भारतीय मानक

Indian Standard

IS 17053 : 2023
IEC 60480 : 2019

विद्युत उपकरणों से ली गई सल्फर हेक्साफ्लोराइड (एस एफ$_6$) की जांच और उपचार के लिए दिशानिर्देश और इसके पुनः उपयोग के लिए विनिर्देश

(पहला पुनरीक्षण)

Guidelines for the Checking and Treatment of Sulfur Hexafluoride (SF_6) Taken from Electrical Equipment and Specification for its Re-Use

(First Revision)

ICS 29.040.20; 29.130.01

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March 2023   **Price Group 14**

Fluids for Electrotechnical Applications Sectional Committee, ETD 03

NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical with IEC 60480 : 2019 ‘Specifications for the re-use of sulphur hexafluoride (SF_6) and its mixtures in electrical equipment’ issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Fluids for Electrotechnical Applications Sectional Committee and approval of the Electrotechnical Division Council.

This standard was first published in 2019 identical IEC 60480 : 2004. This revision has been undertaken to align this standard with the latest version of IEC 60480 : 2019.

The text of the IEC Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words ‘International Standard’ appears referring to this standard, theyshould be read as ‘Indian Standard’.

b) Comma (,) has been used as a decimal marker, while in Indian Standards the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted, are listed below alongwith their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
IEC 60050-192 International electrotechnical vocabulary — Part 192: Dependability	IS 1885 (Part 192) : 2021 Electrotechnical vocabulary: Part 192 Dependability (Superseding IS 1885 Part 39 : 1999)	Identical with IEC 60050-192 : 2015
IEC 60050-212 International electrotechnical vocabulary — Part 212: Electrical insulating solids, liquids and gases	IS 1885 (Part 62) : 2021 Electrotechnical vocabulary: Part 62 Electrical insulating solids liquids and gases (*second revision*)	Identical with IEC 60050-212 : 2010
IEC 60050-441 International Electrotechnical Vocabulary — Part 441: Switchgear, controlgear and fuses	IS 1885 (Part 17) : 1979 Electrotechnical vocabulary: Part 17 switchgear and control gear (*first revision*)	Identical with IEC Pub 60050-441 : 1974

The technical committee has reviewed the provisions of the following international standards referred in this adopted standard and decided that they are acceptable for use in conjunction with this standard.

International Standard	*Title*
IEC 60050-826	International Electrotechnical Vocabulary — Part 826: Electrical installations
IEC 62271-4 : 2013	High-voltage switchgear and controlgear — Part 4: Handling procedures for sulphur hexafluoride (SF6) and its mixtures

Only the English language text has been retained while adopting it in this Indian Standard, and as such, the page numbers given here are not the same as in the IEC Publication.

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Indian Standard

GUIDELINES FOR THE CHECKING AND TREATMENT OF SULFUR HEXAFLUORIDE (SF_6) TAKEN FROM ELECTRICAL EQUIPMENT AND SPECIFICATION FOR ITS RE-USE

(First Revision)

1 Scope

This document provides criteria for the re-use of sulphur hexafluoride (SF_6) and its mixtures after recovery and reclaiming from electrical equipment (e.g. for maintenance, at the end-of-life).

Sulphur hexafluoride (SF_6), nitrogen (N_2) and carbon tetrafluoride (CF_4), are gases commonly used for electrical equipment. Taking into account environmental concerns, particular attention is paid to re-use criteria for SF_6 and its mixtures with N_2 and CF_4 for its use in electrical equipment. Procedures for recovering and reclaiming used SF_6 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 SF_6, and on reclaiming recommendations.

Storage, transportation and disposal of SF_6 and its mixtures are outside the scope of this document and are covered by IEC 62271-4. Procedures to determine SF_6 leakages are described in IEC 60068-2-17 [4][1].

For the purposes of this document, the complementary gases used in SF_6 mixtures will be limited to N_2 or CF_4.

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 (SF_6) and its mixtures*

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[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
SF_6 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
SF_6 mixture
gas mixture formed by SF_6 and a complementary gas, typically N_2 or CF_4

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 SF_6 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

SF_6 recovered from electrical equipment in operation contains several kinds of contaminants. Contaminants in recovered SF_6 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 – SF_6 contaminants

SF_6 situation and use	**Origin**	**Possible contaminant**
Handling and in service	Leaks and incomplete evacuation Desorption	For pure SF_6: Air, oil, H_2O For SF_6 mixtures: Air, oil, H_2O, 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_2F_2 For SF_6 mixtures: HF, SO_2, SOF_2, SOF_4, SO_2F_2, NO_x, NF_X
Switching equipment	Switching arc erosion	Gaseous by-products: HF, SO_2, SOF_2, SOF_4, SO_2F_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_2F_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_2F_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_2F_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 SF_6 by-products in equipment that only have an insulating function

The essential process is the decomposition of SF_6 by partial discharges (e.g. corona) and low energy flashovers and sparkovers. The immediate products are fragments of SF_6, such as SF_5, SF_4 and F, combining with O_2 and H_2O to form compounds, mainly HF, SO_2, SOF_2, SOF_4 and SO_2F_2. Due to low energy of the partial discharges, flashovers or sparkovers, the accumulated quantities of these compounds are usually negligible.

4.4 SF_6 by-products in switching equipment

During current interruption, the existence of high temperature arcs leads to the formation of by-products of SF_6, 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 SF_6 by-products from internal arcs

The occurrence of an internal arc is extremely rare. The expected contaminants in SF_6 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 SF_6 mixtures specific by-products

For SF_6 mixtures, the usual SF_6 by-products mentioned in Table 1 and specific mixture by-products, such as nitrogen oxide(s) and nitrogen fluoride(s) for SF_6/N_2 and fluorocarbon(s) for SF_6/CF_4, are produced. The quantities depend on the mixture composition, contaminants and energy introduced. For typical SF_6 mixtures, the gas decomposition rates are not expected to exceed those for SF_6.

Within the by-products generated in mixtures, SF_6 by-products are generally predominant in terms of quantity and toxicity. Safety procedures related to the presence of the usual SF_6 by-products shall also apply in applications with SF_6 mixtures.

5 Specifications for re-use of SF_6

Table 2 – Specifications for re-use of SF_6

Substance[a]	Concentration
SF_6	> 97 % volume
Air and/or CF_4	< 30 000 µl/l (i.e. 3 % volume)
H_2O	< 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_2S 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 (SO_2 + SOF_2) or in terms of HF with a limit value of 12 µl/l and 25 µl/l respectively.

6 Specifications for re-use of SF_6 mixtures

Table 3 – Specifications for re-use of SF_6/N_2 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_2O	< 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 SF_6/CF_4 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_2O	< 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 SF_6 and SF_6 mixtures

7.1 Feasibility and process

The quality of reclaimed SF_6 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 SF_6 gas (see Table 6).

Table 6 – Typical adsorbents for various SF_6 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_2F_2, HF
Activated charcoal	Oil vapour

If the results of the gas analysis exceed the specifications for re-use of SF_6 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 SF_6 manufacturer or reclaimer.

Figure 1 defines the selection procedure to determine the best use of SF_6 after recovery for potential treatment.

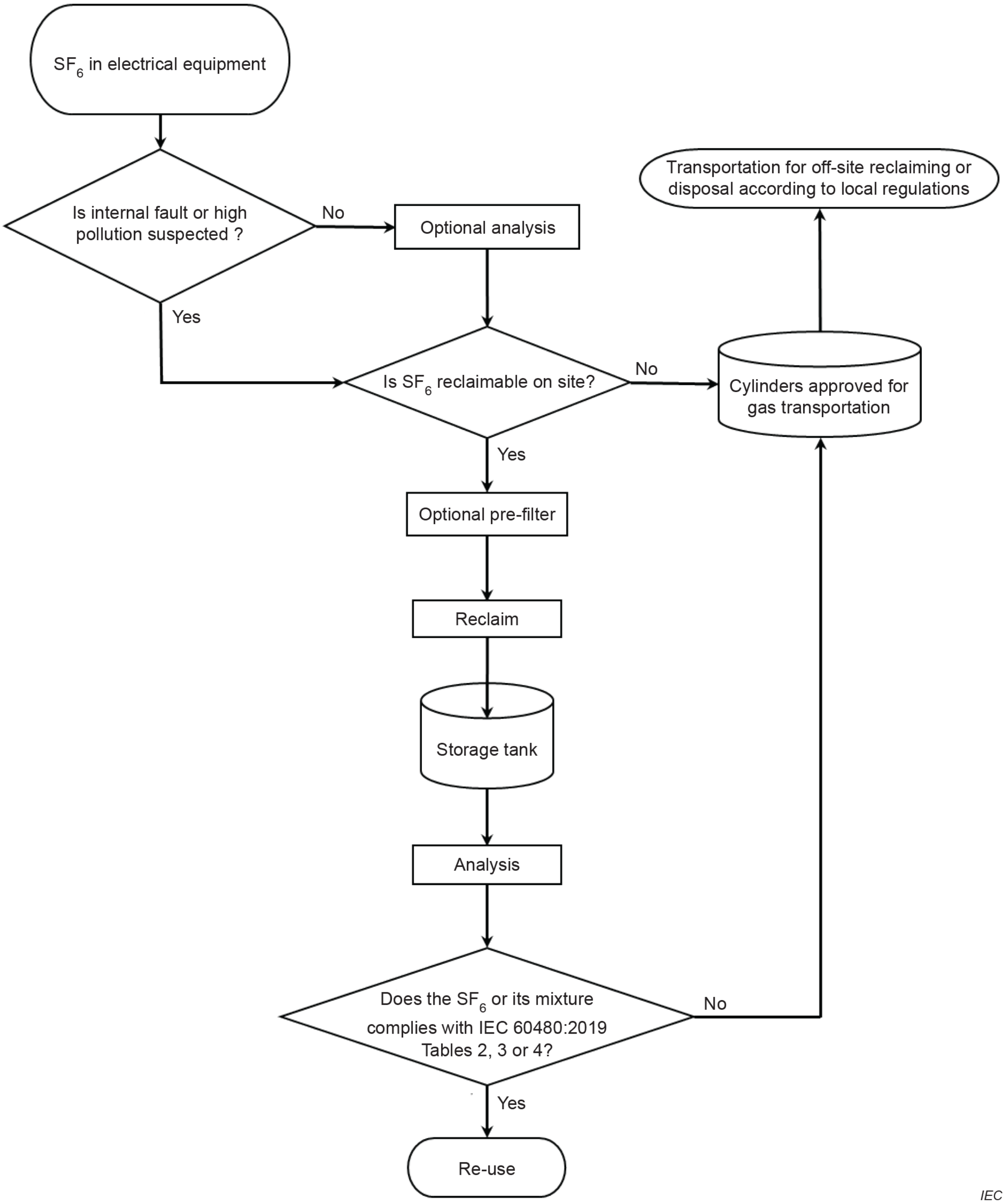

Figure 1 – Decision flow chart for recovered SF_6

- For contaminants of water or by-products, the question of whether the SF_6 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 SF_6 is not reclaimable on-site, then it shall be returned to the SF_6 manufacturer or sent to a reclaiming or disposal company.
- The case of contamination with air, N_2 and/or CF_4 shall be considered separately.
- For non-mixed SF_6, if the concentration of air and/or CF_4 exceeds the maximum acceptable contaminant level as given in Table 2, and if the container from which the sample has been taken contains liquid SF_6, then transfer SF_6 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 SF_6, i.e. only the gas phase, requires the analysis of only one sample for air and CF_4 to determine if it is suitable for re-use or cannot be reclaimed on-site.

- For mixed SF_6, if the concentration of air, N_2 and/or CF_4 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 SF_6 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_2F_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_2F_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 SF_6 electric power equipment.

Table 9 – Measures when working with SF_6 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 SF_6 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 SF_6 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

SF_6 is handled by certified personnel only. Local regulations regarding SF_6 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 SF_6 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-arced 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_2CO_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 SF_6 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 SF_6 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 SF_6 handling potentially contain gaseous or liquid SF_6 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 SF_6 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 SF_6 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 SF_6 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 O_2 or the amount of by-products are at unsafe levels, a respirator is used according to local regulations (e.g. European standard EN 136).
- O_2 concentration measurement device for permanent monitoring of the O_2 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 SF_6 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 SF_6 due to external fire or internal arc fault

General safety recommendations to adopt when working with SF_6 on site are given in 9.1. Subclause 9.2 describes additional safety measures in case of abnormal release of SF_6 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-arced 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 O_2 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 SF_6 and its mixtures can be found in Annex C.

10 Environmental aspects

Most created by-products are recombining to SF_6 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, O_2 and N_2 are most likely present in the gas phase while oil is most likely present in the liquid phase. For pure SF_6, the sample should be taken from the liquid phase in the event a sample from the gas phase does not meet specifications. For SF_6 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 SF_6 in the container.

As far as possible, the components in contact with the SF_6 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 braded 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

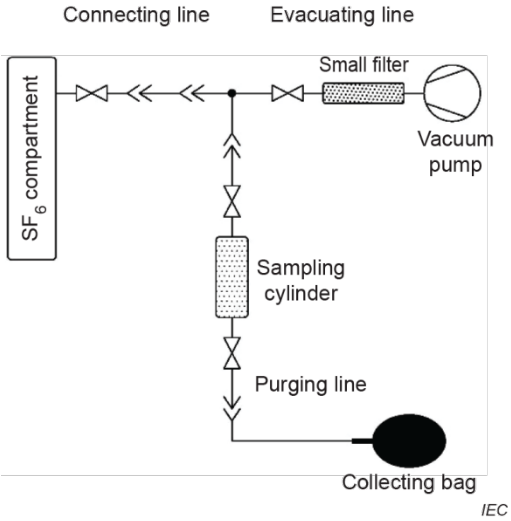

Figure A.1 – One-sampling cylinder method set-up

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 SF_6 into the atmosphere. A few litres of SF_6 can then be passed through the cylinder to condition its internal walls. After analysis, SF_6 samples should be reclaimed.

A.1.4.2 Two-sampling cylinder method

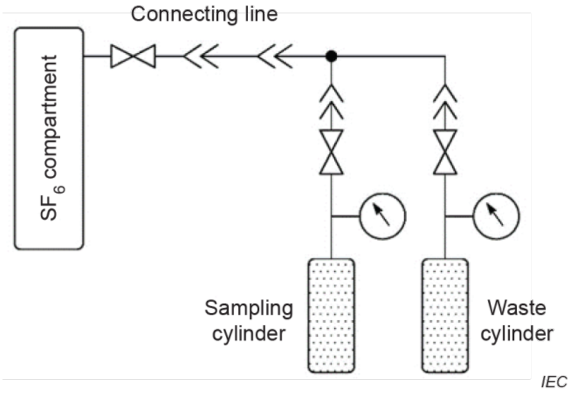

Figure A.2 – Two-sampling cylinder method set-up

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, SF_6 samples and waste materials should be reclaimed.

A.2 On-site analysis

A.2.1 General

Tubes that detect water vapour, HF, SO_2 and mineral oil are available from several manufacturers. The use of such tubes is no longer recommended for analysing SF_6. Other detection systems like SO_2 sensors or multi-detector apparatus are more precise and made for use in SF_6 systems.

A.2.2 SF_6 concentration meter

With this device, the SF_6 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 SF_6 percentage in air with a precision of about ±1 %; however, this accuracy may be affected by the presence of other gases (for example CF_4, 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 µl/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 SF_6 with HF content may damage the equipment. In all cases, the manufacturer's instructions should be followed.

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[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 SF_6 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, CF_4, CO_2, SF_6 and SOF_2 are readily analysed. However, the quantitative measurement of SO_2F_2 and SO_2 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 CF_4, N_2, 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 SO_2 and/or SOF_2 and/or HF in air (or SF_6) 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 H_2); 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 SF_6 contaminants and decomposition by-products is shown in Figure A.3.

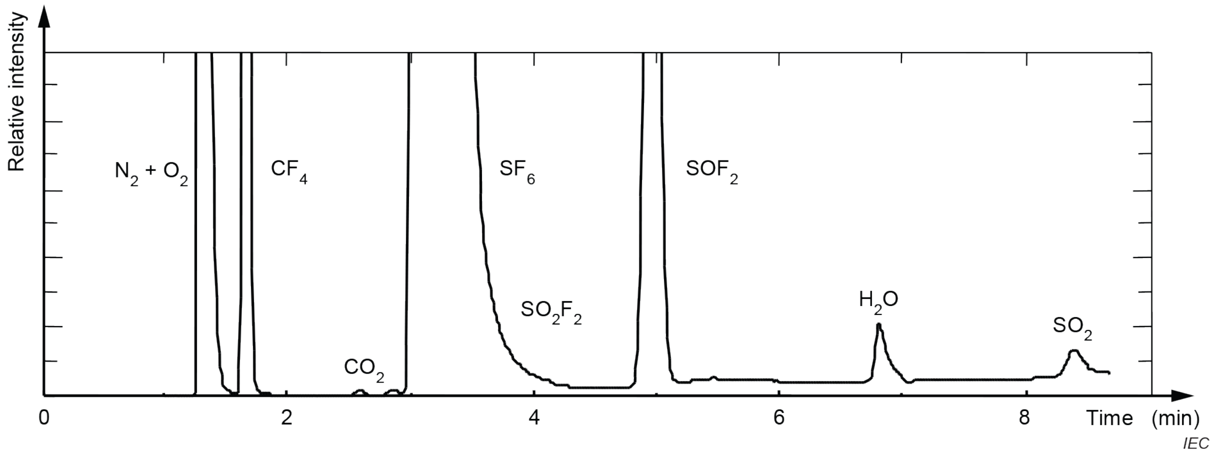


Figure A.3 – Example of a gas chromatogram in one print out showing the different possible by-products after decomposition

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

A.3.1.3.1 General

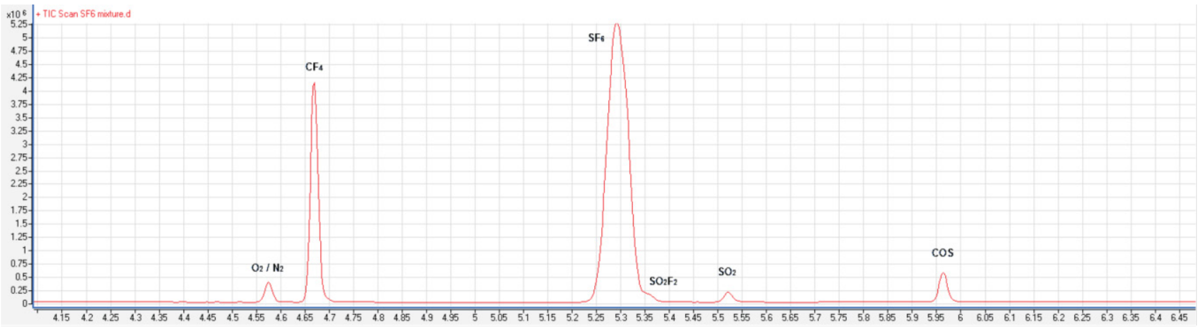
This chromatographic technique allows the quality of the pure SF_6 or SF_6 mixtures to be checked (N_2 or CF_4). It also allows identification and quantification of the following contaminants: CF_4, SO_2, N_2, O_2, COS, SO_2F_2 and SOF_2.

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 SF_6/CF_4 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 SF_6/CF_4 mixture

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

The oil content in SF_6 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 SF_6 will depend on the mass of SF_6 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: SO_2, SOF_2, SO_2F_2, SF_4, 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 SF_6 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 SF_6. The spectral region at frequencies less than 580 cm^{-1} is clear of any SF_6 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 SF_6 and the contaminants of SF_6 are shown in Table A.1.

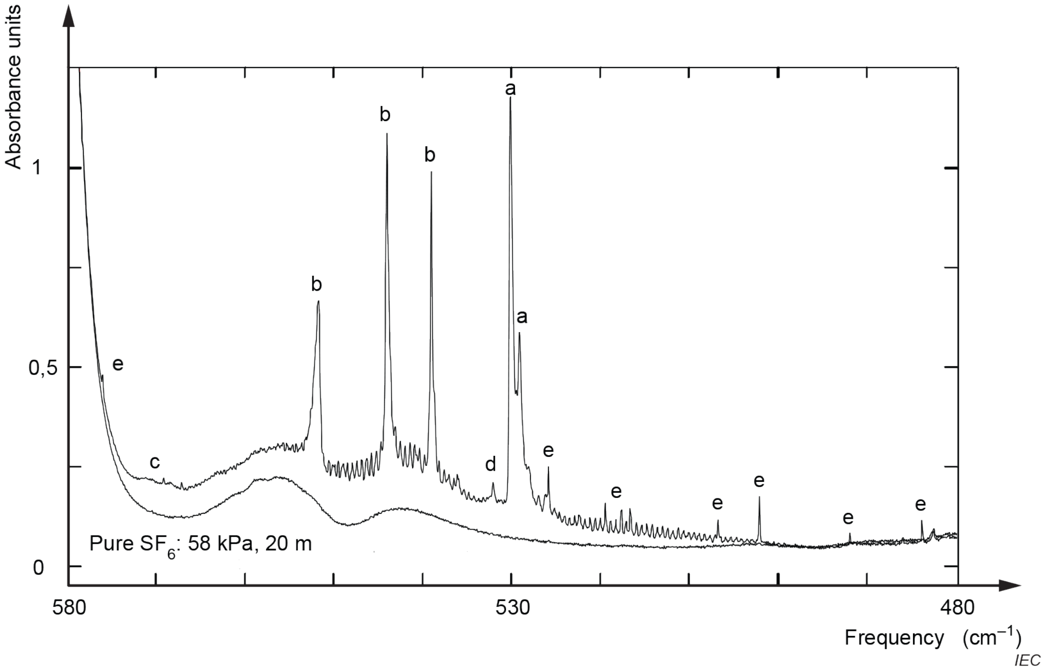
Table A.1 – Peak absorption of SF_6 and contaminants

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_2F_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 SF_6 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.

A typical spectrogram of SF_6 and contaminants recorded in the above conditions is shown in Figure A.5.



Key

a: SOF_2

b: SO_2F_2

c: SOF_4

d: SF_4

e: H_2O

Figure A.5 – IR spectrum of contaminated SF_6

Annex B
(informative)

By-products of SF_6 and its mixtures

B.1 Decomposition of SF_6 and its mixtures

B.1.1 General

For SF_6 mixtures, the usual SF_6 by-products and specific mixture by-products, such as nitrogen oxide(s) and nitrogen fluoride(s) for SF_6/N_2 and fluorocarbon(s) for SF_6/CF_4, will be produced. The quantities will depend on the mixture composition, contaminants and energy introduced. For typical SF_6 mixtures, the gas decomposition rates are expected not to exceed those for undiluted SF_6.

When arcing occurs in SF_6 due either to normal switching operations or fault clearances, or in the unlikely event of an internal arcing fault, different by-products are generated simultaneously in varying quantities [12] [13]. When the SF_6 molecule is stressed by temperature, radiation or electrical discharge and separation of fluorine atoms occurs, a number of radicals, ions, or neutral molecules are produced, depending on the type of excitation and the energy input, according to:

$$SF_6 \xrightarrow{\Delta E} SF_x + (6-x)F, 0<x<6 \qquad \text{(B.1)}$$

When the input of energy ΔE ceases, most of the atoms recombine to form SF_6, whilst others combine with different substances in the system to form a variety of stable end products. Such substances include in particular oxygen and water and also materials used in the construction of the equipment.

These by-products are considered here in relation to the energy delivered to the SF_6.

B.1.2 Behaviour of SF_6 in an electric arc

Heavy current arcing occurs normally during circuit-breaker switching and fault clearance operations, and abnormally during an internal arc fault.

In the presence of arc, SF_6 begins to decompose into its constituent parts, with the degree of decomposition being directly proportional to the quantity of energy converted.

The large quantity of heat absorbed during this process is dissipated away from the arc zone by radiation and convection. As the gas cools down, the atoms recombine or react with other substances, such as vaporized electrode metal, the vessel wall, plastics or contaminants. Gaseous and solid by-products can arise, including metal fluorides and sulphur fluorides, of which the most important are CuF_2, AlF_3, WF_6, CF_4 and SF_4.

These products, generally known as primary by-products, are formed during or shortly after discharge in the less-than-one-second range. Dust-like deposits, which may appear on the surfaces of insulators during normal operation, have no detrimental effect on their dielectric performance.

Some of the by-products are chemically stable; others are very unstable, particularly in presence of water.

In presence of oxygen, by-products can arise as follows:

$$S + O + 2F \text{->} SOF_2 \qquad (B.2)$$

$$SF_4 + O \text{->} SOF_2 + 2F \qquad (B.3)$$

$$SF_3 + O \text{->} SOF_2 + F \qquad (B.4)$$

$$SF_5 + O \text{->} SOF_4 + F \qquad (B.5)$$

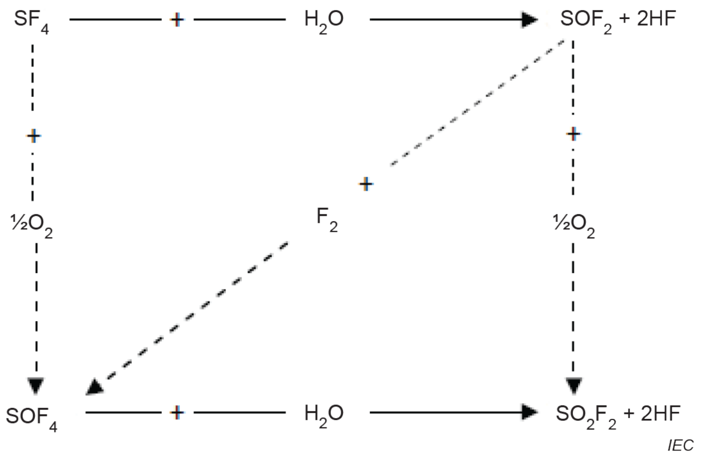
The oxygen involved in the reactions (B.2), (B.3), (B.4), and (B.5) may remain as a result of the evacuation process or may be released by the electrode materials during arcing. SOF_2 is the major by-product.

In the presence of moisture, the following reactions occur:

$$F + H_2O \text{->} HF + OH \qquad (B.6)$$

$$SF_5 + OH \text{->} SOF_4 + HF \qquad (B.7)$$

$$WF_6 + 3H_2O \text{->} WO_3 + 6HF \qquad (B.8)$$



(B.9)

The dashed lines in reaction (B.9) indicate reactions that occur only to a limited degree during SF_6 decomposition in an arc. Whilst the reactions of (B.9) imply the formation of significant quantities of HF, this product has not been reported in large quantities following power arcing in electric power equipment. This is probably because by-product formation is mainly according to (B.2), (B.3) and (B.4). It is also possible that HF formed by the reactions of (B.9) further reacts with metal vapours to produce metal fluorides.

SF_4 is produced in significant quantities but hydrolyses rapidly (B.9) in the presence of moisture.

In the continued presence of moisture, further hydrolysis occurs, leading to:

$$SOF_2 + H_2O \text{->} SO_2 + 2HF \qquad (B.10)$$

Other reactions have been reported in the literature; additional by-products of arcing such as S_2F_{10} may be formed. However, the quantity of S_2F_{10} formed under arcing conditions is extremely low, because SF_5 radicals, produced at high temperatures, form S_2F_{10} only when cooled very rapidly, a condition not likely to apply in the arc [14].

B.1.3 SF_6 decomposition with low current discharges

Whilst operating voltage is applied to equipment containing SF_6, the possibility of low current discharges such as corona, sparking and partial discharges cannot be ruled out. However, the concentrations of by-products resulting from such discharges are likely to be very low. When SF_6 is decomposed in spark discharges, the reactions shown in dashed lines in (B.9) will predominate [14].

B.1.4 Catalytic decomposition of SF_6 (high-temperature behaviour)

SF_6 can be heated to 500 °C in quartz vessels without decomposing. Up to temperatures of about 150 °C, all commonly used materials, such as metals, glass, ceramics, rubber and polyester resin are fully resistant to SF_6. It is only at temperatures higher than 200 °C that some metals begin to have a decomposing effect on the gas, but in the case of the metals and alloys normally used, this effect is not observed to any marked degree until the temperature range of 400 °C to 600 °C is reached [13].

As the maximum operating temperatures inside electric power equipment in the absence of arcing are far below these values, no SF_6 decomposition of this kind is to be expected during operation.

B.2 Corrosion behaviour of SF_6 and its by-products

SF_6 is a completely non-reactive gas. There is no possibility therefore that corrosion will be caused directly by the SF_6 itself. However, the primary and secondary by-products, in the presence of humidity, may form corrosive electrolytes which may damage some of the material used inside the equipment.

The metals commonly used, such as aluminum, steel, copper and brass, are hardly attacked, but materials such as glass, insulation paper and the like are more vulnerable to damage, depending upon the concentration of the corrosive substances concerned. Other insulating materials, such as epoxy resin, polyester, polyethylene, polymethyleneoxide, PTFE and PVC are not significantly affected. It is important that measures be taken in the design to take account of the corrosive properties of the by-products. Corrosion can be prevented by the thorough exclusion of moisture and by using suitable materials.

B.3 Measures for the removal of by-products

Humidity and by-products inside equipment in service can be reduced to acceptable levels by adsorption. Materials such as alumina, molecular sieves or mixtures thereof are suitable for this purpose. They adsorb the gaseous reactive by-products very effectively and practically irreversibly, and at the same time ensure that the gas maintains a low dew point [7]. By-products removed from equipment should be disposed of according to local regulations. The acid compounds (sulphuric acid and hydrogen fluoride) are neutralized by means of alkaline (see 9.1.3 and Table 11).

B.4 Physiological characteristics of by-products

By-products can cause irritation of the skin, eyes and mucous membranes, such as in the respiratory tract, and in high concentrations can cause pulmonary oedema, given sufficient time of exposure. SF_6 containing by-products have an unpleasant pungent smell that is itself associated with an irritant effect. The olfactory thresholds, especially for SOF_2, SO_2 and HF, are of the same order of magnitude as the threshold limit values (TLVs). Because of these characteristics, even small quantities of gaseous by-products may give rise to unmistakable warning indications within a matter of seconds, before any risk of poisoning can arise [12].

Annex C
(informative)

Procedures for evaluating the potential effects on health from by-products of SF_6 and its mixtures

C.1 General

Annex C proposes procedures for the evaluation of the risks to health due to by-products of SF_6 and its mixtures released into the local atmosphere.

Within the by-products generated in mixtures, SF_6 by-products generally are predominant in terms of quantity and toxicity. Safety procedures related to the presence of the usual SF6 by-products shall therefore apply in applications with SF_6 mixtures.

During normal service SF_6 and its mixtures remain inside the electric power equipment and the gaseous by-products formed are neutralized by molecular sieves as well as by natural recombination processes. SF_6 and its mixtures can become present in the atmosphere because of leakage or if a gas filled compartment fails to contain the gas, in case of internal arc fault. It is necessary to differentiate clearly between leakage conditions and internal arc fault situations leading to a sudden release of SF_6, when evaluating health risk.

In case of leakage it is necessary to consider the effects of long-term exposure to the gaseous by-products of SF_6. The concentrations of these by-products in the air should remain low enough to present no threat to unprotected personnel during a normal working period of, for example, 8 h.

In case of a sudden release of SF_6 due to an internal arc fault, the emergency procedure required by the local regulation may imply a momentary exposure. By-product concentrations of higher levels than would be tolerable during for example 8 h can be tolerated if the exposure time is considerably reduced. Clearly in this case account should be taken of all possible sources of toxic emissions and this requires detailed knowledge of all of the by-products formed. In this respect a full treatment should consider contributions from metal vapour, burnt plastics, cable insulation, paint, etc., on an equal footing to those attributable to SF_6.

Clause C.2 provides procedures for calculating the risks associated with the presence of SF_6 by-products in the atmosphere due to leakage and to internal arc fault together with the principles adopted for performing those calculations.

C.2 Formation and health effects of SF_6 by-products

C.2.1 Formation of SF_6 by-products

During high power arcing in SF_6 the arc core reaches temperatures of the order of 10 000 K. At these temperatures, the molecules of the gas are completely broken down into their parent atoms, sulphur and fluorine, as reported in B.1.2. Any contaminants present, such as air or moisture, are dissociated in a similar manner. The result of this is a localized region containing only single atoms of: S, F, H, N, O and diverse ions. The heating of the electrodes and the insulating parts by the arc adds vapours of Cu, W, C and Al to this atmosphere.

After arc extinction or in regions where cooling commences, these atoms start to bind together again and reform mainly SF_6. However, chemical reactions take place with the contaminants present and in particular with moisture and oxygen, giving rise to the so-called arc by-products, as reported in Annex B. The quantities formed are directly related to the power injected into the gas volume.

In case of power arcing, gaseous by-products such as SOF_2, SO_2, HF and also CF_4, SF_4 and SO_2F_2 [15], WF_6 [12], COF_2 [16] and solid by-products such as CuF_2 [12], AlF_3 [12] are the most frequently encountered. In case of low energy electrical discharges, S_2F_{10} is also formed in extremely small quantities [17], [18], [19].

C.2.2 Effects of SF_6 by-products on health

C.2.2.1 General

If SF_6, which has been subjected to arcing or to low-energy discharges, is exhausted into the work place, then the potential health risk will depend on the concentration of each by-product in the air, and hence on the volume of the room containing the equipment. Toxicity estimations should take into account the concentration of each by-product in relation to the permissible concentrations for the appropriate exposure time. Equation (C.1) should be adopted to calculate the total risk associated with the by-products.

$$\mathrm{Risk}_{\mathrm{tot}} = \sum_i \frac{\mathrm{Concentration}\ (\text{by-product})_i}{\mathrm{Threshold}\ (\text{by-product})_i} \leq 1 \tag{C.1}$$

C.2.2.2 Health effects of arc decomposed SF_6

For multi-component mixtures, toxicologists define three general cases:

- Each component acts in a different manner, or on different target organs; the effects are hence not cumulative and each component is separately treated.
- The components act in a similar manner on the same target organs; their effect is thus cumulative and calculations will take this into account.
- One component largely outweighs the contribution of the others; the overall toxic effect should be estimated by studying the concentration of this component alone.

A survey of the majority of the work carried out over the past decades on high power arc decomposition of SF_6 [15], [20] concludes that the total health risk to personnel, due to arc decomposed SF_6, is predominantly dependent on the SOF_2 concentrations generated.

Hydrolysis of SOF_2 may occur in the presence of significant concentrations of moisture, producing SO_2 and HF, as reported in B.1.2. For the time being, no occupational exposure limit (OEL) has been defined for SOF_2. For this reason, it is assumed that hydrolysis always takes place giving HF and SO_2. The potential effect on health is then evaluated based on those gaseous by-products (see Table C.1).

C.2.2.3 Exposure duration and dilution in air

The two key notions essential to any evaluation of risk to health due to toxic substances are:

- concentration within the surrounding volume;
- time duration of exposure, leading to the selection of the appropriate exposure limit.

When a leakage situation is calculated, the OEL concentration, defined as the time weighted average (TWA) over an 8 h per day, 40 h per week exposure limit, should be used.

Under abnormal conditions, for example internal arc fault, personnel immediately leave the room of the electric power equipment and the exposure is hence momentary. Under those conditions, concentrations defined as C (ceiling exposure limit, values never to be exceeded), should be employed. When the C value is not defined, the short term exposure limit (STEL) could be adopted. The STEL refers to an average exposure of 15 min that should not be exceeded during the 8 h working time.

The occupational exposure limits (OELs) defined by the American Conference of Governmental Industrial Hygienists (ACGIH) [21] for SO_2, HF, and S_2F_{10} are given in Table C.1.

Table C.1 – OELs for SO_2, HF, and S_2F_{10}

OEL	SO_2	HF	S_2F_{10}
TWA (µl/l)	2	0,5	Not defined
STEL (µl/l)	5	Not defined	Not defined
C (µl/l)	Not defined	2	0,01
NOTE So far, no OEL has been defined for SOF_2.			

C.2.3 Quantitative estimation of gaseous by-products

C.2.3.1 General

The quantity of gaseous by-products formed during an electrical discharge is generally referred to as the energy in joule dissipated in the gas. This is defined below as production rate, r.

Experimental results are generally quoted in mol/J but another unit widely adopted is l/kJ. The conversion is based on the fact that 1 mol of ideal gas occupies 24,37 l, at 20 °C and at 100 kPa, and is given in Equation (C.2).

$$1\ \text{mol/J} = 24{,}37 \times 10^3\ \text{l/kJ} \qquad \text{(C.2)}$$

The estimation of the production rates of SOF_2 due to arcing and S_2F_{10} due to low energy discharge, both sparking and partial discharges in the gas, is given in C.2.3.2, C.2.3.3 and C.2.3.4, respectively.

C.2.3.2 Estimation of the SOF_2 production rates due to arcing

The SOF_2 production rate is experimentally determined and depends on the electrode material used and the type of discharge considered. Exothermic reactions, which occur with aluminium electrodes, seem to enhance the production rate.

The values used here have been averaged over the range of presently available data found in the literature [15] and [20].

Table C.2 – SOF_2 production rate

Electrode material	SOF_2 production rate (r)	
	mol/J	l/kJ
Cu, Fe, WCu	150×10^{-9}	$3{,}7 \times 10^{-3}$
Al	600×10^{-9}	15×10^{-3}
NOTE Aluminium electrodes are assumed only for GIS bus-bar situations.		

C.2.3.3 Estimation of the S_2F_{10} production rate due to sparking

Manoeuvring disconnectors produces sparking, which is considered as a low energy discharge. Average parameters are: 1 kV arc voltage drop, 0,25 A capacitive current and 1 s time duration, resulting in 0,25 kJ per each sparking event. However, individual sparks may reach up to 3 kA peak current for only a few tens of microseconds.

In the absence of published data, the production rate of $0{,}05 \times 10^{-9}$ mol/J (or $1{,}22 \times 10^{-6}$ l/kJ) is chosen for S_2F_{10} due to sparking, which reflects a situation at the lower end of the spark discharge range but more than 2 000 times greater than for arcs. This has been confirmed as being realistic by measurements on a real disconnector under highly accelerated operation conditions [18].

C.2.3.4 Estimation of the S_2F_{10} production rate due to partial discharges

Single components of high-voltage switchgear and controlgear or sub-assemblies in which they are contained should not exceed the maximum permissible partial discharge level of q = 5 pC (see IEC 62271-203:2011, 6.2.9.102).

At rated voltage U_r, the energy dissipated during each single partial discharge event is therefore:

$$E = q \times \frac{U_r}{\sqrt{3}} \tag{C.3}$$

which always falls into the micro to nano-joule energy range.

The S_2F_{10} production rate due to partial discharges at power frequency is not available in the literature. A value of $0{,}2 \times 10^{-9}$ mol/J (or $4{,}88 \times 10^{-6}$ l/kJ) is obtained by extrapolating the experimental results for sparking in the very low energy region.

C.2.4 Procedures for health risk evaluation

The procedures for the evaluation of the potential effects on health of gaseous by-products are based on the following assumptions:

- the equipment room is completely closed and ventilation is inoperative during the period of interest;
- the adsorbers fitted into the electric power equipment do not reduce the amount of gaseous by-products during the period of interest;
- the gas emitted uniformly mixes with the air in the room containing the electric power equipment in a short time with respect to the working day or exposure duration.

Figure C.1 describes the typical procedure to follow for the evaluation of the potential effects on health of the by-products formed during arcing (either power interruption or capacitive switching) in SF_6. It is applicable to both leakage and internal arc fault cases.

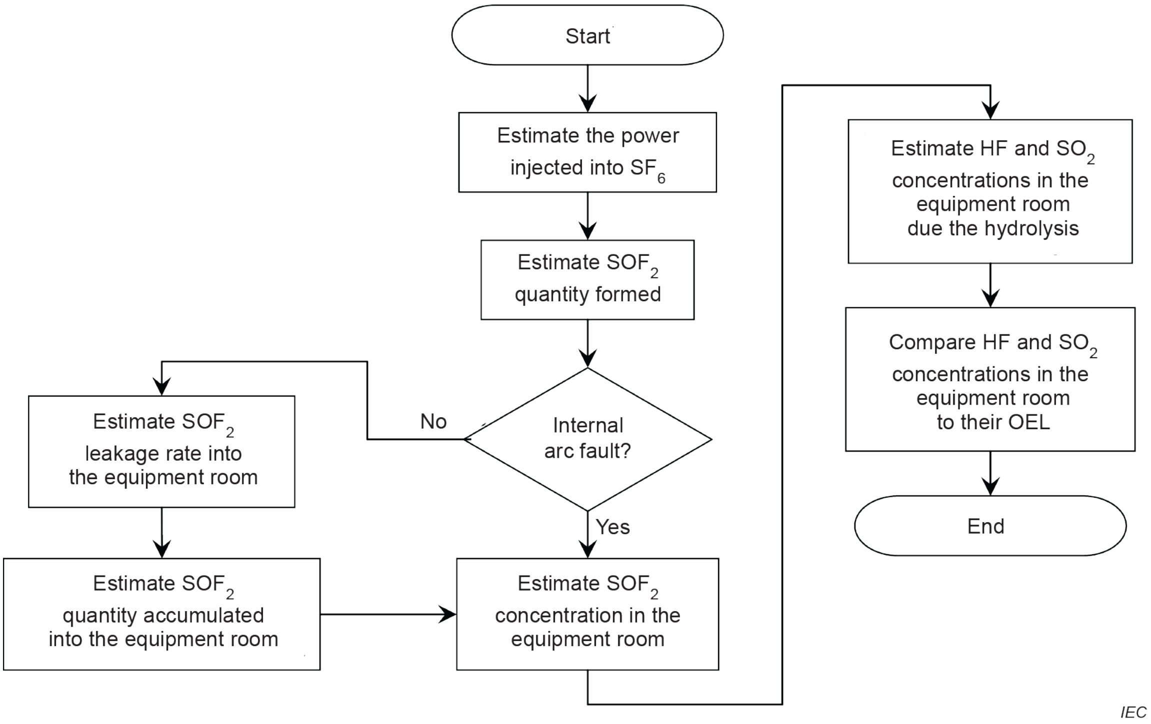


Figure C.1 – Procedure for the evaluation of the potential effects on health due to arcing

The power injected during one single event into the electric power equipment should be evaluated. Depending on the circumstances, for example power arcing, capacitive arcing, corona discharge, this could be thought as arc voltage drop times arc current times arc duration or as voltage drop times charge transferred. The cumulated power is then evaluated multiplying the power of the single event by the number of events taking place during the reference period.

The quantity of SOF_2 formed within the electric power equipment is the power itself times the SOF_2 production rate. The SOF_2 production rate is a non-linear function of the power, the quantity of SF_6, the time duration and the electrode material. Numerical values are determined by experiments (see C.2.3.2.).

In case of internal arc fault, the worst case scenario is when the rupture disk bursts and the entire quantity of SOF_2 is suddenly released into the switchgear room.

In case of leakage, only a small portion of the SOF_2 quantity formed in the electric power equipment transfers to the switchgear room and accumulates over a certain period of time. Under this condition, if $V_{\mathrm{SOF2,equipment}}$ is the SOF_2 quantity formed inside the electric power equipment in litres and $F_{\mathrm{p,rel}}$ is the leakage rate of the electric power equipment in % p.a., the SOF_2 quantity accumulated into the switchgear room during one day is:

$$V_{\mathrm{SOF_2,room}} = \frac{V_{\mathrm{SOF_2,equipment}} \times F_{\mathrm{p,rel}}}{100 \times 365} \qquad \text{(C.4)}$$

The SOF_2 concentration in the equipment room is the SOF_2 quantity in the equipment room divided by the volume of the switchgear room. For outdoor installations, the same evaluation should be performed considering the volume of a virtual equipment room.

Due to hydrolysis in the equipment room, the HF concentration in μl/l is twice the SOF_2 concentration in μl/l and the SO_2 concentration in μl/l is equal to the SOF_2 concentration in μl/l.

The potential effect on health of the by-products is then evaluated comparing the HF and the SO_2 concentrations in the equipment room with the OELs. The total risk R_{tot} should be defined as:

$$R_{tot} = \frac{\text{Concentration}(SO_2)}{\text{TLV}\ (SO_2)} + \frac{\text{Concentration}(\text{HF})}{\text{TLV}\ (\text{HF})} \leq 1 \qquad (C.5)$$

A similar procedure can be adopted for the evaluation of the potential effects on health of S_2F_{10} due to low energy electrical discharges. In this case, no hydrolysis takes place. The procedure is described in Figure C.2.

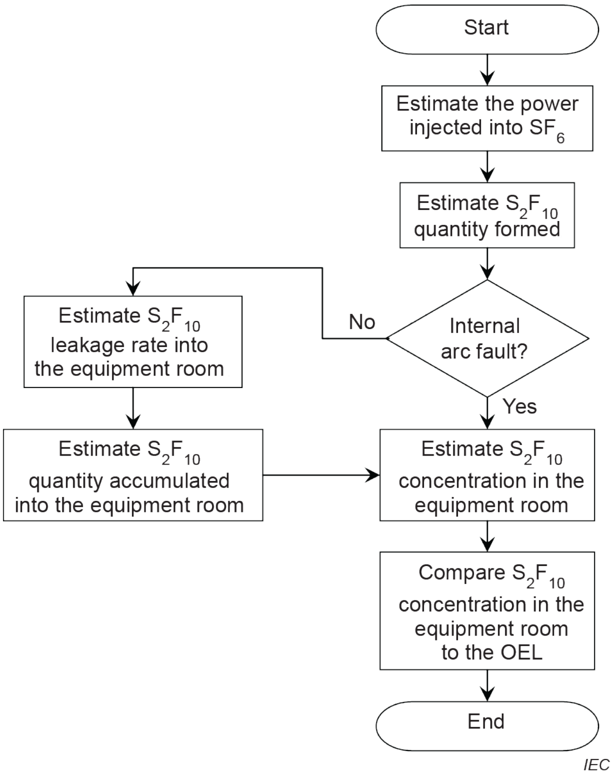


Figure C.2 – Procedure for the evaluation of the potential effects on health due to low energy discharges

C.3 Conclusion

Calculations, based on the state-of-the-art, show that, for leakage situations, there is no risk to health due to exposure to by-products. The by-products, formed by arcing and by low-energy discharges, released due to leakage from SF_6 filled electric power equipment, reach negligible concentrations in the workplace atmosphere. Therefore, there is no cause for concern and no need for precautions other than the normal ventilation practices for low-lying areas. This is still valid even in the case of abnormal leakage situations (a leakage rate, for example, of two orders of magnitude higher than the normal rate).

However, in the unlikely event of an internal fault leading to a release of SF_6, significant concentrations of by-products can occur in the equipment room. In any situation of this sort, basic safety procedures include evacuation rules which are designed to ensure that personnel are exposed to exhausted materials for the shortest possible time. Furthermore, forced ventilation and/or venting ensure the concentration levels can be reduced within minutes.

For outdoor installations, the volume of air into which the arc decomposed SF_6 escapes is large if not infinite. Prevailing winds and the high exhaust velocity also speed up dispersion.

It is thus concluded that, as long as basic safety procedures are followed, the risk specifically associated with the use of SF_6 in electric power equipment is minimized.

Annex D
(informative)

Reclaiming recommendations

D.1 General

On-site reclaiming procedures are based on the absorption of contaminants. Operational contamination should already be absorbed with the user's filter unit. Such filters are already an integral part of the company's SF_6 maintenance devices, or are available as separate filter units.

D.2 Filtering recommendations

The filters should meet the following criteria:

- they should reliably remove the mentioned contaminants;
- the filters should be of cartridge type for safe and easy disposal;
- input and output should be equipped with the same self-sealing couplings;
- changing filters should not require disassembly of any fittings, tubing, or any other connection to eliminate the possibility of leakage;
- changing the filter is recommended for each purification operation.

D.3 Transport of used SF_6 in gas cylinders and containers by road

See IEC 62271-4.

Annex E
(informative)

Cryogenic reclaiming of SF_6

E.1 General

Annex E describes the reclamation of SF_6 gas by a cryogenic technique. The cryogenic technique described below is based on an Australian system [22], [23], [24].

E.2 Applications

The principal use of the cryogenic process is to remove contaminants from used SF_6.

The process can be used to:

- aggregate the remainder of SF_6 from containers that have been used to fill electric power equipment;
- recover SF_6 from electric power equipment to a pressure of below 100 Pa without the need for a recovery compressor;
- restore used SF_6, even heavily arced or contaminated by air to the standard of technical grade SF_6;
- separate SF_6 from a mixture of SF_6 and nitrogen (N_2); all the SF_6 can be recovered and essentially none is released into the atmosphere;
- obtain CF_4 from an SF_6/CF_4 mixture.

E.3 Physical background

The process relies on the variation of the saturated vapour pressure of SF_6 and its typical contaminants with temperature (see Figure D.1).

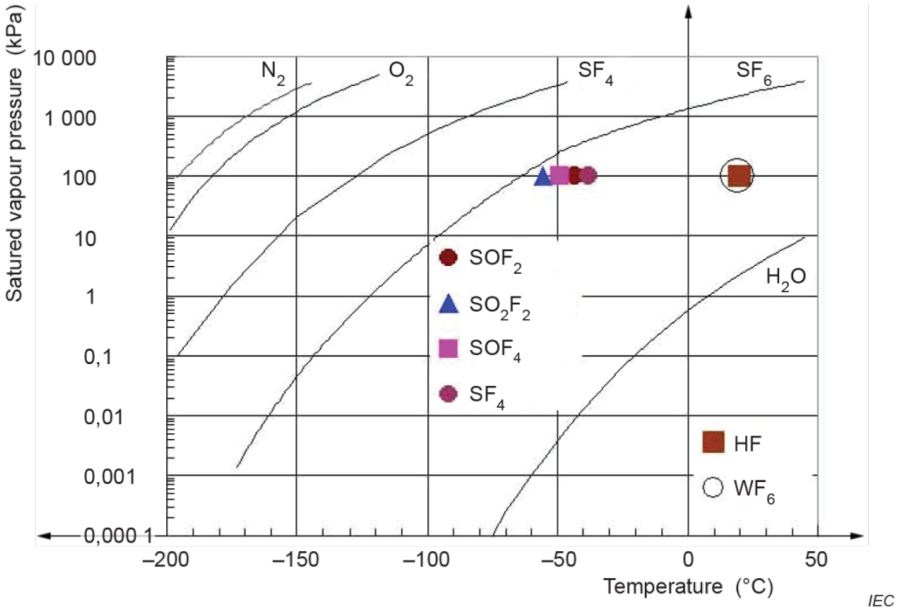


Figure D.1 – Saturated vapour pressure of various gases as a function of temperature

E.4 Cryogenic processes

SF_6 is recovered by means of evacuating and cooling a cylinder called capture cylinder. The pressure differential between the cylinder and the gas-filled compartment or the container containing the SF_6 to be processed causes the SF_6 to be transferred into the cylinder. It will liquefy or solidify due to the low temperatures achieved by liquid nitrogen thereby maintaining a pressure differential until recovery is completed.

The collection process is scalable. A number of cylinders can be connected in parallel to increase collection capacity. Smaller cylinders can be used if the masses of SF_6 involved are small. The speed of mass transfer is mainly limited by the diameter of the connecting pipes and with appropriate sized piping the practical limit given by the speed of sound can be attained.

This process can also be used to separate air from SF_6 as – provided SF_6 is cooled sufficiently – SF_6 will freeze inside the capture cylinder allowing the air to be released or evacuated by a conventional vacuum pump.

The process comprises a second step, where a second cylinder called collection cylinder is cooled by liquid nitrogen and connected to the capture cylinder which is allowed to warm up. The pressure differential between the cylinders causes SF_6 to be transferred into the collection cylinder while the contaminants remain frozen in the capture cylinder. During the SF_6 transfer, the capture cylinder warms up from approximately −150 °C to approximately −45 °C and the pressure in the capture cylinder can be obtained by closing the valve controlling the SF_6 transfer and reading the pressure gauge on top of the capture cylinder. This pressure (see Figure D.1) is representative of the highest temperature portion of SF_6 in the capture cylinder, even though temperature gradients may be present.

The upper temperature limit in the capture cylinder (i.e. approximately −45 °C) is set to limit the concentration (dew point) of moisture transferred to the collection cylinder. This upper temperature limit shall also be low enough to prevent the transfer of reactive gaseous by-products that may be present in the capture cylinder.

The capture cylinder may be partly immersed in a refrigerated bath to allow for a better temperature control.

E.5 Description of a cryogenic reclaimer

A cryogenic reclaimer comprises the following components [23]:

- gas cylinders (see IEC 62271-4) (in addition suitable for cryogenic temperatures);
- insulated dewars meeting local safety requirements and capable of withstanding −220 °C;
- vacuum pump (see IEC 62271-4);
- optional filter, used for reclaiming heavily arced gas (see IEC 62271-4);
- sampling point (see IEC 62271-4);
- valves, fittings, pressure gauges and gas piping (see IEC 62271-4), (in addition suitable for cryogenic temperatures where relevant);
- liquid nitrogen purchased as needed.

The components can be put together in different configurations according to the task at hand.

Figure D.2 is the flow chart of a cryogenic reclaimer suitable for SF_6 recovery on site.

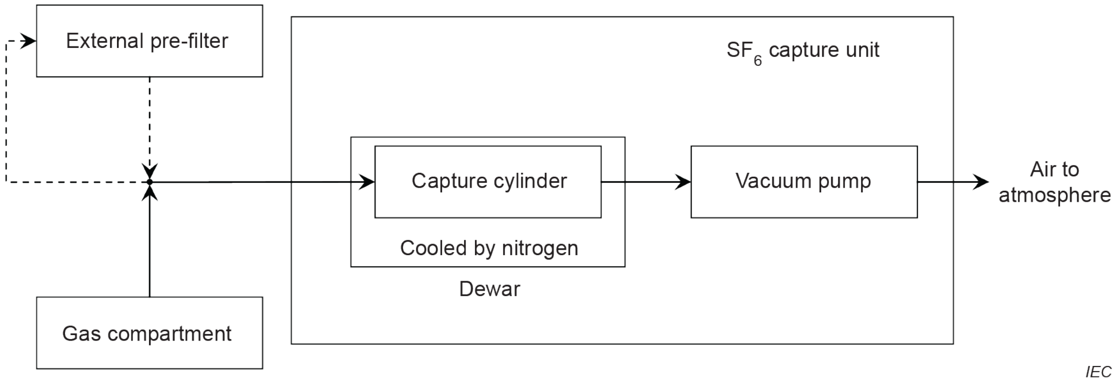


Figure D.2 – Typical cryogenic reclaimer for SF_6 recovery on site

The procedure for operation of the manual process is as follows:

- The whole reclaimer is evacuated with the vacuum pump.
- The capture cylinder is chilled with liquid nitrogen and the vacuum in the cylinder itself is maintained by the freezing of SF_6 which is continuously drawn in.
- The impure gas entering the process can be directed through a filter if it contains a significant amount of solid contaminants as in the case of heavily arced gas.
- Once the gas capture capacity has been reached, the upstream valves are closed and the downstream valves opened.
- The vacuum pump can be used to evacuate and draw off all the gaseous components above the frozen SF_6 i.e. oxygen and nitrogen.
- The downstream valves are closed and the cylinder can be removed, warmed up and transported.

Figure D.3 is the flow chart of a cryogenic reclaimer suitable for removing contaminants.

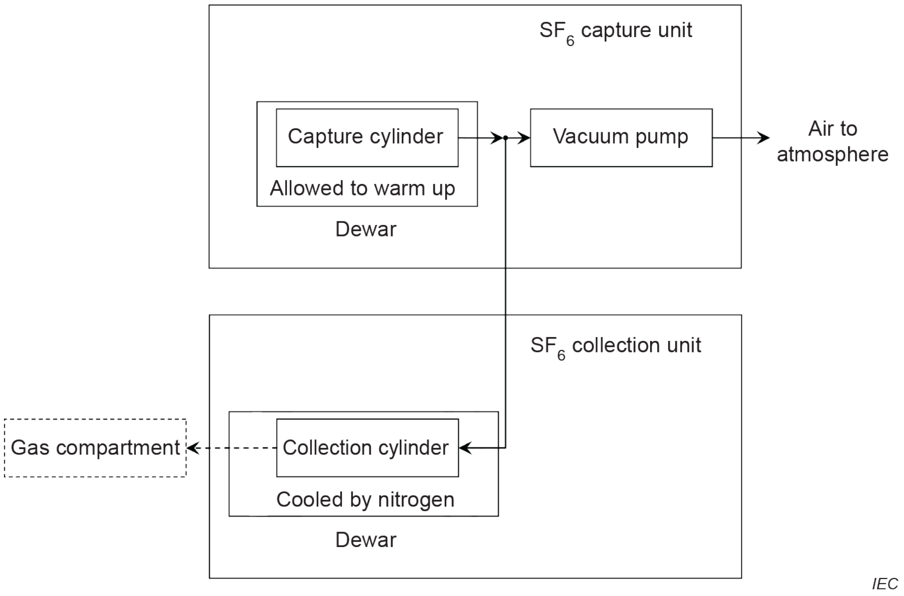


Figure D.3 – Typical cryogenic reclaimer for removing contaminants

The procedure for operation of the manual process is as follows:

- The capture cylinder and the collection cylinder are chilled with liquid nitrogen.

- The vacuum pump can be used to evacuate and draw off all the gaseous components above the frozen SF_6 i.e. oxygen and nitrogen.
- The capture cylinder is then allowed to warm up to the selected upper temperature limit (e.g. −45 °C)
- Pure SF_6 is transferred into the collection cylinder as a consequence of the temperature difference.

The capture cylinder is likely to end up containing solid contaminants from heavily arced SF_6 and requires cleaning after use.

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(*Continued from second cover*)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

This Indian Standard has been developed from Doc No.: ETD 03 (20481).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi