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Long-term performance and decomposition of Fluoronitrile-containing gas mixtures in gas-insulated systems

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SUMMARY

Essential properties of alternatives to SF₆ were investigated. To cover lifetime performance aspects, long-term tests of gas insulated switchgear/line (GIS/GIL) modules under outdoor condition and with simultaneous high voltage and current stress were conducted, applying gas mixtures of Fluoronitrile (C4-FN), CO₂ and O₂. The first test was passed successfully. Although the test setup was stressed with voltage and current without switching actions only, crystalline-shaped decomposition products were found. These were identified to be of two types, Amide and Ligand. While the Ligand remained solid under room temperature conditions, Amide sublimed. During a second test, a flashover occurred. The decomposition process and the potential influence on the GIS operating performance were subject of investigations. The formation of Amide and Ligand requires the presence of C4-FN and some (also low) gas moisture, while temperature and its distribution inside the GIS are of essential impact. The presence of some materials accelerates the process. Based on our experience the formation cannot be fully avoided and therefore some risks were identified concerning handling and safety of gas insulated equipment containing C4-FN. Dependent on temperature conditions, the detectability and quantification of decomposition products in the gas phase is limited or even impossible. The Ligand crystals move in an electric field, but PD measurement requires high sensitivity to detect them. To determine further electric properties and a potential impact on the electric performance of gas-insulated systems, additional investigations are required.

KEYWORDS

Gaseous Dielectrics, Gas-insulated System, SF₆ alternative, Fluoronitrile, C4-FN, Gas mixtures, GWP, Long-term performance, Decomposition, Crystal formation, Amide, Ligand

1 Introduction

Since several decades sulphur hexafluoride (SF₆) is state of the art as insulating and arc extinguishing medium in gas-insulated high-voltage switchgear (GIS) and gas-insulated lines (GIL). Although SF₆ offers excellent technical properties, it requires very careful handling throughout the entire life cycle of GIS/GIL, due to a high global warming potential (GWP). Therefore, intensive research and development of alternatives is being carried out to contribute to the sustainability of the energy supply with SF₆-free switchgear. For some applications, systems are already available and installed [1][2][3].

1.1 Potential alternatives to SF₆

From today's point of view, there is no gas or gas mixture available which combines all the pure technical benefits of SF₆. The most sustainable solution for insulating purpose is the use of compressed synthetic air (Clean Air, 80% nitrogen and 20% oxygen), as this approach allows completely climate-neutral and greenhouse gas-free GIS (GWP = 0) and gives further advantages in terms of gas handling and safety. On the other hand, the insulation properties are not at the same level as SF₆. The addition of fluoro-organic gases to synthetic air or other natural-origin gases (N₂/CO₂) was considered as a potential compromise to achieve approximately the electric strength of SF₆, whereby this is accompanied by an increased GWP, but significantly reduced compared to SF₆. Within government-funded projects, most promising gases were investigated to determine the essential gas properties ([4] to [10]).

1.2 Choice of the gas mixture for further investigations

Within the funded projects, the electric performance of different F-gases was subject of investigations [4][10], particularly mixtures containing C5-Fluoroketone (C5-FK: heptafluoro-3-(trifluoromethyl)-2-butanone, CF₃C(O)CF(CF₃)₂, CAS No. 756-12-7, $3M^{TM}$ NovecTM 5110), C4-Fluoronitrile (C4-FN: heptafluoro-iso-butyronitrile, C₃F₇CN, CAS No. 42532-60-5, $3M^{TM}$ NovecTM 4710) and further gases. The gases need to be mixed with a carrier gas like N₂ or CO₂ to avoid liquefaction at the required minimum temperatures of GIS/GIL. In comparison to the other mentioned alternatives, mixtures containing C4-FN provide the highest electric strength for a temperature and pressure range typically required for GIS/GIL, with the disadvantage of the highest GWP = 500-900, due to a GWP = 2.750 [11] of the pure substance, nevertheless a significant reduction compared to SF₆ (GWP = 24.300 [11]). A gas mixture of 6% C4-FN in 94% CO₂ was chosen for further investigations.

1.3 Material compatibility tests

The compatibility of GIS/GIL materials with the insulating gas and the long-term stability of the gas itself are pre-requisites for its application. As the reduced GWP of fluoro-organic gases goes along with a reduced stability of the molecules in the atmosphere, a – compared to SF_6 – reduced stability under operating conditions might be the consequence. Investigations focused on the influence of various materials on the gas stability [8]. Most of the GIL/GIS materials under test were compatible with C4-FN. Some material adaptations, e.g. related to the desiccant or certain screw alloys, were required.

2 Long-term tests

Gas-insulated systems require a very high reliability during their service life of 40 years and more. To prove this, two long-term tests under realistic in-service conditions were conducted.

2.1 Test setup and test conditions

The tests were performed in the outdoor test lab of the Siemens Energy Switchgear Factory in Berlin / Germany, to test under comparable conditions as gas-insulated systems in service. The test setup consisted of 420 kV GIS/GIL modules, forming a loop of approx. 30 meters in length (Figure 1). Two different gas mixtures were applied. While for most of the modules a gas mixture of C4-FN/CO₂ was chosen, one module was tested with C4-FN/CO₂/O₂. The GIS gas compartments (GC) were equipped with desiccant (4-5 g/l, related to the gas volume), while the GIL modules were left without desiccant, as it is typical in the case of long GIL modules. Details are given in Figure 2.



Figure 1: Test setup



Figure 2: Gas mixtures in the individual gas compartments

In GC2 and GC8 the desiccant was applied in the high-voltage conductor screening covers, while it was placed at the enclosure in GC5 and GC6. A 3\AA zeolite desiccant type was chosen, and the compatibility was pre-tested. Voltage, current, gas pressures and temperatures were monitored. Further, gas samples were taken and analysed. Two current transformers were included to enable the flow of rated current in the high-voltage conductor, simultaneous to high voltage. The current was applied in 24 hours cycles, alternating between rated current (17 hours) and no load (7 hours). The test voltage was two times the service voltage (2 x 420 kV / $\sqrt{3}$). Intermediate impulse voltage tests were performed.

2.2 Test results and observations

A list of the conducted tests is given in Table 1. In the first long-term test, the test setup was stressed with 485 kV AC voltage for 3.160 hours, without any breakdown, neither during AC voltage application nor during impulse voltage tests, conducted as oscillating impulse voltages. Due to the current cycles and outdoor climatic conditions, several temperature cycles were achieved during the test. All temperature values remained within the limits of IEC 62271-1. More details on the tests and the test results are given in [12]. Exemplary results of gas quality checks are given in Table 2.

Test type	Tests and ratings			
Initial test	650 kV AC, 1 min // 1050 kV SI, 15/0 (80%+100%) // 1425 kV LI, 15/0 (80%+100%)			
Long-term test	485 kV = 2 p.u. // 4000 A = 1 p.u. in cycles // 1.873 hours			
Intermediate test	650 kV AC, 1 min // 1050 kV SI, 15/0 (80%+100%) // 1425 kV LI, 15/0 (80%+100%)			
Long-term test	485 kV = 2 p.u. // 4000 A = 1 p.u. in cycles // 1.287 hours			
Final test of long-term test 1	650 kV AC, 1 min // 1050 kV SI, 15/0 (80%+100%) // 1425 kV LI, 15/0 (80%) ¹			
Visual inspection	Test setup after inspection closed w/o desiccant and evacuated, for some weeks w/o voltage and current, under outdoor condition			
HV test with crystals	up to 485 kV AC, \approx 15 min			
Cleaning	Crystals removed, test setup cleaned and refilled with gas			
Long-term test 2	485 kV = 2 p.u. // 4000 A = 1 p.u. in cycles			
	1.500 hours at 485 kV // 3.933 hours at 400 kV $^{\prime}$ // 50 h at 485 kV (flashover in GC4)			

¹ Limitation due to HV test system issues

After the final test of long-term test 1, the insulating gas was removed, and the gas compartments were opened for inspection. At several locations inside the test setup two different types of crystal-shaped solid decomposition products were found (Table 3). An example is shown in Figure 3 (GC8).

Table 2: Gas quality (gas analyzer type Wika GA11)

C4-FN ratio (%vol) / frost point (humidity (calculated))								
GC	start of test	intermediate	end of test					
GC2	6.1% / -60 °C	6.2% / -49 °C	6.1% / -52 °C					
	(1 ppm _v)	(7 ppm _v)	(5 ppm _v)					
GC4	5.7% / -28 °C	5.8% / -23 °C	5.6% / -30 °C					
	(78 ppm _v)	(129 ppm _v)	(63 ppm _v)					
GC8	5.7% / -60 °C	5.8% / -48 °C	5.7% / -56 °C					
	(1 ppm _v)	(8 ppm _v)	(3 ppm _v)					

Table 3: Formation of	crystal-shaped	decomposition products
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	GC2	GC3	GC4	GC5	GC6	GC7	GC8 ^a	
Desiccant	yes	no	no	yes	yes	no	yes	
"Amide" Crystals			Х		X ^b			
"Ligand" Crystals	Х			Х			Х	
^a – gas mixture C4-FN/CO2/O2 ^b – purple dust at the enclosure								

More information on the nature of the two crystal types "Amide" and "Ligand" is given in chapter 3. In the next step, the test setup was closed and evacuated, but remained without gas and desiccant for some weeks. After this period, the gas compartments were opened again. It was found that accelerated crystal formation took place at several locations, as shown in Figure 4 (left). The test setup was refilled with the gas and high voltage up to 485 kV AC was applied, observing the crystals in GC2 during test through a window. The crystals moved and broke into smaller needles (Figure 4).



Figure 3: Crystalline decomposition products (example: GC8)

Figure 4: Crystal growth in GC2 after gas removal and without desiccant, before (left) and after (right) high voltage application

The test setup was cleaned, and the crystals were removed to continue the test. In this second long-term test, a flashover occurred after 5.483 hours at the surface of an (previously cleaned) insulator in GC4 (refer to Table 1). No crystals were found, but the inspection was done at >20 °C ambient temperature and the location was identical to that where Amide type crystals were observed in the first long-term test (Figure 5). So, the correlation is not fully confirmed, but indications of a dependency are given.





Figure 5: "Amide" crystals in GC4 (left) in the first long term test and flashover in the second long term test (right)

3 Decomposition of C4-FN

Despite the absence of partial discharges or gas discharges, solid decomposition products were formed in the long-term test. This formation was reproducible [12]. The investigations resulted in the conclusion, that the presence of C4-FN and some gas moisture is required for the decomposition. Two different types were identified, "Amide" (heptafluoro-iso-butyramide $C_3F_7C(O)NH_2$) and "Ligand" (tetradecafluoro-N-acylamidine, $C_3F_7-C(O)-N=C(NH_2)-C_3F_7$). Violet crystals were also found as decomposition product of C4-FN, and they were identified as square planar Cu(II) complex [13]. Details regarding the formation are given in literature [13] and the reaction path is illustrated in Figure 6.



Figure 6: Reaction path from C4-FN (1) to the copper complex 4 via Amide 2 and Ligand 3

3.1 Literature review

The formation of similar decomposition products is described in a few literature references in the context of laboratory experiments [13][14][15][16] as well as in conjunction with long-term tests [17]. Kessler et al. studied the compatibility of C4-FN with Al, Ag and Cu metal surfaces [13]. In lab-scale autoclave experiments they found the formation of violet crystals in presence of C4-FN, Cu, O₂ and moisture at elevated temperature (Figure 7). The proposed reaction mechanism for the observed multi-step gas-phase reaction is given in Figure 6. As Cu is only relevant in the last synthetic step the formation of "Amide" and "Ligand" can also occur in absence of copper.

Misaka et al. investigated decomposition during arc discharges in C4-FN gas mixtures [14]. Beside discharge experiments and GIS material compatibility tests they also examined the longterm impact of moisture on the gas mixture. They also identified an Amide molecule as the hydrolysis product of C4-FN and water and a "dimer" molecule, interpretated as the conden-



Figure 7: Crystals in the autoclave and microscopic view

sation adduct of two Amide molecules. This corresponds exactly to the "Ligand" found in our tests aside from our interpretation that this is an addition product between an Amide and a C4-FN molecule.

This mechanism is also proposed by Xiaojuan et al. [15] who carried out ab initio calculations of gas phase reactions of C4-FN with water. A tetradecafluoro-N-acylamidine (there designated as formamido imine), similar to the "Ligand" molecule, is postulated as one stable outcome in their study.

The hydrolysis reaction of C4-FN in the presence of moisture to Amide was confirmed in recent literature [16], dependent on temperature. An impact of distinct desiccants on the decomposition was observed. A desiccant was identified without decomposition under the conditions of test (temperature 70-120 °C). The authors stated that the control of humidity inside the equipment is important to ensure gas stability and that comparable humidity limits than in SF₆ switchgear may be applied.

After a more than two years lasting dielectric long-term test of a voltage transformer (VT) under outdoor condition, the formation of gaseous and solid decomposition products was observed [17]. Probably because of hydrolysis Amide was formed and crystals were present.

3.2 Observations in further experiments

The literature review confirmed the formation of crystalline-shaped decomposition products in other investigations also, but further tests were required and conducted. The formation was observed also in conjunction with other tests and test setups. Some examples are given in the next sections.

3.2.1 Gas handling device and gas equipment

During gas handling of the long-term test, an outage of the gas handling device took place under outdoor condition (<10 °C). The subsequent investigation revealed that this outage was due to crystals mechanically blocking a pump, behind the filtering unit.

3.2.2 Autoclave tests with desiccants

As mentioned in section 1.3 and [16], desiccants may be of impact on the decomposition of C4-FN, at least for the "Ligand" formation. To investigate this, autoclave tests were carried out with different desiccant types in granular and powder shape. In the first instance, a zeolite desiccant was stored for 31 days in a dry mixture of 5% C4-FN, 5% O₂, and 90% CO₂. After extensive pre-testing, a temperature of 60 °C was chosen as a compromise between an appropriate reaction rate (which increases with higher temperatures) and adsorption rate (which decreases with increasing temperature). After cooling down the setup with a slow cooling rate, the desiccant was found to be agglomerated and interspersed with crystals (Figure 8, left), identified as "Ligand" based on FTIR and TD-GC-MS. Furthermore, thermal analysis using DSC and TGA was carried out. By TGA the sublimation onset temperature of the "Ligand" crystals was found to be at around 70 °C using open crucibles under normal pressure conditions. However, the sublimation point determined by DSC was in a range of 135 °C to 138 °C, which might be caused by internal pressure effects as hermetically closed crucibles were used.

Following that findings, further tests were carried out using a dry mixture of 5% C4-FN and synthetic air – free of CO₂. Testing was done at 60 °C again; however, the cooling rate after finishing the test was furtherly reduced, because an impact on crystal growth by slow cooling was presumed. Under these conditions large needle-shaped whiskers with a length of several centimetres were found to be constituted both, in the desiccant itself and on the walls of the autoclave (Figure 8, middle and right). It is well known that the growth of whiskers out of the gas phase increases with slow cooling rates. Apparently, this is also the case for gases, which contain decomposition products of C4-FN. As slow temperature changes and gradients can occur in switchgear installations due to ambient and weather conditions, it is strongly recommended that this effect should be carefully considered in practice.



Figure 8: Agglomerated desiccant with crystals after autoclave test in a dry mixture 5 % C4-FN, 5 % O₂, and 90 % CO₂ (left) Large needle-shaped whiskers after autoclave test in a dry mixture of 5% C4-FN and synthetic air (middle + right)

3.3 Evaluation of test results

It was found that the presence of some materials influences the decomposition process. First, gas moisture is essential. Even if desiccants are used the gas will still contain a few ppm of water, potentially being sufficient for the hydrolysis of C4-FN under specific conditions. Under indoor condition crystal formation was not observed, but under outdoor installation, even in gas compartments of low moisture content in the gas (Table 2). An unequal temperature distribution inside the GIS/GIL under specific conditions (e.g. during transition at current cycle start with fast increasing temperature at the inner conductor, while the enclosure is still cold) in conjunction with the moisture distribution provides good conditions for a crystal nucleus formation in the "cold edges". In our test this effect was unintentionally accelerated due to the desiccant position in the "hot" current-carrying high-voltage conductor. Nevertheless, minor formation was observed also in gas compartments with desiccant at the common enclosure position. The accelerated crystal growth during absence of gas was presumably due to remaining solid Amide in the GIS/GIL, subliming and resubliming dependent on the temperature and humidity conditions. For the case of the agglomerated crystals in the GIL gas compartment with subsequent electric breakdown in the second long-term test, minor local moisture ingress at a sealing may have been of importance for the crystal formation. Second, the presence of some other materials influences the process. Purple decomposition products are observed when copper is present [13]. In our long-term test, a purple-coloured dust was found at the enclosure of GC6. Further, the desiccant potentially accelerated the Amide formation and may also contribute to the subsequent reaction of the Amide forming the Ligand molecule. The adsorption of water and/or CO_2 at the desiccant surface may be of impact on this process. Nevertheless, crystal formation was observed in a gas compartment without desiccant also (Amide).

4 Risk assessment

4.1 Safety and handling aspects

Today, the properties of both decomposition products Amide and Ligand concerning health are not well known. OEL (occupational exposure limit) and LC50 (median lethal concentration) values are currently not available. It might be relevant that the Amide readily dissolves in water and, therefore, more easily can enter the body than C4-FN. According to our test experience, especially in the case of long-extended GIS/GIL modules, the decomposition products were not removable by cleaning, so they may remain in the gas compartments despite common cleaning processes.

4.1.1 Amide type

During the tests it was found that the Amide type sublimed at approximately 20 °C under normal pressure conditions, so Amide crystals were observed immediately after opening of the outdoor test setup on a cool morning, while they sublimed over the day at higher temperature (Figure 9).



Figure 9: Amide type crystals in GC4 before (left) and after (right) sublimation

Similar behaviour was observed in other tests also. When the test setup would be opened at higher ambient temperature, the crystals would have not been visible, however with vapours present in the air anyway. The handling requires special care, as the vapour of the Amide type is classified as acute toxic (class 4) and may cause damage to the nervous system.

Respirator masks and protective suits were applied during test setup inspection to avoid inhalation or skin contact. Special care is recommended when working on open gas-insulated equipment containing C4-FN, also when used for pure insulation purpose only.

4.1.2 Ligand type

On the one hand, Ligand type crystals remained stable under ambient conditions at room temperature. In different experiments it sublimed at 70 °C up to >170 °C. The enclosure temperature in the long-term test always remained below these values, so the Ligand type crystals did not evaporate. Extensive tests using TGA and DSC had been carried out on the crystals described in clause 3.2.2.

On the other hand, dependent on temperatures and holding times within DSC, some amount of the "Ligand" crystals obviously transforms to another physical or chemical state. There is some evidence, that different crystals with melting or sublimation temperatures close to room temperature were constituted. The processes are not well understood and still under consideration. As the properties concerning health are unknown, it is recommended to handle them with care and as the Amide type.

4.2 Detectability

4.2.1 Gas analysis

Generally, the decomposition products in gaseous state can be detected with Fourier-Transform Infrared Spectroscopy (FTIR) and Gas chromatography coupled with mass spectroscopy (GC/MS). Limitations are given due the vapour pressure properties of Amide and Ligand, so in several cases both substances are in solid state only and cannot be quantified or even detected in the gas phase. Details are given in [12]. Small gaseous decomposition products of C4-FN (CF₄, C₃F₆, ...) can be separated on a GC column and detected via MS. However, liquid or solid decomposition products need to be evaporated in the GC injector and are usually injected as liquid solution in organic solvents. In presence of Ligand corresponding signals were found in the mass spectra after gas-phase GC/MS measurements. Due to the limited vapour pressure and its decomposition in the injector or on the GC column a quantification of the Ligand in the gas phase is not possible. Although the Amide has a higher vapour pressure (fully sublimes at room temperature) and can be well detected via GC/MS, quantitative measurements remain challenging since the amount of gaseous Amide depends on its vapour pressure and the latter has a temperature dependency. Further details regarding the GC/MS measurements are given in [8] and [12]. Amide and Ligand can be identified by ATR IR spectroscopy showing the following main features: Heptafluoro-iso-butyramide ("Amide"), IR (ATR): 3350 cm-1 (s, NH2, asymmetrical stretch), 3220 cm-1 (s, NH2, symmetrical stretch), 1660 - 1640 cm-1 (s, overlapping C=O stretch and N-H bend), 1520 cm-1 (s, C-N stretch). Tetradecafluoro-N-acylamidine ("Ligand"), IR(ATR): 3430 cm-1 (s, br), 1700 cm-1 (vs).

4.2.2 Partial discharge (PD) measurement

Experiments with Ligand type crystals in an electric field were performed [12] to investigate the movement and detectability with PD measurements, in addition to the experiment within the long-term test (section 2.2). Movement and orientation of crystals in the electric field were observed, but PD of only minor intensity and amplitude were measured. To enable the detection of Ligand type crystals, the movement needs to be strong, and the PD measurement system must be very sensitive.

4.2.3 Visual inspection

Visual inspection of gas-insulated systems is a potential measure to identify crystalline decomposition products inside. This measure may be limited due to the lack of observation windows and the typical locally concentrated formation of Amide and Ligand. Further, Amide type crystals may temporarily disappear due to sublimation, dependent on individual temperature and gas pressure conditions.

4.3 Impact on the electric properties and the system integrity

Reliability and integrity of GIS/GIL in service are of high relevance. Transient overvoltages may stress the systems, and they shall remain without decomposition during its lifetime. The formation of agglomerated and large-sized crystals was observed, reducing the C4-FN content in the gas phase. This reduction was not significant under our test conditions, but this may be different in the long-term range. The electric properties of the crystals itself are of high relevance. Two effects were observed: First, long-extended crystals broke down into smaller pieces during high-voltage application. Obviously, the crystals are of polar nature, moving in an electric field. Second, a flashover occurred at a location where Amide type crystals were found in a previous test (section 2.2). Since comparable conditions have been present in both tests a correlation can be assumed, but slight differences in formation / sublimation may have been of impact on the individual test result. In additional tests [12] up to 400 kV AC voltage no breakdown occurred in a 245 kV GIS, while PD of minor intensity and amplitude were measured. To determine further electric properties (e.g. permittivity) and a potential impact on the electric performance of GIS/GIL (e.g. at impulse voltage), additional investigations are recommended.

4.4 Potential measures to reduce gas decomposition

In our tests, the formation of solid decomposition products in outdoor-installed GIS/GIL could not be fully avoided, despite dry gas with water content below typical limits for SF₆ (200 ppm_v), so the C4-FN application might be preferably indoor. For outdoor installation, the most effective measure is to avoid any moisture in the gas, but this needs to be ensured for the whole lifetime of the gas-insulated system and is improbable in typical installations, due to remaining water molecules in solid insulators as well as at enclosure surfaces, and permeation through sealings. The application of desiccant reduces the gas humidity but may support the hydrolysis of C4-FN and the conversion from Amide to Ligand, dependent on the individual conditions. Full removal of solid Amide (temperature <20 °C) in the GIS/GIL by common cleaning procedures was not possible in our case. Special filters for Amide removal may be a solution, but a long-term reduction of the C4-FN content might be the consequence.

5 Conclusions

Long-term tests of common GIS/GIL modules with gas mixtures of C4-FN, CO₂ and O₂ were conducted under simultaneous application of high voltage and current. The first long-term test was passed successfully, in terms of dielectric and thermal performance. Afterwards crystalline decomposition products were found at several locations. These were identified as Amide and Ligand, resulting from hydrolysis of C4-FN. While the Ligand remained stable, Amide sublimed at room temperature. During a second test, a flashover occurred at the former location of an Amide agglomeration. Further experiments were conducted, and the formation process as well as physical properties of the crystals were determined. The formation requires the presence of C4-FN and some (also very low) gas moisture, while temperature and its distribution are of essential impact. Some materials accelerate the process.

Due to evaporation of the Amide at ≈ 20 °C and its classification as toxic, special care in handling of installations is recommended, also when no crystals are visible. Dependent on temperature conditions, the detectability and quantification of the decomposition products in the gas phase is limited or even impossible. The Ligand crystals move in an electric field, but PD measurement requires high sensitivity to detect them. To determine further electric properties and a potential impact on the electric performance of gas-insulated systems, additional investigations are required.

At the beginning of the government-funded projects in 2016, C4-FN was considered as a potential compromise between technical performance and environmental aspects. In 2022 the European Commission made a legislative proposal to restrict or even ban the use of fluorinated gases in switchgear, including mixtures based on C4-FN [18]. In 2023, some countries proposed the future ban of PFAS substances, including C4-FN, within the European Union [19]. Further, the current single-source [20] manufacturer of C4-FN for switchgear purposes announced to stop the production in 2025. Due to these constraints, also in other countries, and the long service life of gas-insulated systems, gas mixtures containing C4-FN might not be a consistent solution for the future.

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