



Forensic investigations in the case of Mr Louvet: Radioactivity determination in Mr Louvet belongings

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1. Introduction

The Institute of Radiation Physics (thereafter IRA) received the personal belongings of Mr Louvet by the University Centre of Legal Medicine (CURML) for investigating a potential radioactivity contamination with a particular attention to polonium-210 (^{210}Po). Because more than 20 half-lives have passed between a potential contamination by ^{210}Po and the present measurements, it is necessary to apply low level techniques to the determination of unsupported ^{210}Po (^{210}Po not supported by its grandmother ^{210}Pb).

Materials and Methods

Surface contamination measurements

Surface contamination measurements were performed using a Berthold LB-122 monitor in β/γ mode and a Comoro monitor in α mode. All the set of personal belongings was scanned manually for radioactive contamination.

Gamma spectrometry measurements

For gamma spectrometry measurements, personal belongings were pooled into 11 samples as shown in Figure 1. Each sample was then measured on an HPGe gamma spectrometer (measuring time $> 24\text{h}$). Artificial radionuclides, such as ^{60}Co , ^{134}Cs and ^{137}Cs were investigated, as well as ^{210}Pb .

Figure 1. Pictures of Mr Louvet belongings pooled in 11 samples for gamma spectrometry measurements.





Alpha spectrometry measurements

Sampling: we have proceeded to the ^{210}Po determination based on the fact that some of them might have been worn shortly before his death. Thus the first samples are: a sample taken from the collar of a sportswear with strong dirt stains, a sample from the front tights of the same sportswear, a sample from the front tights of a woolen pant, 2 pills each of a mixture of different medicines (about 38 g) and two samples taken from an underwear that appeared obviously worn. One of the two samples has been taken from a stained part of the front of the underwear, identified possibly as a urine stain.

Following the results of the ^{210}Po determination (see results) we have proceeded to the sampling of 6 aliquots from the remaining of this underwear. These samples, as the two before, contain about 3-4 g of cotton wool. The sportswear is a mixture between cotton wool and synthetic fibers.

We have then taken a sample from a hospital cap. This sample has been taken by cutting around a blood stain and weighted 0.7041 g. This 3rd samples batch contained also a sample from another underwear (probably not worn), a second sample from the collar of the sportswear, a sample made of the bristles of two toothbrushes (one manifestly used) and a reactants blank (H_2SO_4 : 10 ml; HNO_3 : 25 ml; NH_4OH : 40 ml, Fe:20 mg).

The forth batch of samples contained two aliquots of a third underwear, an aliquot from a kefieh taken around a stain (probably blood stain, 2.2540 g) and a sample from a "Russian" chapka (internal band in contact with the head) known to have been worn by Mr Louvet shortly before his death.

The 5th batch of samples contained 3 underwears from 3 different IRA collaborators.

The 6th batch of samples contained an aliquot from a sock possibly worn, a large piece from a long johns (112.7529 g), a reactants blank and two new underwears bought directly from a shop (Bon Genie, Lausanne) of the brand Zimmerli (the same as the one of Mr Louvet's underwear) and one underwear of the brand Hanro.

The 7th batch of samples contained an aliquot (far from the blood stain) of the hospital cap, a supplementary aliquot of the Russian chapka, a sample formed by the bristles of the toothbrush of an IRA collaborator and three aliquots of long johns (taken along the leg).

The 8th batch contained a sample formed by 4 pieces of cotton wool taken from a child's drawing sampled around stains (possibly saliva, vomit and blood, total 1.4631 g), a sample taken from the interior and from the superior band (stained) of an old slipper, a sample from a new sock (not worn, still attached by thread) and a sample of reactants blank.

The 9th batch contained two more samples from the collar of the sportswear, an aliquot of the sportswear taken from the back, around a stain (possibly blood, 0.7565 g) and three more aliquots from the interior band in contact with the skull of the Russian chapka.

The 10th and last batch contained smear of personal belongings such as glasses, a blank smear, a samples formed by the bristles of a toothbrush of an IRA collaborator, a sample of the hospital cap and an aliquot of the child drawing (out of the stains) and a reactants blank.

All the sampling has been carried out with the aim to find contaminated samples, thus the choice of "stained" samples. Nevertheless cotton wool may contain a certain amount (not known) of $^{210}\text{Pb}/^{210}\text{Po}$ because of exposition of cotton wool to atmospheric deposition during cultivation because cotton wool balls have a large adsorption surface. We therefore decided to measure some blank cotton (not known to have been exposed to unsupported ^{210}Po).

^{210}Po determination: the method is presented in detail in supplementary material. Briefly, the aliquots are traced with 50 mBq of ^{209}Po and digested by conc. H_2SO_4 at about 70°C to provoke the dehydration of the cotton wool (charred sample), then the charred sample is oxidized cautiously with portions of conc. HNO_3 (strong emission of NO_x vapors). When the NO_x production has reduced, the oxidation is pursued during two more hours under a glass watch on a hot plate. After this treatment aiming to remove a large part of organics by wet ashing, the complete removing of the organic matter is obtained through a digestion in a pressurized microwave digester (MLS Ultraclave IV). A clear solution is obtained. If synthetic fibers are present, the solution must be filtered because they are not soluble. ^{210}Po is co-precipitated from the acidic solution along with iron hydroxide. After centrifugation, the precipitate is dissolved in 80 ml 1 M HCl, 500 mg of ascorbic acid is added and

polonium is spontaneously electrodeposited on a silver disc during 4 hours at 50°C (or overnight at room temperature).

Determination of ^{210}Po supported by ^{210}Pb in the most active samples: the method is presented in detail in supplementary material. Briefly, after ^{210}Po electrodeposition the solution is evaporated to dryness, HNO_3 portions are added to destroy all remaining ascorbic acid. The residue is dissolved in 10 ml 9 M HCl and this solution is passed through an anionic chromatography column (2 g of AG1x8) to extract $[\text{PoCl}_6]^{2-}$. ^{210}Pb will pass in the elution solution which is evaporated to dryness. The residue is dissolved in 80 ml 1M HCl and let in the refrigerator during at least 3 months (36.6% re-growth of ^{210}Po from ^{210}Pb) afterwards, 50 mBq of ^{209}Po is added and polonium is electrodeposited on a silver disc as before.

Biokinetic model of ^{210}Po and urinary excretion

The systemic biokinetic model of polonium proposed by Leggett and Eckerman [1] (see Figure 2) has been implemented in the simulation modeling tool Ecolego in order to calculate the typical retention of ^{210}Po in organs and tissues in case of poisoning. In the latter situation, ingestion is the most probable route of intake. Therefore, the systemic biokinetic model was coupled to the human alimentary tract model of ICRP 100. Absorption of ^{210}Po is assumed to occur exclusively from the small intestine and is characterized by the fractional intestinal absorption $f_1=0.1$ (inorganic form) or $f_1=0.5$ (organic form). The f_1 value is defined as the fraction of the activity leaving the stomach that is subsequently transferred to blood by absorption from the small intestine. Using this model with $f_1=0.1$, the daily urinary excretion after acute ingestion of 1 Bq of ^{210}Po was determined. Based on activity estimates of ingested ^{210}Po in case of the poisoning of Mr. Litvinenko, the level of activity that we might find in urine and belongings, especially underwear, in case of Mr Louvet poisoning was estimated.

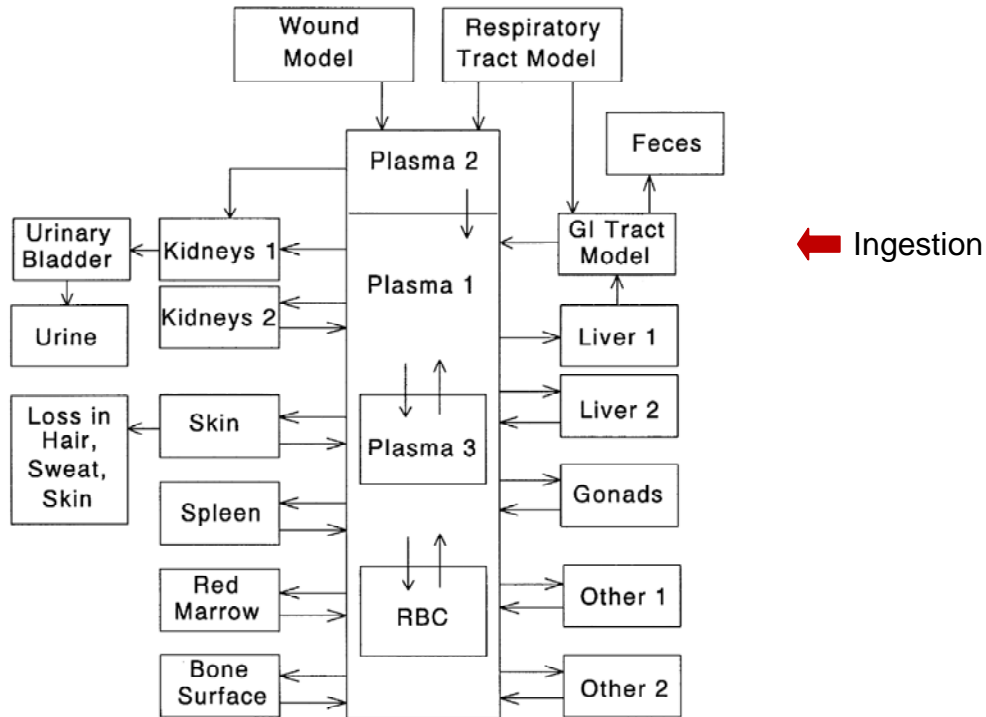


Figure 2. Compartments of the systemic model of polonium and connection with the model of the gastrointestinal tract.

Results and discussion

Surface contamination measurements

Surface contamination measurements did not show the presence of radioactive contamination in the personal belongings.

Gamma spectrometry measurements

Results of gamma spectrometry measurements are given in Table 1. None of the samples did show a measurable activity. We therefore give the detection limits of the measuring facilities for ^{60}Co , ^{134}Cs , ^{137}Cs and ^{210}Pb .

Table 1. Results of activity measurements of Mr Louvet belongings using gamma spectrometry. The symbol "<" means lower than the detection limit of the measuring facility.

#	Description of the sample	Activity in Bq			
		Co-60	Cs-134	Cs-137	Pb-210
1	Kefieh	< 0.6	< 0.6	< 0.6	< 19
2	Sportswear	< 0.2	< 0.2	< 0.2	< 3
3	Hat	< 0.2	< 0.2	< 0.2	< 3
4	Clothing, including underwear	< 0.3	< 0.3	< 0.3	< 4
5	Sports bag (full)	< 2	< 2	< 2	< 30
7	Clothing, including underwear	< 0.7	< 0.7	< 0.7	< 20
8	Russian chapka (3) and cap	< 0.5	< 0.5	< 0.5	< 3
9	Drugs – part 1	< 0.4	< 0.4	< 0.4	< 5
10	Drugs – part 2	< 0.6	< 0.7	< 0.7	< 10
11	Slippers and various objects	< 0.7	< 0.7	< 0.7	< 10
12	Various objets (glasses)	< 0.3	< 0.3	< 0.4	< 5

Since ^{210}Po emits a gamma ray of 803 keV with a low intensity of 0.00107%, its activity can be measured in urine using gamma spectrometry if the level of intake is high and the measurements are performed early enough after intake. Therefore, the results of both measurements of urine performed on 8.11.2004 by the “Laboratoire de contrôle radiotoxicologique des Armées” were reassessed for the presence of ^{210}Po . This complementary analysis of both spectra acquired during 15 hours did not reveal the presence ^{210}Po . Based on the detection limit of ^{54}Mn (γ of 834.8 keV) provided in the report, we estimated the detection limit of ^{210}Po for this facility around 25 kBq/l.

Alpha spectrometry measurements

The results are presented in the Table 2. After the measure of the first batch, it appeared that an aliquot of the underwear with urine stain was particularly high in ^{210}Po (49 mBq/g). We then measured 6 others aliquots and another sample presented an enhanced ^{210}Po activity (9.7 mBq/g). All the others aliquots of the underwear have an activity that may be considered as background activity of 0-4-1.0 mBq/g cotton wool.

Table 2. Activity of ^{210}Po (mBq/g) for aliquots of different samples of Mr Louvet belongings and reference samples (not contaminated) taken from IRA collaborators, classified by order of activity.

Description	m (g) in the analysis	^{210}Po (mBq/g)
Bristles of two toothbrushes	0.4	54
Underwear (A) urine stain	3.69	49
Hospital cap, aliquot 3	0.8193	20
Hospital cap with blood stain	0.704	19.2
Russian chapka-1	3.636	9.7
Underwear (A) ,aliquot 3	6.2667	9.7
Sportswear, back, blood stain	0.7565	6.8
Hospital cap, aliquot 2	0.2895	6.0
Russian chapka-4	1.9403	5.8
Old slipper, interior	0.8906	3.9
Child's drawing, stains	1.4631	3.5
Sportswear, collar, aliquot 1	3.3723	3.2
Sock (worn)	2.1693	2.2
Russian chapka-2	0.3049	2.1
Kefieh, blood stain	2.254	1.6
Sportswear, collar, aliquot 4	3.9710	1.4
Sportswear, collar, aliquot 5	4.1180	1.2
Smear of personal belongings	0.63	1.2
Russian chapka-3	1.7320	1.1
underwear C, aliquot 1	2.5885	1.1
Child's drawing, without stains	2.5781	1.0
Russian chapka-5	2.7055	1.0
Underwear C, aliquot 0	3.2342	0.9
Underwear A, aliquot 2	5.476	0.9
Cotton wool pant	2.7707	0.8
Old slipper, superior band	5.2198	0.8
Underwear A, aliquot 4	3.3384	0.7
Underwear B, aliquot 2	4.6926	0.7
Sportswear, collar without cotton wool phase	2.9461	0.6
Underwear A, aliquot 3	2.3731	0.6
Sportswear, front tight	2.4764	0.6
Underwear A, aliquot 5	4.9678	0.5
Underwear A, aliquot 6	4.3517	0.5
Underwear A, aliquot 1	3.425	0.4
Long johns, aliquot 3	3.5286	0.4
New sock, not worn	1.6164	0.3
Underwear B, aliquot 1	4.0858	0.3
Long johns, aliquot 1	2.2687	0.2
Long johns, aliquot 2	4.1529	0.2
Medicine mixture (2 pills each)	39	0.1
<u>Cotton and reference samples</u>		
Bristles of toothbrush collaborator IRA 1	0.4776	21
Underwear collaborator IRA 2, aliquot 1	3.0715	6.7
Underwear new, brand Zimmerli A	3.3725	2.1
Underwear collaborator IRA 2, aliquot 2	3.2315	1.5
Underwear collaborator IRA 3, aliquot 1	3.7615	1.0
Underwear collaborator IRA 1, aliquot 1	3.1571	0.9
Underwear collaborator IRA 1, aliquot 2	3.5817	0.9
Underwear collaborator IRA 3, aliquot 2	3.3311	0.5
Underwear new, brand Zimmerli A	3.5805	0.5
Underwear new, brand Hanro	4.2094	0.3

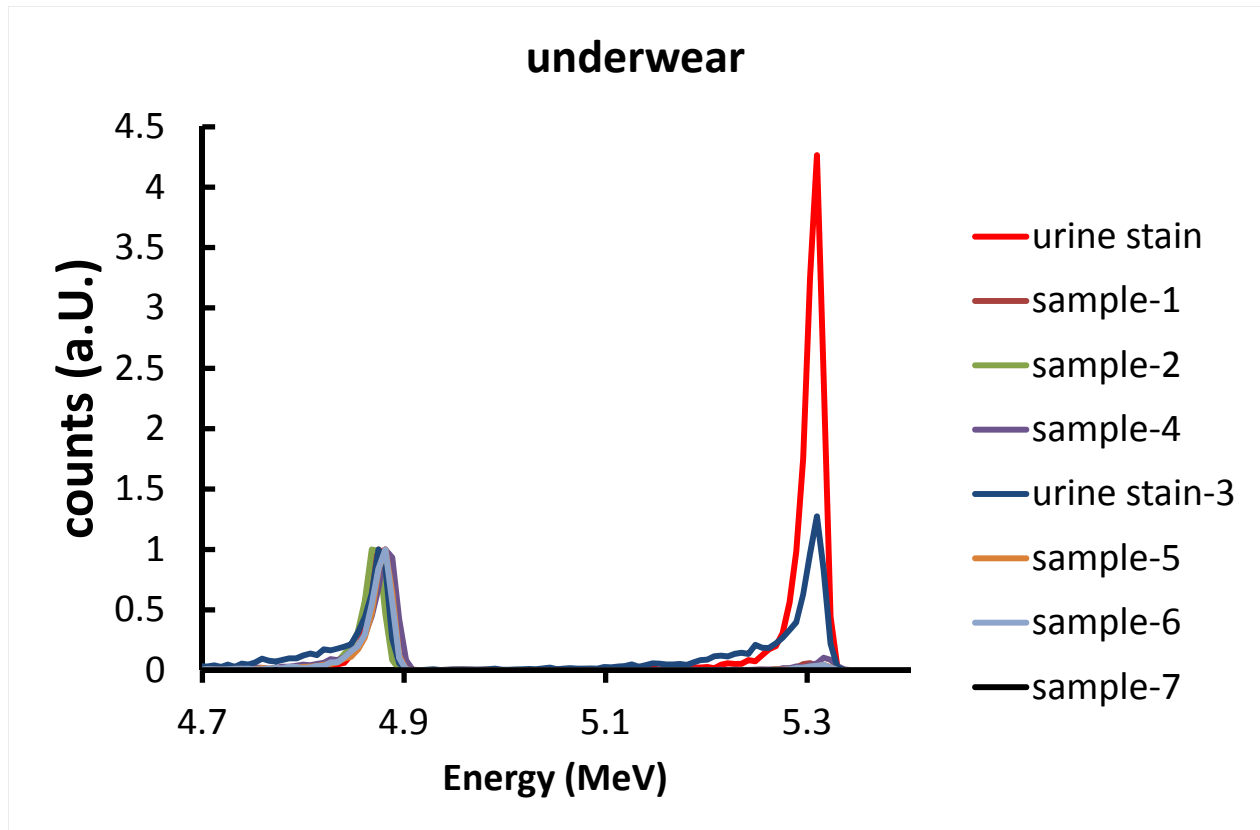


Figure 2. Alpha spectra of polonium normalized to the activity of the ^{209}Po tracer (left peak) for 7 aliquots of an underwear belonging to Mr Louvet. ^{210}Po is estimated by the surface under the right peak. Aliquot named “urine stain” clearly showed stains attributed to urine.

Figure 2 shows the alpha spectra of polonium sources obtained from all the samples taken from the underwear A. We can see that two aliquots are strongly contaminated with ^{210}Po . The most contaminated aliquot is a sample from a part of the underwear showing a stain attributed to urine. Then several aliquots of underwear B and C and also taken from a long johns have been analyzed; none presented an activity above 1.1 mBq/g of cotton wool. This is coherent with the fact that these clothes looked as having not been worn. We suspect these samples as having been bought in an airport free shop during Mr Louvet’s transport to Paris, because the Zimmerli brand of underwear is not commonly found out of luxury shops.

Several aliquots of the sportswear are manifestly contaminated by ^{210}Po . The sportswear showed many dirt stains, especially to the collar, thus we suspect it has been worn by Mr Louvet before his death. Alpha spectra are presented on the Figure 3. We can see that 3 of the 4 aliquots from the collar have ^{210}Po activities much higher than the one sampled from the tight and that the aliquot of the back presenting a suspected blood stain has an activity higher than the two supposedly uncontaminated aliquots.

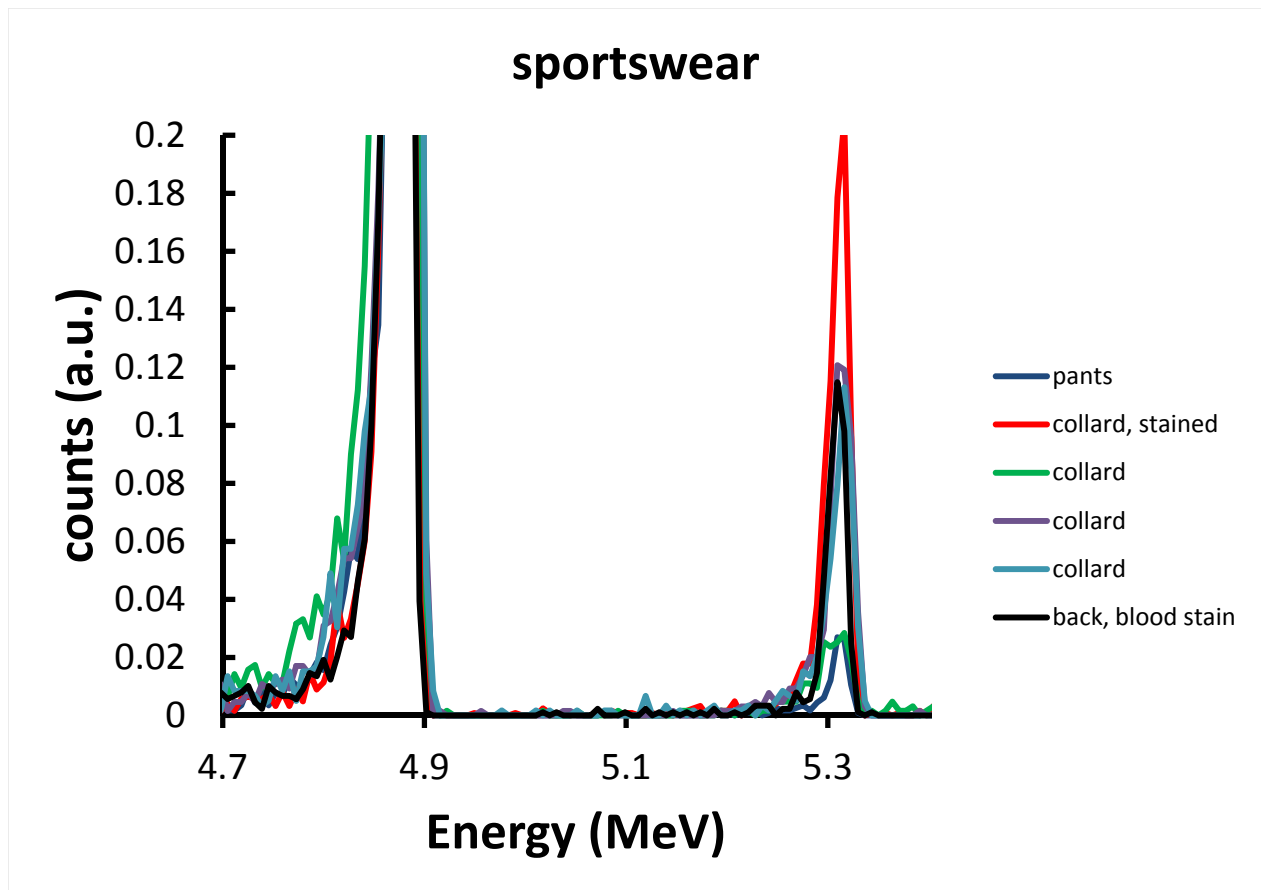


Figure 3. Alpha spectra of polonium normalized to the activity of the ^{209}Po tracer for 5 aliquots of a sportsweat belonging to Mr Louvet.

In the bag of personal belonging, there was a small hospital cap presenting a blood stain. We strongly suspect that this cap has been worn during Mr Louvet hospitalization. We cut a piece of cotton around this blood stain and two others from the cap. Results show that the blood stain and one of the other samples are strongly contaminated by ^{210}Po (Figure 4.)

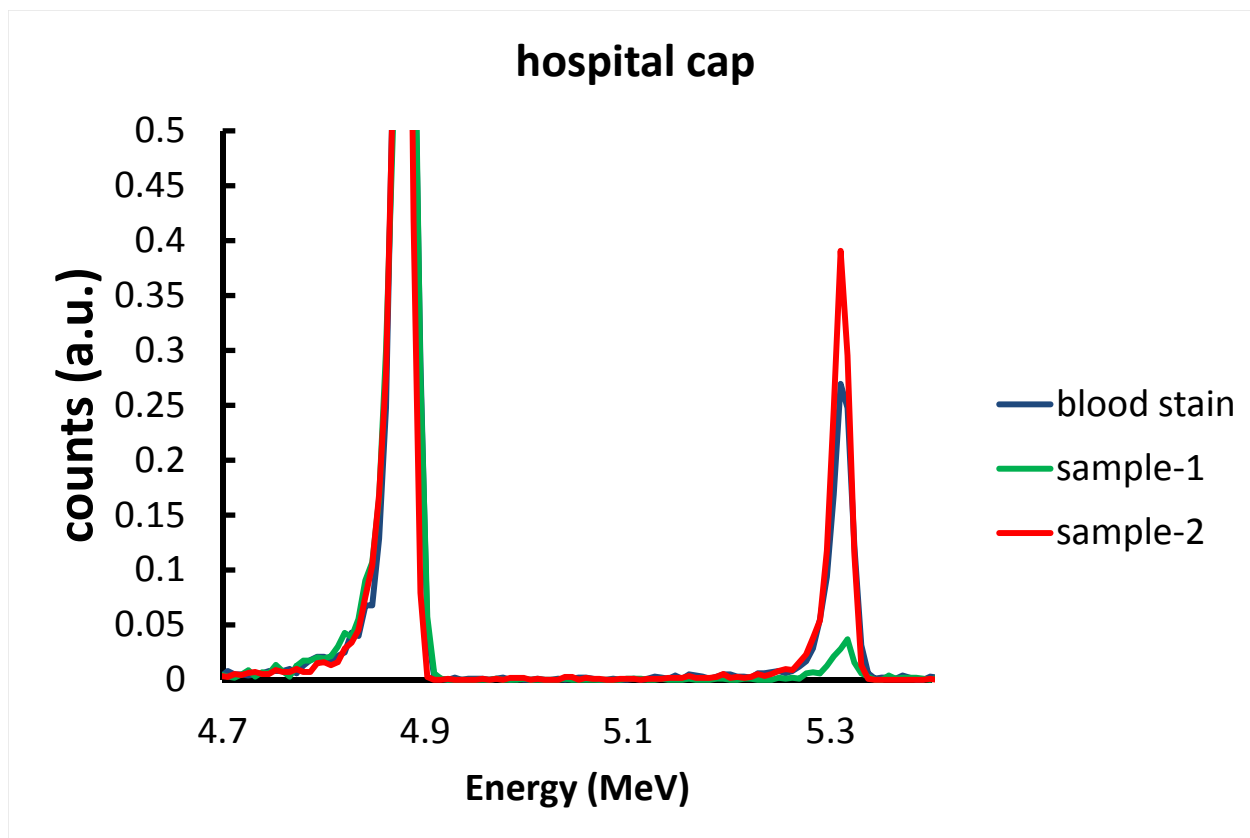


Figure 4. Alpha spectra of polonium normalized to the activity of the ^{209}Po tracer for 3 aliquots of a hospital cap which is strongly suspected to have been worn before Mr Louvet's death.

On a witness photograph of Mr Louvet taken shortly before his transfer to Paris, we can see that he was wearing a Russian chapka. This chapka, along with two others was found in the belongings that we received. We cut several pieces of the internal band in close contact with the skull near the temples. Two of these samples contain significantly more ^{210}Po than the 3 others (Figure 5).

After having measured more than 40 aliquots from Mr Louvet belongings, it was necessary to determine a background value for different samples of cotton wool. These measurements show that most of the samples have a ^{210}Po activity between 0.5-1 mBq/g ($n=6$). Nevertheless, 3 aliquots taken from two different underwears show activities between 1.5 and 6.7 mBq/g. The value of 6.7 mBq/g of underwear collaborator IRA 2 (aliquot 1) is not explained up to now. Nevertheless we observe that the aliquots sampled from Mr Louvet belongings that have been obviously not worn do not contain significant ^{210}Po activity (0.2-0.9 mBq/g, $n=18$). Thus clearly an activity close to 1.0 mBq/g can be considered as a background value for Mr Louvet belongings.

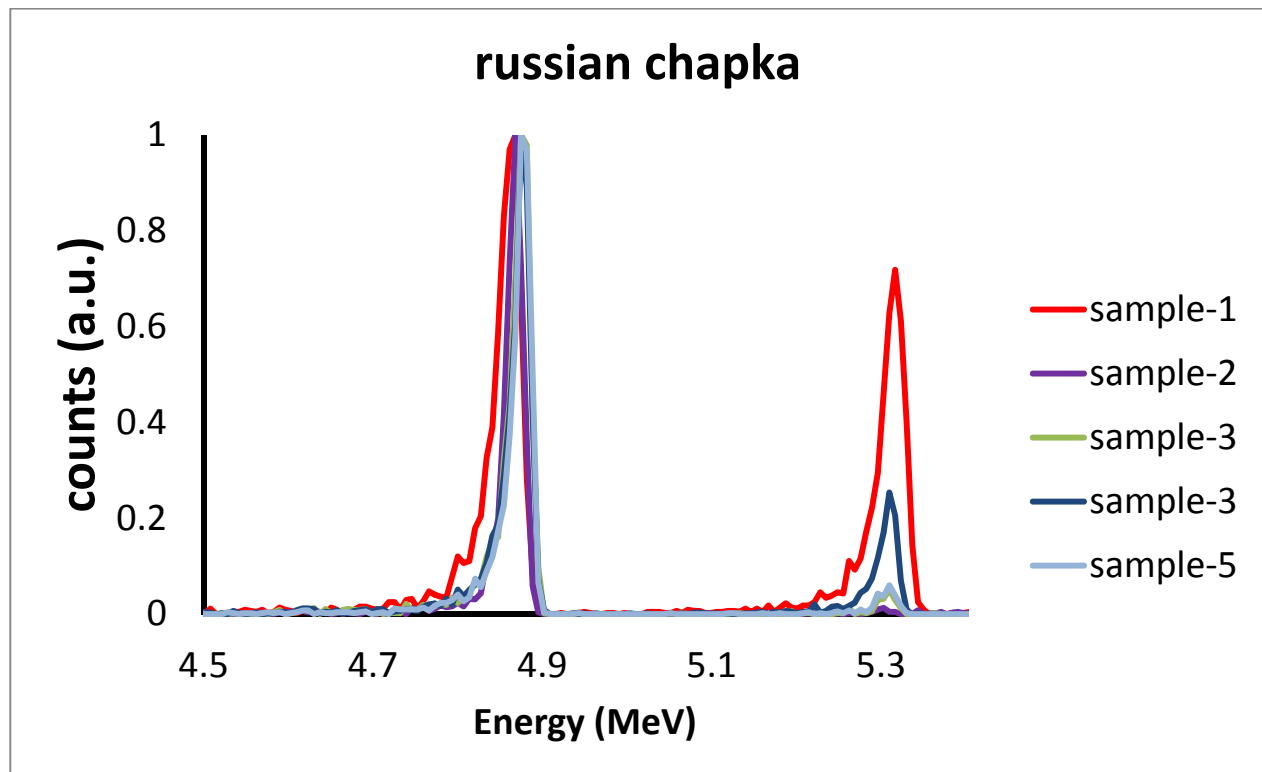


Figure 5. Alpha spectra of polonium normalized to the activity of the ^{209}Po tracer for 5 aliquots of a Russian Chapka which has been worn before Mr Louvet's death.

Bristles from toothbrush present another problem. They appeared clearly contaminated when sampled from Mr Louvet belongings (54 mBq/g). Nevertheless a control measurement carried out on a toothbrush from IRA collaborator 1 also shows a significant activity (21 mBq/g) while a second control measurement on a toothbrush from IRA collaborator 3 presents only a background value (1.0 mBq/g). Thus the measurement of Mr Louvet toothbrush is not conclusive and only the further determination of supported ^{210}Po will give an answer concerning a possible contamination of the toothbrush by unsupported ^{210}Po .

The measurements of reactants blank (all the products used in an analysis without the aliquot) yield activities between 0.2 and 2 mBq/sample. The highest activities (1.2 and 2.0 mBq/sample) have been observed using a glass H_2SO_4 bottle. When a new and plastic H_2SO_4 bottle is used we observe a significant decrease in reactants blank activity (< 1 mBq/sample). Nevertheless we reached the limit of the low activity measurements and some of the aliquots have lower activity than blank samples. In addition, glassware was used all along the chemical process and glass is known to contain significant ^{210}Pb activity. Nevertheless the presence of ^{210}Po in reactants and glassware used in this work cannot account for the ^{210}Po activities found in some of Mr Louvet belongings.

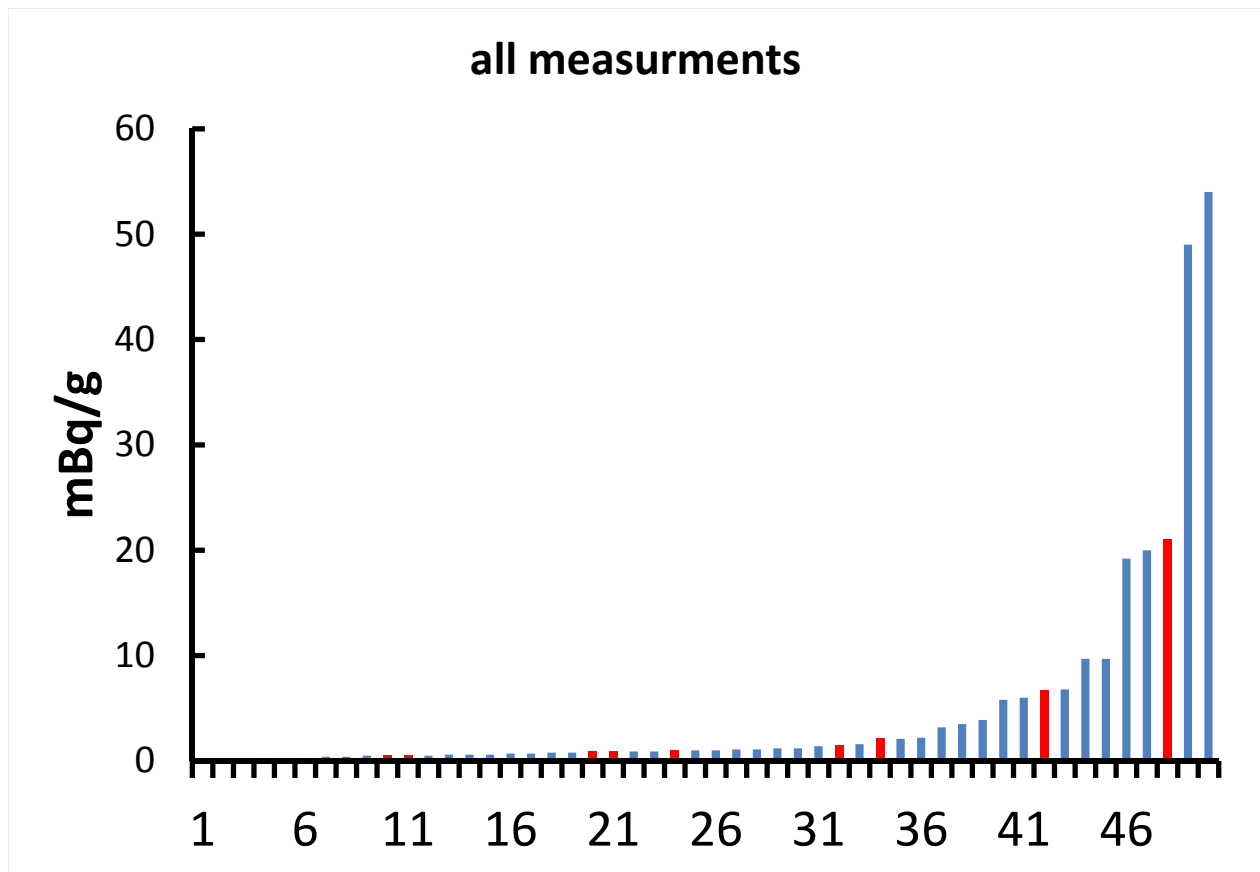


Figure 6. ^{210}Po activity of all the samples measured in this work according to increasing activity. Red bars represent samples taken from IRA collaborator 1,2 and 3 and underwear from a luxury shop. Activities up to 2 mBq/g can be considered as background activities.

On figure 6 we presented the activities of all the samples measured in this work. Activities up to 2 mBq/g can be considered as background activities (here about 33 of 50 measures). We observe clearly samples with increased activities. Nevertheless 2 of the 17 potentially contaminated samples do not come from Mr Louvet belongings, so that the comparison is not fully conclusive. Only the determination of unsupported ^{210}Po can settle the question about a possible poisoning of Mr Louvet by ^{210}Po .

We thus chemically separated ^{210}Pb to determine supported ^{210}Po in 18 of the most contaminated samples and let them settle for more than 3 months for ^{210}Po re-growth in the refrigerator. The list of the samples along with the ^{210}Po current activity is given in Table 3. We plan to measure the supported ^{210}Po at the beginning of June 2012.

Table 3. Activity of supported ^{210}Po (mBq/g), after chemical separation of ^{210}Pb and a re-growth period of ^{210}Po of 3 months. (re-growth level=36.6%).

Type	N°	^{210}Po total (mBq/g)	^{210}Po supported (mBq/g)	% supported
Bristles of two toothbrushes	Po-12-023	54		
Underwear A, urine stain	Po-12-011	49		
Underwear A, aliquot 3	Po-12-015	9.7		
Hospital cap. Blood stain	Po-12-019	19.2		
Sportswear, collar, aliquot 1	Po-12-008	3.2		
Russian chapka-1	Po-12-028	9.7		
Underwear, IRA collaborator 2, aliquot 1	Po-12-030	6.7		
Toothbrush, IRA collaborator 1	Po-12-043	21		
Child' drawing, stains	Po-12-047	3.5		
Sportswear, back, stain	Po-12-054	6.8		
Russian chapka -4	Po-12-056	5.8		
Hospital cap, Aliquot 2	Po-12-061	19.9		
Underwear B aliquot 1	Po-12-020	0.3		
Underwear B, aliquot 2	Po-12-021	0.7		
Sock (worn)	Po-12-032	2.2		
Underwear C, aliquot 1	Po-12-025	1.1		
Old slipper, interior	Po-12-048	3.9		
Kefieh, stain	Po-12-026	1.6		

Biokinetic model of ^{210}Po and urinary excretion

The cumulative urinary excretion and the daily urinary excretion after acute intake by ingestion of ^{210}Po are shown in Figure 7 and 8. A good agreement was found between the values of cumulative urinary excretion reported by Harrison et al.[2] and those calculated in our study (see Figure 6).

Regarding the poisoning of Mr. Litvinenko by ^{210}Po in November 2006 in London, Harrison et al. [2] concluded that 0.1–0.3 GBq or more absorbed to blood of an adult male is likely to be fatal within 1 month. This range would correspond to an intake of 1–3 GBq or more, assuming $f_1=0.1$. Based on their study, Li et al. [3] found that the estimated amount of ^{210}Po ranged from 27 MBq assuming $f_1=0.5$ to 1.4 GBq assuming $f_1=0.1$.

Considering a poisoning by ingestion of 1 GBq of ^{210}Po ($f_1=0.1$), it is expected to find about 500 kBq/day in urinary excretion the first 10 days after intake, about 250 kBq/day between 10 and 20 days and about 150 kBq/day between 20 and 30 days, according to Figure 8. Note that for a daily urinary excretion of about 1000 ml/day, such a concentration of ^{210}Po might have been observed in the gamma spectrometry analysis of 2004 (we estimated the detection limit at 25 kBq/l). Then, assuming that about 2 ml of urine might be found in the underwear with urine stains, we expect that 1 kBq can be found in the underwear if it is worn during the first 10 days after intake. This activity of 1 kBq in October 2004 corrected for radioactive decay of ^{210}Po ($T_{1/2} = 138.4$ d) gives about 1.4

mBq in February 2012. For comparison, an activity of 180 mBq was found for the urine stain of underwear (A) (see Table 2).

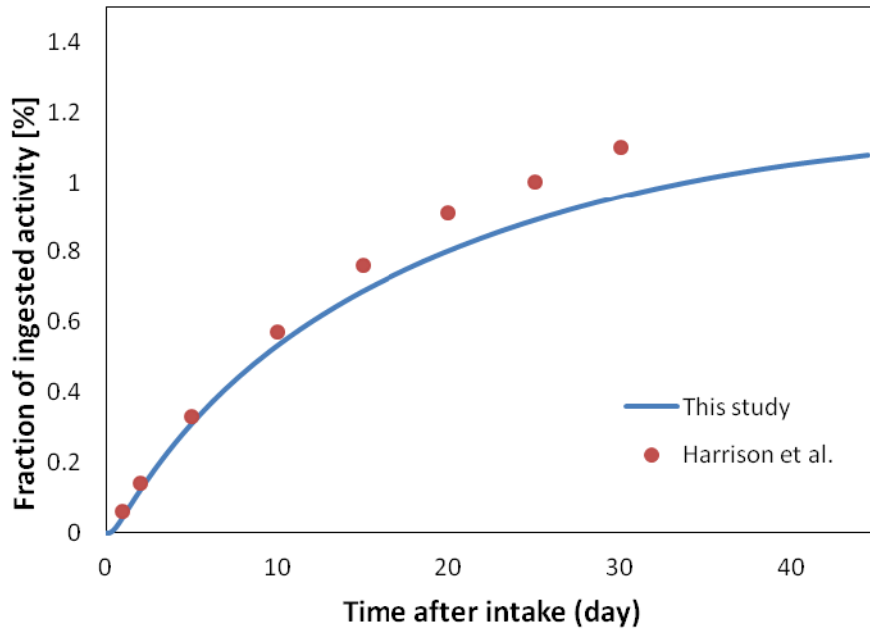


Figure 7. Cumulative urinary excretion of ^{210}Po after a single ingestion ($f_1=0.1$).

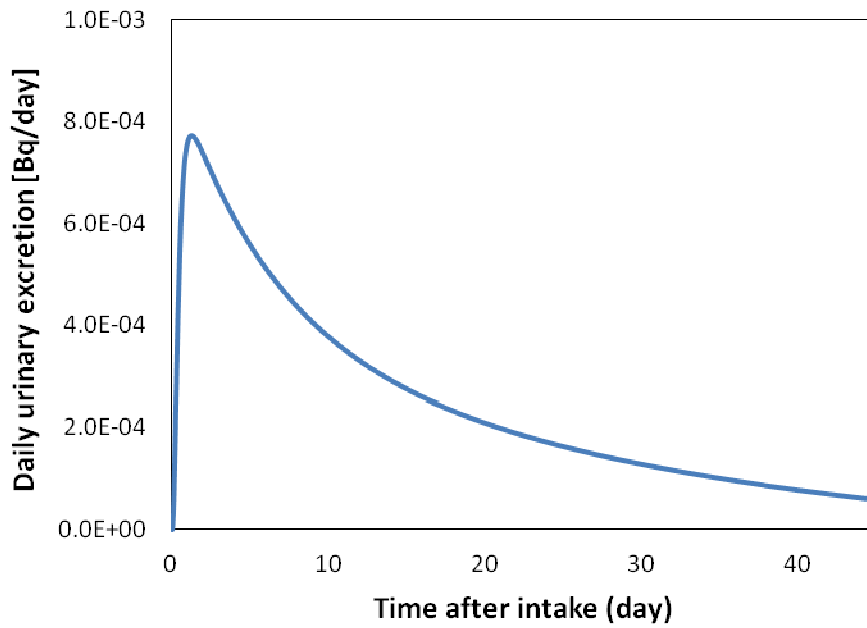


Figure 8. Daily urinary excretion after a single ingestion of 1 Bq of ^{210}Po ($f_1=0.1$).

Conclusions

All the belongings of Mr Louvet were first measured with surface contamination monitors and HPGe gamma spectrometry. We observed no significant activities.

We then evaluated a potential contamination by a lethal quantity of polonium-210 (^{210}Po). Because of the long time between the death (2004) and the present measurements (2012), the potential quantity of ^{210}Po in Mr Louvet's belonging had decreased by a factor of one million (20 radioactive half-lives). We considered the values of the literature and ran our own biokinetic and urinary excretion model. We concluded that, even in case of a poisoning similar to the Litvinenko case, only traces of the order of few mBq (one decay every 1,000 seconds) were expected to be found in year 2012.

We therefore chose to conduct ^{210}Po measurements on samples that had been manifestly worn by Mr Louvet and where stains of residual biological liquids could be suspected by direct eye observation. Some of these samples show ^{210}Po activities that are clearly above the values measured on other samples that had either not been worn or that were not containing visible suspect stains.

This observation alone is however not sufficient to draw a final conclusion because out of the 10 measurements performed on local samples totally unrelated with Mr Louvet's belonging, two show ^{210}Po activities above the value of 2 mBq/g, which can be set as a limit for background value.

To clarify the origin of the measured ^{210}Po , we should take into account that this nuclide is naturally present in the environment as a decay product coming from lead-210 (^{210}Pb): ^{210}Po is said to be supported by ^{210}Pb . Therefore, we propose to wait until the beginning of June 2012 and measure the samples again. If we observe a significant amount of ^{210}Po in the samples with high activities in the first measurements, this would show that ^{210}Po is supported by ^{210}Pb . This would therefore be a very strong argument in favor of a natural origin of the observed ^{210}Po , although such a quantity would be very uncommon. Alternatively, if we do *not* measure a significant amount of ^{210}Po , we should conclude that the high activity measured the first time does not come from the ^{210}Pb decay and therefore is not explainable by a known natural phenomenon.

Finally, and in addition to our alpha-spectrometric analysis, we re-analyzed the raw data of the gamma spectrometry performed on urine samples by the French government before Mr Louvet's death. We did not detect any abnormal gamma activities. In particular, we checked the area around the energy 803 keV that is (weakly) emitted by ^{210}Po . We did not find any evidence of this nuclide in the spectra.

References

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