

NITROUS OXIDE

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SYNONYMS

Nitrogen oxide, Dinitrogen monoxide; INS No. 942

DEFINITION

Chemical names Dinitrogen monoxide

C.A.S. number 10024-97-2

Chemical formula N₂O

Formula weight 44.01

Assay Not less than 97% (v/v)

DESCRIPTION Colourless, odourless gas

FUNCTIONAL USES Propellant, antioxidant, packaging gas, foaming agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) 1 volume dissolves in 1.5 volumes of water (20°, 760 mm Hg)

Flame test A glowing splinter of wood in contact with nitrous oxide bursts into flame (distinction from nitrogen).

Pyrogallol test Passes test (distinction from oxygen).
See description under TESTS

PURITY

Carbon monoxide Not more than 10 µl/l
See description under TESTS

Nitric oxide and nitrogen dioxide Not more than 5 µl/l
See description under TESTS

Halogens and hydrogen sulfide Not more than 5 µl/l
See description under TESTS

Arsine and phosphine Passes test
See description under TESTS

TESTS

IDENTIFICATION TESTS

Pyrogallol test

Collect about 100 ml of the gas under test in a 100-ml tube fitted at the top with a stopcock. Open the stopcock, and quickly add a freshly prepared solution of 500 mg of pyrogallol in 2 ml of water and a freshly prepared solution of 12 g of potassium hydroxide in 8 ml of water. Immediately close the stopcock, and mix. The gas is not absorbed and the solution does not become brown.

PURITY TESTS

NOTE: For the following tests keep the cylinder of gas from which the sample is taken at room temperature for not less than 6 h before carrying out the tests. In all the tests the cylinder is kept in the vertical position with the outlet valve uppermost when delivering the gas. Pass the gas at a steady rate of 4 l/h unless otherwise stated and carry out the tests or calculate the results with reference to the gas at 20° and 760 mm Hg.

Carbon monoxide

Principle: Carry out the test on the first portion of gas issuing from the cylinder. Use 5.0 l of the nitrous oxide in the test and 5.0 l of carbon monoxide-free air as the control. The difference between the volumes of 0.002 N sodium thiosulfate used in the two titrations is not greater than 0.5 ml.

Apparatus: The apparatus consists of the following parts connected in series:

- U-tube containing anhydrous silica gel impregnated with chromium trioxide.
- Scrubber bottle (dreschel type) containing 100 ml of a 40% w/v solution of potassium hydroxide.
- U-tube containing pellets of potassium hydroxide.
- U-tube containing phosphorous pentoxide dispersed on previously granulated, fused pumice.
- Tube containing recrystallized iodic anhydride (I_2O_5) in granules, previously dried at 200° and kept at a temperature of 120°. The iodic anhydride is packed in the tube in 1 cm columns separated by 1 cm columns of glass wool to give an effective length of 5 cm.
- Flask containing 2.0 ml of potassium iodide TS and 3 drops of starch solution TS.

Procedure

Flush the apparatus with 5.0 l of carbon dioxide-free air and, if necessary, discharge the blue colour in the iodide solution by adding the smallest necessary quantity of freshly prepared 0.002 N sodium thiosulfate. Continue flushing until not more than 0.045 ml of 0.002 N sodium thiosulfate is required after passing 5.0 l of carbon dioxide-free air. Pass the gas from the cylinder through the apparatus, using the volume and the rate of flow prescribed in the monograph. Flush the last traces of liberated iodine into the reaction flask by passing through the apparatus 1.0 l of carbon monoxide-free air. Titrate the liberated iodine with 0.002 N sodium thiosulfate.

Carry out a blank assay under the same conditions, using the carbon monoxide-free gas prescribed in the monograph. The difference between the volumes of 0.002 N sodium thiosulfate used in the two titrations is not

greater than the limit prescribed in the monographs.

Nitric oxide and nitrogen dioxide

Principle: Carry out the test after the 5 l used in the test for carbon monoxide have been released from the cylinder. Pass the gas, at a rate of 15.0 l/h, through a solution containing 2.5% w/v of potassium permanganate and 1.2% v/v of sulfuric acid into an evacuated gas sampling tube of 1 l nominal capacity and fill to a pressure about 50 mm below that of the atmosphere, measured on a mercury manometer. Calculate the volume of gas at 20° and 760 mm Hg.

Sulfanilic acid/naphthylenediamine TS

- Solution I: Dissolve 2 g of sulfanilic acid TS in a mixture of 10 ml of glacial acetic acid and 180 ml of water.

- Solution II: Dissolve 0.2 g of naphthylenediamine dihydrochloride in 10 ml of a 50% v/v glacial acetic acid solution heating gently and dilute to 200 ml with water.

Mix 9 volumes of Solution I with 1 volume of Solution II.

Procedure

Introduce 20.0 ml of sulfanilic acid/naphthylenediamine TS into the sampling tube by dipping the end into a dish containing the mixture and carefully opening the tap. Shake the tube and allow to stand for 10 min with occasional shaking. Prepare the reference solution by adding 0.25 ml of 0.00308% w/v sodium nitrite solution to 20.0 ml of the sulfanilic acid/naphthylenediamine TS. Measure the absorbance of a 1 cm layer of the resulting solutions at 550 nm and correct the result to 1.0 l of the gas at 20° and 760 mm Hg. The extinction of 1.0 l of the test substance is not greater than that of the reference solution.

Halogens and hydrogen sulfide

NOTE: For the following test pass the gas into a closed, flat-bottomed glass cylinder fitted with (a) a delivery tube having an orifice of 1 mm internal diameter and reaching to 2 mm from the bottom of the cylinder and (b) an outlet tube. The delivery tube is immersed in the reagent to a depth of 12 to 14 cm.

Pass 10.0 l through 49 ml of water containing 1 ml of silver nitrate TS. The solution does not darken. After 5 min, any opalescence produced is not more intense than that obtained with a mixture of 1 ml of silver nitrate TS, 20 ml of chloride standard solution (5 mg of Cl per l), 0.15 ml of dilute nitric acid TS and sufficient water to make 50 ml of solution and left to stand for 5 min.

Arsine and phosphine

Silver DDC/Quinoline TS: Dissolve 50 mg of finely powdered silver nitrate in 100 ml of quinoline and add 0.2 g of silver diethyldithiocarbamate. The reagent should be freshly prepared.

Procedure: Pass 10.0 l at a rate of 1.0 l/min through a glass tube packed with lead acetate TS on cotton wool and a gas distribution head of the domed, sintered type, of porosity 100, into a tube of about 2.5 cm internal diameter, containing 5 ml of Silver DDC/Quinoline TS, the domed head being almost in contact with the bottom of the tube. The colour of the Silver DDC/Quinoline TS is not changed.

METHOD OF ASSAY

Use a gas burette (see Figure below) of 100 ml capacity having as its upper end a two-way tap connected to two capillaries, one of which (tube A) is used to introduce the gas into the apparatus, the other (tube B) connected to the condenser (C) and the manometer (M). The lower part of the gas burette has a one-way tap connected to a mercury reservoir by a rubber tube. The gas burette is graduated from 0 to 5 ml in tenths of a ml in the upper part and from 99.5 to 100.5 ml in tenths of a ml in the lower part. The capillary tube (B) is connected to a vertical capillary arm to form a four-way junction. The descending arm of the junction is connected to a condenser (C) of about 60 ml capacity. The right arm of the junction is connected to a mercury manometer (M). A tap (D) on the upper vertical arm of the junction opens to the air.

Close the three taps and immerse the condenser in liquid nitrogen, keeping the level slightly above the upper part of the condenser. By manipulating the two-way tap and the mobile reservoir create a partial vacuum in the apparatus, choosing an arbitrary pressure P_0 between 50 and 60 mm Hg, accurately measured. This pressure must remain constant for 10 min to demonstrate that the apparatus is gas-tight.

Open the two-way tap to tube (A) and completely fill the burette and tube (A) with mercury. Close the two-way tap. Connect a rubber tube to the exit valve of the nitrous oxide cylinder through a suitable pressure relieving device and pass a current of nitrous oxide through the rubber tube for 1 min. Whilst the gas is still flowing, connect the rubber tube to the end of tube (A) and immediately open the two-way tap to tube (A). Allow 100 ml of nitrous oxide to enter the burette by lowering the mercury reservoir. Close the two-way taps.

Raise the mercury reservoir slightly above the tube (A) and lower the level of the liquid nitrogen to the middle of the condenser. Carefully open the tap of the burette to connect with the condenser and allow the mercury to rise in the burette until it reaches the tap. Close the tap. Raise the level of the liquid nitrogen so as to totally immerse the condenser. Read the pressure and wait until it remains steady for 2 min.

Place the mercury reservoir in its bottom position, open the tap of the burette to make connection with the condenser. Move the mercury reservoir until the manometer reading is the same as the initial pressure P_0 . Close the tap of the burette and by means of the mercury reservoir bring the pressure of the gas in the burette to atmospheric pressure. The number of ml of gas represents the non-condensable volume in 100 ml of nitrous oxide. The volume of non-condensable gas must not exceed 3 ml. After each series of 10 determinations allow atmospheric air to enter by opening the tap (D), remove the liquid nitrogen from the condenser and allow the condenser to warm to room temperature.

