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Nuclear energy — Determination of carbon compounds and fluorides in uranium hexafluoride infrared spectrometry

Énergie nucléaire — Détermination des produits carbonés et fluorures dans l'hexafluorure d'uranium par spectrométrie infrarouge

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16794 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

Nuclear energy — Determination of carbon compounds and fluorides in uranium hexafluoride infrared spectrometry

1 Scope

This International Standard specifies a test method for the determination of hydrocarbons, chlorocarbons and partially or completely substituted halocarbons or halohydrocarbons contained as impurities in uranium hexafluoride (UF_6) by infrared (IR) spectrometry.

This method cannot be used for compounds giving IR rays with interference by UF_6 (for example CF_4).

The test method is quantitative and applicable in the mole fraction from 0,000 1 % or 0,001 0 %, depending on the type of impurity, up to 0,100 %.

The test method can also be used for the determination of hydrofluoric acid (HF) and several elements existing as fluorides; boron in BF_3, silicon in SiF_4, phosphorus in PF_5, molybdenum in MoF_6 and tungsten in WF_6.

2 Principle

A liquid-phase UF_6 sample is collected through a manifold and introduced into a 60 °C thermostatically controlled infrared gas cell.

Carbon compound impurities are recorded as a spectrum of peaks.

The scanned infrared spectrum ranges from 4 000 cm^{-1} to 700 cm^{-1}.

3 Apparatus

3.1 Infrared spectrometer

A Fourier transform spectrometer is recommended but not essential, if a standard scattering instrument has sufficient performance.

IR range:

in cm^{-1}	4 000 to 400	(wave number)
in µm	2,5 to 25	(wave length)

Resolution:

in cm^{-1}	2

Background:

in O.D.	5×10^{-4}	(optical density, O.D.)

3.2 Liquefaction and filling manifold

The IR spectrometer may be connected either

— directly onto a uranium enrichment device, at the drawing-off point (for cylinder filling), or

— onto a subsampling bench, or

— onto a specific manifold enabling connection of one or several vessels of types 1 S, 2 S, ANSI-14 or CEA 23 D.

In all cases, connection networks permit the following operations:

— evacuating the pipes (< 2 Pa);

— emptying the system into a cooled cylinder followed by removal of final traces of UF_6 into a chemical trap;

— cleaning all pipe networks after operations (maintenance, replacement of defective parts).

3.3 Infrared cell

The optical path length is 0,2 m (200 mm).

The infrared cell is a scanner (two valves), and shall be chemically inert to fluorinated products.

Windows are made of silver chloride (AgCl) and metal parts are made of nickel or Monel.

The IR cell is permanently heated at 60 °C ± 1 °C, within an enclosure equipped with a thermostat.

4 Calibration

The apparatus is calibrated in pure gas with the same IR cell as that used for analyses, at a temperature of 60 °C ± 1 °C.

Particular attention shall be given to interferences when several components are simultaneously present.

It is advisable to check such effects through repeated analyses under the same conditions (identical temperature and pressure) as applied to samples, on pure gas mixtures used for calibration (UF_6 and halohydrocarbons).

5 Operating procedure

5.1 Sample liquefaction

Vessels, in the upside-down position, are connected to the manifold. After evacuation of the connection network and routine leak tests, the whole assembly is heated to a temperature of 80 °C.

5.2 Rinsing connection networks and infrared cell

After reaching 80 °C, UF_6 is first introduced under a $10^{-4} \pm (5 \times 10^{2})$ Pa pressure, then removed, and the whole network, including the IR cell, is primary evacuated. The removed UF_6 is either collected in a liquid nitrogen-cooled vessel, or reprocessed in secondary networks for on-line analysis devices.

5.3 Measurement

While the apparatus is drained, the background of an empty cell scanned over the range from 1 430 cm^{-1} to 700 cm^{-1}, shall not exceed 5×10^4 (in optical density).

The UF_6 sample withdrawn in the liquid phase is then released into the IR cell under pressure ranging from 8×10^4 Pa to 10^5 Pa. The same pressure ($\pm 4 \times 10^2$ Pa) is used for each sample.

Once the method offering the most sensitive results has been chosen, the infrared spectrum is recorded between 4 000 cm^{-1} and 400 cm^{-1}.

A confirmation analysis is carried out, in the gas phase, at ambient temperature, for each component detected. The sensitivity for interference is greater in the gas phase.

A component with a wave number listed in Table 1 is considered to be detected when its amplitude reaches twice the background expressed in optical density.

5.4 Expression of results

The results are expressed as percentages.

The Lambert-Beer law enables the determination of partial pressures of carbonaceous products that may be present in UF_6, as impurities.

The mole fraction x_c, expressed as a percentage, is derived from the ratio of the partial pressure to total pressure measured in the IR cell:

$$x_c = \frac{p_p}{p_t} \times 100$$

where

p_p is the partial pressure, expressed in pascals;

p_t is the total pressure, expressed in pascals.

6 Performance

6.1 Detection limit

The detection limits are displayed in Tables 1 and 2 for selected carbon compounds and several other elements existing as fluorides. Other carbon compounds could be determined, provided that a calibration is performed. Impurities are identified through their wavenumbers expressed as reciprocal centimetres. Mole fractions are expressed as percentages. Optical densities are displayed for information and are only valid for a specific instrument.

6.2 Repeatability

At a cell pressure of (8×10^4) Pa $\pm$ (4×10^2) Pa and at a temperature of 60 °C ± 1 °C, the relative standard deviation is

— 20×10^{-2} for mole fractions, expressed as percentages, ranging from 0,000 1 to 0,002 0,

— 10×10^{-2} for mole fractions, expressed as percentages, ranging from 0,002 0 to 0,010 0,

— 5×10^{-2} for mole fractions, expressed as percentages, ranging from 0,010 0 to 0,100 0.

7 Test report

The test report shall include the following information:

a) the method used by reference to this International Standard;

b) identification of the sample;

c) the results and the form in which they are expressed;

d) any unusual features noted during the test;

e) any operations not included in this International Standard, or regarded as optional.

Table 1 — Limit of detection of carbon compounds in UF_6 by IR. Total pressure 8 × 10 Pa

Freons	Formula	Wavenumber cm^{-1}	Related optical density	Limit of detection mole fraction %
F11	$CFCl_3$	1 087	$9,4 \times 10^{-4}$	0,000 6
F12	CF_2Cl_2	1 107	$9,0 \times 10^{-4}$	0,000 4
F13	CF_3Cl	1 215	$8,8 \times 10^{-4}$	0,000 2
F13B	CF_3Br	1 208	11×10^{-4}	0,000 1
F113	$CF_2Cl\text{-}CFCl_2$	1 121	$9,0 \times 10^{-4}$	0,000 5
F114	$CF_2Cl\text{-}CF_2Cl$	1 052	$8,3 \times 10^{-4}$	0,000 4
F115	$CF_3\text{-}CF_2Cl$	1 242	10×10^{-4}	0,000 2
F142	$CH_3\text{-}CF_2Cl$	885	$9,3 \times 10^{-4}$	0,000 7
F21	$CHFI_2$	1 087	$8,5 \times 10^{-4}$	0,000 6
F23	CHF_3	1 379	$6,1 \times 10^{-4}$	0,001 0
F318	$CF_3CF{=}CFCF_3$	1 198	$9,6 \times 10^{-4}$	0,000 2
FC318	$CF_2\text{-}CF_2$ $CF_2\text{-}CF_2$	962	12×10^{-4}	0,000 2
Total				0,005 1

Table 2 — Limit of detection of fluorides in UF_6 by IR. Total pressure 8 × 10 Pa

Gas	Wavenumber cm^{-1}	Related optical density	Limit of detection mole fraction %
SiF_4	1 028	0,02	0,000 2
PF_5	955	0,02	0,000 7
BF_3	1 443	0,02	0,000 8
MoF_6	741	0,01	0,000 1
HF	3 976	0,02	0,000 1