

INTERNATIONAL STANDARD

NORME INTERNATIONALE



**Specifications for the re-use of sulphur hexafluoride (SF₆) and its mixtures
in electrical equipment**

**Spécifications pour la réutilisation de l'hexafluorure de soufre (SF₆)
et des mélanges contenant du SF₆ dans le matériel électrique**



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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**SPECIFICATIONS FOR THE RE-USE OF SULPHUR HEXAFLUORIDE (SF₆)
AND ITS MIXTURES IN ELECTRICAL EQUIPMENT**

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International Standard IEC 60480 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This third edition cancels and replaces the second edition, published in 2004. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- specifications for the re-use of SF₆ have been confirmed;
- specifications for the re-use of SF₆ mixtures, namely SF₆/N₂ and SF₆/CF₄ mixtures are included;
- as a result of a new repartition of annexes in IEC 60376, IEC 60480 and IEC 62271-4, this new edition now contains the following five annexes:
 - Annex A: Description of methods of analysis (on-site and laboratory);
 - Annex B: By-products of SF₆ and its mixtures;

- Annex C: Procedure for evaluating the potential effects on health from by-products of SF₆ and its mixtures;
- Annex D: Reclaiming recommendations.
- Annex E: Cryogenic reclaiming of SF₆;

The text of this International Standard is based on the following documents:

FDIS	Report on voting
10/1075/FDIS	10/1080/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

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SPECIFICATIONS FOR THE RE-USE OF SULPHUR HEXAFLUORIDE (SF₆) AND ITS MIXTURES IN ELECTRICAL EQUIPMENT

1 Scope

This document provides criteria for the re-use of sulphur hexafluoride (SF₆) and its mixtures after recovery and reclaiming from electrical equipment (e.g. for maintenance, at the end-of-life).

Sulphur hexafluoride (SF₆), nitrogen (N₂) and carbon tetrafluoride (CF₄), are gases commonly used for electrical equipment. Taking into account environmental concerns, particular attention is paid to re-use criteria for SF₆ and its mixtures with N₂ and CF₄ for its use in electrical equipment. Procedures for recovering and reclaiming used SF₆ and its mixtures are outside the scope of this document and are described in IEC 62271-4.

This document provides several annexes on the description of the different methods of analysis, on by-products, on the procedure for evaluating the potential health effects from by-products, on cryogenic reclaiming of SF₆, and on reclaiming recommendations.

Storage, transportation and disposal of SF₆ and its mixtures are outside the scope of this document and are covered by IEC 62271-4. Procedures to determine SF₆ leakages are described in IEC 60068-2-17 [4]¹.

For the purposes of this document, the complementary gases used in SF₆ mixtures will be limited to N₂ or CF₄.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050-192, *International Electrotechnical Vocabulary – Part 192: Dependability* (available at <http://www.electropedia.org>)

IEC 60050-212, *International Electrotechnical Vocabulary – Part 212: Electrical insulating solids, liquids and gases* (available at <http://www.electropedia.org>)

IEC 60050-441, *International Electrotechnical Vocabulary – Part 441: Switchgear, controlgear and fuses* (available at <http://www.electropedia.org>)

IEC 60050-826, *International Electrotechnical Vocabulary – Part 826: Electrical installations* (available at <http://www.electropedia.org>)

IEC 62271-4:2013, *High-voltage switchgear and controlgear – Part 4: Handling procedures for sulphur hexafluoride (SF₆) and its mixtures*

¹ Numbers in square brackets refer to the bibliography.

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60050-192, IEC 60050-212, IEC 60050-441 and IEC 60050-826, and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

electrical equipment

item used for such purposes as generation, conversion, transmission, distribution or utilization of electrical energy, such as electric machines, transformers, switchgear and controlgear, measuring instruments, protective devices, wiring systems, current-using equipment, insulated bushings, surge arresters

[SOURCE: IEC 60050-826:2004, 826-16-01, modified – "insulated bushings, surge arresters" has been added.]

3.2

container

vessel (cylinder) suitable for the containment of pressurized gases either in gaseous or liquid phase, according to local and/or international safety and transportation regulations

3.3

used sulphur hexafluoride

SF₆ which has been introduced into electrical equipment

3.4

reclaiming

process of contaminants removal from an insulating liquid or gas

3.5

recovery

process of transferring gas from electrical equipment to an alternate container

3.6

SF₆ mixture

gas mixture formed by SF₆ and a complementary gas, typically N₂ or CF₄

3.7

contaminant

foreign substance or material in an insulating liquid or gas which usually has a deleterious effect on one or more properties

[SOURCE: IEC 60050-212:2010, 212-17-27, modified – "or solid" has been deleted.]

3.8

by-products

contaminants which are formed by the degradation of SF₆ and its mixtures by electrical arcs or sparks

3.9

ambient air

normal atmosphere surrounding the equipment

[SOURCE: IEC 60079-29-2:2015, 3.1.1.]

4 Contaminants and their sources

4.1 General

SF₆ recovered from electrical equipment in operation contains several kinds of contaminants. Contaminants in recovered SF₆ come both from gas handling and from use.

Table 1 summarizes the main contaminants and their sources. Additional information is available in Annex B.

Table 1 – SF₆ contaminants

SF ₆ situation and use	Origin	Possible contaminant
Handling and in service	Leaks and incomplete evacuation	For pure SF ₆ : Air, oil, H ₂ O
	Desorption	For SF ₆ mixtures: Air, oil, H ₂ O, N ₂ , CF ₄
Insulating function	Partial discharges (e.g. corona) and low energy flashovers and sparkovers	Gaseous by-products: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂
		For SF ₆ mixtures: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂ , NO _x , NF _x
Switching equipment	Switching arc erosion	Gaseous by-products: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂ , SF ₄ , CF ₄ , WF ₆ Solid by-products: Metal dusts, particles, AlF ₃ , FeF ₃ , WO ₃ , CuF ₂ For SF ₆ mixtures: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂ , NO _x , NF _x
	Mechanical erosion	Metal dusts, particles
Internal arc	Melting and decomposition of materials	Gaseous by-products: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂ , SF ₄ , CF ₄ , WF ₆
		Solid by-products: Metal dusts, particles, AlF ₃ , FeF ₃ , WO ₃ , CuF ₂ For SF ₆ mixtures: HF, SO ₂ , SOF ₂ , SOF ₄ , SO ₂ F ₂ , NO _x , NF _x

4.2 Contaminants from handling and use

Filling and recovering gas leads to the additional contamination with ambient air and water (humidity).

Moisture desorbs from internal surfaces of the equipment and from polymeric parts. Oil from handling equipment (pumps and compressors) may also be inadvertently introduced.

When using gas mixtures, the possibility of cross contamination shall be considered (contaminating one gas mixture by another).

4.3 SF₆ by-products in equipment that only have an insulating function

The essential process is the decomposition of SF₆ by partial discharges (e.g. corona) and low energy flashovers and sparkovers. The immediate products are fragments of SF₆, such as SF₅, SF₄ and F, combining with O₂ and H₂O to form compounds, mainly HF, SO₂, SOF₂, SOF₄ and SO₂F₂. Due to low energy of the partial discharges, flashovers or sparkovers, the accumulated quantities of these compounds are usually negligible.

4.4 SF₆ by-products in switching equipment

During current interruption, the existence of high temperature arcs leads to the formation of by-products of SF₆, vaporized electrode metal, polymeric materials and contaminants. In addition, chemical reactions take place among the products formed (see Table 1).

The quantity of these by-products depends on the number of operations, the cumulative short circuit current, the design of equipment and the use of adsorbers (solid adsorbents).

Switching equipment may also contain particles and metal dust coming from the rubbing of contacts.

4.5 SF₆ by-products from internal arcs

The occurrence of an internal arc is extremely rare. The expected contaminants in SF₆ in faulted equipment are similar to those normally found in switching equipment. The difference lies in the quantity of compounds, which create a potential toxic risk (see Clause 9). In addition, significant vaporization of metallic material occurs and creates additional reaction products such as dust.

4.6 SF₆ mixtures specific by-products

For SF₆ mixtures, the usual SF₆ by-products mentioned in Table 1 and specific mixture by-products, such as nitrogen oxide(s) and nitrogen fluoride(s) for SF₆/N₂ and fluorocarbon(s) for SF₆/CF₄, are produced. The quantities depend on the mixture composition, contaminants and energy introduced. For typical SF₆ mixtures, the gas decomposition rates are not expected to exceed those for SF₆.

Within the by-products generated in mixtures, SF₆ by-products are generally predominant in terms of quantity and toxicity. Safety procedures related to the presence of the usual SF₆ by-products shall also apply in applications with SF₆ mixtures.

5 Specifications for re-use of SF₆

Table 2 – Specifications for re-use of SF₆

Substance ^a	Concentration
SF ₆	> 97 % volume
Air and/or CF ₄	< 30 000 µl/l (i.e. 3 % volume)
H ₂ O	< 200 µl/l (i.e. 200 ppmv)
Mineral oil	< 10 mg/kg ^b (i.e. 10 ppmw)
Acidity	< 50 µl/l total (i.e. 50 ppmv) or 12 µl/l (i.e. 12 ppmv) for (SO ₂ +SOF ₂) or 25 µl/l (i.e. 25 ppmv) HF
Key ppmv = part per million by volume ppmw = part per million by weight ^a H ₂ S and CO have been considered irrelevant due to lack of valuable data. ^b If gas handling equipment (pump, compressor) containing oil is used, it may be necessary to measure the oil content of the SF ₆ . If all equipment in contact with the SF ₆ is oil-free, then it is not necessary to measure the oil content.	

For the determination of total acidity, the sum of all acidic compounds is reported as one value. Alternatively, total acidity can be measured in terms of (SO₂ + SOF₂) or in terms of HF with a limit value of 12 µl/l and 25 µl/l respectively.

6 Specifications for re-use of SF₆ mixtures

Table 3 – Specifications for re-use of SF₆/N₂ mixtures

Substance	Concentration
N ₂	As per OEM specifications
SF ₆ percentage	±5 % volume of the specified percentage ^a
Air and CF ₄	< 30 000 µl/l (i.e. 3 % volume) ^a
H ₂ O	< 200 µl/l (i.e. 200 ppmv)
Mineral oil	< 10 mg/kg ^b (i.e. 10 ppmw)
Total acidity	< 50 µl/l total (i.e. 50 ppmv) or 12 µl/l (i.e. 12 ppmv) for (SO ₂ +SOF ₂) or 25 µl/l (i.e. 25 ppmv) HF
Storage conditions	
Shall comply with IEC 62271-4:2013, Clause J.7 in order to prevent liquefaction of SF ₆ .	
Key ppmv = part per million by volume ppmw = part per million by weight ^a Or unless otherwise specified by the original equipment manufacturer (OEM). ^b If gas handling equipment (pump, compressor) containing oil is used, it may be necessary to measure the oil content of the SF ₆ . If all equipment in contact with the SF ₆ is oil-free, then it is not necessary to measure the oil content.	

Table 4 – Specifications for re-use of SF₆/CF₄ mixtures

Substance	Concentration
CF ₄	As per OEM specifications
SF ₆ percentage	±5 % volume of the specified percentage ^a
Air and N ₂	< 30 000 µl/l (i.e. 3 % volume) ^a
H ₂ O	< 200 µl/l (i.e. 200 ppmv)
Mineral oil	< 10 mg/kg ^b (i.e. 10 ppmw)
Total acidity	< 50 µl/l total (i.e. 50 ppmv) or 12 µl/l (i.e. 12 ppmv) for (SO ₂ +SOF ₂) or 25 µl/l (i.e. 25 ppmv) HF
Storage conditions	
Shall comply with IEC 62271-4:2013, Clause J.7 in order to prevent liquefaction of SF ₆ .	
Key ppmv = part per million by volume ppmw = par per million by weight ^a Or unless otherwise specified by the original equipment manufacturer (OEM). ^b If gas handling equipment (pump, compressor) containing oil is used, it may be necessary to measure the oil content of the SF ₆ . If all equipment in contact with the SF ₆ is oil-free, then it is not necessary to measure the oil content.	

7 Reclaiming of SF₆ and SF₆ mixtures

7.1 Feasibility and process

The quality of reclaimed SF₆ shall meet the requirements of this document.

All occurring contaminants are formed in normal operation and can generally be eliminated on-site. Table 5 lists methods recommended for removing the contaminants as given in Table 1.

Table 5 – General contaminants and methods for their removal

Contaminant	Humidity (water vapour)	Gaseous by-products	Solid by-products	Air, N ₂ , CF ₄	Mineral oil
Removal method	Adsorption with molecular sieve	Adsorption with activated aluminium oxide	Retaining with solid filters	Separation by cryogenic process or membrane filtration	Adsorption with activated charcoal filter
For SF ₆ and its mixtures, these gaseous contaminants cannot be removed easily on-site. In each situation, an evaluation of the reclaiming options should be done to determine if the SF ₆ and its mixture could be reclaimed on-site.					

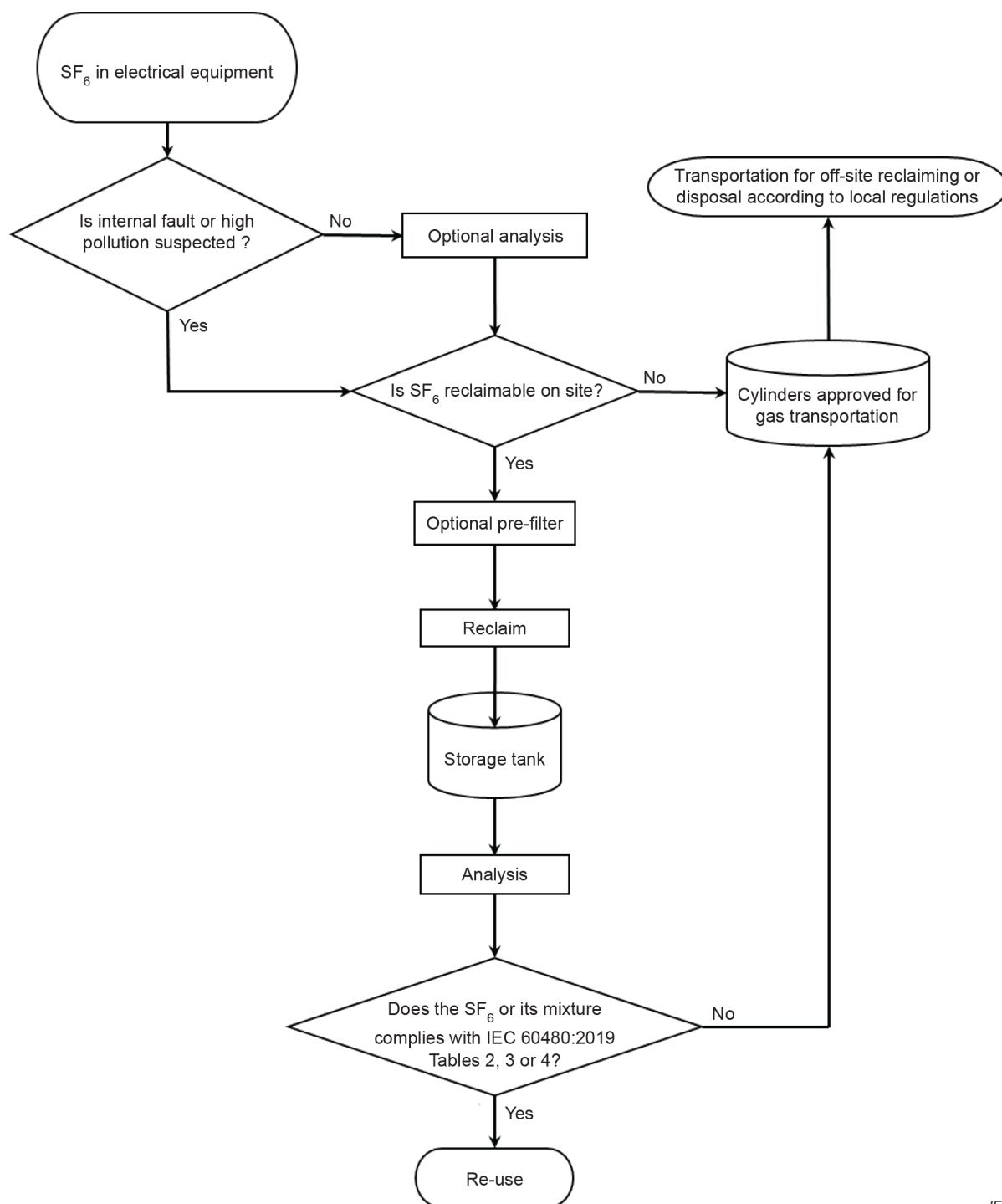
Various types of adsorbent materials are available to remove contaminants from SF₆ gas (see Table 6).

Table 6 – Typical adsorbents for various SF₆ contaminants

Adsorbent	Contaminants removed
Molecular sieve 4A	Water, SO ₂ , SOF ₂ , SF ₄
Molecular sieve 13X	Water, SO ₂ , SOF ₂ , SF ₄ (also adsorbs some SF ₆)
Activated aluminium oxide	Water, SO ₂ , SOF ₂ , SF ₄ , HF
Soda lime (CaO-NaOH)	Water, SO ₂ F ₂ , HF
Activated charcoal	Oil vapour

If the results of the gas analysis exceed the specifications for re-use of SF₆ and its mixtures given in Table 2, Table 3 or Table 4, a decision regarding the reclaiming method has to be made depending on the level and type of contamination. In general, re-purifying the gas on-site with a service device plus a separation device will be the most favourable way. However, if re-use is not possible, reclaiming by the gas manufacturer or disposal will be necessary. In this case, the gas shall be sent to the SF₆ manufacturer or reclaiming.

Figure 1 defines the selection procedure to determine the best use of SF₆ after recovery for potential treatment.



IEC

Figure 1 – Decision flow chart for recovered SF₆

- For contaminants of water or by-products, the question of whether the SF₆ is reclaimable on-site depends only on the performance of the filters available. The addition of external pre-filters may be required to increase the efficiency of the reclaiming process. If the SF₆ is not reclaimable on-site, then it shall be returned to the SF₆ manufacturer or sent to a reclaiming or disposal company.
- The case of contamination with air, N₂ and/or CF₄ shall be considered separately.
- For non-mixed SF₆, if the concentration of air and/or CF₄ exceeds the maximum acceptable contaminant level as given in Table 2, and if the container from which the sample has been taken contains liquid SF₆, then transfer SF₆ from the gas phase into a second container. The transfer should be continued until a sample from the first reservoir satisfies the maximum acceptable level. The contents of the second container cannot be

reclaimed on-site. Any container that contains no liquid SF₆, i.e. only the gas phase, requires the analysis of only one sample for air and CF₄ to determine if it is suitable for re-use or cannot be reclaimed on-site.

- For mixed SF₆, if the concentration of air, N₂ and/or CF₄ exceeds the maximum acceptable contaminant level as given in Table 3 or Table 4, an evaluation of the reclaiming options should be done to determine if the mixture could be reclaimed on-site.

7.2 Detection techniques for checking the quality of the gases

7.2.1 General

The detection techniques shall be used on gas samples. Details of the procedures for obtaining a sample and of the analytical methods are provided in Annex A.

7.2.2 On-site analysis

If on-site analysis systems are used, they shall be equipped with a gas recovery system. The release of SF₆ into the atmosphere shall be avoided and the safety of personnel shall be ensured. On-site available analytical methods are shown respectively in Table 7 and Annex A.

In case of high concentration of by-products (see Clause 4), care should be taken to avoid damage to the measuring instrument.

Table 7 – On-site methods

Substance	Detection technique
SF ₆	Portable gas chromatography with thermal conductivity detector (GC-TCD) Infrared absorption Condensation method Speed of sound method
N ₂ in SF ₆ mixture	Portable gas chromatography with thermal conductivity detector (GC-TCD) Infrared absorption (NOTE 1) Speed of sound method (NOTE 1) (NOTE 2)
CF ₄ in SF ₆ mixture	Portable gas chromatography with thermal conductivity detector (GC-TCD) Infrared absorption Speed of sound method (NOTE 1) (NOTE 2)
By-products: SO ₂ , SOF ₂ , SO ₂ F ₂	Portable gas chromatography with thermal conductivity detector (GC-TCD) Infrared absorption Electrochemical sensor
HF	Infrared absorption Electrochemical sensor
Air and CF ₄	Density meter (for the % of SF ₆) Portable gas chromatography with thermal conductivity detector (GC-TCD) (NOTE 3) Infrared absorption
Water (humidity)	Electronic hygrometer Chilled mirror hygrometer Infrared absorption Electrochemical sensor
Mineral oil	Tube for mineral oil
NOTE 1 Indirect quantification done by the subtraction of the SF ₆ content.	
NOTE 2 Applicable only to known mixture composition.	
NOTE 3 Gas chromatography is only used to determine the concentration of N ₂ or CF ₄ in SF ₆ mixtures.	

7.2.3 Laboratory analysis

If no equipment is available on-site, the following recommended techniques should be used (see Table 8). Laboratory methods are intended to provide a quantitative assessment of the contaminants in a gas sample.

Water is the only contaminant that cannot be determined accurately from a sampling cylinder. The water content of a sample in a cylinder taken from a container is not representative of the water content in the container because water adsorbs on all surfaces. Therefore, the water analysis shall always be carried out directly on the container on-site. There is no recommended order of analysis.

Table 8 – Laboratory methods

Contaminants	Methods available
SF ₆	Gas chromatograph (NOTE 2). Infrared absorption
N ₂ in gas mixture	Gas chromatograph (NOTE 2)
CF ₄ in gas mixture	Gas (NOTE 2). Infrared absorption
Air: oxygen and nitrogen	Gas chromatograph (NOTE 2)
CF ₄ as contaminant	Gas chromatograph (NOTE 2). Infrared absorption
Oil	Infrared absorption Gas chromatograph (NOTE 2)
Decomposition products (total acidity): SO ₂ , SOF ₂ , SO ₂ F ₂ , SF ₄ , HF (NOTE 1)	Gas chromatograph (NOTE 2) Ion chromatography Infrared absorption Wet chemistry
NOTE 1 Ion chromatography, infrared absorption and wet chemistry are the only methods available to quantify HF.	
NOTE 2 Gas chromatography can be performed by using adequate detectors for the determination of contaminants or to control the mixing gas concentrations.	

More information about reclaiming recommendations can be found in Annex D. Information about cryogenic reclaiming, an example of reclaiming technique, can be found in Annex E.

8 Handling, storage and transportation (informative)

Refer to IEC 62271-4.

9 Safety and first aid

9.1 General safety rules

9.1.1 General

Before starting any maintenance/service work on electric power equipment, a detailed inspection of the state of the equipment shall be carried out and documented. In addition to the local safety regulations, at least the following general safety rules shall be followed:

- switch off and isolate;
- secure against re-closing;
- verify that equipment is de-energized;
- earth and short-circuit the equipment;
- cover or fence off nearby live parts.

Written documents giving permission to work on the electric power equipment should be agreed and signed by both the owner/user of the equipment and the service provider.

Table 9 lists the major issues to consider when working with SF₆ electric power equipment.

Table 9 – Measures when working with SF₆ electric power equipment

Item	Work in the vicinity of equipment (operation of equipment, visual check, room-cleaning)	Filling, recovery, evacuation of gas compartments	Opening of gas compartments, work on open compartments
Material safety data sheet/operational manuals	Not required	Applicable	Applicable
Training	Applicable ^a	Applicable	Applicable
SF ₆ handling equipment	Not required	Applicable	Applicable
Cleaning/neutralizing equipment	Not required	Not required	Applicable
Personal protection equipment	Not required	Not required	Applicable
^a General information should be specified according to the type of work and installation, in accordance with local safety regulations.			

A notice stating that open fire, naked flames (e.g. matches), smoking, use of heat engines, heating to more than 200 °C and welding without special precautions are prohibited and instructions for giving first-aid (see 9.3) should be displayed while SF₆ is being handled in any location.

When a gas compartment is opened after the electric power equipment has been in service, in order to avoid contact with the fine solid by-products, which may be present, personnel should wear suitable protective clothing. Particular attention should be given to protecting the eyes and the respiratory tract. Personnel working in or near to opened gas compartments, which have contained normally arced or heavily arced SF₆ should:

- use suitable tools and equipment;
- wear suitable protective clothing (see Table 10 and 9.1.5);
- observe high standards of personal hygiene;
- clean themselves and their equipment using disposable materials, before leaving the work area;
- remove protective clothing and wash them thoroughly as soon as possible after having left the work area;
- ensure that clothing, tools and components that have been in contact with by-products are securely packed in sealed bags or other sealed containers and are subsequently treated to neutralize any residues.

9.1.2 Protection of personnel

SF₆ is handled by certified personnel only. Local regulations regarding SF₆ handling may apply. Different training modules may be adopted, depending on the kind of work to be performed on the electric power equipment.

Specific training covering the recovery of used SF₆ from a gas-filled compartment is required for personnel opening or entering the gas compartment. The operating instruction manual of the equipment provided by the original equipment manufacturer should be strictly followed.

Table 10 gives an overview of the potential risks, safety precautions as well as safety equipment and tools required when opening or entering a gas compartment. No substantial difference between indoor and outdoor conditions is given as the operator opens or enters the compartment.

Table 10 – Safety measures when opening or accessing gas compartments

Item	Any compartment which contained normally or heavily arced SF ₆	Any compartment which contained non-arc'd SF ₆
Potential risk	Fumes of cleaning substances O ₂ starvation Remaining used SF ₆ Residual reactive gaseous by-products Solid by-products and adsorber materials	Fumes of cleaning substances O ₂ starvation Remaining used SF ₆ or other gas from production process
Safety precaution	Removal of solid by-products and adsorber materials Ventilation Measurement of O ₂ concentration when entering Wear personal protective equipment Protect solid by-products against hydrolysis	Ventilation Measurement of O ₂ concentration before entering
Safety equipment and tools	Suction ventilator or vacuum cleaner O ₂ concentration measuring device Single use protective overalls, protective footwear, hair cap Acid proof safety gloves Full face mask (preferred) or, at least, breathing protective mask Protective goggles Environmental protection against rain or wind (outdoors only)	Suction ventilator or vacuum cleaner O ₂ concentration measuring device

If the oxygen concentration falls below 160 ml/l (16 % by volume) then the risk of asphyxiation arises. As a consequence, to mitigate the risk, the oxygen content in the gas compartment and the confined spaces should be checked prior to accessing or entering.

Eating, drinking and smoking are prohibited when accessing or entering a gas compartment. It is recommended that clothes are changed and the skin is washed as soon as possible after the work to prevent potential danger of irritation or burns.

9.1.3 Handling of contaminated safety equipment and tools

Equipment and tools, which have been in contact with solid by-products or adsorber materials are considered to be contaminated. They are collected afterwards and placed in plastic bags. The plastic bags are sealed with tape and labelled. Disposal is done according to the local regulations.

Reusable equipment and tools should be washed and neutralized in a water/soda solution with 10 % by weight liquid soda or equivalent and then rinsed with clean water. Examples of neutralizing solutions are given in Table 11.

Disposal of both the waste water/soda solution and the waste washing water is done according to the local regulations.

Table 11 – Neutralizing solutions

Active agent	Formula	Concentration kg/100 l	T1 ^a h	T2 ^b h	Reference ^e
Lime	Ca(OH) ₂	Saturated	Not applicable	24	[9]
Sodium carbonate (washing soda)	Na ₂ CO ₃	1, 1 3 10 ^c 10 to 14 ^c 3	Not applicable Wash not applicable 1 Not applicable	24 Not applicable 0,25 48 Not applicable	[10] [9] [11] [12] [13]
Sodium bicarbonate	NaHCO ₃	1 ^d	Not applicable	Not applicable –	[9]

^a Reusable safety equipment, tools, gas compartments, and internal parts of compartments which contained normally arced SF₆ should, where practicable, be treated with a neutralizing solution for a time period T1. They should then be rinsed with clean water.

^b Reusable safety equipment, tools, gas compartments, and internal parts of compartments which contained heavily arced SF₆ should, where practicable, be treated with a neutralizing solution for a time period T2. They should then be rinsed with clean water.

^c When using alkaline solutions at such high concentrations, care should be taken to avoid contact with the skin and eyes.

^d Recommended for washing the skin.

^e Numbers in square brackets refer to the Bibliography.

9.1.4 Pressurized equipment and tools or measuring devices

As with any pressurized gas, a sudden volume expansion results in a local temperature drop and may cause freezing. Suitable insulated gloves (e.g. leather, not latex) and protective goggles should be worn when working on high-pressure piping, valves or connectors, during filling operations.

All equipment and tools used during SF₆ handling potentially contain gaseous or liquid SF₆ under high pressure. They should be handled with care.

9.1.5 Personal safety and protective equipment

The use of personal safety and protective equipment is not related to the presence of the SF₆ electric power equipment itself. Standard safety shoes, helmet, and protective goggles may be required according to local regulations when working in the vicinity of the switchgear.

In case of abnormal release of SF₆ due to external fire or internal arc fault, additional safety rules apply for entering a switchgear room, according to 9.2.

Depending on the type of work to be performed on site and according to Table 10, workers engaged in handling SF₆ are provided with the following personal safety and protective equipment:

- Protective gloves: suitable acid resistant gloves made of for example neoprene, PVC, rubber.
- Protective goggles: chemical type industrial goggles according to local regulations (e.g. European standard EN 166).
- Breathing protective mask: to be used in conjunction with protective goggles, the breathing protective mask helps to protect mouth and nose against dust and should be used in case of exposure to minor dusty areas only.

- Full face mask: for short-term inspection and work where ventilation can be provided but where the concentration of by-products may exceed the appropriate maximum level, a face mask with changeable active charcoal filter is used according to local regulations. For example, European standards EN 140, EN 14387 and EN 143 specify masks, gas filters and particle filters, respectively;

NOTE Combined filters of type A2/B2/E2/K2/P3 manufactured to these standards are available and are able to provide protection against by-products including particles with a diameter greater than 1 µm.

- Single use protective overall: dust proof protective clothes to wear over normal clothes, shoe covers, hair cap. Pocket-less, hooded, non-permeable (e.g. bonded polypropylene) disposable industrial grade overalls having elastic ankle and wrist grips, overlapping the footwear and gloves.
- Respirator: when entering indoor applications after major leakage or internal arcing when the concentration of O₂ or the amount of by-products are at unsafe levels, a respirator is used according to local regulations (e.g. European standard EN 136).
- O₂ concentration measurement device for permanent monitoring of the O₂ content in the environment.
- Environmental protection for outdoor work locations: temporary shelter to prevent the ingress of rain and the wind dispersing solid by-products (if any) while the gas compartment is open.
- Vacuum cleaner: a high efficiency dedicated vacuum cleaner, equipped with a filter capable of trapping particles in the range of 1 µm, and a non-metallic open-ended nozzle according to local regulations (e.g. a type H machine in accordance with BS 5415-2-2, Supplement No. 1, 1986).
- Suction ventilator: equipment for forced ventilation of enclosed spaces and other inaccessible areas. Such equipment might be portable or permanently installed, depending on the size of the installation.

9.1.6 Facilities and services

Where gas compartments containing used SF₆ have to be recovered and opened, it is desirable that adequate washing facilities for workers be available, and a supply of water for preparing cleaning solutions may be required.

9.2 Additional safety measures in case of abnormal release of SF₆ due to external fire or internal arc fault

General safety recommendations to adopt when working with SF₆ on site are given in 9.1. Subclause 9.2 describes additional safety measures in case of abnormal release of SF₆ due to external fire or internal arc fault.

Under these circumstances, personnel trained on modules C1 or C2 (see IEC 62271-4:2013, Annex C) are allowed to enter and clean the switchgear room or to access the electric power equipment. Table 12 gives an overview of the potential risks, safety precautions as well as safety equipment and tools required.

Fire fighters entering the switchgear room should do so in accordance with local regulations.

Table 12 – Additional safety measures

Item	Abnormal release of heavily arced SF ₆	Abnormal release of non-arc'd or normally arced SF ₆
Potential risk	Fumes of cleaning substances O ₂ starvation SF ₆ abnormally released Residual reactive gaseous by-products Solid by-products	Fumes of cleaning substances O ₂ starvation SF ₆ abnormally released
Safety precaution	Removal of solid by-products Ventilation Measurement of O ₂ concentration when entering Wear personal protective equipment	Ventilation Measurement of O ₂ concentration when entering
Safety equipment and tools	Suction ventilator or vacuum cleaner O ₂ concentration measuring device Single use protective clothes, shoe covers, hair cap Acid proof safety gloves Full face mask (preferred) or, at least, breathing protective mask and protective goggles	Suction ventilator or vacuum cleaner O ₂ concentration measuring device

The same rules apply to areas below the level at which the release occurred, poorly ventilated or unventilated areas (e.g. cable trenches, inspection pits, drainage systems). Outdoor equipment needs standard measures for outdoor conditions to be applied (e.g. rain protection, wind protection). Natural ventilation is normally enough to prevent the potential risk of O₂ starvation.

A second person being in continuous visual and audible contact should be present when entering areas that may have a low oxygen concentration.

9.3 First aid equipment and treatment

9.3.1 General

First aid equipment includes:

- normal industrial first-aid equipment including eyewash equipment containing a saline solution;
- means for contacting emergency services;
- guidance for medical doctors.

The application of the general safety rules (see 9.1) should minimize the likelihood of accidents. In case of an accident, first aid treatment should be applied as described in 9.3.2 to 9.3.4.

9.3.2 Irritation of the skin

If skin irritation is observed, all personnel shall be evacuated from the area. Contaminated clothing shall be removed and the affected area of the skin shall be washed with cool running water.

NOTE While seeking professional medical advice, the affected part can be treated with calcium gluconate gel (HF antidote gel) as a remedy, after washing, for fluoride acid on skin.

9.3.3 Irritation of the eyes

In case of signs of eye irritation, the personnel shall evacuate the area. Irrigate the eye or eyes until a medical professional advises the patient to stop.

9.3.4 Breathing difficulty

All personnel should immediately evacuate the affected area and move into the fresh air. Contaminated clothing on a person with breathing difficulties should be removed and this person covered with a blanket and kept still and under continuous observation. Emergency medical assistance shall be summoned without delay. If the patient stops breathing, artificial respiration shall be immediately commenced by trained and qualified personnel.

More information about procedures for evaluating the potential effects on health from by-products of SF₆ and its mixtures can be found in Annex C.

10 Environmental aspects

Most created by-products are recombining to SF₆ inside the gas compartment or are transferred to solid by-products, some remain as toxic gaseous by-products but are trapped by molecular sieve. At the end of life all gaseous by-products and molecular sieve are recovered from the gas-filled compartment and shall not be released into the environment.

Major failures causing gas releases are extremely rare as records from 50 years of experience show. The quantities released in such extreme cases are again very limited by the fact that standard design of products is compartmented, limiting the fault to the place where it originates.

Annex A (informative)

Description of methods of analysis (on-site and laboratory)

A.1 Sampling

A.1.1 General

A sample should be representative of the gas composition in a container. The concentration of contaminants can be different between the liquid and gas phase. Humidity, O₂ and N₂ are most likely present in the gas phase while oil is most likely present in the liquid phase. For pure SF₆, the sample should be taken from the liquid phase in the event a sample from the gas phase does not meet specifications. For SF₆ mixtures, there should be no liquid phase. If there is no liquid phase in the container, sampling the gas phase is a true representation of the gas composition.

Before taking a sample from the container, the volume of all the connections from the container to the analytical equipment should be evacuated with a vacuum pump. In the case of a container that is connected to a sampling port, via a long section of narrow piping, the pipe section should be purged with gas from the container. This will ensure that the sample is representative of the SF₆ in the container.

As far as possible, the components in contact with the SF₆ should be constructed of stainless steel or other chemically resistant material to minimize reactions and contamination during the sampling process. The sample to be analysed shall not pass through an active filter (molecular sieve, aluminium oxide) that could alter the gas composition. However, a particle filter may be necessary to ensure that the sample is not contaminated with particles.

A.1.2 On-site sampling connection

A direct connection is required for all on-site methods of analysis. The connecting line should be stainless steel braided PTFE tubing type as short as possible. The ends of the line should have self-sealing fittings or valves to seal the line from ambient air contamination when not in use.

A.1.3 Sample cylinder for laboratory analysis

The cylinder volume should be in the range of 150 ml to 500 ml (a larger volume, up to 1 000 ml, may be needed for the Fourier transform infrared analysis (FTIR)). All materials of the cylinder and valve in contact with the sample should be stainless steel or chemically resistant materials, such as PTFE (polytetrafluoroethylene). Ideally the sampling material should be internally treated to create an inert surface. The cylinder and connections need to be rated for use at high pressure up to 5 MPa.

The following procedure is recommended in an effort to prepare a sampling cylinder prior to use (Figure A.1):

- heat the cylinder and evacuate for minimum 1 h (primary vacuum);
- close the valve and allow the cylinder to cool to room temperature before use.

A.1.4 Sampling methods for laboratory analysis

A.1.4.1 One-sampling cylinder method

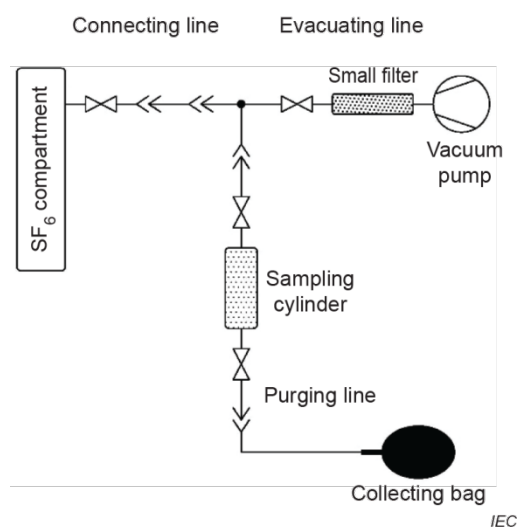


Figure A.1 – One-sampling cylinder method set-up

Figure A.1 shows an example of set-up for the one-sampling cylinder method. Connect the set-up to the sampling port of the container as for a direct connection. Fill the cylinder once and wait 1 min to allow the conditioning of the inside wall, purge, evacuate and fill it again. The use of self-sealing fittings facilitates all aspects of sample handling.

When purging, a collecting bag or similar equipment should be used in order to avoid any release of SF₆ into the atmosphere. A few litres of SF₆ can then be passed through the cylinder to condition its internal walls. After analysis, SF₆ samples should be reclaimed.

A.1.4.2 Two-sampling cylinder method

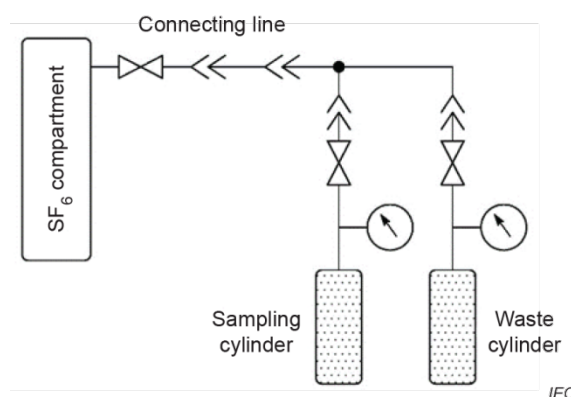


Figure A.2 – Two-sampling cylinder method set-up

Figure A.2 shows an example set-up for the two-sampling cylinder method. Two (2) cylinders are connected in parallel with tubing and distribution block to the sampling port of the container as for a direct connection. Each cylinder is equipped with a manometer in order to confirm that the cylinders are under vacuum before the sampling. Open the valve of the waste cylinder in order to purge the tubing and distribution block. Pressure in the waste cylinder will increase according to the pressure inside the sampled equipment. Close the valve of the waste cylinder when pressure is stabilized. Open the valve of the sampling cylinder. Pressure in the sampling cylinder will increase according to the pressure inside the sampled equipment. Close the valve when the pressure is stabilized.

No evacuation and collection bag or similar equipment is necessary on-site. The cylinders have to be prepared according to the procedure described in A.1.3. The tubing, manometer and valve connected to the cylinders has to be properly designed and assembled with care in order to keep the cylinders under vacuum for a long period until their utilization. After analysis, SF₆ samples and waste materials should be reclaimed.

A.2 On-site analysis

A.2.1 General

Tubes that detect water vapour, HF, SO₂ and mineral oil are available from several manufacturers. The use of such tubes is no longer recommended for analysing SF₆. Other detection systems like SO₂ sensors or multi-detector apparatus are more precise and made for use in SF₆ systems.

A.2.2 SF₆ concentration meter

With this device, the SF₆ concentration is determined by a density meter which measures the speed of sound in a gas sample. This type of meter is usually calibrated for measuring pure SF₆ percentage in air with a precision of about ± 1 %; however, this accuracy may be affected by the presence of other gases (for example CF₄, by-product).

In all cases the manufacturer's instructions should be followed.

A.2.3 Hygrometers

The following types of apparatus are available:

Chilled mirror hygrometer

A chilled mirror hygrometer determines the dew point or frost point by measuring the temperature at which condensation in the form of dew or frost occurs on a mirror. The equilibrium temperature at which the mirror maintains a stable condensation layer is called the dew point temperature above 0 °C and frost point temperature below 0 °C. The measured dew or frost point temperature is then mathematically converted to water vapour pressure and then to any desired units such as $\mu\text{l/l}$ (ppmv)².

Electronic hygrometer

An electronic hygrometer utilizes a sensor with characteristics that are affected by partial pressure of water vapour in a gas mixture, generally by a change in capacitance as the gas passes through a semi-permeable layer. The capacitance or other changing characteristic is usually calibrated in terms of dew point.

Electrochemical sensor hygrometer

The electrochemical hygrometer determines the humidity by measuring the steady current needed to electrolyse the water in the gas flow.

WARNING: Measurements of SF₆ with HF content may damage the equipment. In all cases, the manufacturer's instructions should be followed.

² ppmv = part per million by volume

A.3 Laboratory analysis

A.3.1 Gas chromatography

A.3.1.1 General

Chromatography is used to analyse SF₆ and its mixtures in order to identify and quantify the composition of the gas and its contaminants.

Chromatography is a separation technique used for chemical substances (homogeneous liquid or gaseous mixture) which is based on differences in behaviour between a current mobile phase (e.g. gas) and a stationary phase (an analytical column).

Each different compound requires a different time to traverse the column which is related to its chemical interaction with the latter. The result is a chromatogram, which is a time series of peaks corresponding to the components of the sample. For peak detection, different detector types are available. The thermal conductivity detector (TCD) is recommended for multi-purpose measuring and stability. Other detectors are available and can be used too for example discharge ionisation detectors (DID) or electron capture detectors (ECD) (Figure A.3).

A.3.1.2 Gas chromatography with detector systems with linear accuracy over minimum three decades

A.3.1.2.1 General

An example of a detector system with linear accuracy is the thermal conductivity detector (TCD). Air, CF₄, CO₂, SF₆ and SOF₂ are readily analysed. However, the quantitative measurement of SO₂F₂ and SO₂ may be difficult. The analysis of HF is not possible, as it is not detectable with this chromatographic technique.

Typical accuracy for the quantification of CF₄, N₂, and air are given in IEC 60376 [3]. The accuracy of the recommended method as described in IEC 60376 [3] can be improved as follows:

- An automatic injection unit, using a sampling loop (0,1 ml to 1 ml) and a multi-port switching valve, is preferred to the manual injection with a glass syringe. The chromatograph should be equipped with an inlet port to which the sampling bottle is attached.
- An improved separation of trace contaminants is obtained with a temperature gradient (typically 60 °C to 180 °C), rather than by isothermal. In this case, a peak corresponding to the water vapour present in the gas sample can be seen in the chromatogram. However, moisture content can only be determined semi-quantitatively in this case.
- It is important to note that this chromatographic method will produce adequate quantitative measurements if, and only if, the chromatograph systems are well conditioned. This means that the instrumentation should be kept running and used routinely. Otherwise, prior to use, it will need a thorough preconditioning by injecting suitable gas mixtures containing several hundreds of ppmv of SO₂ and/or SOF₂ and/or HF in air (or SF₆) followed by a series of calibration runs.

A.3.1.2.2 Representative chromatograph set-up

Reliable results are expected if using the following parameters:

- Carrier gas:
helium or hydrogen (10 ml/min to 25 ml/min, a slightly better resolution can be obtained with H₂); flow rate should be optimized for the type of column used;
- Analytical column:
stainless steel tube packed by porous polymers (80/100 mesh), 3 m to 4 m × 3 mm;

wide-bore capillary, 20 m to 30 m, 0,53 mm

- Oven programme:
initial temperature: 60 °C to 80 °C;
final temperature: 120 °C to 180 °C;
heating rate: 10 °C /min to 20 °C/min.

A chromatogram of SF₆ contaminants and decomposition by-products is shown in Figure A.3.

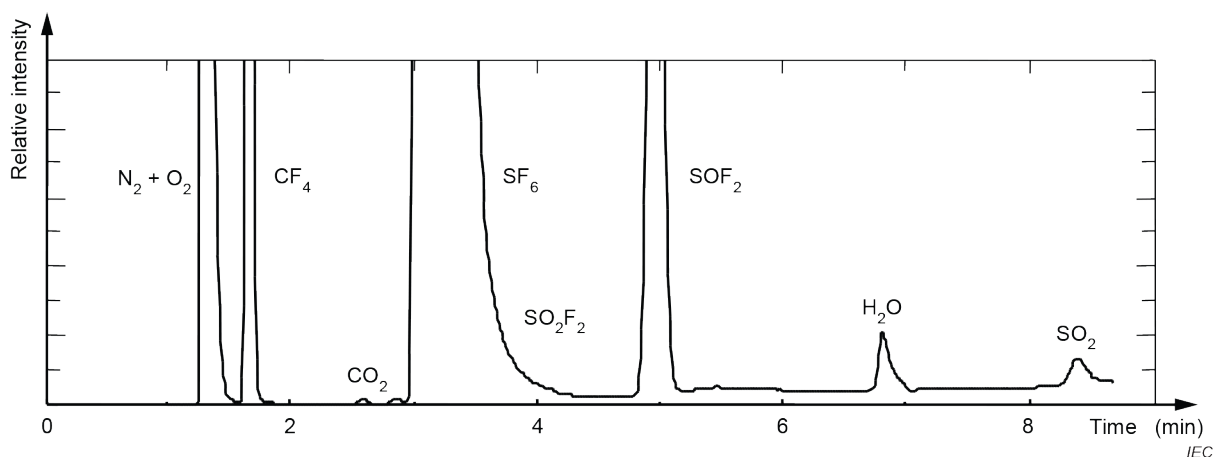


Figure A.3 – Example of a gas chromatogram in one print out showing the different possible by-products after decomposition

A.3.1.3 Example of gas chromatography with mass spectrometer detector (MS)

A.3.1.3.1 General

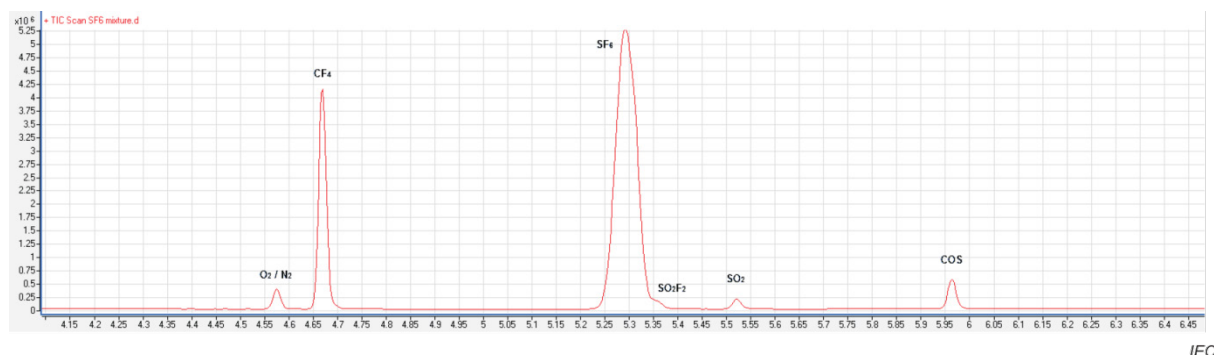
This chromatographic technique allows the quality of the pure SF₆ or SF₆ mixtures to be checked (N₂ or CF₄). It also allows identification and quantification of the following contaminants: CF₄, SO₂, N₂, O₂, COS, SO₂F₂ and SOF₂.

A.3.1.3.2 Representative chromatograph set-up

Reliable results are expected if using the following parameters:

- Carrier gas: helium (flow rate: 2,5 ml/min (40 cm/s)); flow rate should be optimized for the type of column used;
- Analytical column: low sulphur, 60 m, 0,32 mm;
- Oven programme:
 - initial temperature: 30 °C;
 - final temperature: 105 °C;
 - heating rate: 25 °C/min;
- Interface GC/MS temperature: 250 °C;
- Detector:
 - source temperature: 230 °C;
 - quadrupole temperature: 150 °C.

A typical chromatogram of SF₆/CF₄ mixture contaminants and decomposition by-products is shown in Figure A.4.



SOURCE: HYDRO-QUÉBEC, reproduced with the permission of the authors.

Figure A.4 – Typical GCMS chromatogram of decomposed SF₆/CF₄ mixture

A.3.1.3.3 Example for the determination of oil by gas chromatography with flame ionization detector (FID)

The oil content in SF₆ can be measured with the following method, which is applicable to the analysis of C₁₀ to C₅₀ petroleum hydrocarbons. The oil may be obtained from a filter placed in a transfer line, or directly from a container, by washing with cyclohexane. With a high temperature flame ionization detector, the minimum detection limit is about 4 µg oil per 1 ml of cyclohexane. The minimum detection limit of oil in SF₆ will depend on the mass of SF₆ that passed through the filter or was in the container and the volume of cyclohexane used to extract the oil.

The following conditions represent one example that will achieve the desired results:

- Analytical column: Stainless steel megabore column, 5 m in length × 0,53 mm inner diameter, with a 0,15 µm film thickness.
- Carrier and make-up gas: Ultra-high purity helium at 15 ml/min each, for a total flow rate to the flame ionization detector of 30 ml/min.
- Flame ionization detector gas: Ultra-high purity hydrogen at 30 ml/min, and air at 300 ml/min.

The chromatogram is analysed by peak grouping to measure the area of the C₁₀-C₂₄ and the C₂₄-C₅₀ petroleum hydrocarbon ranges. These two ranges are calibrated with n-hexadecane (n-C₁₆) and n-hexatriacontane (n-C₃₆), respectively.

A.3.1.4 Ion chromatography

Ion chromatography is based on ion exchange columns. It can be used for the analysis of the following decomposition products: SO₂, SOF₂, SO₂F₂, SF₄, HF. Carbonate eluent has very low background yield and excellent sensitivity. This method has been validated for a variety of environmental matrices using the ION PAC AS4A. Further details can be found in US EPA catalogue [11].

A.3.2 Infrared spectroscopy

A.3.2.1 Principle

A beam of infrared light directed through a material sample to a light sensitive detector is attenuated. The ratio of the transmitted light to the incident as a function of wavelength forms the infrared absorption spectrum of the material sample.

The infrared absorption spectrum of a gas sample will exhibit peaks at the absorption wavelength of the gas. The size, shape and location of peaks in a spectrum can be used to identify the presence and quantity of a gas sample.

Most of the contaminants of SF₆ can be identified by infrared absorption except oxygen and nitrogen; diatomic gases and monatomic gases (e.g. argon) do not have a significant infrared absorption. The presence of some contaminants may be obscured by the spectrum of SF₆. The spectral region at frequencies less than 580 cm⁻¹ is clear of any SF₆ interference.

A.3.2.2 Fourier transform infrared spectrometer (FTIR)

FTIR spectrometers provide high resolution with a fast response. The whole infrared spectrum is measured many times and averaged to reduce the effect of noise. Spectra should be obtained at a resolution sufficient to resolve the absorption bands to identify and quantify the sample components.

A.3.2.3 Absorption cell

The gas sample is introduced into an absorption cell within the spectrometer for analysis. The cell is preferably made of stainless steel to minimize the reaction with the contaminant, HF, of the sample. The cell should be fitted with KBr windows for infrared transmission down to, at least, 500 cm⁻¹. The optical path length of the cell should be at least 10 cm. Path lengths of several metres are obtained by path-folding mirrors within the cell to minimize the total volume of the cell. The optimum path length depends on the signal-to-noise ratio of the spectrometer at the absorption frequencies of interest and on the minimum detection limit required.

A.3.2.4 Analysis

The method of spectral analysis is based on the linear relationship between absorbance and concentration. The linearity domain depends upon the equipment used and the absorptivity.

A.3.2.5 Absorption frequencies of gases

The principal frequencies of absorption peaks for SF₆ and the contaminants of SF₆ are shown in Table A.1.

Table A.1 – Peak absorption of SF₆ and contaminants

Gas	Maxima of absorption cm ⁻¹	Peak absorptivities x 10 ⁻⁶ (kPa x ml/l x m) ⁻¹
SO ₂	491, 494, 497, 500, 503, 506	~ 1,1 each
SOF ₂	530, 808	12, 46
SO ₂ F ₂	539, 544, 552	21, 25, 15
SOF ₄	570, 752	4, 8
SF ₄	532, 730	9, 80
CF ₄	1 283, 2 186	550; 2,2
HF	3 644, 3 693	Lines too narrow to quantify
Mineral oil	2 930	Not available
SF ₆	610, 860, 950, 1 260, 1 560 (broad bands)	Not given

The presence of water vapour can be observed but is hard to be quantified by FTIR.

The peak absorptivities in Table A.1 are provided as a guide to select and/or calibrate the conditions of pressure and path length for the spectra of the samples. Note that the presence of SF₆ does modify the appearance of the spectra, broadening and in some cases shifting the peaks. Reference spectra of contaminants should be obtained in conditions similar to the conditions for samples.