Implementation Of Treatment & Recovery Of the SF_6 Gas Containing A High Amount Of Decomposition Products Due To High Voltage Electrical Interruptions.

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1/ Abstract

After several years of use in substations, the SF_6 gas was subjected to several types of arcing due to current interruptions in High Voltage Switchgear equipment. These short circuit interruptions have changed the composition of the pure SF_6 gas, and decomposition products are polluting it. This paper described the tests performed at site by the AREVA T&D team and the recovery of a polluted gas by AVANTEC in their factory, removing the decomposition products. Generally, this paper describes the treatment and the recycling of a used gas.

2/ Introduction

Fig.1. Typical 245 kV dead tank circuit breakers using SF6 gas as internal insulation and interrupting medium. This is Air Insulated Switchgear (AIS) as described later.

Fig. 2. Typical Gas Insulated Substation (GIS) 245 kV using SF6 gas as insulation and interrupting medium. This is Gas Insulated Switchgear (GIS) as described later.

The High Voltage electrical industry uses SF_6 gas because it is an excellent dielectric, which in addition has very good electrical arc switching characteristics. The gas is chemically inert, non-toxic, and non-flammable. However, the SF_6 gas due to its high Global Warming Potential (GWP), has been included in the basket of gases of the KYOTO Protocol that many European countries have signed.
For this reason, and even if the SF_6 gas from electrical equipment represents a very small portion of the anthropic greenhouse gasses in the atmosphere, the electrical industry worldwide has undertaken many initiatives to reduce the SF_6 gas emissions. In particular, measures concerning the recycling of polluted SF_6 have been considered.
In this context, AREVA T&D, a major world producer of high voltage switchgear, and AVANTEC, a company specialized in performance chemicals, have been collaborating for many years in order to develop a new approach to treat SF_6 containing a high amount of decomposition products due to electrical normal interruption.
AVANTEC has developed a SF_6 gas treatment technology, which aims at avoiding its destruction by a proper recycling of the polluted SF_6 gas, as it is described in this paper. A pilot installation was set, based on selective adsorption of chemicals in the liquid phase, able to decrease the amount of decomposition products contained in the polluted SF_6 gas. In parallel, AREVA T&D has developed an analytical method for on site analysis of polluted SF_6 gas based on Gas Chromatography technique.

3/ The Impact of SF_6 On The Environment

3.1/ Environmental Issues for Switchgear.

Due to its high GWP and its long atmospheric lifetime, the SF_6 gas was included in the basket of greenhouse gases of the KYOTO protocol, which aims to limit the emission of greenhouse gases. For the period 2008-2012, the reduction should be 5% compared to 1990's or 1995's emissions.
The relative contribution of SF_6 to the man-made global warming effect can be calculated by taking into account the SF_6 concentration in the atmosphere and its GWP. This value is approximately 0.1% [1]. When searching only the SF_6 gas coming from the world electrical industry, many people estimated it at 0.012% [2]. For Europe, SF_6 from the electrical industry contributes only to 0.008% of the whole Global Warming [3].

Fig. 3. Row of high-voltage circuit breakers with internal SF_6 gas insulation and connections to the bus air insulated with large energy losses on the buses.

On another hand, many attempts have been done to evaluate the environmental impact of SF_6 from electrical equipment via environmental Life Cycle Assessment (LCA). For example, when considering the LCA of High Voltage Circuit Breakers, it has been proven that SF_6 losses due to leakage in service or during manufacturing and commissioning are not the major contributors to Global Warming. In fact, most of the global warming impact is due to the energy losses during the life of the apparatus [4]. On a more global approach, full LCA of Medium Voltage networks has been done. For example, the impact of AIS (insulation by air) and GIS (insulation by SF_6) products included in rural or urban networks were studied [5-6]. These studies showed that the total environmental impact of AIS is higher than for GIS equipment (using SF_6). More over, it was made clear that in such networks, the switchgear represents less than 10% of the total networks Global Warming impact, which is mostly due, here again, to the energy losses within the network.

3.2/ Electrical Industry Initiatives To Reduce SF_6 Emissions

If the SF_6 gas represents a very good technical solution, even from the environmental point of view, electrical equipment manufacturers are making efforts to reduce the SF_6 emissions. Indeed, since 1995/1996 (when SF_6 became known as a gas having a high GWP), the emission of SF_6 from the electrical industry has decreased, showing the promptness of the electric industry's reaction in response to the problem [7]. However, the electrical industry in co-ordination with its suppliers is doing the best to put the environmental impact as low as possible [8].
For that purpose, improvement of the gas handling procedures, systematic gas re-uses and voluntary emission reduction programs have been set-up [9-10].
Concerning handling and re-use, for example CIGRE WG B3.02 (formerly WG 23.10), published a guide related to SF_6 Practical Handling, which became an "SF_6 recycling guide" giving appropriate recycling procedures, describes recycling equipment, and the origins and quantities of contaminants [11]. This group is also working on tightness prescriptions and test procedures [12]. This is a major technical contribution to the management of the emissions, as unfortunately, during the life cycle of this equipment, SF_6 emissions can occasionally occur. Other recommendations are also given in an IEC technical report about the handling of SF_6, and the end of life of SF_6 filled equipment [13]. The Power Industry is following these guides in North America in regards to recycling and leak rate and will be included in the new ANSI/IEEE Standards.
In addition to this, the electro-technical industry is also involved in the revision of IEC standards concerning the specification of the SF_6 [14-15] as well as for the checking and treatment of SF_6, which will facilitate the re-treatment and the re-use of this gas [16-17]. The new versions of these two documents, namely IEC 60 376 Ed. 2 to be published early 2005 and IEC 60 480 Ed. 2 that was issued October 2004, specify impurity levels that, without creating any problem, will appear more

practical and will allow and ease the re-use and re-cycling of the polluted gas (A summary is included in Table 1).
AVANTEC and AREVA T&D are following worldwide the recommendations of IEC and CIGRE for the work to be described in the following paragraphs.

Fig. 4 Summary of Standard and technical Organizations that Switchgear Manufacturers are working with to reduce emissions

As a contribution to the global management of SF_6, the electrical industry is also deeply involved in the setting up of strategies for the end of life of SF_6 [18].
In fact, when the SF_6 gas contains too much decomposition products, due to arc switching, the reclaiming of the gas, on site, with the help of the movable devices is not possible. In this case the gas has to be treated in another way and many reclaiming techniques have been developed worldwide that could avoid destruction of the gas.
For example, in Japan, gas-reclaiming equipment has been developed to separate SF_6 gas from mixed gas. This system concentrates SF_6 gas, which is sent into a gas liquefaction system. Then a filter refines the SF_6 [19]. Another type of equipment has also been developed based on different filtering stages, a liquefaction system and also analytical system (Gas Phase Chromatography in particular) [20].
In Germany too, a reuse concept has been developed, based on permanent quality control and a device for purifying and storing of the used SF_6 gas [21].
Other systems using separative membranes for the purification of the SF_6 have also been developed. These systems could be found in France, Germany [22], and numerous references can be found in the catalogue of guidelines and standards for the handling and management of Sulphur Hexafluoride from the US EPA [23].

IEC 60376 Ed.1	**Standard**	**IEC 60376 Ed. 2 (Proposal)**	**IEC 60480 Ed.1**	**IEC 60480 Ed.2**	
				Rated absolute pressure <200kPa	**Rated absolute pressure >200kPa**
<0.05%w	Air and/or CF_4	Air<1%vol CF4<0.4%vol	No maximum acceptable impurity levels specified	<3%vol	<3%vol
15 ppmw	H_2O	25 ppmw		95 ppmw	25 ppmw
10 ppmw	Mineral oil	10 ppmw		10 ppmw	
Acidity expressed in HF:0.3 ppmw Hydrolysable fluorides expressed as HF:1.0 ppmw	Total gas decomposition products	Acidity expressed in HF:6 ppmvol		50 µl/l total or 12 µl/l for (SO2+SOF2) or 25 µl/l HF	

Table 1: Old and New Proposal for IEC Standards

4/ The AREVA T&D / AVANTEC Procedure for SF_6 Recovery & Recycling.

Taking into account the experience of both companies, the original concept consists of taking into account the whole process from the site to the end of life of the gas. It covers all stages of the life cycle of the SF_6, from the recovery on site, on the circuit breaker pole or bay, to the final re-use or destruction, including the transport phase and the re-treatment itself (figure 5). Of course, in order to insure it's efficiency, this original procedure for the re-use of SF_6 requires a strict and deep collaboration between the two partners which proved to be operational between AREVA T&D and AVANTEC.

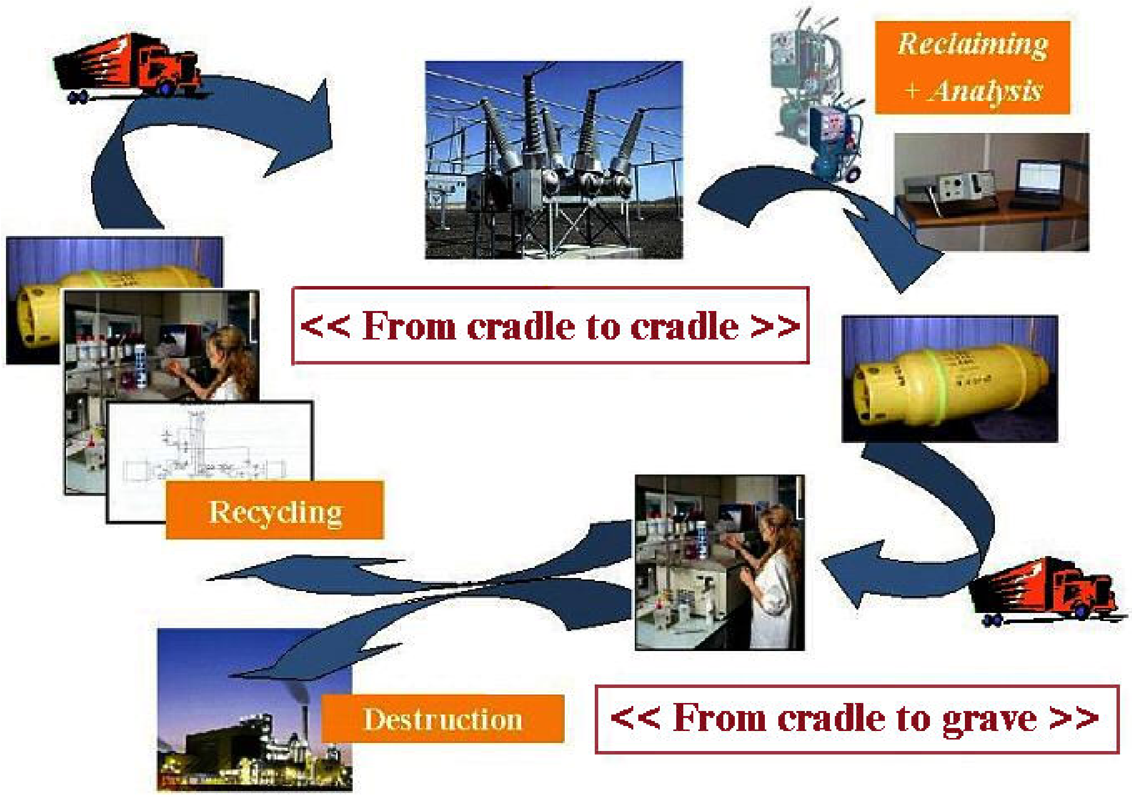

Fig. 5. Schematic diagram showing the recycling of SF_6 and its re-use or the disposal of SF_6 by destruction according to the results of the tests performed in laboratory.

Basically, the procedure consists of a succession of operations, some of them being optional. Indeed, the normal current interruptions in switchgear are leading to the formation of decomposition products of SF_6. Such decomposition products are numerous and various (HF / SO_2 / SiF_4 / SOF_4 / SF_4/ S_2F_{10} / CO_2/ CF_4 / SO F_2/ SO_2F_2 / H_20) as well as oxides and various powders either free or combined with metals (figure 6).

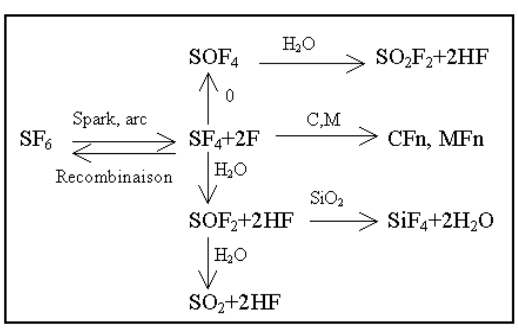

Fig. 6: Reaction diagram showing the formation of stable decomposition products in SF_6,. M represents a metal and C indicates carbon [24].

When SF_6 has lost its properties of dielectric agent, it is usually taken away from the equipment, and filtered on site in order to take away the humidity and the particles.
At this stage, directly on the electrical equipment, the SF_6 can be analyzed to determine whether it is polluted or not. Nevertheless, after a certain period of being subjected to important electrical arcs, it can no longer be purified and the filtration does not allow the user to meet the minimal specifications that are required.
Then the gas is reclaimed and can be analyzed again with on site analytical method to determine whether it can be re-used (if the reclaiming device was efficient enough to filter it) or if it has to be send to the recycling unit. To be purified with the purification unit, AVANTEC Company requires that the gas exhibit acidity level less than 4,000 ppm, expressed in HF, and non-condensable gases content less than or equal to 7,500 ppm (weight).
After the recycling and the control of the batches of regenerated product, experience shows that the "typical values" are far below the limits indicated in the specifications in the future IEC 60480 Edition 2 standard. So this purification method appears to help the limitation of SF_6 emissions and limits the destruction of SF_6.

5/ Gas Analyses

As presented in figure 5, chemical analyses are used all along the recovery and the recycling procedure. The control of the gas is primordial to know if the gas contains any toxic or corrosive by-products that would impose specific handling. More over, the chemical analysis allows knowing if the gas can be recycled.
Depending on the chemicals, many analytical methods are available to check humidity, acidity and decomposition products. By example, humidity can be checked with electronic hygrometer or chilled mirror hygrometer. On site, acidity can be checked with reactive tubes for HF whereas in the laboratory, a bubbling method is used. The decomposition products (SO_2, SOF_2, SO_2F_2) can be analyzed on site using reactive tubes too for SO_2+SOF_2.
In order to get very precise methods even on site, AREVA T&D and AVANTEC have developed two types of Gas Phase Chromatography (GPC) that are described below, of which the results have been crosschecked. These SF_6 gas analyses are done on site and at various stages of the process.
5.1/ On site Gas Analysis
This type of analysis can be done when the gas is taken out from the circuit breaker to be introduced into a storage tank. The gas can be re-checked with this method before being sent for recycling.
In order to intervene easily and rapidly on site, with reliable analytical method, AREVA T&D has developed a specific portable Gas Phase Chromatography (GPC) (Agilent P200). This GPC is used with a laptop to facilitate transportation on site, associated with software named "Ezchrom 200". It is a portable analyzer with two different columns i.e. a Poraplot U and an OV1.

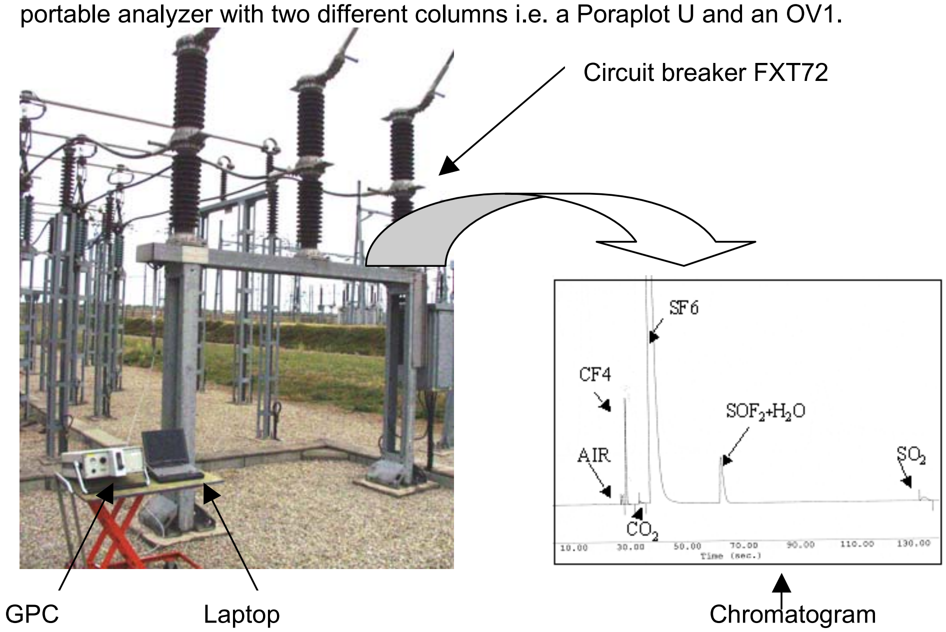

Fig. 7 Gas Phase Chromatography (GPC) set up developed by AREVA T&D with laptop.

The Poraplot U column of 8 meters is used to test air, CF_4, CO_2, COS and SO_2, whereas the OV1 column of 14 meters tests the concentration of SO_2F_2 and SOF_2. It is a very compact system (Figure 7) which enables to measure the concentrations of the air, CF_4, CO_2, COS, SO_2F_2 and SOF_2. The precision is very good for detecting even the smallest levels of by-products (10ppmv).
To develop these methods, AREVA T&D is using specific calibration gases from Air Products. These gases are containing decomposition products i) at very small-defined concentrations or ii) at high and defined concentrations. Prior to any set of measurements, the calibration gases are first analyzed. Then it is possible to integrate measurement peaks with great precision, and therefore to analyze the samples more precisely two analytical methods have been developed, one for the analysis of highly polluted SF_6, and a second one for lightly polluted gases. For these two different methods, the procedure is mainly the same, but the parameters of the integration of the different peaks from the chromatogram are different. The validation of the technique is made by testing the SF_6 gas to determine the by-products levels at pre/post treatment. An example of the analysis is presented in the table 2.

Air (% v)	**CF_4 (% v)**	**CO_2 (ppmv)**	**COS (ppmv)**	**SO_2 (ppmv)**	**SO_2F_2 (ppmv)**	**SOF_2 (ppmv)**	**S_2F_{10} (ppbw)**	**Acidity (ppmv HF)**
0.676	0.215	88	35	2270	/	2289	25	1200

Table 2: Result of an analysis as example.

5.2/ Laboratory Analysis

Fig 8. Analysis by GPC

Once the used SF_6 container arrives at AVANTEC's plant, a sample of the SF_6 is taken on the liquid phase. The product is then analyzed in order to check the rate of contaminants, and to decide what to do with the product, recycling or destruction by incineration. This first control allows checking of the purity of SF_6 (by GPC), the acidity in terms of HF, CF_4 content, non-condensable gases content.The testing of the water content and the acidity level in "Hydrogen Fluor compounds" (HF) is carried out by AVANTEC according to the IEC 60376 standard. SF_6 content is tested using the Gas Phase Chromatography (GPC) (figure 8). In particular, the S_2F_{10} concentration is determined by using a Perkin Elmer-Auto system XI with two columns (Chromosorb O with Kel F and Chromosorb P with BEES phase). The relevant software used is the "turbochrom software".

After the recycling of the SF_6, controls are processed in order to check if the gas purity is in accordance to the future standard IEC 60480 Ed.2, or, better, with the future specification for technical grade SF6, IEC 60376 Ed.2.
Each batch of recycled SF_6 is carefully analyzed in order to determine:

- the purity of SF_6 (GPC method MO 561) : A SHIMADZU (PERKIN HELMER) integrator is used.
- The water content (KARL FISHER method MO 620) : This operation is done with KARL FISHER METHOD, thanks to a "titrator" equipped with a microprocessor, which makes a coulometric proportioning of the water in liquids, solids or gases ; A TACUSSEL AQUAPROCESSOR is used.
- The non condensable gases (GPC method MO 561) : same method as for the determination of the purity of SF6.
- The acidity in terms of HF (method FI 709) : HF is taken by bubbling. The determination of the content of ions F- in the soda solution has been done by potentiometry because of an electrode, which is specific to the ion fluorides. This work is processed with several equipment

such as test tubes of 50 ml precision, gas cans of 5ml precision, micrometric valves and chemicals. All the process is done under a range hood.

- The $S_2 F_{10}$ content (method MO 625): This operation is done by GPC. The method allows the separation of S_2F_{10} from SF_6 and of S_2F_{10} O in a first column (KEL F) at 45°C and the collection of the flushed product in a metallic loop, with introduction of S_2F_{10} in a second column (EES).
- The non-volatile residues: by precision balance method.
- The oil content: by infrared absorption.

6/ Handling of the SF_6

When the gas is sent for purification or destruction, it contains toxic and corrosive by-products. So, in order to prevent from any accident, the following handling procedures are followed.

6.1/ Filling Procedure

First, specific containers dedicated for gas recovery are used. These types of containers are specially provided by AVANTEC to AREVA T&D, and are especially studied for transport, and for containing used SF_6, which may contain TOXIC and CORROSIVE impurities (figure 9). For that purpose, they are designed with specific corrosion protection, including internal preparation and steel valves fittings. Then, during the filling operation of the containers with the used SF_6, the following procedure, which is compulsory, is followed:

Fig. 9. Recovery container

- Checking of the latest testing date of the container (in Europe these containers have to be re-tested every 5 years).
- Checking of the filling ratio of the container (for SF_6 filled in packaging tested at 70 bar, the filling ratio is 1.04 KG/LT).
- Use of a weighing machine in order to control the weight filled in the container and to avoid overfilling.

Fig. 10: Gas recovery unit.

Once filled, the recovery packaging will be fitted with protection caps for both valves (liquid and gas phase).
The use of adapted gas transfer units, which is highly recommended, helps to minimize the losses of SF_6 during that operation (figure 10). This equipment includes an oil-less compressor and a vacuum pump. They allow to charge the SF_6 gas in the electrical equipment or to recover before maintenance operations. The compressor draw a vacuum on equipment while pumping against high pressures, and the vacuum pump allows to vacuum the electrical equipment, the hoses and transfer unit itself after operation. The vacuum is done below 0.5 bar and this allows to limit drastically the emission of SF_6 into the atmosphere. This allows more SF_6 to be recovered and stored for purification and re-use.

6.2/ Procedure For The Return Of Packaging Containing Used SF_6 To The Plant

Before remitting the packaging to the transportation company for the shipment to AVANTEC's plant, the containers are treated in accordance to the "PROCEDURE OF RETURN OF THE CONTAINERS". This procedure is the official French method, which allows AREVA T&D to transfer the responsibility of the waste product towards AVANTEC, which is then responsible of the destruction or the recycling the used products. It includes the following points:

- The packaging is labeled with stickers where the legal information for a transport of waste products by road will be indicated (figure 11).
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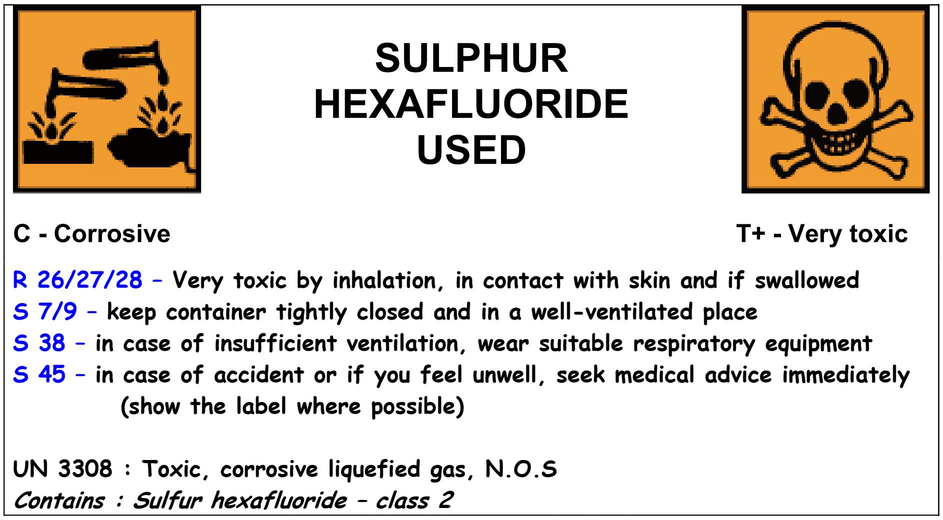

Fig. 11: Label used for transport of polluted SF_6

- A legal document, in France the BSDI (bulletin for the follow up of industrial waste products) is then established by AREVA T&D. The document will include a reference number. This reference number is allocated by AVANTEC upon receipt of the SHEET OF IDENTIFICATION OF THE WASTE PRODUCT. The BSDI document will have to travel together with the used gas. This document will also have to be filled and signed by the transportation company.

After the receipt of the used product in AVANTEC's plant, the packaging is weighted again and the exact weight of SF_6 received is determined. This weight is written on the BSDI document, signed by AVANTEC and sent to AREVA T&D.

7/ SF_6 Purification Techniques

As a pre-treatment, when the content of non-condensable gases in SF_6 is too high (more than 10,000 ppm weight), a process of distillation has to be used by separation between the gas phases of air and of SF_6 at very low temperature.

Then the purification is run on the equipment that has been studied and designed by AVANTEC. It allows the regeneration of SF_6 in the liquid phase. A large column is filled with successive layers of various adsorbents like soda ash, activated alumina, active charcoal, molecular sieves, in order to come to an optimal adsorption. Tissue rings separate these adsorbents and are displayed according to a certain order and precise quantities. In addition to the filtration capacity of the absorbents, a 5 microns particles filter is added at the end of the column. The polluted SF_6 in liquid form is pushed with a pneumatic pump, then rises inside the column, passes through adsorbents layers, is filtered through the safety 5 microns filter and is recovered. At the end of the equipment, a valve allows a direct sampling in order to determine the level of purity of the recovered product.

Fig. 12: SF_6 containers – AVANTEC INDUSTRIEL SITE

Finally, this device, an improvement of a micro pilot unit already available in the factory, has been optimized by a special choice of i) the quality of the adsorbents, including the dedicated choice of the suppliers, ii) the order of placing the absorbents in the column, iii) the quantity of each component, iv) the dimensions of the several layers, v) the speed of the liquid SF_6 through the various layers, and vi) the pressure and its control.
From time to time, when the performance of the process decreases, the adsorbents are taken away, are placed in tight containers, and are sent, for destruction to a specialized company certified for this type of destruction.

8/ Destruction

When the SF_6 gas is mixed with more than 4,000 ppmv of acidity, expressed in HF, it is too highly corrosive to be passed through the purification device. Also, when the SF_6 contains more than 7,500 ppmw of air the efficiency of the purification process is very poor.
In these cases, the gas must be destroyed using industrial waste treatment equipment. Indeed, heated at above 1,000°C, SF_6 starts to dissociate into reactive fragments, which interact with appropriate partner chemicals, mainly hydrogen and oxygen to form SO_2 and HF. The SF_6 gas can thus be destroyed with a removal efficiency greater than 99% when the thermal process operates at 1,200°C. The products of the reaction (HF and SO_2) are removed by passing through a calcium hydroxide solution in order to neutralize the acids and to form solid sulfates and fluorides.

9/ Assessment

The target for the recycling operation was to meet the requirements of the future standard IEC 60480 Ed. 2, and even better, of the future specification for technical grade SF_6 (IEC 60376 Ed. 2) (table 1).
Experience showed that, after the recycling, the typical concentrations of impurities were much better than the limits given by the IEC 60480 Edition 2. In some cases, we could observe that the gas sometimes conforms to the draft IEC 60 376 Edition 2. As the matter of fact, the micro pilot gave good purification performances, and the amount of SF_6 destroyed was reduced.
The portable GPC has been developed to allow for on site testing and especially for SF_6. So with this analysis technique and the AVANTEC micro-pilot put together, one can conclude that AVANTEC and AREVA T&D are working successfully together for the effective treatment of SF_6.
After 2 years of experience of a joint action between AREVA T&D and AVANTEC it can be seen that tens of tons of used SF_6 have been treated and recycled, allowing a second use of the product. During these operations of process, the total losses are below 2% of the quantities originally recovered. AREVA T&D and AVANTEC try to optimize the process in order to continue to decrease these emissions (target below 0.5%).
The recycling of SF_6 has also a positive aspect in terms of cost. It allows avoiding the destruction cost of the SF_6 and allows a selling price of the recycled SF_6 as nearly 10% less than the market price of brand new SF_6.

10/ Conclusion

As it has been seen above, AREVA T&D and AVANTEC have developed original concept and procedure for the SF_6 recovery and recycling. After three years of experience, it can be confirmed that this concept is adapted for the regeneration of heavily polluted SF_6, and that the handling of such gas is covered by proper procedures, including analysis, that insure SF_6 releases to be as low as possible.
In particular, on site analyses by Gas Chromatography as developed by AREVA T&D, are available and proved to be reliable in the detection of SF_6 by products. The recycling of SF_6, using a liquid

phase filtration technology developed by AVANTEC, proved to be very efficient. Laboratory analytical methods, also developed by AVANTEC, appeared to be very precise and reliable to guaranty the final composition of the purified gas.

Finally it can be confirmed that such procedure allow excellent purification performances by producing a gas in conformity with the requirements of the future standard IEC 60480 Ed. 2, and even with the future specification for technical grade SF_6 (IEC 60376 Ed. 2). Such a concept practically allows reducing SF_6 destruction and emissions in an important way, and brings qualitative, quantitative and financial improvements as well as better environmental protection.

This concept, now running in France for three years is going to be deployed in Europe. A further deployment on the American continent via AREVA T&D and AVANTEC subsidiaries and its partners is under consideration.

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