

International Standard



5918

H-25-25

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Chlorofluorohydrocarbons for industrial use — Determination of inert gas content — Gas chromatographic method — General principles

*Hydrocarbures chlorofluorés à usage industriel — Dosage des gaz inertes — Méthode par chromatographie en phase gazeuse —
Principes généraux*

First edition — 1982-11-01

UDC 661.723 : 543.544 : 546.29

Ref. No. ISO 5918-1982 (E)

Descriptors : industrial products, halohydrocarbons, fluorochlorinated hydrocarbons, chemical analysis, determination of content, gases, oxygen, nitrogen, argon, methane, carbon monoxide, gas chromatographic analysis.

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5918 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in March 1981.

It has been approved by the member bodies of the following countries :

Austria	Germany, F. R.	Philippines
Belgium	Hungary	Poland
Brazil	India	Portugal
China	Italy	Romania
Czechoslovakia	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Mexico	Thailand
France	Netherlands	USSR

The member bodies of the following country expressed disapproval of the document on technical grounds :

United Kingdom

Chlorofluorohydrocarbons for industrial use — Determination of inert gas content — Gas chromatographic method — General principles

1 Scope and field of application

This International Standard gives general principles in establishing a gas chromatographic method for the determination of the inert gas content of chlorofluorohydrocarbons for industrial use.

It is applicable to products having inert gas contents equal to or greater than 0,005 % (V/V).

2 References

ISO 2209, *Liquid halogenated hydrocarbons for industrial use — Sampling.*

ISO 3427, *Gaseous halogenated hydrocarbons (liquefied gases) — Taking of a sample.*

3 Definition

For the purpose of this International Standard, the following definition applies.

inert gas : The permanent gases (oxygen, nitrogen, argon, carbon monoxide and methane) collectively present in a chlorofluorohydrocarbon.

4 Principle

Collective detection of inert gas from test portions taken from the liquid or the gaseous phase and analysis by means of gas chromatography under conditions such that none of the gases are retained irreversibly by the stationary phase.

5 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

5.1 Carrier gas

Hydrogen, minimum purity 99,9 % by volume.

WARNING — Highly flammable; explosive when mixed with air at concentrations varying approximately from 4 to 75 % (V/V).

5.2 Calibration reagents

Pure chlorofluorohydrocarbons, containing not more than 0,005 % (V/V) of inert gas.

5.3 Material for preparation of the column

Porapak Q, of particle size 180 to 300 µm, or any other equivalent, suitable material.

6 Apparatus

NOTE — Column dimensions, column packing material and experimental conditions are generally not critical. They should be selected, however, to ensure that the inert gas peak is fully separated from the adjacent peaks.

6.1 Gas chromatographic system

Any suitable gas chromatograph, fitted with a thermal conductivity detector, may be used.

6.1.1 Characteristics of the system

6.1.1.1 Column temperature : typically 75 °C.

6.1.1.2 Detector temperature : typically 75 °C.

6.1.2 Carrier gas flow rate

The carrier gas flow rate shall be 40,0 ml/min. It is usually measured at column outlet and at ambient temperature.

6.2 Injection equipment

Stainless steel gas **sample valve** with loops of 1 or 2 ml capacity, operating under the following conditions :

- temperature : ambient to 50 °C;
- pressure : atmospheric.

6.3 Column

6.3.1 Construction

The column may be made of any suitable material that does not react with the test portion, for example glass, stainless steel or copper. Typical dimensions are as follows :

- a) length : according to the test method used but typically 1 to 5 m;
- b) diameter : internal diameter approximately 4 mm; external diameter approximately 6 mm;
- c) shape : adapted to the oven geometry. If coiled, the coil diameter shall be at least ten times the outer diameter of the tube.

6.3.2 Packing

6.3.2.1 Composition

Porapak Q (5.3)

6.3.2.2 Method of packing

Use a standardized procedure to give a packed column capable of separating the inert gas, as a single peak, from the chlorofluorohydrocarbons.

6.3.3 Thermal conductivity detector, of such sensitivity that the height of the inert gas peak to be measured at the limit of detection is not less than twice the peak-to-noise level.

6.3.4 Potentiometric recorder, having the following characteristics :

- a) range : compatible with the chromatograph used [typically 0 to 1 mV full scale deflection (fsd)];
- b) chart width : not less than 200 mm;
- c) response time : not greater than 1 s for fsd;
- d) linearity : better than 0,5 % for fsd;
- e) dead band : not greater than 0,2 % for fsd;
- f) chart speed : variable.

NOTE — A suitable electronic data handling system may be used instead of the potentiometric recorder.

7 Sampling

Ensure that the laboratory sample used for the determination of the inert gas content is the first to be taken, sampling from the liquid phase or from the gaseous phase, as required and following the indications given in the International Standards listed in clause 2.

8 Procedure

8.1 Setting up the chromatograph

Set up the chromatograph as specified in clause 6.

8.2 Calibration

8.2.1 Preparation of calibration mixtures

Using a volumetric metering device, prepare calibration mixtures containing 0,005 to 0,5 % (V/V) of inert gas (air) in the chlorofluorohydrocarbons (5.2).

NOTE — Detailed prescription for the preparation of the calibration mixtures cannot be given here, because the most diversified chemico-physical features of the various chlorofluorinated hydrocarbons (with their relatively large range of boiling points) do not allow a single preparation method to be used for all these products.

According to each case it can be useful to employ the International Standards that will be published by ISO/TC 158, *Analysis of gases*, concerning the preparation of calibration gas mixtures.

8.2.2 Determination of calibration factor

Flush the gas inlet system with about 100 ml of calibration mixture (8.2.1). To avoid systematic sampling errors, the flow rate of the gas should be at least 30 ml/min but not more than 100 ml/min in order to avoid cooling and condensation. Close the sample valve, introduce the calibration mixture entrained in the carrier gas (5.1) and obtain the chromatogram.

At a specified bridge current, the calibration factor, k_i , is given by the formula

$$\frac{V}{A_i \times R_i}$$

where

V is the concentration of inert gas, expressed as a percentage by volume, in the calibration mixture;

A_i is the area of the peak, in square centimetres, corresponding to the inert gas in the calibration mixture;

R_i is the recorder range, in millivolts, used during the determination.

8.3 Procedure

8.3.1 Test portion

Take the test portion from the liquid or the gaseous phase as required, of a sample that has not been used for other determinations. (See clause 7).

8.3.2 Determination

Introduce the test portion (8.3.1) in gaseous form, using the procedure specified in 8.2.2 and obtain the chromatogram.

The deviation of the bridge current between the calibration and the determination should be less than 0,1 % of the adjusted value.

9 Expression of results

The inert gas content, expressed as a percentage by volume, is given by the formula

$$A \times R \times k_i$$

where

A is the area of the peak, in square centimetres, corresponding to the inert gas in the test portion;

R is the recorder range, in millivolts, used during the determination;

k_i is the calibration factor (8.2.2).

NOTE — A typical chromatogram, obtained by determining the inert gas content of the gaseous phase of a sample of chlorodifluoromethane (R 22), is shown in the figure.

10 Test report

The test report shall include the following information :

- a) an identification of the sample;
- b) the reference of the method used;
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

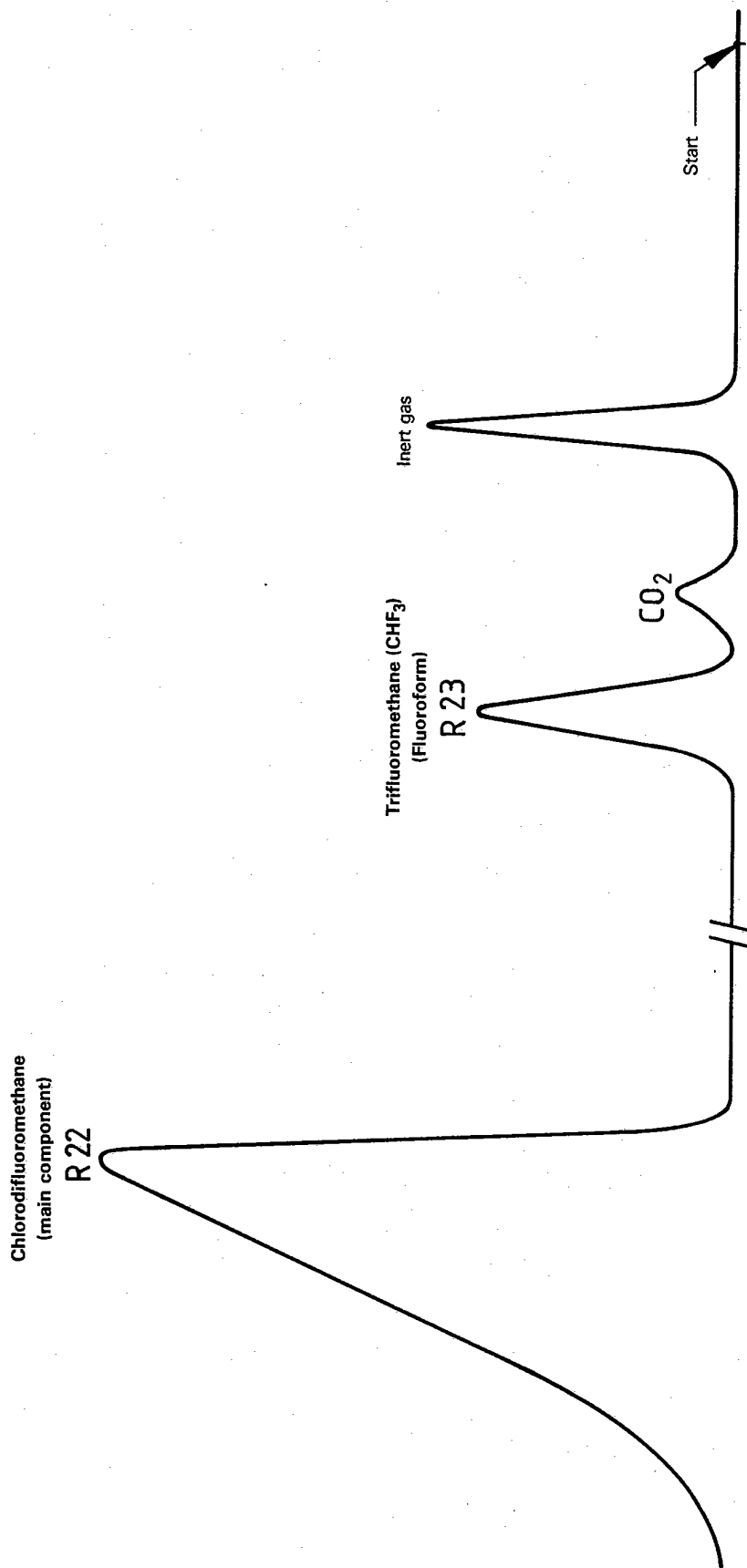


Figure — Typical chromatogram [obtained by analysis of chlorodifluoromethane (R 22) for inert gas content using a Porapak packing]