min; carbon dioxide: 0.8 min; hexane: 14.4 minutes.

### H.3 Procedure

Inject in triplicate 1.00 ml of the standard preparation into the gas chromatograph, and average the peak area responses. The relative standards deviation should not exceed 5.0%. Similarly, inject in triplicate 1.00 ml of sample, sum the average peak areas as the individual peaks, except the carbon dioxide peaks.

### **H.4 Calculation**

Volatile hydrocarbon, 
$$\mu l/l$$
, max =  $S\left(\frac{A_u}{\Delta}\right)$ 

Where:

S = calculated concentration of methane in ppm in the standard preparation (approximately 50  $\mu$ l/l,

 $A_u$  = the sum of the averages of the individual peak area response in the sample,

 $A_s$  = the sum of the averages of the standard preparation area response.

### Annex I

### (Normative)

### Determination of oil

### I.1 Apparatus

- I.1.1 Two gauge Bubblers shown in Figure 6.
- I.1.2 Dreschel Bottle 250 ml capacity.
- I.1.3 Flow meter to measure 200 to 2 000 ml of carbon dioxide per minute.
- I.1.4Wet gas meter calibrated for 1 or 2.5 litres per revolution.
- I.1.5 Infrared Spectrometry and accessories to measure at 3.46µ.





### I.2 Reagents

**I.2.1** Carbon Tetrachloride – Distilled, analytical reagent quality. The infra-red absorption at 3.46  $\mu$  of each fresh supply of this reagent should be determined before the use to ensure that it is of suitable quality.

**1.2.2** *Standard Oil Solution* – Dissolve 0.0200 g of liquid paraffin, in carbon tetrachloride and dilute to exactly 100 ml with more carbon tetrachloride. One millilitre of this solution contains 200µg of oil.

#### I.3 Procedure

**I.3.1** About 800 to 1000 g of material is needed for each determination. A set of small sampling cylinders of 1.5 to 2 Kg capacity with one valve at each end may be used for containing this amount of gas.

Note: A set cylinders should preferably be retained for sampling for oil and not used for another purpose.

**1.3.2** *Preparation of the test solution.* Place 100 ml of the carbon tetrachloride in the dreschel bottle and 25 ml in each gage bubbler. Connect the weighed sampling cylinder, supported vertically with the dip tube at the top of the evaporator and thence to absorption train, flow meter and wet gas meter in series using short rubber connections. Alternatively, for this determination the sample cylinder may be supported vertically with the dip tube valve at the bottom; when this is done, omit the evaporating device and connect the absorption train to the top valve of the cylinder so that gaseous carbon dioxide can be withdrawn.

**I.3.3** Totally expand the contents of the cylinder through the absorption train at the rate of about 1000 ml per minute and read the volume of the gas passed; this serves as a check on the amount of carbon dioxide used for test.

NOTE: Some evaporation of the carbon tetrachloride will take place. If this is excessive, stop the gas flow and refill the absorption bottles noting the volume of carbon tetrachloride added.

**I.3.4** Reweigh the sampling cylinder taking care to use the same valve fittings as the first weighing. Dismantle the lower cylinder valve and wash the inside of the cylinder with 25 ml of carbon tetrachloride. Wash the interior of the evaporating device and valves with carbon tetrachloride and combine this solution with the cylinder washings and the bubbler contents. Adjust the combined solutions to some suitable definite volume, after previous evaporation, if this is considered necessary for the particular infra-red spectrometer to be used. This is the test solution.

**1.3.5** Preparation Control – Evaporate a volume of carbon tetrachloride equal to the volume (including any additions) of carbon tetrachloride used in the absorption train plus washings to the volume of the combined solution and subsequently treat it in the same way as the test solution. If there is definitely no blank on the carbon tetrachloride, it is sufficient to prepare a control of volume equal to that of the final test solution.

**I.3.6** Preparation of the Calibration Chart – Prepare suitable dilutions of the standard oil solution to cover the range of values within which the weight of oil in the sample is expected to be found. Measure optical densities of this series of solutions as described above. Prepare a calibration chart by plotting the weights of oil against the corresponding optical densities.

**1.3.7** Measurement of Infra-red Absorption of the Test Solution – Following the manufacturer's instructions for the operation of the particular instrument used, determine the optical densities of the test solution and control at the wavelength of maximum absorption (approximately 3.46  $\mu$ ). From the calibration graph, compute the weights of oil present in the total volume of both test solution and control.

### **I.4 Calculation**

oil content, ppm by weight = 
$$\frac{m_1 - m_2}{m_2}$$

Where

 $m_1$  = mass in µg of the oil in test solution,

 $m_2$  = mass in µg of the oil in control and

 $m_3$  = mass in g of the sample taken.

### Annex J

### (informative)

### Determination of odour

**J.1** Carefully open the container valve to produce moderate flow of gas. Do not direct the gas stream towards the face, but deflect portion of the stream toward the nose: no appreciable odour shall be discernible.

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