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Installation and Test Run of Comprehensive Analysis System for SF_6 in Power Equipment

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Abstract

After SF_6, which is being used in power equipment as an insulating material, is classified as one of the 6 major greenhouse gases, the maintenance and the refinement of used SF_6 started to get attention. In regard to this, KEPCO Research Institute (KEPRI) is developing SF_6 recovery and refinement technology starting with establishing a comprehensive SF_6 analysis system. With the analysis system, qualitative and quantitative analyses of the purity and the impurities of SF_6 before and after recovery, and before and after refinement have been carried out. The analysis system is comprised of GC-DID (Gas Chromatograph -Discharge Ionization Detector) for trace impurities analysis, GC-TCD (Thermal Conductivity Detector) for analyses of SF_6 purity and major impurities concentration from several hundred ppm up to percent range, GC-MSD (Mass Selective Detector) for analyses of impurities not included in standard gas, FT-IR (Fourier Transform-Infrared) Spectrometer for analysis of HF and SO_2, and moisture analyzer for analysis of moisture below 100 ppm. With this analysis system, complete analysis method of SF_6 has been established. This analysis system is being used in the maintenance of power equipment and the development of SF_6 recovery and refinement technologies. In this paper, the analysis results of four samples - gas and liquid phase SF_6 samples from a SF_6 refinement system before and after refinement are presented.

Keywords: Gas Chromatography, FT-IR, SF_6 Refinement, SF_6 Analysis, Gas Insulated Switchgears (GIS)

I. INTRODUCTION

Currently, heavy electrical equipment switches, circuit breakers and power equipment including some of transformers in Korea are gas insulated with SF_6. Especially, Gas insulated switchgears (GIS) installed and operated in substations are using large amount of SF_6. In case of GIS, when routine detailed inspection is performed all SF_6 in GIS is extracted before the inspection. The extracted SF_6 is discarded and not reused to avoid degradation of insulation due to contamination and new SF_6 is used to fill the GIS. This policy is applied to other SF_6 insulated power equipment's when they are inspected, replaced, or discarded. As SF_6 is regulated globally, the current trend is toward developing and adopting alternative insulating materials. However, gas insulation with SF_6 is still being used locally and globally for now.

Due to its property to capture free electrons to form low mobility heavy ions, SF_6 makes the development of electron avalanches very difficult. Consequently, it has high breakdown voltage (three times higher than air), high thermal interruption capabilities (ten times higher than air), and high heat transfer capabilities (two times higher than air). Since early 1960's it has been successfully used as an insulating gas in power equipment such as high voltage and medium voltage switchgears, GIS, ring main, automatic circuit breaker, transformer, and cable. SF_6 is nontoxic, no potential acute or chronic ecotoxicity reported, and not hazardous to surface water and underground water due to its low solubility to water [1]-[7].

It is also not hazardous to ecosystem because it does not accumulate biologically and is not a carcinogen nor a mutagen. Table 1 shows the impurities and their cause of occurrence in SF_6 insulated power equipment [8]-[11].

Table 1. Origin of SF_6 impurities (IEC 60480)

Source of impurities	Possible impurities
Leakage, vacuum, desorption	Air, Oil, H_2O
Partial discharge (corona, spark)	HF, SO_2, SOF_2, SOF_4, SO_2F_2
Corrosion due to switching arc	H_2O, HF, SO_2, SOF_2, SO_2F_2, CuF_2, WO_3, CF_4, AlF_3
Corrosion of equipment	Metal dusts, Particles
Decomposition	Air, H_2O, HF, SO_2, SOF_4, SO_2F_2, SF_4, CF_4, Metal dusts, Particles, AlF_3, WO_3, CuF_2

On the other hand, the global warming potential (GWP) of SF_6 is very high; 23,900 times higher than CO_2, and its life duration in air is 3,200 years making it environmentally very important to not release SF_6 into the air and reuse it. Therefore, during the processes of research, production, maintenance, and reclaim of SF_6, SF_6 should be released into the air conforming environmental regulations. Even though SF_6 is nontoxic, it should be maintained to minimize the release into the air and recovered and reused through separation and refinement when it is used in power equipment since it is one of the 6 major greenhouse gas and expensive. KEPCO defined SF_6 purity and impurity limits according to International Electrotechnical Commission (IEC) guideline. In Table 2 and Table 3, KEPCO's management standards are presented for new and used/recycled SF_6 respectively. In Table 3, the maximum acceptable moisture level is the same as that of IEC 60376, and the maximum acceptable SO_2 level is the same as the maximum acceptable HF level of IEC 60376 [8].

And also, Internationally, IEC 60376 specifies that SF_6 of 0.997 mole/mole or higher purity is suitable for switches and

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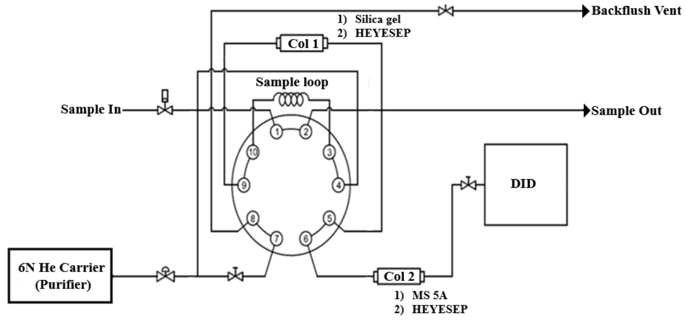


Fig. 1. Valve configuration of GC-DID.

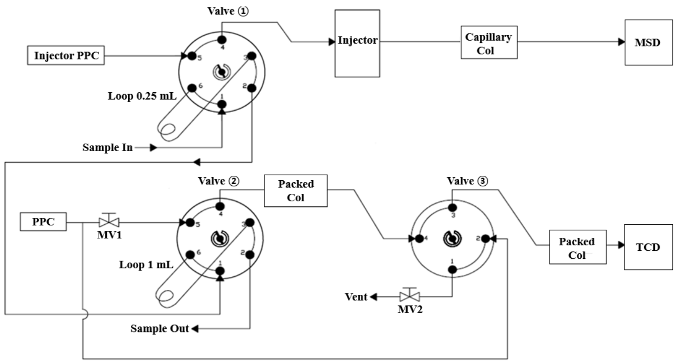


Fig. 2. Valve configuration of GC-MSD/TCD (MV: Main Valve, PPC: Programmable Pneumatic Control).

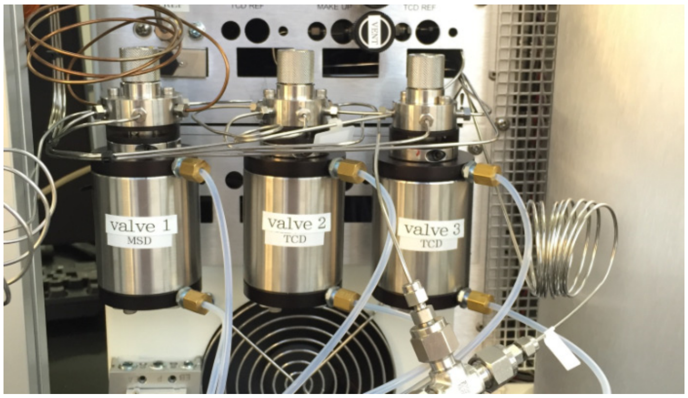


Fig. 3. Three valves installed at the back of GC-MSD/TCD.

suggests the maximum acceptable level of impurities such as air, CF_4, H_2O, oil, and acidity in SF_6, and their analysis method and precision [8][9].

Presently KEPCO is developing separation/refinement and decomposition technologies for used SF_6 in power equipment. Prior to this developments, SF_6 analysis system has been installed. This paper presents the analysis results of SF_6 samples before and after refinement using the analysis system.

II. EXPERIMENTAL

A. Samples

In KEPCO's power equipment such as 22.9 kV GIS, bus is filled with SF_6 gas from several liters to tens of liters and maintained on a regular basis. For GIS, switches, and bus, the life time is 22 years. When their lifetime expires, they are subject to replacement preferentially. And they are replaced according to the budget. Currently in Chungbuk headquarters is carrying out recovery, refinement, and reuse of used SF_6 from power equipment. SF_6 gas from power equipment such as 22.9 kV GIS'es and buses in KEPCO's local headquarters are recovered with SF_6 recovery equipment (SLR-600 Woosung Vacuum Co. Ltd.) and stored in gas cylinders to be transported to separation/refinement facility. The recovery equipment operates at 30 kg/cm^2, -20~40°C and comprised of a compressor, a vacuum pump, a storage tank, a cooler, a heater, and controllers. The SF_6 gas from the recovery equipment is pumped into cylinders at the pressure of 24~28 bar varying with the atmospheric temperature.

Table 2. Management standard for new SF_6

Item	Maximum acceptable level	Action
Purity	over 99.7%	Replace gas if level exceeds
H_2O	below 200 ppmv	
SO_2	below 1 ppmv	

Table 3. Used and recycling SF_6 management standard

Item			Concentration
H_2O	equipment where SF_6 decompose	managing value	150 ppm
		acceptable value	300 ppm
	equipment where SF_6 do not decompose	managing value	300 ppm
		acceptable value	1,000 ppm
SO_2			below 1 ppm
Purity			over 99.7%

Table 4. GC-DID analysis condition

Specification	Condition
Oven temperature	100~110°C
Detector temperature	80°C
Carrier gas, flow rate	He, 40 mL/min
Sample, flow rate	50 mL/min

B. Analysis equipment

A portable analyzer (SF6-6100 pump back analyzer, Rapidox) is used on site. And precise analysis is performed with FT-IR (Fourier Transform-Infrared Spectrometer, MIDAC I1801, MIDAC Corp.), GC-DID (Gas Chromatograph-Discharge Ionization Detector, Novachrom 1000, AGC), GC-MSD (Gas Chromatograph-Mass Selective Detector, Clarus SQ 8T, Perkin-Elmer), GC-TCD (GC-Thermal Conductivity Detector, Clarus 680, Perkin-Elmer), and Moisture Analyzer (DF-745SGMAX, Servomex). The GC-DID uses two SilcoSteel packed columns (Molecular sieve 5A, Heyesep) as shown in Fig. 1. The first column (Col 1) identifies the main peak and impurity peaks. And then only impurities go into the second column (Col 2) and the rest is vented. The second column (Col 2) separates and analyzes the minute amount of impurities qualitatively and quantitatively. The analysis conditions are summarized in Table 4. The samples to be analyzed are mainly SF_6 gas with trace of impurities. To analyze the minute amount of impurities, back flush method - one of valve cut methods are adopted to analyze without the interference of SF_6 peak since SF_6 peak comes out last. Since molecular sieve column cannot separate CO_2 and Heyesep column cannot separate N_2 and O_2, both are used to analyze H_2, CH_4, CO, CO_2, CF_4, O_2, and N_2.

To analyze the purity of SF_6 and impurities without standard samples, TCD and MSD are used. TCD, which detects the thermal conductivity difference between sample and carrier gas, uses He which has high thermal conductivity as a carrier gas to analyze O_2 and N_2, which comprises most of the impurities [12]-[16]. The oven temperature is 60°C, and the sample is injected at

Table 5. GC-MSD/TCD analysis condition

Specification	Condition	
	MSD	TCD
Oven temperature	200°C	60°C
Carrier gas (He), flow	1 mL/min	1 mL/min
Sample flow	1 mL/min	1 mL/min

Table 6. FT-IR analysis condition

Specification	Condition
Scan time	0.8 sec.
Resolution	1 cm^{-1}
Cell temperature	80 °C

Table 7. Moisture analyzer analysis condition

Specification	Condition
Flow	500~600 mL/min
Purge time	60 min
Analytical time	30 min

a flow rate of 1 mL/min. In general, purity of SF_6 in percent is estimated by subtracting the concentrations of impurities from 100. The estimation is cross checked by analyzing SF_6 concentration with TCD using standard samples of 85% (air balance) and 99.99% purity. As shown in Fig. 2, sample is injected into valve 1, and passes through 0.25 mL sampling loop and capillary column (Gas pro, 60 m) sequentially and is analyzed with TCD. Part of the sample is injected into channel 1 of valve 2, and passes through 1 mL sampling loop, the first packed column (Porapak Q), valve 3, and the second packed column (Molecular sieve 5A), and is analyzed with TCD. The actual 3 valves installed at the back of GC is shown in Fig. 3.

GC-MSD/TCD analysis condition is shown in Table 5. Since the sensitivity of GC-TCD is lower than GC-DID, it is used to the analysis of main components - SF_6, air, and N_2. GC-MSD is used to the analysis of chemicals whose standard samples are not exist or hard to find. And it is also used to qualitative and quantitative analysis of CF_6, C_2F_6, CO_2, SO_2, etc. whose concentration in SF_6 is over 20 ppm: not suitable for GC-DID analysis.

HF and SO_2, which are the major decomposition products of SF_6, and other decomposition products are analyzed with FT-IR and the analysis condition is shown in Table 6. FT-IR (Fourier Transform-Infrared spectroscopy) is a very valuable equipment for SF_6 impurity analysis, which can scan the whole spectrum range at a fast 0.8 scan/sec, identify more than 60 chemicals in 650~4,500 cm^{-1} range, analyze HF and sulfur compounds [17]-[20]. For the SF_6 analysis system, the FT-IR's cell length is 10 m and filled with 1.9 L gas (@ 1 atm.; 25°C). The cell pressure during the analysis is about 1 bar. The detector is cooled with liquid nitrogen to enhance the analysis sensitivity. Since the detector detects the small heat caused by infrared absorption, it is better to have low background temperature to widen the heat sensing range. To minimize the effect of other chemicals the resolution is set to 1 cm^{-1}. ZnSe is used for beam splitter, which splits the infrared ray from the source and sends to fixed mirror and moving mirror. Before the sample analysis, the cell is purged with high purity (99.999%) N_2 to remove moisture and impurities. Then non existence of possible residual impurity peaks such as CO (2,300~2,350 cm^{-1}) and H_2O (4,0004,500 cm^{-1}, 1,400~2,000 cm^{-1}) is assured and the cell is cooled, background temperature is measured before the sample is injected.

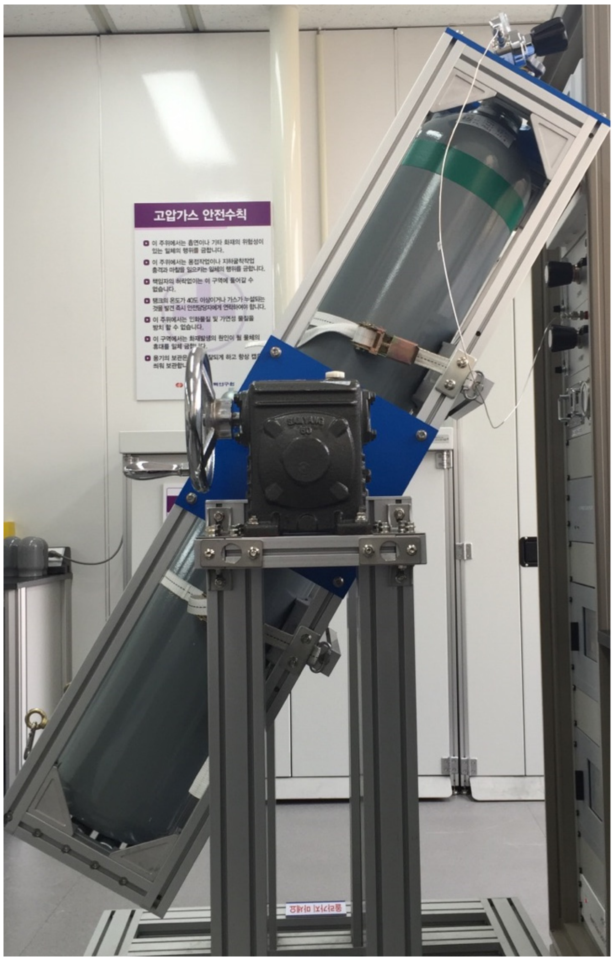


Fig. 4. Cylinder inverter.

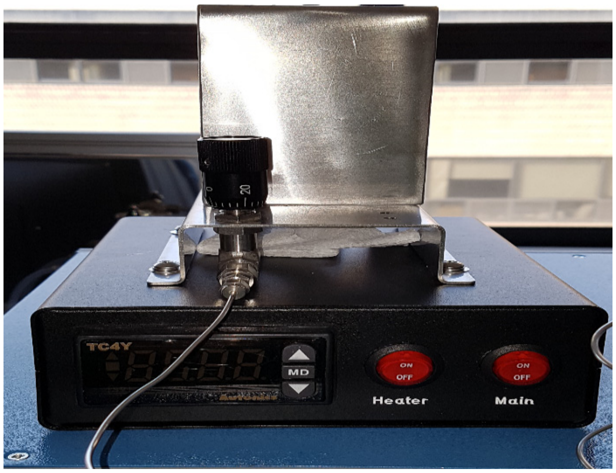


Fig. 5. Vaporizer.

To analyze the moisture in the range of several ppb to 20 ppm, diode laser adsorption spectroscopy type was selected. To analyze ppm level moisture, sufficient purging is required. After the moisture peak is stabilized, wait more than 30 minutes before taking the measurement to get correct value. The analysis condition is shown in Table 7.

C. Supplementary equipment

1) Cylinder Inverter

Since SF_6 is an easily liquefiable gas under pressure, it is partly liquefied in the sample cylinders at the pressure of about

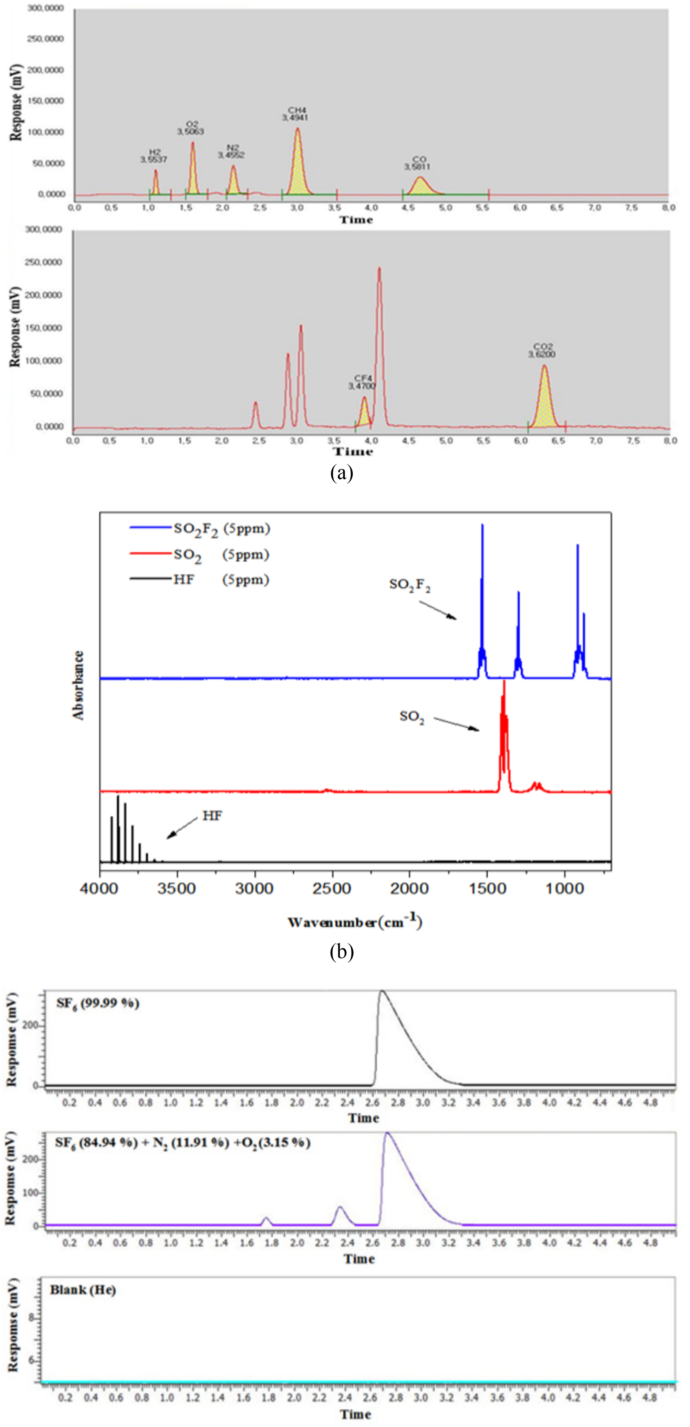


Fig. 6. Chromatograms for standard samples. (a) GC-DID. (b) FT-IR. (c) GC-TCD.

20 bar. Therefore, both liquid and gas phase sample should be analyzed separately for precise qualitative and quantitative analysis. For this purpose, a siphon may be installed in the cylinder. However, we opted to fabricate a cylinder inverter to turn the cylinder upside down to sample liquid phase as shown in Fig. 4 to be used for various cylinders. The inverter has been designed with consideration for stability since SF_6 filled cylinder is quite heavy (about 100 kg including 50 kg of SF_6). Especially the ratchet located in the middle of the inverter should withstand 100 kg [12].

Table 8. Certificate of reference materials

Components	Concentration (μmole/mole)	Uncertainty (μmole/mole)
H_2	3.54	0.01
O_2	3.53	0.03
N_2	3.50	0.05
CH_4	3.49	0.00
CO	3.57	0.07
CO_2	3.62	0.00
CF_4	3.47	0.00
He	Balance	

2) Vaporizer

It is preferable to vaporize liquid SF_6 samples before they are injected into analysis equipment. In our analysis system, sampling lines from the sample cylinder to the analysis equipment are not long enough to vaporize liquid samples before they reach the sample inlets. Thus a vaporizer with a small heater, as shown in Fig. 5, is installed in the sampling line instead of heating the sampling line with a water bath. The heater temperature is set to 60°C.

D. Standard samples

A custom-made standard sample having similar composition to used SF_6 gas from power equipment was purchased from a local company (RIGAS, Korea). Two more SF_6 gas samples having 84.97% and 99.999% purity, HF sample (5.15 μmol/mole, He balance) and SO_2 sample (5.00 μmol/mole, He balance) were also purchased. Small amount of SO_2F_2 was provided in a Tedlar bag. The information of the standard sample is shown in Table 8.

III. RESULTS & DISCUSSION

A. Analysis result of low concentration SF6 standard sample

To obtain reliable analysis results, standard samples were analyzed before samples were analyzed. Fig. 6(a) shows the peaks of 7 chemicals of the standard sample from GC-DID analysis and they were clearly identifiable in 3~5 ppm range. The analysis result showed that the measurement errors of H_2, O_2, N_2, CH_4, CO, CF_4, CO_2 concentration were maximum 0.07 μmole/mole. It was speculated that large variation of retention time, which is typical characteristics of gas samples, was the cause of large error. Gas and liquid phase SF_6 samples before and after refinement were analyzed after the analysis of the standard sample. Fig. 6(b) is the analysis result of the standard sample with GC-TCD. A blank sample was measured with high purity He followed by measurement of standard SF_6 sample of 84.94% with 11.91% N_2 and 3.15% O_2. Then 99.99% SF_6 standard sample was measured, and qualitative and quantitative analyses of samples were performed. Fig. 6(c) is the analysis result of 5 ppm HF, SO_2, and SO_2F_2 for quantitative analysis. Peaks of HF, SO_2, and SO_2F_2 were observed at 4,000~3,500 cm^{-1}, 1,500~1,000 cm^{-1}, and 1,500~100 cm^{-1} respectively.

B. Analysis results of gas and liquid phase SF6 samples before and after refinement

1) Analysis result of gas phase sample before refinement

Analysis result of gas phase sample before refinement under the condition specified in 'II. Experimental' is shown in Table 9. Analysis procedure was set up such that analysis result is cross-

Table 9. Analysis results of gas and liquid phase sample before refinement

Components	Concentration (μmole/mole)		Analyzer
	Gas phase	Liquid phase	
H_2	39.01	9.68	GC-DID
O_2	4.07% (mole fraction)	0.39% (mole fraction)	GC-TCD
N_2	16.20% (mole fraction)	1.65% (mole fraction)	GC-TCD
CH_4	54.24	5.88	FT-IR
CO	100.47	10.71	FT-IR
CO_2	226.88	112.49	FT-IR
CF_4	130.61	8.39	GC-DID
C_2F_6	666.53	N.D (Detection limit < 0.1)	GC-MSD
C_5F_{10}	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
H_2O	13.47	7.00	Moisture Analyzer
HF	0.71	N.D (Detection limit < 0.1)	FT-IR
SO_2	2.97	N.D (Detection limit < 0.2)	FT-IR
SOF_2	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SO_2F_2	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SOF_4	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SF_6	79.60% (mole fraction)*	97.95% (mole fraction)*	

* Assay is by difference, after impurity content is determined.

Table 10. Analysis results of gas and liquid phase sample after refinement

Components	Concentration (μmole/mole)		Analyzer
	Gas phase	Liquid phase	
H_2	0.62	N.D (Detection limit < 0.1)	GC-DID
O_2	23.25	10.05	GC-DID
N_2	24.02	10.91	GC-DID
CH_4	1.46	0.59	GC-DID
CO	1.32	0.37	GC-DID
CO_2	1.31	1.24	GC-DID
CF_4	10.06	6.35	GC-DID
C_2F_6	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	GC-MSD
C_5F_{10}	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
H_2O	1.00	1.00	Moisture Analyzer
HF	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SO_2	N.D (Detection limit < 0.2)	N.D (Detection limit < 0.2)	FT-IR
SOF_2	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SO_2F_2	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SOF_4	N.D (Detection limit < 0.1)	N.D (Detection limit < 0.1)	FT-IR
SF_6	99.99% (mole fraction)*	99.99% (mole fraction)*	

* Assay is by difference, after impurity content is determined.

checked with GC-DID and GC-TCD when impurity concentration was in the range of 100～1,000 ppm. It is expected that gas phase sample before refinement would have considerable amount of impurities. With GC-DID only H_2 (Gain×1) was measured in analyzable range. Since SF_6 could be contaminated with air or N_2 during the power equipment operation or recovery process, O_2 and N_2 concentrations in SF_6 could be in % range so that GC-TCD was used to analyze them. C_2F_6, CF_4, and SO_2 were analyzed with GC-MSD even though they could be analyzed with FT-IR. Other impurities were analyzed with FT-IR. N_2 or air were measured at about 20%. HF was low at 0.71 ppm. Moisture was 13.47 ppm, which is below the reuse standard. The SF_6 purity was estimated to be 79.60%.

2) Analysis result of liquid phase sample before refinement

The analysis result of liquid phase sample before refinement is also shown in Table 9. O_2 and N_2 were analyzed with GC-TCD - same as gas phase sample. Most of impurities (H_2, CH_4, CO, CO_2, CF_4, Gain ×4) were able to be analyzed with GC-DID due to their low concentration. Moisture content was 7.00 ppm. Other impurities were undetectable with FT-IR and GC-MSD.

3) Analysis result of gas phase sample after refinement

It is expected that impurities in sample after refinement would be very low. Chemicals from H_2 to CF_4 in Table 10 could be analyzed with GC-DID. Other impurities were undetectable and moisture content was 1 ppm.

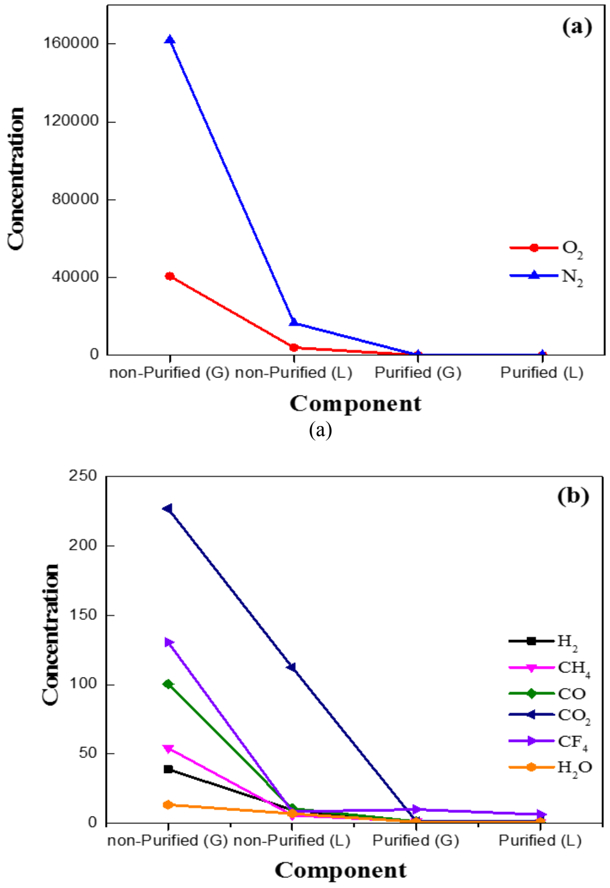
4) Analysis result of liquid phase sample after refinement

The impurity concentration in liquid phase sample after refinement was expected to be much lower than 3 previous samples. Chemicals from H_2 to CF_4 in Table 10 were able to be analyzed with GC-DID - the same as the gas phase sample after refinement. Other impurities were undetectable and moisture content was 1 ppm.

The major impurity concentrations of above samples were compared in Fig. 7. Fig. 7(a) shows O_2 and N_2 which comprised most of impurities, and Fig. 7(b) shows the rest of impurities in ppmv. As discussed above, the impurity concentration was highest in the gas phase sample before refinement and there were almost no impurities in the gas and liquid phase samples after refinement.

IV. CONCLUSION

A comprehensive analysis system for SF_6 in power equipment was established. The following results were found through the analysis of 4 samples - gas and liquid phase samples before and after refinement - with analyzers in the system: GC-



(a)

(b)

Fig. 7. Comparison of impurity concentrations in ppmv.

DID, GC-MSD/TCD, FT-IR, and moisture analyzer.

(1) Most of the impurities in samples before refinement were nitrogen and/or air.

(2) Major impurities in liquid phase sample before refinement and gas/liquid phase samples after refinement could be analyzed with GC-DID at the same time in a short time.

(3) HF, SO_2 and other sulfur compounds could be analyzed with FT-IR.

(4) Since liquid phase samples have the same or lower impurity concentrations than gas samples, only gas sample analysis is needed to figure out the purity of SF_6 to reduce analysis time. Further analysis of liquid samples would be performed if required.

(5) The analysis result confirmed that SF_6 after refinement can be reused since it meets the KEPCO management standard shown in Table 3.

Presently proper sampling method are being sought using gas sampling bag, gas sampling cylinder, Tedlar bag other than normal gas cylinder. And method of injecting samples to analyzers from this sampling device will be established. These current activities will be presented soon.

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