



Designation: C1883 – 19

Standard Practice for Sampling of Gaseous Enriched Uranium Hexafluoride¹

This standard is issued under the fixed designation C1883; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the methods for withdrawing samples of enriched uranium hexafluoride (UF_6) during and after a transfer occurring in the gas phase in order to determine compliance with Specification C996. It is applicable to the filling of a transport UF_6 container (30B cylinder or 1 m³ vertical container) at a centrifuge enrichment facility that has been fed with UF_6 that complies with Specification C787.

1.2 Since UF_6 sampling is taken during the filling process, this practice does not address any special additional arrangements that may be agreed upon between the buyer and the seller when the sampled bulk material is being added to residues already present in a container (“heels recycle”). Such arrangements will be based on QA procedures such as traceability of cylinder origin (to prevent, for example, contamination with irradiated material).

1.3 This practice is only applicable when the transfer occurs in the gas phase. When the transfer is performed in the liquid phase, Practice C1052 should apply.

1.4 The scope of this practice does not include provisions for preventing criticality incidents.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.02 on Fuel and Fertile Material Specifications.

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2. Referenced Documents

2.1 ASTM Standards:²

C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C787 Specification for Uranium Hexafluoride for Enrichment

C859 Terminology Relating to Nuclear Materials

C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ²³⁵U

C1052 Practice for Bulk Sampling of Liquid Uranium Hexafluoride

C1441 Test Method for The Analysis of Refrigerant 114, Plus Other Carbon-Containing and Fluorine-Containing Compounds in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy

C1842 Test Method for The Analysis of Boron and Silicon in Uranium Hexafluoride via Fourier-Transform Infrared (FTIR) Spectroscopy

C1838 Practice for Cleaning for 1S and 2S Bottles

2.2 ISO Standards:³

ISO 7195:2005 Nuclear energy—Packaging of uranium hexafluoride (UF_6) for transport

3. Terminology

3.1 Terms shall be defined in accordance with Terminology C859, except for the following:

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *container, n*—a vessel either holding or receiving by transfer, the UF_6 to be sampled; it may consist of, for example, a fixed vessel in a UF_6 handling plant or a cylinder to be used for the transport of UF_6 .

3.2.2 *sample vessel, n*—the small vessel into which the sample of UF_6 is withdrawn for analysis in the laboratory for characterization. It can be a 1S or 2S bottle or a PCTFE

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

(polychlorotrifluoroethylene)/PTFE (polytetrafluoroethylene) pot or tube or any other type of cylinder compatible with UF₆.

4. Summary of Practice

4.1 The method is based on three different steps for UF₆ characterization, namely: (1) continuous on-line analysis by mass spectrometry (MS) for isotopic measurement, (2) sequential withdrawals during filling to produce a representative composite sample, (3) gas sampling of container after the filling is complete, to determine volatile impurities.

4.2 The conformity of the container is determined by sampling (1) for isotopic composition; by sampling (3) for HF to determine UF₆ concentration, for very volatile impurities compared to UF₆, such as BF₃, SiF₄, and low molecular weight hydrocarbon, chlorocarbon, and partially substituted halohydrocarbon compounds; and by sampling (2) for other requirements: Pu, Np, fission products, ⁹⁹Tc, if they are necessary. The sample can also be used for counter-analyses or supplied to the customer. Additionally, the sample may be used to determine UF₆ concentration. Figs. 1 and 2 summarize the principle of the three sampling steps.

4.3 Determining concentration of ²³⁵U (²³⁴U, ²³⁶U) in a batch of product extracted is carried out according to the results of hourly mass-spectrometry isotope analysis. Weighted average value calculated over the entire control period is assigned to a batch of transport UF₆ containers processed simultaneously.

4.4 Content of radioactive admixtures (²³²U, ²³⁷Np, Pu isotopes, ⁹⁹Tc, gamma-emitting uranium fission products) shall be determined according to analysis results related to a sample collected from the process flow intermittently one time per hour during the entire period UF₆ batch is extracted. Refer to Test Methods C761 for test methods.

4.5 Quality control of UF₆ by UF₆ concentration is carried out using an indirect method based on performed IR-spectrometry analysis for HF content in gas phase of container once it is filled.

4.6 Compliance of UF₆ quality with Specification C996 by content of boron, silicon, and hydrocarbon, chlorocarbons, and partially substituted halohydrocarbons is confirmed according to the results of IR-spectrometry analysis of samples collected from gas phase of container (reservoir) once it is filled.

4.7 The intention of this practice is to avoid liquid UF₆ sampling once the cylinder has been filled. For safety reasons, manipulation or large quantities of liquid UF₆ should be avoided when possible. This practice is applicable only if the process of gas phase filling is sufficiently stable over time, ²³⁵U concentration fluctuations in the process flow are characterized by the relative standard deviation equal to 0.2 % rel. This practice does not address cylinders filled during transitional regime of the cascade.

4.8 In case of the presence of volatile impurities close to the specification (for example, 80 %) or in case of HF concentration close 0.5 % or less, a confirmation using liquid sampling may be necessary.

4.9 It is recommended to validate the gas sampling using a comparison on several cylinders with liquid sampling after filling. Statistically significant sampling basis and requirement should be established. Adequacy shall be demonstrated by quality assurance procedures.

4.10 The presence of residues may have significant implications for the quality of the UF₆. For safety and quality reasons, cylinders and bottles shall be clean, dry, and empty before filling.

4.11 Cross-contamination may occur between subsequent samples taken using the same equipment, and appropriate precautions must be taken to prevent this. It is therefore recommended that, before taking definitive samples, the equipment is flushed through with an aliquot of the material to be sampled. This is normally accomplished by taking an initial volume which is then rejected and not used for definitive

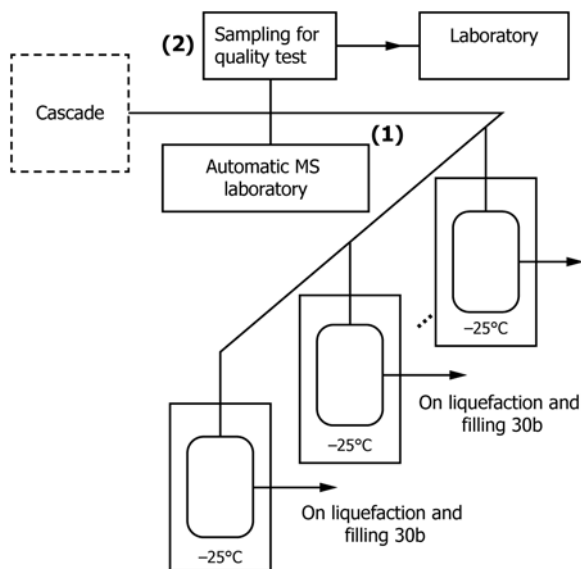


FIG. 1 Schematic Arrangement for Online Filling (3) Measurement (1)

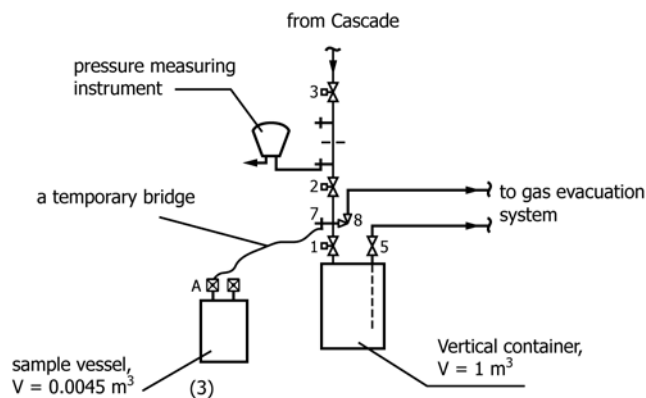


FIG. 2 Schematic Arrangement for Gas Sampling After and Composite Sample (2)

analysis. Alternative procedures to prevent cross-contamination are possible and should be validated individually.

5. Significance and Use

5.1 The standard method for taking representative UF₆ sample from a cylinder is collecting from UF₆ liquid phase. Homogeneity of material is obtained by the basic indicator, that is, content of uranium isotope. Representativeness of a sample by other indicators such as content of highly volatile admixtures (boron fluoride, silicon fluoride, organic impurities), admixtures generating non-volatile fluorides may not be satisfactory owing to differences between physical properties of admixture and UF₆. Nevertheless, such sampling is widely applied during the product quality control due to the fact that liquid phase process is applied when filling transport containers both for feedstock UF₆ and enriched one. Refer to Practice C1052 for UF₆ liquid phase sampling practice.

5.2 Uranium hexafluoride is normally produced and handled in large (typically 1 to 14-ton) quantities and must, therefore, be characterized by reference to representative samples (see ISO 7195:2005). The samples are used to determine compliance with the applicable commercial Specification C996. The quantities involved, physical properties, chemical reactivity, and hazardous nature of UF₆ are such that for representative sampling, specially designed equipment must be used and operated in accordance with the most carefully controlled and stringent procedures. This practice can be used by UF₆ enrichers to review the effectiveness of existing procedures or as a guide to the design of equipment and procedures for future use.

5.3 It is emphasized that this practice is not meant to address conventional or nuclear criticality safety issues.

6. Hazards

6.1 Because of its chemical, radiochemical, and toxic properties, UF₆ is a hazardous material.

6.2 Uranium hexafluoride is very reactive and corrosive. It reacts readily with water, atmospheric moisture, certain metals, and many organic materials. For reasons of safety and to avoid contamination, precautions must be taken to avoid contact with such materials. The sampling equipment is therefore fabricated

to appropriate high standards of vacuum and high temperature integrity, and components in direct contact with UF₆ are made from nickel, high-nickel alloys, or materials having equivalent resistance to UF₆ corrosion. The formation of an inert fluoride layer is often an important feature of UF₆ corrosion resistance, and hence, internal surfaces are generally conditioned with a suitable fluorinating agent, sometimes UF₆ itself.

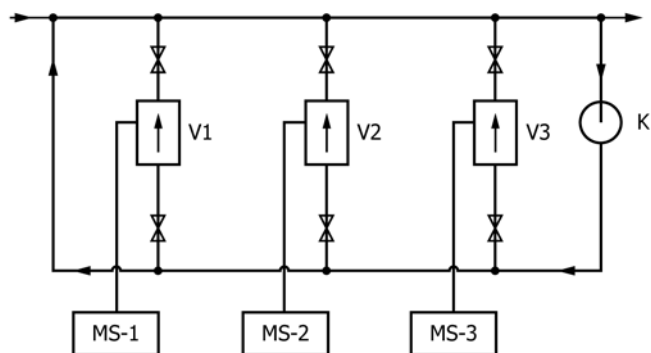
6.3 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Familiarization and compliance with the Safety Data Sheet is essential.

7. Procedure for Continuous On-line Isotopic Analysis by MS

7.1 Determining concentration of ²³⁵U (²³⁴U, ²³⁶U) in a batch of product extracted is carried out according to the results of mass-spectrometry isotope analysis of gas flow admitted for condensation. Concentration of uranium isotopes in UF₆ shall be monitored with frequency of single measurement per hour (hourly). To ensure measurement conducted is reliable and precise, monitoring is carried out simultaneously using two magnet sector four-manifold “gas” mass-spectrometers operating in automatic mode. Precision measurement is conducted using referent samples of uranium isotope composition in the form of UF₆. Refer to Fig. 3 for connection diagram of mass-spectrometers and process flow. During the measurement period UF₆ flow via measuring vessels shall be blocked.

7.2 Weighted average values calculated over the entire control period are assigned to a batch of containers (reservoirs) processed simultaneously. Calculation of uranium isotope mass fractions C_i, %, in a product batch extracted at a constant value of flow to the cylinders is performed based on calculated arithmetical average using Eq 1:

$$C = \frac{\sum C_i}{N} \quad (1)$$



K – centrifugal compressor ensuring 5–7 kPa pressure drop (40–50 mm Hg). MS-1, MS-2, MS-3 – automatic “gas” mass-spectrometers, (MS-3 – fallback). V₁, V₂, V₃ – measuring vessels placed in delivery system of mass-spectrometers.

FIG. 3 Example of Mass-Spectrometer Connection Diagram

where:

C_i = current results for determining content of i^{th} uranium isotope, and

N = number of analyses performed.

When extracting product with changing value of flow to the cylinders (as an example by changing pressure before meter nozzles), weighted average value of mass fraction of ^{235}U in commercial product batch C_{wa} , %, is calculated considering pressure change using Eq 2:

$$C_{wa} = \frac{\sum_{i=1}^n P_i \cdot T_i \cdot C_i}{\sum_{i=1}^n P_i \cdot T_i} \quad (2)$$

where:

P_i = pressure maintained before meter nozzles on flow during T_i time.

7.3 To determine content of ^{232}U , ^{237}Np , Pu isotopes, ^{99}Tc , gamma-emitting uranium fission products (when required) a portion of sample shall be used to be collected as per Section 8.

7.4 Values for content of radioactive admixtures obtained when analyzing this sample are assigned to a batch of containers to be filled simultaneously.

8. Procedure for Sequential Sampling to Produce a Composite Sample

8.1 To prove quality of UF_6 when extracting a batch of containers, a representative sample shall be collected for subsequent generating of customer’s sample and a reference sample. A portion of this sample⁴ is used to control quality of UF_6 as per 7.3. Representative sample shall be collected using UF_6 partial condensation method from measuring vessel V₁ (V₂) into sampling reservoir, for instance, 2S or another similar one while cooling the latter with nitrogen. UF_6 condensation shall be performed following each hourly measurement of UF_6 isotope composition. Volume of measuring vessel V₁ (V₂) shall be selected so as to ensure condensation of ~1 g of UF_6 during one of procedure at pressure reduce in the vessel by 4000 Pa

(30 mm Hg) from initial pressure. While the weight of representative sample over the time UF_6 is extracted will be 300 to 400 g. This sample shall be divided into three portions when homogenization in the liquid phase is conducted: (1) for conducting quality control as per 7.3; (2) customer’s sample; and (3) reference sample.

8.2 The conditions for collecting an average sample when extracting product described above guarantee the material taken is representative by such indicators as: ^{235}U , ^{234}U , ^{236}U , ^{232}U , ^{99}Tc , Pu isotopes, ^{237}Np , gamma-emitting fission products.

9. Procedure for Gas Sampling after Filling the Container

9.1 When filling of container (reservoir), impurities with a high vapor pressure are concentrated in the gas phase. To control the quality of product with respect to compliance with Specification C996 by such indicators as UF_6 concentration, the content of boron, silicon, organic compounds (OC): hydrocarbon, chlorocarbons, and partially substituted halohydrocarbon sampling is performed into sampling bottles having 4 to 6 dm³ capacity from gas phase of each cylinder (container). Sampling is performed once control is conducted whether pressure in container complies with Specification C996 at a room temperature. Sample analysis shall be performed using IR-spectrometry method and IR Fourier spectrometer with MCT type detector completed with 10 m gas sample cell. The device shall be calibrated using certified mixtures prepared based on clear UF_6 and compounds of admixtures. Refer to 9.4 for sampling diagram (Fig. 4) and sampling technique. Sampling bottles shall be prepared according to Practice C1838. Refer to Test Methods C1441 and C1842 for test methods. The comparison of measurement results by this method and liquid sampling as per Practice C1052 and calculation example are given in Appendix X1.

9.2 Quality control of UF_6 concentration is carried out using indirect method based on performed IR-spectrometry investigations for HF content in gas phase of container (reservoir). Concentration of UF_6 , C_{UF_6} , g $\text{UF}_6/100$ g of sample is calculated using Eq 3:

$$C_{\text{UF}_6} = 100 - 1.3 \cdot 10^{-9} \cdot P_{\text{UF}_6} \cdot C_{\text{HF,mol,\%}} \quad (3)$$

⁴ Composite sample can also be used to determine or confirm U isotopes.

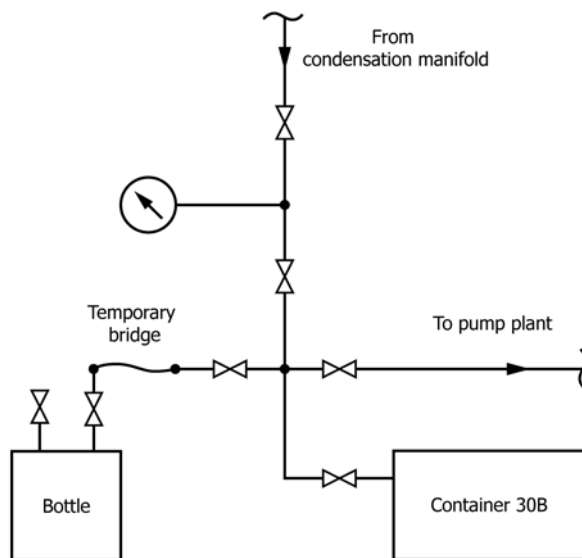


FIG. 4 Bottle Connection Diagram

where:

P_{UF_6} = pressure in container (reservoir), Pa, and
 $C_{HF, mol, \%}$ = HF molar concentration measured by IR spectrometry method.

9.2.1 Calculated value shall be rounded to four significant figures after comma.

9.2.2 Calculation example is given in Appendix X3.

9.3 Content of boron, silicon, and organic compounds (OC) to determine compliance with requirements of Specification C996 according to the results of analysis of sample collected from gas phase of container (reservoir) is calculated using Eq 4-6:

$$C_{\text{Boron}}, \mu\text{g/g U} = 8 \cdot 10^{-6} \cdot P_{\text{cont}} \cdot C_{\text{BF}_3} \quad (4)$$

$$C_{\text{Silicon}}, \mu\text{g/g U} = 2 \cdot 10^{-5} \cdot C_{\text{SiF}_4} \cdot P_{\text{cont}} \quad (5)$$

$$C_{\text{OC}}, \text{mol, \%} = 2 \cdot 10^{-8} \cdot C_{\text{OCGP}} \cdot P_{\text{cont}} \quad (6)$$

where:

P_{cont} = pressure in container (reservoir), Pa, and
 C_{BF_3} , C_{SiF_4} , C_{OCGP} = molar concentration of compounds measured by IR spectrometry method in gas phase of container (reservoir).

9.3.1 Examples of calculation of content of boron, silicon, and organic compounds according to the results of IR-spectrometry analysis are given in Appendix X1 and Appendix X2.

9.3.2 Compliance of UF_6 quality with Specification C996 by content of “light” admixtures (boron, silicon, and organic compounds) may be confirmed in accordance with Table 1 by

TABLE 1 Impurities and Permissible Content Limits

Element (compound)	Specification C996 Requirements	Permissible Content of Compound in “Gas” Sample, % mole
1	2	3
B (BF_3)	4 $\mu\text{g/g}$ of uranium	3.5
Si (SiF_4)	250 $\mu\text{g/g}$ of uranium	84
OC (in terms of CH_4)	0.01 % mole	4

comparing the results of “gas” sample IR-spectrometry analysis with calculated permissible values. In case of measured value for content of admixtures shown in Table 1 is below the permissible value (column 3), UF_6 complies with Specification C996 by content of “light” admixtures. The following values may be recorded in quality and quantity certificate for each specific 30B container (reservoir): boron < 4 $\mu\text{g/g}$ of uranium, silicon < 250 $\mu\text{g/g}$ of uranium, OC < 0.01 % mole. Otherwise, measures to reduce them shall be taken, for instance, by pumping out gas phase.

9.4 Sampling into Bottle:

9.4.1 Gas sample shall be collected using pressure equalization method in a bottle previously depressurized and container. Bottle shall be connected to container (reservoir) using temporary bridge according to the diagram in Fig. 4.

10. Keywords

10.1 bulk sampling; nuclear material; uranium hexafluoride

APPENDIXES

(Nonmandatory Information)

**X1. CALCULATION OF BORON AND SILICON CONCENTRATION IN URANIUM HEXAFLUORIDE FOR 30B CYLINDERS:
C_B, μg/g OF URANIUM, C_{Si}, μg/g OF URANIUM**

X1.1 Input Data

X1.1.1 Weight of uranium hexafluoride in the Model 30B cylinder – M_{UF₆}: 2277 kg;

X1.1.2 Density of solid UF₆, ρ_{solid}: 5.0 kg/dm³ (g/cm³) at 25°C;

X1.1.3 Free volume of the Model 30B cylinder – V_{free}: 280 dm³ (736 – 2277/5.0);

X1.1.4 Weight of boron impurities – M_B, g; silicon impurities – M_{Si}, g in the cylinder gaseous phase;

X1.1.5 Pressure in the Model 30B cylinder at temperature T = 25°C – P_{cont}, Pa;

X1.1.6 Atomic mass – μ, g/atom for boron or silicon;

X1.1.7 Gas Constant R = 8.3 J/cal·K;

X1.1.8 Molar concentrations of BF₃ – C_{BF₃}, %mol; SiF₄ – C_{SiF₄}, %mol, determined using the IR spectroscopy method with analysis of the sample taken from the cylinder gaseous phase.

X1.2 The following equation shall be used to determine the weight M_B, M_{Si}:

X1.2.1

$$P \cdot V = \frac{M}{\mu} \cdot RT \quad (X1.1)$$

$$M_B = \frac{P_{cont} \cdot C_{BF_3} \cdot V_{free} \cdot \mu_B}{100 \cdot R \cdot T} \quad (X1.2)$$

$$M_B = \frac{280 \cdot 10^{-3} \cdot 10.8 \cdot P_{cont} \cdot C_{BF_3}}{100 \cdot 8.3 \cdot 298} = \frac{1.22 \cdot 10^{-5} \cdot P_{cont} \cdot C_{BF_3}}{100 \cdot 8.3 \cdot 298} \quad (X1.3)$$

$$C_B = \frac{M_B \cdot 10^6}{M_{UF_6} \cdot 0.676 \cdot 10^3} = \frac{1.22 \cdot 10^{-5} \cdot P_{cont} \cdot C_{BF_3} \cdot 10^6}{2277 \cdot 0.676 \cdot 10^3} = 8 \cdot 10^{-6} \cdot P_{cont} \cdot C_{BF_3}, \mu g/g \ U \quad (X1.4)$$

Example: P_{cont}: 2·10⁴ Pa; C_{BF₃}: 0.005 % mol. C_{Boron} = 8·10⁻⁶·2·10⁴·0.005≅8·10⁻⁴, μg/g U.

X1.2.2

$$M_{Si} = \frac{P_{cont} \cdot C_{SiF_4} \cdot V_{free} \cdot \mu_{Si}}{100 \cdot R \cdot T} \quad (X1.5)$$

$$M_{Si} = \frac{280 \cdot 10^{-3} \cdot 28 \cdot P_{cont} \cdot C_{BF_3} \cdot 3.2 \cdot 10^{-5} \cdot P_{cont} \cdot C_{SiF_4}}{100 \cdot 8.3 \cdot 298} \quad (X1.6)$$

$$C_{Si} = \frac{M_{Si} \cdot 10^6}{M_{UF_6} \cdot 0.676 \cdot 10^3} = \frac{3.2 \cdot 10^{-5} \cdot P_{cont} \cdot C_{SiF_4} \cdot 10^6}{2277 \cdot 0.676 \cdot 10^3} = 2 \cdot 10^{-5} \cdot P_{cont} \cdot C_{SiF_4}, \mu g/g \ U \quad (X1.7)$$

Example: P_{cont}: 2·10⁴ Pa; C_{SiF₄}: 0.06 % mol. C_{Si} = 2·10⁻⁵·2·10⁴·0.06≅0.024, μg/g U.

X1.3 Results of calculations for boron and silicon content according to the data of IR-spectroscopy analysis and their comparison with results of liquid phase sampling are shown in

Table X1.1. <https://standards.iteh.ai/catalog/standards/sist/7125510c-610a-51098b80304c/astm-c1883-19>