Study Committee B3

Substations and Electrical Installations

11081_2022

End-of-life procedures and gas reclamation of SF_6 alternative gas mixtures

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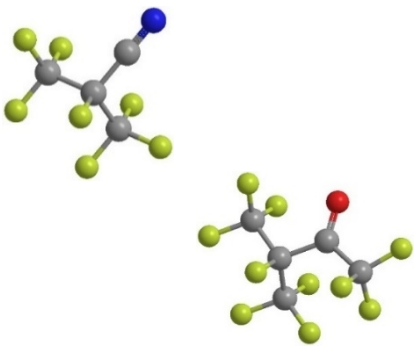
Motivation

- The compounds 2,3,3,3-tetrafluoro-2-(trifluoro-methyl)-propanenitrile (C4-FN) and 1,1,1,3,4,4,4-heptafluoro-3-(trifluoromethyl)-butan-2-one (C5-FK), used in gas mixtures are widespread alternatives to SF_6 in high and medium voltage equipment.
- Despite having significantly lower GWPs, gas mixtures with C4-FN and C5-FK should not be intentionally released into the atmosphere. Discharges during operations may produce toxic or environmentally harmful decomposition products or both.
- For ecological and economic reasons they should be handled using closed-loop processes in order to reclaim the used material.



Objects of investigation

- The used gas mixtures containing C4-FN with only minor impurities of decomposition products (carbon monoxide < 100 ppm) have mainly to be separated from oxygen, nitrogen and carbon dioxide.
- In the present example approx. 100 kg of gas mixture with C4-FN were reclaimed.

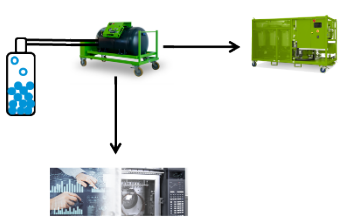
Cryogenic distillation

- The "cryogenic distillation" is a process of separation of gas mixtures, using simple distillation, at high pressure and low temperature. In conventional distillation, the gas mixtures are separated based on the difference in their boiling points.
- Due to the large difference in boiling points and volatility of C4-FN and C5-FK compared to the other main components in the mixture (CO_2, N_2, O_2) the cryogenic distillation is advantageous.

Compound	CAS no.	Molar mass [g/mol]	Boiling point [°C]
C4-FN	42532-60-5	195.04	−4.7 [1]
C5-FK	756-12-7	266.04	+26.9 [2]
N_2	7727-37-9	28.01	−195.79
CO_2	124-38-9	44.01	−78.50
O_2	7782-44-7	32.00	−183.0
SF_6	2551-62-4	146.06	−50.8
CO	630-08-0	28.01	−191.5

Test setup & test results

- Since complete separation is most efficient from the gaseous phase, the gas mixture was first homogenised into gaseous form. From this state, initial analyses are carried out at an early stage in order to detect impurities that cannot be processed (SF_6, C5-FK in C4-FN mixtures and vice versa).
- Following the initial analysis, moisture is removed from the gas mixtures via dry filters. This is necessary to prevent condensation and freezing in the buffer vessel.
- After drying, the C4-FN is then separated from the remaining gases in a multi-stage process with different temperature levels.
- After the complete separation of the gas mixtures, about 16 kg of C4-FN were reclaimed and analysed. This corresponds to a purification conversion of approx. 97 %.



[1] 3M, Electronics Materials Solutions Division, Technical Data Sheet: 3M™ Novec™ 4710 Insulating Gas, 3M Company, https://multimedia.3m.com/mws/media/1132124O/3m-novec-4710-insulating-gas.pdf

[2] 3M, Electronics Materials Solutions Division, Technical Data Sheet: 3M™ Novec™ 5110 Insulating Gas, 3M Company, https://multimedia.3m.com/mws/media/1132123O/3m-novec-5110-insulating-gas.pdf

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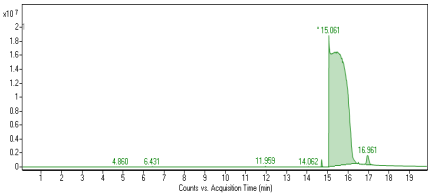
Quality analysis of reclaimed C4-FN

- For the final quality analysis of the reclaimed compounds, an overview of all by-products contained in the gas is required.
- Unlike SF_6, where the main by-products after reclamation to 99.9 mol-% are tetrafluoromethane (CF_4) and nitrogen, the by-products after separating C4-FN or C5-FK mixtures are typically nitrogen, carbon dioxide and 1,1,1,2,3,3,3-heptafluoropropane (C_3HF_7).
- The final quality of the reclaimed C4-FN/C5-FK analysed in laboratory also determined the sum of the by-products. The necessary analytical method is chromatography (GC) in conjunction with various detectors like mass spectrometry or thermal conductivity detectors (TCD).



TIC-Chromatogram of the liquified phase of reclaimed C4-FN.

Purity of reclaimed gas

- The sum of all by-products of the reclaimed C4-FN is less than 1000 ppm which is equal to a quality of 99.9 mol-%.

Compound	Retention time	CAS no.	Concentration liquid phase [ppm]
N_2	4.860	7727-37-9	< 100
CO_2	6.431	124-38-9	160
C_3F_6	11.959	116-15-4	55
C_4F_8	14.062	360-89-4	14
C_3HF_7	14.709	431-89-0	430
C4-FN	15.061	42532-60-5	balance
n-C4-FN	16.961	375-00-8	143
Total			~ 902

- Since the purity of the reclaimed gases depends on the quality of the gases put in, the ratio of by-products in the reclaimed gases may vary.
- Considering the results of all reclamations carried out to date, a quality of over 99 mol-%, as in the example, was consistently achieved in the liquid phase. The major deviations were the concentration of the residual carrier gases and the main by-products.

Conclusion

- Since SF_6 alternative gases are spreading more and more, the demand for a complete end-of-life solution is increasing.
- The latest findings stipulate that technical solutions for reclaiming gas mixtures with C4-FN or C5-FK are already available.
- The quality of reclaimed fluoronitriles and fluoroketones is similar to new C4-FN/C5-FK and is therefore suitable for the use in gas mixtures.
- The state-of-art reclamation is the key factor for a completely closed handling of alternative gases based on C4-FN/C5-FK. This closed-loop process ranges from mixing to handling in switchgear with on-site analysis to reclamation without additional emissions through combustion at the end of the gas life.