ENTE PER LE NUOVE TECNOLOGIE, L'ENERGIA E L'AMBIENTE

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LABORATORIO NAZIONALE PER LA CARATTERIZZAZIONE DEI RIFIUTI RADIOATTIVI

L.E.T. Project

8° Report

by the Head of the Section

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1.0 Background

This document reports the results of the test with Uranium, done on May 21, 1998, and May 25, 1998, using materials supplied by Eucan Tech.

1.1 Characterisation of materials

Two packages, shipped from Eucan:
- FMKN (270 g)
- FMAN (270 g)
were mixed, sampled and analysed.

The five packages with no marking containing the "grey lead" (730g), were mixed, sampled and analysed.

Also the Uranium nitrate was sampled and analysed.

The composition of the three kind of starting materials resulted as reported below (table 1).

Table 1: Composition of starting materials

	FMKN/AN	Mix A	U nitrate
Elements	%	%	%
Ag	0,01	22,09	
Pb	0,01	40,10	
Sb	0,00	0,01	
Ir	0,00	0,00	
Pt	0,00	0,00	
Rh	0,00	0,00	
Ru	0,00	0,00	
Au	0,00	0,00	
Pd	0,00	0,00	
Ni	0,00	0,01	
Sc	0,00	0,00	
U	0,00	0,00	53,42
Th	0,00	0,00	

2.0 The Test with U (May 21, 1998)

The materials above characterised were used in the following formulation:

Components	Amounts (g)
U nitrate	10,02592
Mix A	733,25
FMKN/AN	540,08
TOTAL	**1280,36**

The mixture was homogenised for 4 hours and after that a sample of about 10 g was taken and analysed.
The results showed, in the remaining 1270,01 g of firing mixture, a composition in compliance with that calculated (table 2), as awaited.

Table 2 : Composition of input materials

	IGNIT. PW Calculated	IGNIT. PW Analysed
Elements	(g)	(g)
Ag	141,04	132,10
Pb	246,89	226,10
Sb	0,04	0,07
Ir	0,01	0,01
Pt	0,01	0,02
Rh	0,01	0,04
Ru	0,02	0,02
Au	0,01	0,00
Pd	0,01	0,01
Ni	0,14	0,23
Sc	0,00	0,00
U	5,32	4,39
Th	0,01	0,05
TOTAL Me	**393,50**	**363,06**

The mixture was poured into reactor. The smoke traps were filled both with 50 litres of distilled water. The reaction was ignited with 41,5 g of FMKN/AN and left to be completed itself.
The reaction lasted about 4÷ 5 minutes.
The day after the reactor was opened and 160,88 g of slag and 399,98 g of metal cupel were recovered. The water of traps was recovered and stored.

The SLAG was finely ground and sampled. Two samples were attacked several times with perchloric acid until complete dissolution and analysed. The final undissolved residues was negligible.

The metal cupel was treated with 2 litres of nitric acid 8 M, under eating. The solution (called cup 1) was recovered and the white residues still remaining was attacked with 2 litres of ammonium acetate. The obtained solution (called cup 2) was recovered and the residues still remained was treated with 0,1 of perchloric acid (obtaining solution cup 3). The final undissolved residues was negligible.

All the recovered solutions (cup 1, cup 2, cup 3) were analysed.

With the data obtained from the analysis it has been made the following balance:

OUTPUT

Balance IN/OUT

	Cupel	Slag	W trap 1	W trap 2	TOTAL OUT	TOTAL IN	Element
Element	(g)	(g)			(g)	(g)	
Ag	127,57	3,79	n. a.	n. a.	131,36	132,10	Ag
Pb	185,94	21,09	n. a.	n. a.	207,03	226,10	Pb
Sb	0,00	0,02	n. a.	n. a.	0,02	0,07	Sb
Ir	0,00	0,00	n. a.	n. a.	0,00	0,01	Ir
Pt	0,00	0,00	n. a.	n. a.	0,00	0,02	Pt
Rh	0,00	0,01	n. a.	n. a.	0,01	0,04	Rh
Ru	0,00	0,01	n. a.	n. a.	0,01	0,02	Ru
Au	0,01	0,00	n. a.	n. a.	0,01	0,00	Au
Pd	0,00	0,03	n. a.	n. a.	0,03	0,01	Pd
Ni	0,01	0,06	n. a.	n. a.	0,07	0,23	Ni
Sc	0,00	0,00	n. a.	n. a.	0,00	0,00	Sc
U	0,00	3,07	n. a.	n. a.	3,07	4,39	U
Th	0,00	0,03	n. a.	n. a.	0,03	0,05	Th

n.a. : not analysed.

3.0 The Test with U (May 25, 1998)

The materials above characterised were used in the following formulation:

Components	Amounts (g)
U nitrate	10,0214
Mix A	730,01
Silica	50,15
FMKN/AN	540,05
TOTAL	**1330,23**

The mixture was homogenised for 4 hours and after that a sample of about 10 g was taken and analysed.
The results showed, in the remaining 1320,19 g of firing mixture, a composition in compliance with that calculated (table 2), as awaited.

Table 2 : Composition of input materials

Elements	IGNIT. PW Calculated (g)	IGNIT. PW Analysed (g)
Ag	135,71	136,41
Pb	237,57	228,59
Sb	0,04	0,04
Ir	0,01	0,01
Pt	0,01	0,01
Rh	0,01	0,01
Ru	0,02	0,01
Au	0,01	0,00
Pd	0,01	0,00
Ni	0,13	0,13
Sc	0,00	0,00
U	5,12	4,56
Th	0,01	0,04
TOTAL Me	**378,63**	**369,80**

The mixture was poured into reactor. The smoke traps were filled both with 50 litres of distilled water (the same of previous test). The reaction was ignited with 87 g of FMKN/AN and left to be completed itself.
The reaction lasted about 4÷ 5 minutes.
The day after the reactor was opened and 219,92 g of slag and 372,62 g of metal cupel were recovered. The water of traps was recovered and stored.

The SLAG was finely ground and sampled. Two samples were attacked several times with perchloric acid until complete dissolution and analysed. The final undissolved residues was negligible.

The metal cupel was treated with 2 litres of nitric acid 8 M, under eating. The solution (called cup 1) was recovered and the white residues still remaining was attacked with 2 litres of ammonium acetate. The obtained solution (called cup 2) was recovered and the residues still remained was treated with 0,1 of perchloric acid (obtaining solution cup 3). The final undissolved residues was negligible.

All the recovered solutions (cup 1, cup 2, cup 3) were analysed.

h the data obtained from the analysis it has been made the following balance:

	OUTPUT				Balance IN/OUT		
	Cupel	Slag	W trap 1	W trap 2	TOTAL OUT	TOTAL IN	Element
Element	(g)	(g)			(g)	(g)	
Ag	127,17	5,36	0,07	n. a.	132,6	136,41	Ag
Pb	161,94	45,73	0,43	n. a.	208,1	228,59	Pb
Sb	0,00	0,03	≤ 0,01	n. a.	0,04	0,04	Sb
Ir	0,00	0,00	≤ 0,01	n. a.	0,00	0,01	Ir
Pt	0,00	0,00	≤ 0,01	n. a.	0,01	0,01	Pt
Rh	0,00	0,01	≤ 0,01	n. a.	0,01	0,01	Rh
Ru	0,00	0,01	≤ 0,01	n. a.	0,01	0,01	Ru
Au	0,01	0,00	≤ 0,01	n. a.	0,01	0,00	Au
Pd	0,00	0,00	≤ 0,01	n. a.	0,00	0,00	Pd
Ni	0,01	0,06	≤ 0,01	n. a.	0,07	0,13	Ni
Sc	0,00	0,00	≤ 0,01	n. a.	0,00	0,00	Sc
U	0,01	2,49	≤ 0,01	n. a.	2,50	4,56	U
Th	0,00	0,02	≤ 0,01	n. a.	0,02	0,04	Th

n.a. : not analysed.

4.0 CONCLUSION

Taking into account that all the analytical determinations have a reproducibility of about 5÷10 %, the balance seems to demonstrate, except for Uranium, that all the input materials are recovered with any changes.

Nevertheless it has to be specified that:

- The difficulties to ignite the powder made necessary to use some of FMKN/AN on the top of ignition powder, opening several time the reactor;
- The reaction had "strange" behaviour and the slag was found not only on the top of the cupel, but spread in all the part of the reactor (mainly in the second test);
- The recovery of slag was difficult and perhaps not quantitative.

In any case the test procedures have to be validate with good materials (that make sure ignition) and in a formulation that make possible the slag recovery in a simple way.

Foot note: The composition of slag was exactly the same of the previous tests. Therefore, if we would envisage that the slag has not quantitatively recovered and his weight was more or less the same of previous test, the balance of Uranium should be correct. It is a "strange" result that need to be investigated.

5.0 Next tests

For the next tests, if it will be, it is necessary to start with standard and certified materials.