

## Research on Rapid Analysis for Purity of SF<sub>6</sub> via Ion Mobility Spectrometry

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**Abstract.** The detection of partial discharge and analysis of sulfur hexafluoride (SF<sub>6</sub>) purity in gas-insulated switchgears (GIS) is important for diagnosis and operating state assessment of the electric device. This paper introduced a method for rapid purity detection of SF<sub>6</sub> based on one-way ion mobility spectrometry. The influence of flow rate, temperature and purity of SF<sub>6</sub> on the drift time of SF<sub>6</sub> were investigated in details. The drift time of SF<sub>6</sub> was found to increase with the rising of impurity in SF<sub>6</sub>. A time of flight mass spectrometer was used for monitoring of decomposition products in SF<sub>6</sub> from partial discharge. SO<sub>2</sub>, SOF<sub>2</sub>, SO<sub>2</sub>F<sub>2</sub>, SOF<sub>4</sub> and SiF<sub>4</sub> were found to be the main discharging decomposition products, which lead to the decrease of purity of SF<sub>6</sub>.

### Introduction

Sulfur hexafluoride (SF<sub>6</sub>) has been widely used in gas-insulated switchgears for its excellent insulating and arc-suppression properties [1]. However, due to the presence of partial discharge, SF<sub>6</sub> decomposes into various by-products according to discharge scenarios, which lead to a significant decrease in its insulating performance. Therefore, the monitoring of SF<sub>6</sub> purity is very important to guarantee the operating state assessment of power equipment [2,3]. Presently, the main method used for purity detection was gas chromatography (GC) based on the measurement of decomposition products in SF<sub>6</sub> from partial discharging [1]. However, owing to its size and weight as well as the complexity in separation process, GC can be rarely applied for on-site purity detection of SF<sub>6</sub>. Recently, on-line monitoring method and instrument has been developed for rapid purity analysis of SF<sub>6</sub> such as carbon nanotube gas sensor [4], ion mobility spectrometry, and so on [5,6].

Ion mobility spectrometry (IMS) has been recognized as a promising technique for its attractive features such as atmospheric pressure operation, high sensitive, short response time, and low cost. It has been widely employed for on-site detection of trace amount of chemical warfare agents (CWA), explosives, narcotics and toxic industrial chemicals [7-9]. Based on IMS, Baumbach investigated the effect of purity of SF<sub>6</sub> on the drift time and discovered that the drift time increased with the decrease of SF<sub>6</sub> purity in gas insulated switchgears [10,11], and gave a method to detect the quality of SF<sub>6</sub> based on the variations of its drift time [12,13]. However, rather scarce work has been performed to investigate the reasons why the drift time of SF<sub>6</sub> changed with the variations of the SF<sub>6</sub> purity and what factors that could contribute to the variation of drift time.

This paper introduced a method for the rapid detection of the SF<sub>6</sub> purity via one-way IMS. The influence of flow rate, temperature and purity of SF<sub>6</sub> on the drift time of SF<sub>6</sub> were investigated in details. Moreover, the decomposition products in SF<sub>6</sub> due to partial discharge were monitored using a time of flight mass spectrometry.

### Experimental

The schematic diagram of IMS working in negative mode was shown in Fig. 1, which has been described previously [14-19]. Therefore, only the main feature of the apparatus was outlined in Table 1. The IMS tube was constructed by a series of stacked stainless steel guard rings separated by teflon insulating rings. The reaction region was 22 mm long with inner diameter 14 mm and the drift region was 120 mm long with inner diameter 15 mm. The guard rings were located 5.5 mm apart from each

Table 1 Parameters for the ion mobility spectrometer

Parameters	Values
$\text{Ni}^{63}$ source(mCi)	15
Electric field(V/cm)	300
Drift tube(cm)	12
Ionization region(mm)	20
Inner diameter of drift tube(mm)	15
Sampling frequency(Hz)	25
Injection pulse width (ms)	0.2

other and connected by a series of 1 M $\Omega$  resistors to form the electric gradient. The ionization source is radioactive  $^{63}\text{Ni}$  source with radioactivity of 15 mCi. The ion gate was a Bradbury-Nielson type with an injection pulse width of 200  $\mu\text{s}$  under current conditions. Ions were separated in the drift region and detected by the faraday plate at the end of the drift tube. The ion current received by the Faraday plate was amplified with a gain of  $10^9$  V/A, and then fed to a computer via an A/D converter. The spectra was recorded when the drift time stabilized. The  $\text{SF}_6$  gas samples were conducted into the IMS through the inlet port near the faraday plate, and were extracted through the outlet port near the ionization source. The gas flow rates were controlled by mass flow controllers.

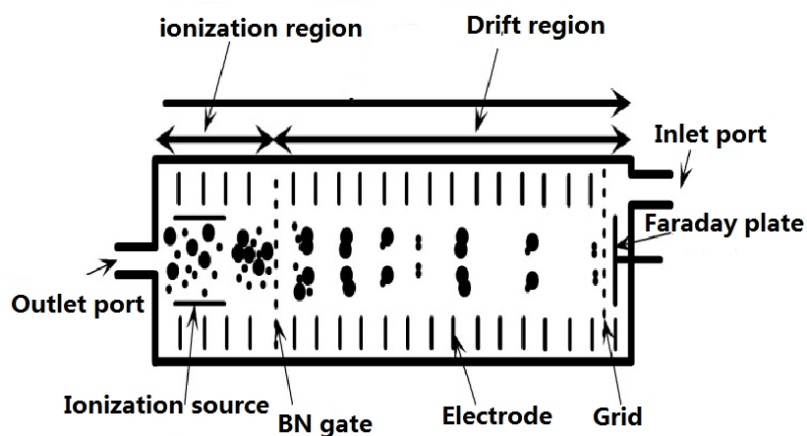


Fig. 1. Schematic diagram of ion mobility spectrometer

## Results and discussion

### The effect of gas flow on the drift time of $\text{SF}_6$

The variation of drift time of  $\text{SF}_6$  as a function of flow rate at room temperature was shown in Fig.2. It is seen that the drift time decreased from 27.8 ms at 60 ml/min to 25.2 ms at 600 ml/min, while the full width at half maximum increased from 1 ms to 2.5 ms. It can be due to the fact that simple ions clusters with similar mobility were formed under a lower flow rate, while mixture of ion clusters with different mobility were formed in the case of a higher flow rate. Therefore, a higher resolution and sensitivity could be obtained at a lower flow rate and a flow rate of 60 ml/min was selected for further study.

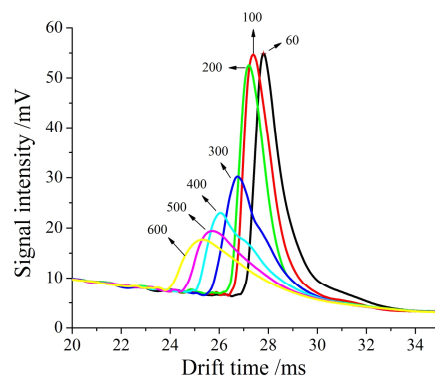


Fig. 2. Influence of flow rate on the drift time of SF<sub>6</sub> samples

### The effect of temperature on the drift time of SF<sub>6</sub>

The effect of temperature of the drift tube on the drift time of SF<sub>6</sub> was illustrated in Fig. 3. It is seen that the drift time of SF<sub>6</sub> decreased from 24 ms at 45 °C to 17.8 ms at 100 °C, which could due to the increase of velocity of the ions. The velocity of the ions  $V_d$  is proportional to their mobility  $K$ , which can be described by the following function.  $V_d = KE$ , where  $E$  is the strength of the electric field. The mobility of the ions  $K$  is proportional to the reduced mobility  $K_0$  as  $K = K_0 (T / 273)$ , where  $T$  is the temperature of the drift tube. Therefore, the velocity of the ions increased while the drift time decreased with the increasing temperature, and a temperature of 45 °C was selected for further study.

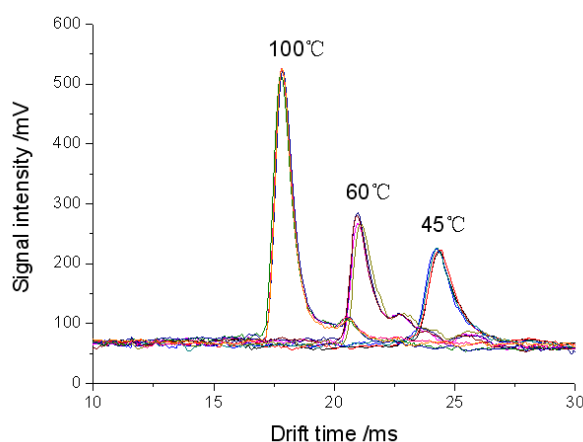


Fig. 3. Influence of temperature on the drift time of SF<sub>6</sub> samples

### The Effect of SF<sub>6</sub> purity on the drift time of SF<sub>6</sub>

The effect of SF<sub>6</sub> purity on the drift time of SF<sub>6</sub> was investigated at a temperature of 45°C and a flow rate of 60 ml/min, and was shown in Fig. 4. The purities of SF<sub>6</sub> gas samples were measured with the method based on GC [20]. It is seen that the drift time increased from 21.84 to 26.32 ms with decreasing SF<sub>6</sub> purity. In order to investigate the relation between the drift time and the impurity content, the experimental data were fitted as shown in Fig. 5. It is seen that the experimental data were well liner fitted with correlation coefficient of 0.992. Therefore, the purity of SF<sub>6</sub> could be extracted by measuring the drift time of the SF<sub>6</sub> gas samples.

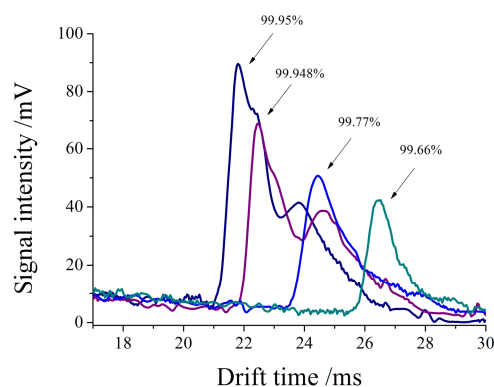


Fig. 4. Influence of SF<sub>6</sub> purity on the drift time of SF<sub>6</sub> samples

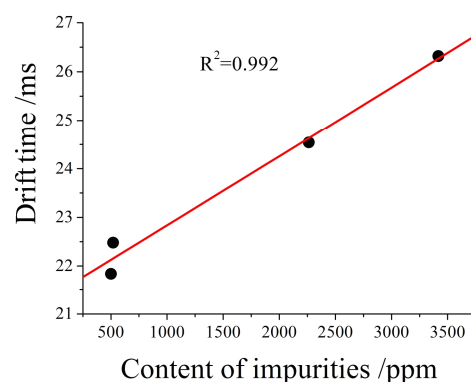


Fig. 5. Linear calibration curve for drift time and content of impurities

### Detection of decomposition products in SF<sub>6</sub>

In order to investigate the detection mechanism for SF<sub>6</sub> purity with ion mobility spectrometer, a home-made time of flight mass spectrometer was employed for monitoring of discharging decomposition products of SF<sub>6</sub>. A point discharging system was used for simulation of partial discharge in GIS, and SF<sub>6</sub> gas sample with purity of 99.95% was used as the discharging gas. The decomposition products were introduced into the time of flight mass spectrometer with a capillary inlet. The monitoring curve of discharging decomposition products of SF<sub>6</sub> was demonstrated in Fig. 6.

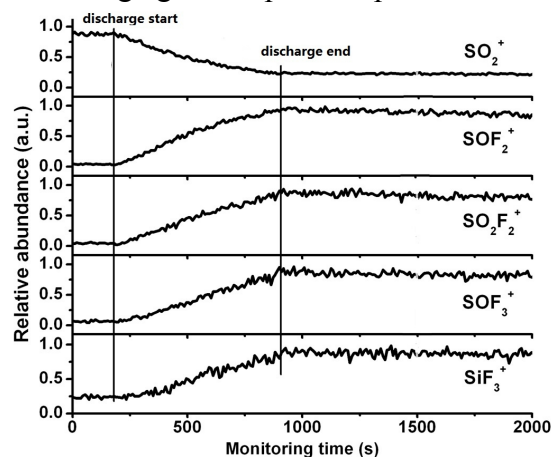


Fig. 6. Monitoring curves for the main discharging decomposition products of SF<sub>6</sub>

It is clear that the content of  $\text{SOF}_2$ ,  $\text{SO}_2\text{F}_2$ ,  $\text{SOF}_4$  and  $\text{SiF}_4$  increased gradually with the discharging process, while the content of  $\text{SO}_2$  decreased. And a plateau was observed for all the decomposition products after the discharging process ended. It was proposed that the increasing drift time with decreasing  $\text{SF}_6$  purity could be due to the decomposition products. Some of the decomposition products would attach the core ions to produce new ions clusters with bigger cross sections, which decreased the velocity of the ions in IMS.

## Conclusion

This paper introduced a method for rapid analysis of  $\text{SF}_6$  purity based on one-way IMS. The purity of  $\text{SF}_6$  can be deduced by measuring its drift time. The drift time of  $\text{SF}_6$  increased with decreasing flow rate, drift tube temperature and purity of  $\text{SF}_6$ . And a higher resolution and sensitivity could be obtained at a lower flow rate. The product for partial discharge in  $\text{SF}_6$  was monitored with a time flight mass spectrometry. And it is found that  $\text{SO}_2$ ,  $\text{SOF}_2$ ,  $\text{SO}_2\text{F}_2$ ,  $\text{SOF}_4$  and  $\text{SiF}_4$  were found to be the main discharging decomposition products, which lead to the decrease of purity of  $\text{SF}_6$ .

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