

# UF<sub>6</sub> Sampling Method using Alumina

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## **Abstract:**

*The ABACC-Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials has developed a UF<sub>6</sub> sampling method for enrichment determination (named ABACC-Cristallini Method) which uses a fluorothene P-10 tube type containing alumina pellets that absorb and hydrolyze UF<sub>6</sub> directly during the sampling. The alumina pellets retain up to few hundreds milligrams of U (in a solid compound – UO<sub>2</sub>F<sub>2</sub>) without the need of using liquid nitrogen during sampling. This new method has advantages compared to the actual method that uses a Hoke tube as: the UF<sub>6</sub> sample content left at the installation (archive sample) will be lower and less reactive, the laboratory procedures for manipulating the sample will be much easier, the residual uranium retained at the laboratory will be much lower, the sampling device is less expensive, there will be saves in transport cost as well, and it is relatively safer concerning radiological protection aspects during transportation.*

*This paper describes the physical principle of the new method, the experiments carried out at laboratory taking into account different process parameters foreseen in real cases, and the development of a procedure for recovering the uranium retained inside the alumina pellets for the U enrichment measurement. The behavior of the new method is compared to the traditional one, showing no loss of accuracy for the enrichment determination with real UF<sub>6</sub> samples taken from enrichment plants. The qualification strategy applied to the new method for routine safeguard application at the enrichment plants is presented in this paper as well.*

**Keywords:** UF<sub>6</sub> sampling; enrichment plant safeguard; measurement techniques and standardisation

## **1. Introduction**

The ABACC-Brazilian-Argentine Agency for Accounting and Control of Nuclear Materials performs safeguard inspections jointly with the IAEA-International Atomic Energy Agency at enrichment plants in Brazil and Argentina. Particularly, at enrichment plants in Brazil that use centrifuge enrichment process, routine and unannounced inspections are performed and UF<sub>6</sub> samples are taken from process lines and cylinders to verify the uranium enrichment conformity with design/operator declarations.

A Hoke tube type is normally used for UF<sub>6</sub> sampling. Sampling dwells up to one hour, with the tube immersed in liquid nitrogen, and up to 10 grams of UF<sub>6</sub> are collected. The samples taken in Brazil are sent by ABACC to a Network Laboratory in Argentina for mass spectrometry analysis. The UF<sub>6</sub> sample is hydrolyzed and a very small quantity (some milligrams) is used for the enrichment determination. The residual quantity of UF<sub>6</sub> retained at the laboratory is very large compared to the needs for the enrichment measurement. It is also a costly sampling system, as the Hoke tube type is expensive, it

has to be cleaned-up before reutilization, and additional costs are added to the transportation of cleaned tubes from Argentina to Brazil.

Due to the disadvantages of the actual UF<sub>6</sub> sampling method, ABACC has developed a method (named ABACC-Cristallini Method) of sampling UF<sub>6</sub> for enrichment determination. The new method uses a fluorothene P-10 tube type containing alumina pellets that absorb and hydrolyze UF<sub>6</sub> directly during the sampling. The alumina pellets retain up to few hundreds milligrams of U (in a solid compound – UO<sub>2</sub>F<sub>2</sub>) without the need of using liquid nitrogen during sampling. With this new method the UF<sub>6</sub> sample content left at the installation (archive sample) will be lower and less reactive as the actual, the laboratory procedures for manipulating the sample will be much easier (no need for hood, gas sampling, vacuum system, nitrogen cleaning, etc), the residual uranium retained at the laboratory will be much lower, the sampling device is less expensive, there will be saves in transport cost as well, and it is relatively safer concerning radiological protection aspects during transportation. Figure 1 shows the two types of UF<sub>6</sub> sampling device and the alumina pellets.

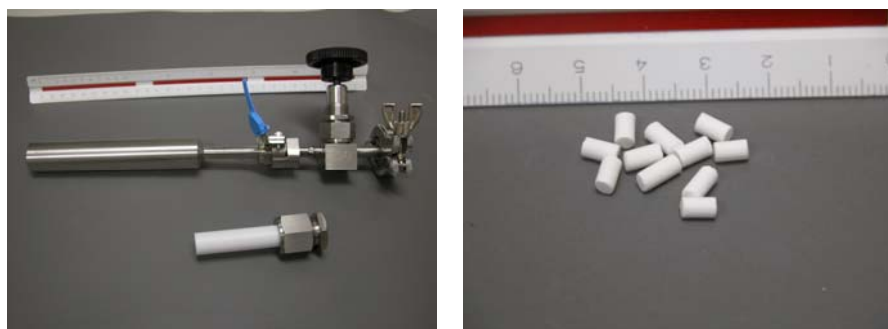


Figure 1: UF<sub>6</sub> sampling devices – Hoke tube / fluorothene P-10 tube and alumina pellets

### 1.1 Reaction Mechanism

Chemical traps are commonly employed in enrichment plants to remove UF<sub>6</sub> from various gas streams of the process. The effectiveness of the chemical trap largely depends on the particular absorbing material that is used to fill the trap. As these traps are the final barriers between the process and the environment, they should assure the total elimination of UF<sub>6</sub>. The materials normally used in these traps are alumina (Al<sub>2</sub>O<sub>3</sub>) and sodium fluoride (NaF). The election of the appropriate absorber is made comparing different performance factors as: reaction kinetics, absorber regeneration / uranium recovery requirements, absorbing capacity, pressure losses, and effects of other system components such as HF and F<sub>2</sub> [1,2]. These factors do not have the same importance in every application. For the UF<sub>6</sub> sampling method proposed in this paper the predominant factors are reaction kinetics and uranium recovery.

The sodium fluoride trapping mechanism involves the reaction of UF<sub>6</sub> to form a solid complex, according to the following reaction:



The uptake of uranium hexafluoride by sodium fluoride is a chemisorption process forming an UF<sub>6</sub>·2NaF complex. This reaction can be readily reversed heating the complex to around 350°C and recovering the uranium as UF<sub>6</sub>. Also, it can be dissolved in water, obtaining a solution with the uranium and a high NaF saline content. If the NaF, with the retained complex, is dissolved in water, the high fluoride quantity present in the resulting solution should be eliminated completely by successive evaporations, because its presence affects the isotopic analysis.

On the other hand, the effectiveness of the alumina relies on the hydrolysis of the UF<sub>6</sub> with the available lattice water. The reaction is the following:



The  $\text{UF}_6$  alumina trapping mechanism depends on the hydrolysis reaction with subsequent retention of the uranyl fluoride ( $\text{UO}_2\text{F}_2$ ) in the porous structure. The  $\text{UO}_2\text{F}_2$  is a non-volatile solid and soluble in water. This facilitates the preparation of an adequate solution to carry out the isotopic analysis.

Due to the characteristics described above, alumina pellet was chosen as the material to be used for  $\text{UF}_6$  sampling.

Some initial quantity of water is essential for the alumina loading mechanism, being a content of 3 to 4% near the optimum [1]. However, if the alumina, in the form of pellet, contains too much water, greater than 7 to 8%, it will be so reactive that the pores leading to the interior of the pellet will become prematurely plugged, and the uranium loading is largely confined to the periphery of the pellet. In this case, the average load can be considerably lower than otherwise expected. These facts shall be taken into account for the material specification and sampling setup.

## 1.2. Alumina Characteristics

The tested aluminum oxide is type gamma, used as catalyst support, bimodal, with a very high specific surface. The material has form of pellets of 1/8", (cylinders of 3 mm diameter and 5-6 mm high – see Figure 1) with an apparent density of  $0.39 \text{ g/cm}^3$  and a total pore volume of  $1.14 \text{ cm}^3/\text{g}$ . The specific area is around  $250 \text{ m}^2/\text{g}$  measured by the BET Method.

The absorbed water was determined as 0.07% by heating at  $120^\circ\text{C}$  for 2 hours. The crystallization water was determined as 4.5% by heating at  $1200^\circ\text{C}$  for 2 hours. Considering the high specific area of the pellets, the material was not exposed to the atmosphere and maintained in its original and hermetic container in order to low the absorbed humidity.

To verify that the alumina did not contribute with any impurity, especially uranium, which could interfere or cause error in the determination of the U isotopic composition, a blank pellet was analyzed. Several washes with distilled water and  $\text{NO}_3\text{H}$  1M, as is applied to the recovery of the  $\text{UO}_2\text{F}_2$ , were carried out. The blank solutions were measured using the Total Reflection X Ray Fluorescence (TXRF) technique. The impurities detected by TXRF in the blank were the following:

- Iron:  $0.15 \text{ }\mu\text{g/ml}$ , equivalent to  $1.8 \text{ }\mu\text{g/g}$  in the alumina;
- Potassium and Calcium:  $0.5 \text{ }\mu\text{g/ml}$ , equivalent to  $6 \text{ }\mu\text{g/g}$  in the alumina;
- Uranium was not detected, being  $0.02 \text{ }\mu\text{g/ml}$  in the blank, equivalent to  $0.2 \text{ }\mu\text{g/g}$  in the alumina, the detection limit of the method.

Due to the presence of HF generated during the hydrolysis, the alumina presents certain solubility that increases with time and heating. Under the conditions selected for  $\text{UO}_2\text{F}_2$  recovery, it was determined that the total alumina dissolution amounts to 0.05%. Such a low quantity does not cause any problem to the isotopic analysis by mass spectrometer.

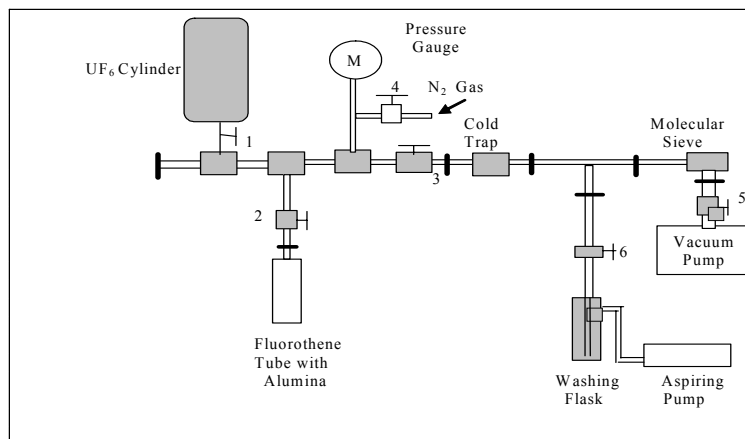
## 2. Experimental setup

Figure 2 shows the equipment setup scheme used for the  $\text{UF}_6$  sampling with alumina pellets at the Laboratorio de Control Químico y Físico – Combustibles Nucleares – Centro Atómico Constituyentes – Comisión Nacional de Energía Atómica - Argentina.

The  $\text{UF}_6$  cylinder and the fluorothene tube loaded with the alumina pellets are connected to the manifold. The loaded fluorothene tube, with its plug and nuts, are tare before its connection to the system in order to estimate the quantity of uranium retained in each experiment.

The manifold is dried out before starting each test by applying vacuum and heating ( $80\text{-}90^\circ\text{C}$ ) during 30 minutes. Then, the  $\text{UF}_6$  cylinder valve is opened in order to obtain the desired pressure and valve 2 is opened to allow that the alumina pellets enter in contact with the  $\text{UF}_6$ . After having elapsed the elected time for the test, valve 1 and 2 are closed. To remove the  $\text{UF}_6$  from the system, valve 3 is opened, being  $\text{UF}_6$  retained by the cold trap with liquid nitrogen. Valve 2 is also opened to be sure that there are no detectable gases in the fluorothene tube. Valve 4 is then opened to allow the entrance of nitrogen gas for equalizing the internal pressure to the atmospheric pressure. Finally valve 2 is closed and the fluorothene tube is disconnected from the manifold. It is closed with the respective plug and

nuts and weighted to know the uranium mass retained in the alumina. In general, the procedure described above was applied on all experiments, with some variants according to the kind of test being done.



**Figure 2:** equipment setup for  $UF_6$  sampling with alumina pellets

### 3. Experiments

Various experiments with fluoroethene tube containing alumina pellets were carried out using  $UF_6$  samples, which were obtained from ABACC's inspections, having U enrichments between 0.31 to 4.05wt%. The first 5 experiments were carried out to set up the system. During these experiences some smaller losses took place, and the data was not reliable, but gave evidence that the uranium was retained by the alumina. Then the experiments were carried out at several constant pressures in the system to evaluate the quantity of uranium retained at a fixed time. Data were also collected when the  $UF_6$  tubes were becoming empty, giving knowledge about the alumina behavior. In all the experiments, the fluoroethene tube was loaded with 1g of alumina pellets without any previous treatment. Some representative experiments are described below.

#### 3.1. Experiments carried out at decreasing pressure

Experiments with decreasing pressure in the experimental setup are presented in Table 1. Figure 3 shows the evolution of pressure versus time for one specific test.

The ideal gas equation was used ( $P.V = n.R.T$ ) to estimate the free  $UF_6$  mass in the system that could be absorbed by the alumina. This value is compared to the measured retained mass, and was calculated with the following data:

- System Volume: 0.103 liters (it is the volume of the equipment between the valves 1, 2, 3 and 4 - see Figure 2);
- Test Temperature: 80°C;
- Pressure (mb) is the difference between the initial and final pressure of the system.

Test Number	Alumina Mass (g)	Initial Pressure (mb)	Final Pressure (mb)	Contact Time (min)	Calculated U Mass (mg)	Retained U Mass (mg)	U Mass / $Al_2O_3$ Mass Ratio
#14	1.072	30	2.9	30	227	193 (85%)	0.18
#18	1.033	15	2.4	12	105	75 (71%)	0.07
#20	1.028	108	26.1	80	686	465 (68%)	0.45

**Table 1:** experiments carried out at decreasing pressure

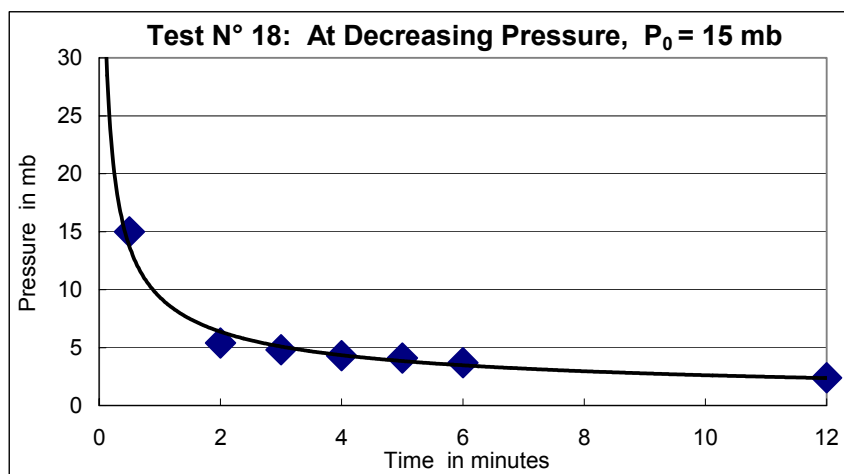


Figure 3: experiment at Decreasing Pressure

Test #14 shows that a high percentage of  $UF_6$  (85%) was retained by the alumina pellets. Test #18 showed a smaller retention (71%), but the contact time was 12 minutes compared to 30 minutes in Test #14. Test #20 showed 68% retention for 80 minutes, but the final pressure of the system was 10 times higher than the other experiments because the alumina was coming closer to its loading limit.

According to Schultz [2] the maximum saturation load, expressed as the uranium to alumina mass ratio is between 0.4 and 0.5. The maximum values obtained in this work were something superior (up to 0.6 for all tests).

It is necessary to highlight that in Test #18, with an initial pressure as low as 15 mb, the pressure had a quickly decreasing because the tube of  $UF_6$  was almost empty, but in only 12 minutes 75 mg of uranium were retained by the alumina pellets. This quantity of uranium is more than enough to carry out an isotopic analysis. This is an indicative that it is possible to sample  $UF_6$  from almost empty containers or plant pipes that have very low pressure.

### 3.2. Experiments carried out at constant pressure

Several constant pressures in the system were set to evaluate the amount of uranium retained in a fixed time and the maximum uranium load for the alumina saturation as well. Table 2 presents some relevant data of the tests performed and Figures 4 and 5 show the pressure evolution as function of time.

The pressure of the system was maintained as much as possible constant by regulating the  $UF_6$  Hoke tube valve. After 60 minutes, the valve was closed but the pressure recording continued for 20 minutes more. It is clearly observed that the pressure keeps falling during the following 20 minutes in the case where the alumina is less loaded (Test #12, Figure 4), indicating a continuous  $UF_6$  absorption. In opposite, Test #16 (Figure 5) shows that the alumina pellets seem to be practically saturated, with a very low decrease of the system pressure, and uranium to alumina mass ratio of 0.62.

Test Number	Alumina Mass (g)	Initial Pressure (mb)	Final Pressure (mb)	Contact Time (min)	Calculated U Mass (mg)	Retained U Mass (mg)	U Mass / $Al_2O_3$ Mass Ratio
#12	1.031	10	3.1	60 + 20	315	213	0.21
#17	1.067	25	17.1	60 + 20	662	447	0.42
#13	1.018	50	42.3	60 + 20	773	523	0.51
#16	1.040	100	95.3	60 + 20	947	640	0.62

Table 2: Experiments carried out at constant pressure

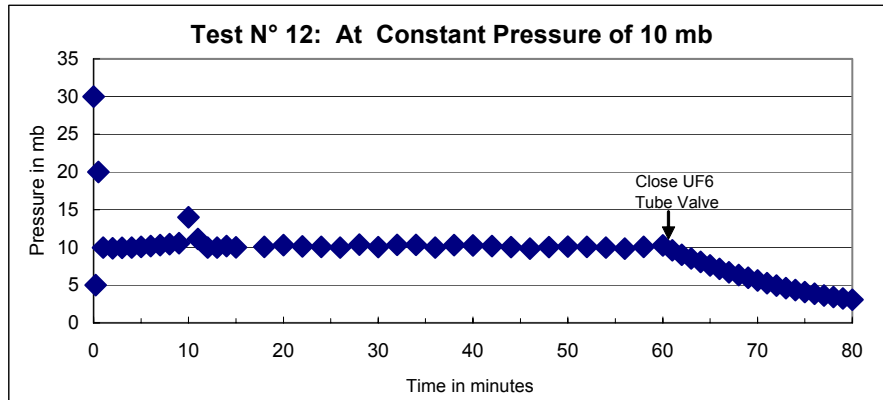


Figure 4: experiment at constant pressure test#12

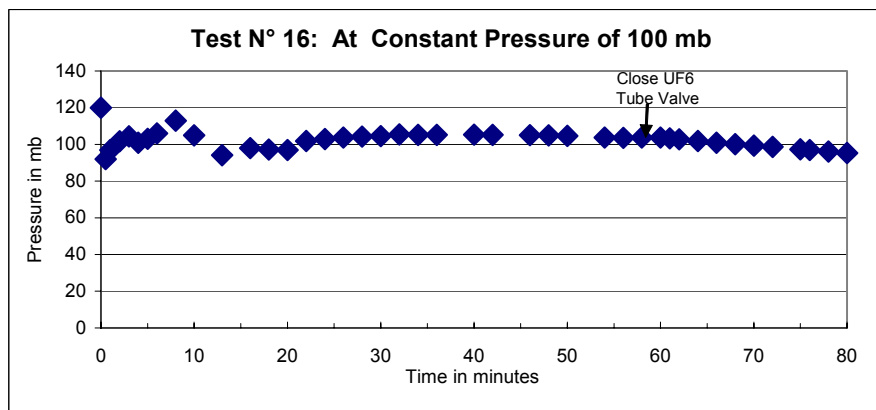


Figure 5: experiment at constant pressure test#16

It is evident that the reaction is quite quick despite of the several steps that are involved in this kind of gas-solid reactions:

- diffusion of the  $UF_6$  molecule from the bulk gas stream to the external pellet surface;
- diffusion into the porous structure;
- adsorption on the interior surfaces;
- reaction with the absorbing material;
- diffusion of the  $UF_6$  molecule through the complex layers to unreacted absorbers.

In Test #12 at a very low pressure of only 10 mb, within 60 minutes, 213 mg of uranium were retained. The fourth part of this U mass is more than enough to perform an isotopic analysis. Therefore, 15 to 30 minutes seems to be an appropriate time interval for sampling lines with  $UF_6$  pressures among 10-100 mb, and using only 1gram of alumina pellets as specified by this method.

### 3.3. Evaluation of the uranium mass retained as function of the system pressure

Figure 6 shows the uranium mass retained by 1 gram of alumina versus the  $UF_6$  pressure inside the system. In this graph the value obtained at constant pressure tests were used (see Table 2). A similar curve can be obtained plotting the  $U/Al_2O_3$  mass ratio versus the  $UF_6$  pressure. The retained uranium was determined by the weight difference of the fluorothene tube loaded with the alumina pellets, before and after each test. Some data were also checked by measuring the uranium content by Davies & Gray Method.

The saturation value of  $U/Al_2O_3$  mass ratio seems to be next to 0.62, which is the highest value obtained by Test #16, carried out at 100 mb of pressure during 80 minutes.

This is a high uranium retention value obtained with the alumina pellets used. This value is superior to the one obtained by Schulz [2], where values of 0.6-0.7 are consigned for the  $UF_6/Al_2O_3$  mass ratio saturation, which are equivalent to 0.4-0.5 for the  $U/Al_2O_3$  mass ratio.

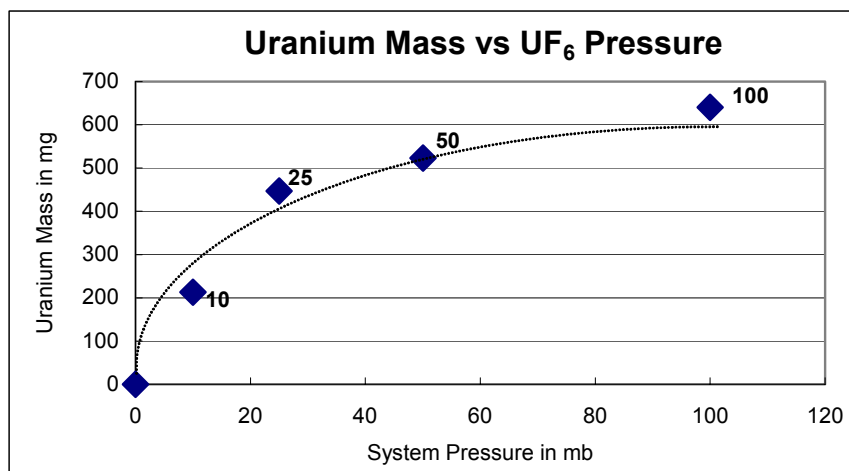


Figure 6: uranium mass retained in function of the system pressure

### 3.4 Recovery of the uranium retained in the alumina

As the uranyl fluoride ( $UO_2F_2$ ) is a very soluble salt, the uranium retained in the alumina pellets can be removed with distilled water without any effort. However, due to the high material porosity, several washes with small water volumes and even some washes with 1M nitric acid are necessary for complete uranium dissolution. A high solution acidity, intense heating or prolonged contact times are not recommended, because the alumina is partially dissolved and the final solution will have a high aluminum content that shall affect the isotopic analysis procedure.

After some tests, the uranium recovery procedure adopted for its simplicity and acceptable efficiency is the following:

- the pellets loaded with the  $UO_2F_2$  are placed in an Erlenmeyer, added 5 ml of distilled water, stirred during 3 minutes and then left to settle other 5 minutes;
- the solution so far obtained is separated and the extraction process is repeated 3 times more, the first one with distilled water and the two remaining with 1M  $NO_3H$ ;
- with these 4 serial washing it is possible to recover around 85% of the original uranium, without dissolving an appreciable quantity of alumina. As the solution has certain turbidity due to the presence of very fine alumina in suspension, it is necessary to centrifuge the solution to separate the alumina.

This solution or an aliquot of it is evaporated to eliminate the fluoride that interferes in the isotopic analysis, and finally the dried product is dissolved in 1M  $NO_3H$  to obtain a solution containing around 5 mg U/ml for the isotopic analysis.

### 3.5. Comparison of the isotopic analysis data

Available  $UF_6$  samples were sub-sampled [3,4] in order to hydrolyze them directly for isotopic analysis. The isotopic analysis from the solution obtained by the  $UF_6$  direct hydrolysis method is compared to the isotopic analysis obtained from the alumina pellet method.  $UF_6$  standard samples were also utilized for this comparison between the two methods. Table 3 presents the results of this comparison.

No significant differences in the isotopic analysis by mass spectrometer were found between the direct hydrolysis method and the alumina pellet method. This is a logical result as the blank alumina pellet analysis by TXRF did not detect the presence of uranium. The presence of small quantities of alumina dissolved in the solution did not generate any difficulty in the isotopic analysis as well.

Sample	U isotopic results actual method (UF <sub>6</sub> direct hydrolysis) (wt %)	U isotopic results new method (UF <sub>6</sub> alumina pellets sampling) (wt %)	Difference in the U-235 (wt%)
#1	<b>U-235 = 1.468 ± 0.002</b> U-234 = 0.0092 ± 0.0002 U-236 < 0.0007	<b>U-235 = 1.467 ± 0.002</b> U-234 = 0.0091 ± 0.0002 U-236 < 0.0007	<b>-0.001</b>
#2	<b>U-235 = 2.168 ± 0.003</b> U-234 = 0.0146 ± 0.0003 U-236 < 0.0005	<b>U-235 = 2.168 ± 0.002</b> U-234 = 0.0145 ± 0.0003 U-236 < 0.0005	<b>0</b>
#3	<b>U-235 = 4.046 ± 0.005</b> U-234 = 0.0378 ± 0.0002 U-236 < 0.0005	<b>U-235 = 4.045 ± 0.004</b> U-234 = 0.0378 ± 0.0002 U-236 < 0.0005	<b>-0.001</b>
#4	<b>U-235 = 1.751 ± 0.005</b> U-234 = 0.011 ± 0.001 U-236 < 0.001	<b>U-235 = 1.753 ± 0.003</b> U-234 = 0.011 ± 0,001 U-236 < 0.001	<b>0.002</b>
#5	<b>U-235 = 1.624 ± 0.002</b> U-234 = 0.011 ± 0.001 U-236 < 0.003	<b>U-235 = 1.621 ± 0.002</b> U-234 = 0,012 ± 0,001 U-236 < 0.003	<b>-0.003</b>
#6	<b>U-235 = 1.187 ± 0.002</b> U-234 = 0.009 ± 0.001 U-236 < 0.003	<b>U-235 = 1.183 ± 0.003</b> U-234 = 0.009 ± 0,001 U-236 < 0.003	<b>-0.004</b>
IRMM 020	<b>U-235 = 0.210 ± 0.001</b> U-234 < 0.003 U-236 = 0.029 ± 0.001	<b>U-235 = 0.209 ± 0.001</b> U-234 < 0.003 U-236 = 0.029 ± 0.001	<b>-0.001</b>
IRMM 022	<b>U-235 = 0.720 ± 0.002</b> U-234 = 0.005 ± 0.001 U-236 < 0.003	<b>U-235 = 0.720 ± 0.003</b> U-234 = 0.005 ± 0.001 U-236 < 0.003	<b>0</b>
IRMM 023	<b>U-235 = 3.274 ± 0.004</b> U-234 = 0.033 ± 0,001 U-236 < 0.003	<b>U-235 = 3.268 ± 0.003</b> U-234 = 0.033 ± 0,001 U-236 < 0.003	<b>-0.006</b>
IRMM 029	<b>U-235 = 4.173 ± 0.006</b> U-234 = 0.080 ± 0.001 U-236 = 0.989 ± 0.001	<b>U-235 = 4.165 ± 0.004</b> U-234 = 0,079 ± 0,001 U-236 = 0.989 ± 0.002	<b>-0.008</b>

**Table 3:** Isotopic analysis data comparison

#### 4. Future Work

As demonstrated in the previous section, the UF<sub>6</sub> sampling method using alumina pellets improves the safeguard measurement procedure applied to enrichment facilities.

Before implementing the new method as a routine procedure the following steps are foreseen as necessary:

- to perform a demonstration exercise at the enrichment facility in order to the operator evaluate its impact to the systems and operation;
- to certify the method by an independent international laboratory;
- to agree with IAEA, Operators and National Authorities for implementing the proposed UF<sub>6</sub> sampling method for U isotopic determination.

The first step is already being done at one of the enrichment facility laboratories in Brazil. The preliminary results indicate that the operator is comfortable with the method and is reproducing the positive results obtained previously at laboratory.

The second step will be performed by a laboratory out of South America that performs nuclear material measurements and is a nuclear material certifier.

The third step will be done after the conclusion of the two previous ones.



## 5. Conclusion

ABACC proposed a new method of sampling UF<sub>6</sub> for enrichment determination (ABACC-Cristallini Method) using a fluorothene tube containing alumina pellets that absorb and hydrolyze UF<sub>6</sub> directly during the sampling process.

The method was demonstrated at laboratory where an experimental system was set up.

The alumina pellets used are commercial catalyst support, and one gram of these pellets without any previous treatment was determined as enough for sampling UF<sub>6</sub> for enrichment determination.

The experimental results show that 10 to 30 minutes of contact of the UF<sub>6</sub> with the pellets would retain enough quantities of uranium for isotopic analysis, even for system pressures lower than 10 mb.

The recovery of the uranium is simple and quick. It does not require any special equipment and it can be done in a radiochemical hood as well as in a laboratory bench, since no gas is liberated when the fluorothene tube is open.

The method shall be further qualified by the enrichment facility operator, and certified by an international independent laboratory.

The method responds appropriately and its implementation as routine procedure to enrichment facility safeguard does not present any foreseen difficulty.

It is evident the advantages that the new method presents compared to the actual method as: the UF<sub>6</sub> sample content left at the installation (archive sample) will be lower and less reactive, the laboratory procedures for manipulating the sample will be much easier, the residual uranium retained at the laboratory will be much lower, the sampling device is less expensive, there will be saves in transport cost as well, and it is relatively safer concerning radiological protection aspects during transportation.

## 6. References

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