# CARBON DIOXIDE

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## SYNONYMS

INS No. 290

## DEFINITION

| | |
|---|---|
| C.A.S. number | 124-38-9 |
| Chemical formula | $CO_2$ |
| Formula weight | 44.01 |
| Assay | Not less than 99% of $CO_2$, by volume |

## DESCRIPTION

Colourless, odourless gas, 1 litre of which weighs about 1.98 g at 0$^{o}$ and 760 mm of mercury. Under a pressure of about 59 atmospheres it may be condensed to a liquid, a portion of which forms a white solid (Dry Ice) upon rapid evaporation. Solid carbon dioxide evaporates without melting upon exposure to air. Commercial carbon dioxide is shipped and handled in pressurized cylinders or low pressure bulk liquid systems, or in solid blocks.

## FUNCTIONAL USES

Propellant, freezing agent, carbonating agent, preservative, extraction solvent

## CHARACTERISTICS

The following specifications apply to gaseous carbon dioxide as produced from its condensed liquid or solid phase by evolution to the gas phase at normal environmental conditions. Additional specifications may be applied to liquid or solid forms of carbon dioxide by vendors or by specific users of commercial carbon dioxide products.

### IDENTIFICATION

Precipitate formation

When a stream of the sample is passed through a solution of barium hydroxide, a white precipitate is produced which dissolves with effervescence in dilute acetic acid.

Detector tube test

Passes test
See description under TEST

### PURITY

Acidity

Transfer 50 ml of water, previously boiled and cooled to room temperature, into a Nessler tube. Introduce 1,000 ml of the sample into the water through a tube (1 mm internal diameter) keeping the opening of the tube within 2 mm from the bottom of the vessel. Add 0.1 ml of methyl orange TS. The red colour produced is not darker than the colour of an identical control solution to which has been added 1.0 ml of 0.01 N hydrochloric acid instead of the carbon dioxide.

Phosphine, hydrogen sulfide, and other organic reducing substances

Transfer 25 ml of silver ammonium nitrate TS and 3 ml of ammonia TS into a Nessler tube. In the absence of light, introduce 1,000 ml of the sample in the same manner as in the test of Acidity. No brown colour is produced.

Carbon monoxide

Not more than 10 µl/l
See description under TESTS

Non-volatile hydrocarbons

Not more than 10 mg/kg
See description under TESTS

Volatile hydrocarbons

Not more than 50 µl/l
See description under TESTS

Water

Passes test
See description under TESTS

## TESTS

### IDENTIFICATION TESTS

Detector tube test

Pass 100±5 ml, released from the vapour phase of the contents of the container, through a carbon dioxide detector tube (see below) at the rate specified for the tube: The indicator change extends throughout the entire indicating range of the tube.

Detector tube is a fuse-sealed glass tube (Draeger or equivalent) that is designed to allow gas to be passed through it and that contains suitable adsorbing filters and support media for the indicators hydrazine and crystal violet. (The Draeger Reference Number is CH 30801, National Draeger Inc., P.O. Box 120, Pittsburgh, PA 15205-0120, USA; the measuring range is 0.01% to 0.30%).

### PURITY TESTS

Carbon monoxide

Principle
Carry out the test on the first portion of gas issuing from the cylinder. Use 5.0 l of the sample mixed with an equal volume of carbon monoxide-free nitrogen and 10 l of carbon monoxide-free nitrogen 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 phosphorus pentoxide dispersed on previously granulated, fused pumice.
-Tube containing recrystallized iodic anhydride ($I_2O_5$) in granules, previously dried at 200$^o$ and kept at a temperature of 120$^o$. 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.

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 using 10 l of carbon monoxide-free nitrogen. The difference between the volumes of 0.002 N sodium thiosulfate solution used in the two titrations should not be more than 0.5 ml.

### Non-volatile hydrocarbons

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 the 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 to evaporate, using heating to 104° until the watch-glass or petri dish is at a constant weight. Determine the weight of the residue by difference. The weight of the residue does not exceed 5 mg.

### Volatile hydrocarbons

#### Standard preparation

Flush a 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 min 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 min 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 volumes of the tubes.

#### Chromatographic system

The gas chromatograph is equipped with the 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 a flow rate of 30 ml/min. The injector temperature and the detector temperatures both are maintained at 230°. The column temperature is programmed according to the following steps: It is held at 70° for 1 min, then increased to 200° at a rate 20°/min, and then held at 200° for 10 min. The parameters for the detector are sensitivity range: $10^{-12}$ A/mV; attenuation: 32. The concentration of volatile hydrocarbons is reported in methane equivalents. The various gas chromatographic 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 min.

Procedure

Inject in triplicate 1.00 ml of the standard preparation into the gas chromatograph, and average the peak area responses. The relative standard deviation should not exceed 5.0%. Similarly, inject in triplicate 1.00 ml of sample, sum the average peak areas of the individual peaks, except for the carbon dioxide peaks, and calculate the concentration v/v in the sample by formula:

$\mu l/L = S(A_U/A_S)$,

where

S is the calculated ppm of methane in the standard preparation (approximately 50 μl/l)

$A_U$ is the sum of the averages of the individual peak area responses in the sample

$A_S$ is the average area of the standard preparation area responses

Water

Water vapour detector tube. A fuse-sealed glass tube (Draeger or equivalent) that is designed to allow gas to be passed through it and that contains suitable absorbing filters and support media for the indicator, which consists of a selenium sol in suspension of sulphuric acid. (The Draeger Reference Number is CH 67 28531, National Draeger Inc., P.O. Box 120, Pittsburgh, PA 15205-0120, USA; the measuring range is 5 to 200 mg/m$^3$).

Pass 24,000 ml of the gas sample through a suitable water-absorption tube not less than 100 mm in length, which previously has been flushed with about 500 ml of the sample and weighed. Regulate the flow so that about 60 min will be required for passage of the gas. The gain in weight of the absorption tube does not exceed 1.0 mg.

## METHOD OF ASSAY

Transfer a 1 in 3 potassium hydroxide solution into a gas pipette of adequate volume. Measure accurately about 1,000 ml of the sample into a gas burette containing a 1 in 10 sodium chloride solution. Transfer the sample into the gas pipette and shake well. When the volume of gas remaining unabsorbed is constant (V ml), the content of carbon dioxide is calculated by:

$$\text{Content v / v\%} = \frac{\text{Vol of sample ml} - \text{V ml}}{\text{Vol of sample ml}} \times 100$$