DRAFT TANZANIA STANDARD

Gaseous nitrogen – technical grade – Specification TANZANIA BUREAU OF STANDARDS

2<sup>nd</sup> edition

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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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The Organization marked with an asterisk (\*) in the above list, together with the following were directly represented on the technical committee entrusted with the preparation of this standard:

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### 0.FOREWORD

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

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

IS 1747:1972 Specification for Nitrogen, published by the Bureau of Indian Standards,

BS 4366:1968 Specification for nitrogen industrial, published by the British Standard Institution.

This second edition cancels and replaces the first edition of (**TZS 218:2017**), which has been technically revised

**0.3.** 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.

## 1. SCOPE

This Tanzania Standard prescribes requirements, sampling and test method for gaseous nitrogen of technical grades.

Gaseous nitrogen of technical grade is used as a freezing agent, propellant, packaging gas, purging and general purposes.

It doesn't intend to be used for packaging of food or food products.

### 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

3.1.1. Gaseous nitrogen of technical grade shall be odourless and colourless.

### 3.2. Specific requirements

When tested according to the methods prescribed in Annex A, gaseous nitrogen shall comply with the specific requirements in Table 1.

## Table 1- Requirements for gaseous nitrogen - technical grade

S/ No.	Characteristic	Requirement	Test method
(i)	Nitrogen, percent by volume, min.	99.5	Annex A
(ii)	Oxygen, percent by volume, max.	0.5	Annex B

### 4. Sampling

# 4.1. Preparation of test samples

**4.1.1.** Samples of compressed nitrogen shall be taken from a steel cylinder containing the gas direct to the appropriate apparatus for all determination. The valve and connecting lines shall be carefully purged before taking a sample.

**4.1.2.** When samples of liquid nitrogen are taken, efforts should be made to ensure complete vaporization of the sample.

# 4.2. Scale of sampling

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

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

# 4.3. Method of sampling

**4.3.1.** 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	
301 and above	25	

Table 2– Number of cylinders to be selected

**4.3.2.** The cylinders shall be selected at random and to ensure randomness of selection, a random number table as agreed 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 each of the cylinders selected according to **4.3.2** 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

The 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 or deionized water of equivalent purity shall be used for the appropriate tests.

# 8. Packaging and marking

# 8.1. Packaging

The nitrogen of technical grade shall be packaged in compressed gas cylinder(s) or low pressure bulk liquid system.

# 8.2. Marking

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

### Annex A

### (normative)

# Method of determination of nitrogen by gas chromatograph

## A.1 Column

A.1.1 Material - stainless steel of 2 m length with 2 mm diameter

**A.1.2** *Packing Material* – appropriate molecular sieve capable of absorbing molecules up with diameters up to 0.5 nm.

# A.2 Carrier

A.2.1 Gas- helium gas of not less than 99.995% (v/v) of He.

A.2.2 Detector- thermal conductivity detector.

A.2.3 Injector- loop injector.

A.2.4 Column temperature- 50°C.

A.2.5 Detector temperature- 130°C.

A.2.6 Reference gas (a) –ambient air.

**A.2.7** *Reference gas (b)* –nitrogen (not less than 99.999% of N<sub>2</sub>, less than 1 ppm CO, less than 5 ppm O<sub>2</sub>).

# A.3 Procedure

**A.3.1** Inject reference gas (a). Adjust the injected volumes and operating conditions so that the height of peak due to nitrogen in the chromatogram is at least 35% of full scale of the recorder. The assay is not valid unless the chromatograms obtained show a clear separation of oxygen and nitrogen.

**A.3.2** Inject the gas to be examined and the reference gas (b). In the chromatogram obtained with the gas to be examined, the area of the principal peak is at least 99.0% of the area of the principal peak in the chromatogram obtained with reference gas (b).

## Annex B

(normative)

# Method of test for oxygen for technical grade nitrogen

## **B.1 Apparatus**

**B.1.1**The assembly of the apparatus shall be as shown in Figure 1. Lubricate all taps with suitable vacuum tap grease, Silicon greases are unsuitable

**B.1.2** Test the apparatus for leaks by filling the burette with the water closing tap *F* and lowering the levelling bottle. On standing, the level of the water shall not fall.



Figure 1 - Apparatus for the determination of oxygen and other active contaminants

## **B.2 Reagents**

**B.2.1** *Pyrogallol solution* - Dissolve 350 g of pyrogallol oxide in 1000 ml of water. Keep the stock solution in amber coloured bottles.

**B.2.2***Potassium hydroxide solution* - Dissolve 1000 g of potassium hydroxide in 1000 ml of water.

**B.2.3**Confining liquid - Approximately 1 ml sulphuric acid containing a few drops of methyl orange to facilitate reading

# **B.3 Procedure**

**B.3.1** Pour 140 ml pyrogallol solution and 100 ml of potassium hydroxide solution into Woulfe's bottle A through opening J. Immediately after introducing the solutions, close the opening with rubber bung and shake the bottle A with the solutions, fill measuring burette C with water by raising levelling bottle D, closing one way tap and turning three way tap F, to position 2. When the water in the burette reaches the zero mark, turn three way tap Fto positional I and replace bottle D, in set.

**B.3.2** The burette shall be capable of measuring to an accuracy of 0.05 ml.

**B.3.3** Open one way to G and lower bottle D to draw the liquid into absorption pipette B from bottle A. When burette C becomes full of gas, close one-way tap G and turn three-way tap F to position 2 and raise bottle D until burette E is full of water again. Turn three way top F to position A and repeat the foregoing operations until the level of the solution reaches mark L. Perfect control of the level may be obtained by raising or lowering bottle D slowly.

**B.3.4** Connect the supply of gas to inlet of a three way tap F, the tap being in position 2, purge the inlet tube with the sample and then turn tap F to position 2. Bring the level of water in bottle D in line with graduation mark 100 on burette C. Turn tap F to position 3 and raise water bottle D to expel all the gas to atmosphere. Again turn tap F to position 2 and lower water D in line with graduation mark 100 on burette C. Repeat this process of purging burette C and the manifold connections up to G and F once again.

**B.3.5** Turn tap F to position I again. Allow water to drain down the sides of burette C and read the volume keeping water level same in burette C and bottle D. Open tap G, and raise bottle D until burette C is just full of water. Close tap G, replace bottle D in a set and leave gas to be tested in absorption pipette B for a few minutes so that oxygen and other active contaminants are absorbed from it.

**B.3.6** Open tap *G* lower bottle *D* until the level of the solution again reaches mark *L*. To ensure complete absorption, raise the bottle again until burette*C* isfull of water, close tap *G*, replace bottle*D* Inset for another few minutes. Open tap *G* and lower bottle *D* until the level of the solution again reaches mark*L*. Close tap G and finally allow water to drain in the burette *C* and read the volume of gas, keeping the water level same in *Cand D*.

B.3.7 Repeat the steps outlined in B.3.5 and B.3.6 until two consecutive readings agree

**B.4 Calculation** 

Oxygen, percent by volume =  $\frac{V - V_1}{V} \times 100$ 

Where;

/ = Initial volume of gas (see B.3.5) and

V<sub>1</sub>= Volume of gas after absorption (see B.3.7)