
International Standard**5921**

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

Chlorofluorohydrocarbons for industrial use — Analysis by gas chromatography — General principles

Hydrocarbures chlorofluorés à usage industriel — Analyse par chromatographie en phase gazeuse — Principes généraux

First edition — 1982-11-15

UDC 661.723 : 543.544**Ref. No. ISO 5921-1982 (E)**

Descriptors : industrial products, halohydrocarbons, fluorochlorinated hydrocarbons, chemical analysis, determination, volatile matters, gas chromatographic analysis.

Price based on 7 pages

Foreword

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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 5921 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in April 1981.

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

Austria	Hungary	Portugal
Belgium	India	South Africa, Rep. of
China	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Philippines	USSR
Germany, F. R.	Poland	

No member body expressed disapproval of the document.

Chlorofluorohydrocarbons for industrial use — Analysis by gas chromatography — General principles

Scope and field of application

This International Standard establishes the standard layout for, and gives general information relating to, methods for the determination of volatile components in chlorofluorohydrocarbons by gas chromatography.

It takes into account the general layout of methods of chemical analysis given in ISO 78/2 and the recommendations given in ISO 2718.

Reference

ISO 78/2, *Layouts for standards — Part 2 : Standard for chemical analysis.*

ISO 2718, *Standard layout for a method of chemical analysis by gas chromatography.*

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

Standard layout for methods of analysis by gas chromatography**Title****Scope and field of application****References****Definitions****Principle****Reagents and materials****Apparatus****Sampling****Procèdure****Expression of results****Notes on procedure, particularly with reference to hazards****Test report****Annexes****Bibliography**

Notes on the application of the standard layout to a general method of analysis of chlorofluorohydrocarbons by gas chromatography

NOTE — Clauses contained in the standard layout, but which are irrelevant to the general method, have been omitted. It may nevertheless be necessary, in writing specific methods, to include different combinations of clauses.

1 Scope and field of application

This clause shall identify the component(s) to be determined and the range(s) of concentration for which the method is suitable.

2 Principle

This clause shall give a concise summary of the method used.

3 Reagents and materials

During the analysis, unless otherwise stated, only reagents of recognized analytical grade should be used.

3.1 Carrier gases and auxiliary gases

3.1.1 Helium, minimum purity 99,9 % by volume, for use with katharometer (thermal conductivity) detectors, or, as an alternative.

3.1.2 Hydrogen, minimum purity 99,9 % by volume.

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

3.1.3 Nitrogen, minimum purity 99,9 % by volume, for use with katharometer (thermal conductivity) detectors or flame ionization detectors.

3.2 Auxiliary gases for flame ionization detectors

3.2.1 Hydrogen, minimum purity 99,5 % by volume.

3.2.2 Air, minimum purity 99,5 % by volume.

3.3 Calibration reagents, minimum purity 99,5 % by volume.

3.4 Internal standards, minimum purity 99,5 % by volume.

4 Apparatus

4.1 Gas chromatograph

Any suitable instrument may be used. The mode of temperature control (isothermal or temperature programming) and the type of detector required (thermal conductivity or flame ionization) should be specified.

The temperature, gas flow rate and pressure and electrical characteristics of the chromatograph and its auxiliary devices should have a performance specification to allow

- a) the peak retention time to be repeatable to ± 2 % or better;
- b) the peak heights or areas to be repeatable to ± 2 % or better.

4.2 Injection devices

4.2.1 Liquids

A microsyringe (typical capacity 1 to 10 μ l) is suitable. The test portion should be injected into an injection port heated to a temperature at least 20 °C above the boiling point of the main component.

4.2.2 Gases

Gaseous test portions should be injected through a stainless steel gas sample valve with a choice of loops of 0,1, 1 or 5 ml capacity, which should be capable of being heated to a temperature of at least 75 °C.

NOTE — For certain determinations, temperatures of up to 150 °C may be necessary.

4.3 Column

The column may be constructed of any suitable material, for example glass, stainless steel or copper, that does not react with the sample. The following details shall be specified :

- a) length : according to the method specified (typically 1 to 5 m);
- b) tube diameter : internal and external diameters about 4 mm and 6 mm respectively;
- c) shape : to suit the oven geometry. If the column is coiled, the coil diameter should be at least 10 times the outer diameter of the tube.

4.4 Column details

The following details shall be specified.

4.4.1 Packing material, suitable for the separation required (for example a stationary phase on a support, adsorbents, or porous polymer beads, for example "Porapak") :

- a) support : type, particle size and pretreatment;

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b) stationary phase : type, packing and method of coating the support.

4.4.2 Filling and conditioning the column : special procedures to ensure compact and uniform filling.

4.4.3 Column efficiency, expressed as the length equivalent to a theoretical plate for a specified compound.

4.4.4 Carrier gas flow rate, determined directly by means of a flow meter. This measurement is usually made at column outlet and at ambient temperature.

4.5 Detector

The following details shall be specified.

4.5.1 Detector type

A thermal conductivity cell of sufficient sensitivity or a flame ionization detector is suitable.

4.5.2 Required sensitivity

The height of the smallest peak to be measured should be not less than twice the noise level.

4.6 Recorder

A potentiometric recorder is generally used, the following characteristics being specified :

- a) range : appropriate to the chromatograph (0 to 1 mV full scale deflection is typical);
- b) response time : not greater than 1 s for full scale deflection;
- c) linearity : better than 0,5 % of full scale;
- d) dead band : not greater than 0,2 % for full scale deflection;
- e) variable chart speed.

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

5 Sampling

5.1 Chlorofluorohydrocarbons liquid at ambient temperature and pressure

The test portion should be taken and injected as usual using the microsyringe (4.2.1), which may be cooled if necessary.

NOTE — In the case of too high ambient temperature, place the sample in a bottle previously cooled to between 5 and 10 °C and cool the syringe and its needle to approximately 0 °C.

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5.2 Chlorofluorohydrocarbons gaseous at ambient temperature and pressure

The laboratory sample should be used whenever possible, the sample being taken from the liquid phase.

For taking the sample, use a sample cylinder of type A (see ISO 3427).

The liquefied gas should be vaporized by passage through a fine control valve and then, in order to ensure complete volatilization, passed through either an empty 50 ml bubble indicator (see the note) or, for samples with higher boiling points, through a heated copper helix connected to the sample valve (see the figure).

The temperature of the fine control valve should be lower than that of the sample cylinder in order to avoid premature evaporation. This may be achieved by flushing the liquid phase through the valve momentarily before connecting the sample cylinder to the heating coil.

NOTE — The bubble indicator compensates for variations of concentration at the point of evaporation.

The gas flow rate through the sample valve should be adjusted to between 100 and 200 ml/min, the fine control valve closed and, after waiting 5 s for the pressure to reach equilibrium, the sample should be injected into the gas chromatograph (4.1).

5.3 Chlorofluorohydrocarbons which are solid at ambient temperature and at normal pressure

The sample may be taken either by heating the container and then transferred in a bottle maintained at the same temperature or taken at the solid state by means of a spatula and successively liquefied. The microsyringe and its needle shall be heated to the same temperature before the sample is injected in the usual manner.

6 Procedure

6.1 Setting up the chromatograph

The chromatograph should be adjusted to the conditions appropriate to the particular method. Specifications should include :

- a) the temperature of the column is set for the desired chromatographic performance, but shall be high enough to avoid condensation of any component; the temperature of the liquid injection port should be at least 20 °C higher than the boiling point of the main component; the temperature of the detector is generally 30 to 50 °C higher than the injection port, to ensure no deposits;
- b) gas inlet temperature : a specific temperature in the range 75 to 150 °C, to be maintained during the calibration and determination, should be specified;
- c) settings of the electrical equipment associated with the detector;

d) carrier gas flow rate : the flow rate should be adjusted to the specified value by means of bubble flow meters connected to the exit of the detector following the column.

6.2 Peak identification

The peaks should be identified by comparison with the retention times of reference samples.

7 Calibration

7.1 By means of absolute response factors obtained from determinations using gaseous standards

7.1.1 Procedure

The same test conditions should be used for the calibration and the determination. At least two calibration measurements should be performed and the calibration factors should be checked every day.

7.1.2 Calibration for high concentrations

The undiluted calibration reagents (3.3) should be used.

7.1.3 Calibration for low concentrations

Known dilutions of the calibration reagents (3.3) should be used.

7.1.4 Calibration data (gas addition)

The calibration factor, $k_{i(\text{gas})}$, should be calculated by means of one of the following formulae, as appropriate.

7.1.4.1 Area measurement

$$k_{i(\text{gas})} = \frac{A_1 \times R_1 \times 100}{V_1}$$

where

A_1 is the area of the peak corresponding to the pure component being calibrated;

R_1 is the recorder range during the measurement;

V_1 is the quantity of the component being calibrated, expressed as a percentage by volume of the sample injected.

7.1.4.2 Measurement of integrator counts (without attenuation)

$$k_{i(\text{gas})} = \frac{N_1 \times 100}{V_1}$$

where

N_1 is the integrator count of the pure component being calibrated;

V_1 is the quantity of the component being calibrated, expressed as a percentage by volume of the sample injected.

7.2 Internal calibration for determinations using liquid standards

The calibration factor, $k_{i(\text{liquid})}$, should be calculated by means of one of the following formulae, as appropriate.

7.2.1 Area measurement

$$k_{i(\text{liquid})} = \frac{A_1 \times c_0 \times R_1}{A_0 \times c_1 \times R_0}$$

where

A_1 is the area of the peak corresponding to the pure component being calibrated;

A_0 is the area of the peak corresponding to the internal standard;

c_1 is the concentration, expressed as a percentage by mass, of the pure component being calibrated in the calibration mixture;

c_0 is the concentration, expressed as a percentage by mass, of the internal standard in the calibration mixture;

R_1 is the recorder range associated with the measurement of A_1 ;

R_0 is the recorder range associated with measurement of A_0 .

7.2.2 Measurement of integrator counts (without attenuation)

$$k_{i(\text{liquid})} = \frac{N_1 \times c_0}{N_0 \times c_1}$$

where

N_1 is the integrator count of the pure component being calibrated;

N_0 is the integrator count of the internal standard;

c_1 is the concentration, expressed as a percentage by mass, of the pure component being calibrated in the calibration mixture;

c_0 is the concentration, expressed as a percentage by mass, of the internal standard in the calibration mixture.

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8 Expression of results — Methods of calculation

8.1 Gaseous samples

The content of component i , expressed as a percentage by volume, is given by the formula

$$\frac{A_i \times R_i \times 100}{k_i (\text{gas})}$$

where

A_i is the area of the peak corresponding to component i ;

R_i is the recorder range during the measurement;

$k_i (\text{gas})$ is the calibration factor.

8.2 Liquid samples

The content of component i , expressed as a percentage by mass, is given by the formulae

a) in the case of area measurements :

$$\frac{A_i \times R_i \times c_0 \times 100}{A_0 \times R_0 \times k_i (\text{liquid}) \times (100 - c_0)}$$

or

b) in the case of measurement of integrator counts :

$$\frac{N_i \times c_0 \times 100}{N_0 \times k_i (\text{liquid}) \times (100 - c_0)}$$

where

A_i is the area of the peak corresponding to component i ;

A_0 is the area of the peak corresponding to the internal standard;

c_0 is the concentration, expressed as a percentage by mass, of the internal standard in the test mixture;

$(100 - c_0)$ is the concentration, expressed as a percentage by mass, of the sample in the test mixture;

$k_i (\text{liquid})$ is the calibration factor of the component i ;

R_i is the recorder range for the measurement of component i ;

R_0 is the recorder range for the measurement of the internal standard;

N_i is the integrator count for component i ;

N_0 is the integrator count for the internal standard.

9 Test report

The test report shall include the following information :

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

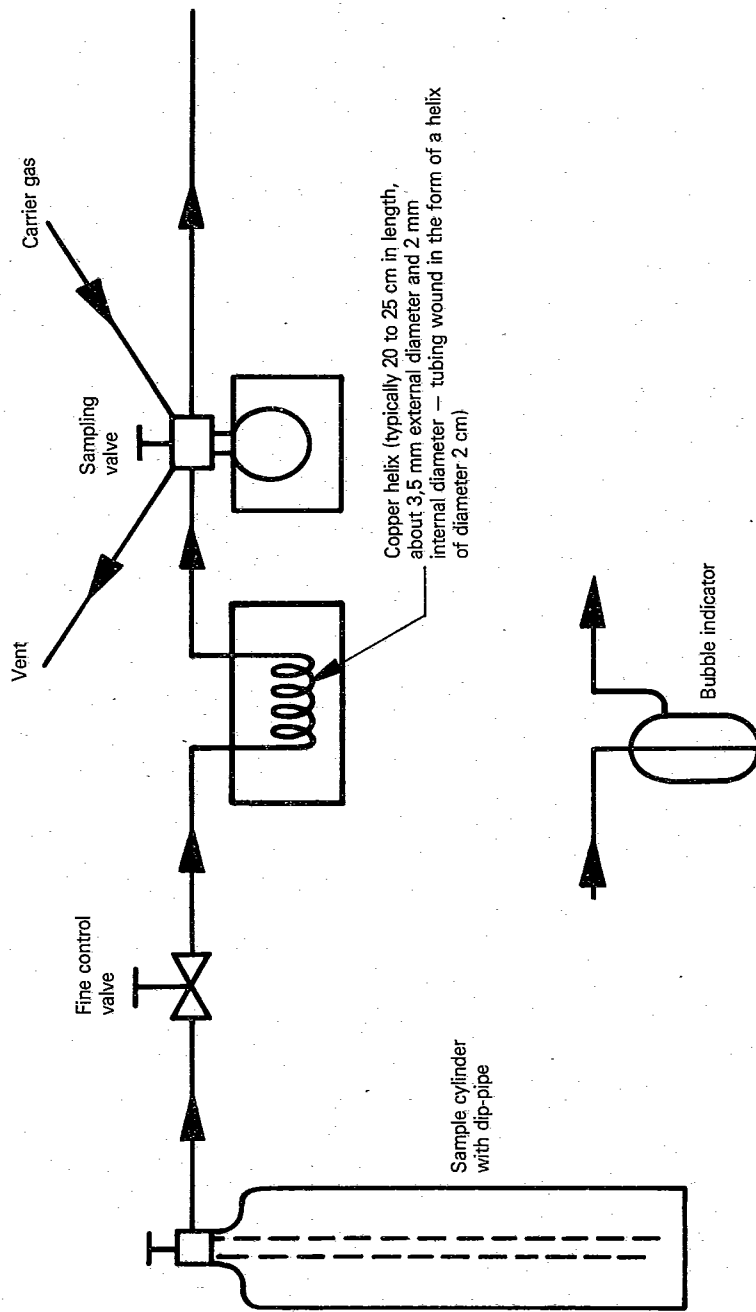


Figure — Sampling system for chlorofluorohydrocarbons gaseous at room temperature and atmospheric pressure