

Decontamination of Tritium Contaminated Surfaces Using Strippable Polymeric Gel

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Abstract. The purpose of the present paper consists in finding a more efficient and less expensive decontamination method for surfaces contaminated with Tritium-labelled compounds, being able to provide information for nuclear field specialists. This paper studies the polymeric hydrogel DeconGel, presenting the methods and facilities used, as well as the obtained results from the experiments and tests. The decontamination factor of DeconGel type 1108 for the analysed surfaces (contaminated with a mixture of tritium labelled compounds) can take values in the range of 76%-93%, while in the case of DeconGel type 1102 the values of the decontamination factor for the analysed surfaces (contaminated with tritiated oil) can vary between 76% and 98%, results far greater than the ones obtained with the classical method of wet wiping. Because the results were more than satisfying, this paper concludes by recommending the implementation of this new method.

Keywords: surface decontamination, tritium contamination, radiochemistry, radioisotope, polymeric hydrogel, clean-up

1.Introduction

This new study has the purpose of searching and developing better radiation protection and decontamination measures for laboratories that are working with tritium, with the intention of, eventually, implementing them. The idea came from some everyday problems encountered in a radiochemistry laboratory, in this particular case in the TRITIULAB (Tritium Laboratory) NIPNE, Romania [1-2].

Tritium is a more special radioisotope, being a pure beta-emitting radionuclide, with a maximum energy of about 18.5 keV and a half-life of 12.36 years. The characteristics of the emitted radiation make it impossible to determine the tritium content in solid samples. Tritium is dangerous for humans especially through internal contamination (ingestion or inhalation). Tritium, in the gas form, in water or in labelled compounds, migrates differently and non-homogeneous especially in solid materials, having an uneven distribution. From the radiation protection point of view, tritium (beta emitter) is one of the current problems of the research laboratories as well as nuclear plants, an element that migrates rapidly and leads to its spread on larger surfaces. Due to these special characteristics of tritium, the work, the handling, the measurement of the activity of the sources with tritium and the storage are imposed special conditions. This laboratory is specifically designed for the work and research of compounds marked with tritium and is the sole in our country. The Tritium Laboratory was commissioned in 1975 as a part of Radioisotopes Production Center (now Department of Radioisotopes and Radiation Metrology). The laboratory focuses on the problem of the safe management of waste. The treatment, characterization and conditioning are still under study and this is why tritium management is of particular interest. Information about TRITIULAB can be found on the Erris platform (http://erris.gov.ro/main/index.php: TRITIULAB). Since 2007 Tritium Laboratory has been refurbished.

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The refurbishment strategy for this laboratory involved 2 steps: the first step represented the cleanup and the other was the upgrading stage. The cleanup step included area measurements for inventory of radioactive wastes resulted from refurbishment activities, decontamination and dismantling of the used equipment and characterisation and storage of radioactive wastes. In the case of this laboratory the strategy of cleanup also included the identification of the removable surface contamination (the values obtained for contamination are correlated with quantity of resulted wastes volume) for equipments, removal apparent ceiling, utilities, walls. The refurbishment was started after decontamination of the laboratory was finished.

Considering that the laboratory was put into use in 1975 and so far the decontaminations undergone were the usual ones, strictly of the facilities and laboratory equipment without the infrastructure, and that it worked so much with tritium gas, tritiated water and tritium-labeled organic compounds (with specific or general labelling) and the small number of those involved in tritium research, we have chosen the method of strippable coating polymeric gel.

Decontamination practices are not generally applicable and must be customized depending on the architecture and destination of the spaces and interior arrangements, and especially on the type and activities of the radioisotopes that have been worked or will be worked on. Good practices from the nuclear field require decontamination and decommissioning of nuclear facilities. IAEA Agency from Vienna (for its member States, which include Romania) and National Commission for Nuclear Activities Control (CNCAN) recommend the optimization of these activities [3-17]. The removable surface contamination (RSC) is one of the most important risk factor that workers from a tritium laboratory are facing. [18]. The classic method applied on laboratory for determination of RSC has been carried out by smear test using extruded polystyrene smears followed by the measurements of activity at LSC (Liquid Scintillations Counter). For studying this problem, we used a new material, DeconGel, a polymeric hydrogel. Furthermore, we developed some testing protocols in order to validate its use as a proper decontamination method. The article refers strictly to the stage of decontamination of some existing types of surfaces from our laboratory, presenting the experiments and decontamination tests performed with the chosen method in order to confirm if it is suitable for the needs of the laboratory and if it can be implemented. The problems raised are related to the difficulties of the decontamination process and the limited storage space for the resulted radioactive wastes [19-36].

From the point of view of waste, it is considered ordinary household waste; if the dry gel incorporated toxic or radioactive materials only then would it be treated as a specific waste and comply with the rules for that type of waste in the respective country. The volume of waste has not been presented in this study, but it can be said to have decreased significantly because many decontaminated objects could be reused or released unrestrictedly as simple non-radioactive waste, thus reducing the costs for modernizing the laboratory. We have also taken into consideration the requirements regarding the protection of the health of workers, people and environment, which in fact implies the reduction of the radiological hazard (risk) and the efficient management of radioactive waste. We chose this polymeric hydrogel because it presents more advantages: easy to be handled and used, clean peeling, environment friendly, can be applied on both horizontal and vertical surfaces, it reduces the exploitation costs and storage space. Moreover, the polymeric gel has the advantage of being appliable on rough and deformed surfaces with cracks. It can cover the whole surface and bind the contaminants, especially useful in the case of old facilities, laboratory furniture, walls, etc. [37-41]. For the implementation of a laboratory-level decontamination technique and its validation, it must be commercially available from one or more suppliers and previously demonstrated in other nuclear units [42-55]. The paper describes the surfaces selected for the study, the obtaining process of the contamination agents and the process of controlled contamination of the surfaces selected for the study.

The figures of this paper are original, being either photographies or illustrations, some of the later being realised in Free CAD Application and were afterwards imported in Microsoft Word.



2.Materials and methods Materials

The two types of gel were tested only in terms of efficiency of decontamination, in order to make a significant contribution for tritium contaminated surfaces. For this experiments realised we chose DeconGel type 1108 and type 1102, were procured from CBI Polymers (USA). In the experiment we used two types of tritium contaminants: (1) ethanol solution of hydrophilic and hydrophobic tritium labelled compounds mixture and (2) tritiated oil from broken vacuum pumps from which the volatile component was removed. The following types of surfaces have been selected and prepared, these surfaces being often found within TRITIULAB: type 316L austenitic stainless steel with a smooth surface; type 316L austenitic stainless steel with a matte surface; sheet laminated steel coated with antistatic paint (new false ceiling); rusty laminated steel; aluminium; glossy glass; PVC tiles (old false ceiling); PVC Mipolan Esprit 500 GELSOR (pavement for laboratories and access halls); PVC type Mipolan Mural Club N (PVC wallpaper from TRITIULAB). From the selected surfaces, 10 x 10 cm samples were prepared.

Method

The chosen method is a combined method that removes in particular radioactive contaminants (in this case tritium) as well as chemical contaminants (organic compounds, solvents, inorganic compounds, metals, etc., considering that the tritium laboratory is par excellence a radiochemistry laboratory). The methods of decontamination can be physico-mechanical (blasting, polishing, cutting, scarifying, cleaning with water jet at low pressure or at high pressure, steam cleaning, dry cleaning by vacuum) or by chemical methods (exfoliating foam and gels, chelatization, organic acids, mineral acids, oxide-reducing agents, cementation) [56-58]. Aggressive decontamination methods, which damage the surface of the material, can be appliable if they (the materials) are no longer used [59]. Opting for one method or another or for the combined use of decontamination methods depends on the measurement techniques of both surfaces and the characterization of radioactive waste. The theory of formulation of these gels, the description of their composition and properties, the different types, as well as their applications are described in the book Developments in Surface Contamination and Cleaning –Applications of Cleaning Techniques, volume 11, Rajiv Kohli and KLMittal, Elsevier (2018), which also grants a subchapter of radioactive decontamination by this method. Here the DeconGel is aslo described, (chapter 2). The principle of action of this decontaminant is to be able to bind by trapping in the polymer matrix and encapsulate a broad spectrum of radioisotopes and chemicals contaminants by forming chemical complexes at the nano- or molecular level [60]. It can be applied in thin layer, without the use of solvents, which can emit vapors, thus eliminating the risk of fire, poisoning, allergies, air pollution. The gel is ready-made and can be used directly from the container if the ambient temperature is between 4-32°C. The drying time of the polymeric hydrogel can vary from a few hours to 24 hours or even longer, depending on the ambient conditions, namely humidity, temperature, type of substrate (glossy or very porous, oily, dry or wet), the application type (with brush or spray) and the thickness of the applied layer. The laboratory experiments studied the degree of decontamination after the procedures were finished depending on the residual surface contamination. For this step, the unfixed contamination was determined for each surface by wiping them with smears, their activity being then mesured with a TRICARB TR2800 liquid scintillator counter (LSC).[61] The measurements of tritium's activity were the results of the collective work and experience of our team and a group from RML (Radiations Metrology Laboratory) from NIPNE [62-70]. The scheme of decontamination for the used protocol is presented in Figure 1. In the figures from 2-7 there are shown different stages from the experiments for different types of surfaces.





Figure. 1. How to use the polymeric hydrogel DeconGel in our experiments



Figure. 2. DeconGel layer on glass surface (colour online)



Figure 3. Decongel layer on PVC pavement and wallpaper surface





Figure 4. DeconGel layer on sheet



Figure. 5. DeconGel layer on the stainless-steel surface



Figure. 6. DeconGel layer on rusty laminated steel





Figure. 7. DeconGel layer on PVC tiles (old)

Obtaining of the Tritiated Labelled Compound Solutions

A stock solution consisting of a tritiated organic compound mixture in ethanol solution was prepared using ENT FU-T-G [71], Phenyl acetylene derivatives [72], Testosterone-1,2-T [73], [5-3H]Uracil Nucleoside Analogue [74], Acyclovir-T-G [75]. The radioactive concentration of stock solution has been determined using a TRICARB TR 2800 LSC and the Triple to Double Coincidence Ratio (TDCR) method at the Laboratory of Radionuclides Metrology [76-77]. 0.1 mL from stock solution have been introduced into glass vials, Econo-glass type which contain 10 mL scintillation cocktail (12.5 g 3,5 biphenyl oxazole (PPO) scintillation grade Loba Feinchemie, 1.25 g 2,2'-(1,4-phenylene) bis-(5phenyl) oxazole (POPOP) scintillation grade Riedel de Haen dissolved in 2.5 L Toluene pa Merck) with predetermined background. The measurement protocol at LSC was: Radionuclide: ³H, Count Time: 5 min., Repeat Sample Count: 5.

Obtaining of the Contaminated Oils

The tritiated oil wastes were processed for volatile components' removal using the facility showed in Figure 8. The removing process of the labile and volatile tritium (LVT) was: bath temperature 80°C, vacuum $<10^{-2}$ mbar, ethanol as LVT exchange agent, number of LVT removal steps: 10. After the removal stage, the radioactive concentration of tritiated oil was determined by liquid scintillation technique using the previously described protocol [78].



Figure 8. The Labile Tritium (volatile) Removal Facility scheme

TB-Thermostatic bath; EF-Evaporation flask; RSC-Rotation speed controller; LLD-Lowering-lifting device; TBC-temp erature bath controller, EE –Electrical engine; C-Vertical condenser; DLK-cooler; T-Thermostat; LNT-Liquid Nitrogen Trap; CF1 and CF2-Collecting flask; A/ST-Air/protic solvent inlet tube; S-Support; AV-Admission valve; M-Manometer; PVP-preliminary vacuum pump; ETF-Electric tubular furnace; FC-Furnace controller; CuOB-CuO bed; HTORS-HTO retention system; TGM-Tritium Gas Monitor; GB-Glove Box; ALC-air-lock chamber

Rev. Chim., 71 (3), 2020, 269-281



The obtained stock solutions (mixt solution of tritiated labelled compounds and tritiated oil from vacuum pumps) were characterised from the point of view of radioactive concentration using the LSC technique. The radioactive concentrations of stock solutions were 12.348 ± 0.486 kBq/mL in the ethanol labelled mixt compounds case and 3.127 ± 0.162 kBq/mL in tritiated oil case.

Controlled Contamination of the Selected Surfaces cm)

Analysed surfaces with a 100 cm² (10 x 10 area, were contaminated in a controlled manner with a solution made out of a mixture of tritium labelled compounds and respectively tritiated oil. The controlled contamination was made by deposition of 10μ L of the solution described above for each square centimetre of analysed surface using an aluminium grill (see fig10). Using this protocol, the conventional deposited activities onto 100 cm² surfaces are equivalent with radioactive concentration expressed in Bq/mL.



Fig.9 - Controlled contamination grill.

The contaminated surfaces were conditioned by deposition in Radionuclide fume cupboards (type Kottermann) at room temperature for 12 h, then at 80°C for 30 min in oven type UFE 400 Memmert and again at room temperature for at least 20 h.

The Determination of the Decontamination Factor

The selected controlled contaminated (with labelled compounds) surfaces were fully covered with a relatively uniform layer of DeconGel 1108. The exfoliation depends only on three factors: substrate, substrate adhesion and layer thickness. For the contaminated surfaces with tritiated oils a layer of DeconGel 1102 was applied. In both cases the average thickness of the layer was 1.5-2 mm. The samples were left at room temperature for at least 12 h for gel drying. After that, the resulted layers of dried gel were removed by peeling. The application and removal of DeconGel type 1102 and 1108 did not affect or damage the surfaces they were tested on. Because the DeconGel is insoluble in all scintillation liquids, the residual surface contamination (RSC) was determined by wiping. The RSC was determined using the following protocol:

- predetermination of the sampling factor for each surface;
- determination of the RSC of sampling factors for surfaces with known sampling factor;
- smear's introduction in glass vials ECONO GLASS with 10 ml of liquid scintillator
- (based on toluene with predetermined background);
- determination of the sampled activity at LSC;
- correlation between the sampled activity, the predetermined sampling factor and the total surface activity determination.

The Sampling Factor Determination

The RSC sampling factors were determined for each controlled contaminated surface described in above paragraph No 2.1 and 2.3. Determination of sampling factor was achieved by wiping the contaminated surface with smears made of extrudate polystyrene moistens in 50 L isopropyl alcohol, followed by activity measuring at LSC. The smears used were made of polystyrene materials because it has a good solubility in liquid scintillation cocktails, although it presents a lower sampling factor



than desired due to its hydrophobic properties. The values of sampling factor (f) were obtained using equation:

$$f = 100 x (SA/RCA)$$
(1)

where: SA is the activity determined at LSC expressed in Bq, RCA (Real Conventional Activity) represents the activity of the contaminant, controlled deposited onto analysed surface. For the rectification of the experimental values obtained in the determination of the residual activities (after application of the DeconGel) it was used the average value of the sampling factor obtained for ten replicates.

Determination of the residual contamination

The residual RSC of the decontaminated surfaces with DeconGel were determined by wiping using extruded polystyrene smears and their activity determined at LSC. The obtained values at LSC were rectified using predetermined average value of the sampling factor [79].

Determination of the decontamination factor

The decontamination efficiency of the DeconGel has been quantified as decontamination factor (DF), which represent the ratio between tritium activity incorporated in gels and total tritium activity deposited onto the analysed surface. The values of each decontamination factor (DF) were determined using next equation:

$$DF = 100 x (RCA - RA) / RCA$$
(2)

respectively,

$$DF = 100 \text{ x} (RCA - (ALSC \text{ x fav}) / 100) / RCA$$
 (3)

where: RCA represent the activity of the contaminant, controlled deposited onto analysed surface expressed in Bq, RA represent residual activity after treatment with DeconGel expressed in Bq, ALSC is the activity of the smear determined at LSC and f_{av} represent the predetermined mean value of the sampling factor.

3. Results and discusions

The sampling factors agents were determined by controlled contamination of analysed surfaces with two contaminant agent types followed by removing of the activities by wiping using extruded polystyrene smears and determination of activities at LSC. The obtained average values of sampling factor (f_{av}) for each analysed surface controlled contaminated with the two contaminants were presented in Table 1 and Table 2.

Real conventional Sampled activity (SA) f av determined at LSC f = 100 x (SA/RCA)Surface type activity (RCA) [kBq] [kBq] [%] Stainless Steel 316L smooth surface 12.348 ± 0.486 9.254 ± 0.584 74.94 Stainless Steel 316L matte surface 12.348 + 0.486 70.35 8.687 <u>+</u> 0.642 Steel coated with antistatic paint 12.348 + 0.486 5.987 ± 0.362 48.49 8.531 ± 0.536 12.348 + 0.48669.09 Rusty laminated steel 8.293 <u>+</u> 0.512 12.348 ± 0.486 67.16 Aluminium

Table 1. Average sampling factor (f _{av}) for analysed surfaces contaminated with ethanol solution of mixture of tritium-labelled compounds

Rev. Chim., 71 (3), 2020, 269-281



Glossy glass	12.348 <u>+</u> 0.486	11.284 <u>+</u> 0.542	91.38
PVC tiles	12.348 <u>+</u> 0.486	5.967 <u>+</u> 0.367	48.32
PVC Mipolan Esprit 500 GELSOR	12.348 <u>+</u> 0.486	6.987 <u>+</u> 0.452	56.58
PVC type Mipolan Mural Club N	12.348 <u>+</u> 0.486	6.687 <u>+</u> 0.423	54.15

Surface type	Real conventional activity (RCA) [kBq]	Sampled activity (SA) determined at LSC [kBq]	f = 100 x (SA/RCA) [%]
Stainless Steel 316L smooth surface	3.127 <u>+</u> 0.162	2.458 <u>+</u> 0.163	78.61
Stainless Steel 316L matte surface	3.127 <u>+</u> 0.162	2.246 <u>+</u> 0.153	71.83
Steel coated with antistatic paint	3.127 <u>+</u> 0.162	1.623 <u>+</u> 0.098	51.90
Rusty laminated steel	3.127 <u>+</u> 0.162	2.197 <u>+</u> 0.138	70.26
Aluminium	3.127 <u>+</u> 0.162	2.213 <u>+</u> 0.121	70.77
Glossy glass	3.127 <u>+</u> 0.162	2.965 <u>+</u> 0.163	94.82
PVC tiles	3.127 <u>+</u> 0.162	1.583 <u>+</u> 0.098	50.62
PVC Mipolan Esprit 500 GELSOR	3.127 <u>+</u> 0.162	1.862 <u>+</u> 0.124	59.55
PVC type Mipolan Mural Club N	3.127 <u>+</u> 0.162	1.785 <u>+</u> 0.113	57.08

Determination of the decontamination factor

The decontamination efficiency of DeconGel 1108 was determined for selected surfaces and for a tritium labelled compound mixture used as contaminant agent. The DeconGel 1102 was analysed from the point of view of decontamination efficiency for selected surfaces contaminated with tritiated oils. The obtained average values of decontamination factor (DF) for each analysed surface are presented in Table 3 and 4.

Table 3. The average value of decontamination factor (df_{av}) of decongel 1108 for analysed surfaces
contaminated with ethanol solution of tritium-labelled compounds mixture

Surface type	Real conventional activity (RCA) [kBq]	Residual activity (ALSCxf _{av}) [kBq]	Residual activity after treatment with DeconGel determined at LSC [kBq]	DF _{av} [%]
Stainless Steel 316L smooth surface	12.348 <u>+</u> 0.486	1.103 <u>+</u> 0.061	1.472 <u>+</u> 0.081	88.08
Stainless Steel 316L matte surface	12.348 <u>+</u> 0.486	1.204 <u>+</u> 0.081	1.711 <u>+</u> 0.115	86.14
Steel coated with antistatic paint	12.348 <u>+</u> 0.486	1.436 <u>+</u> 0.095	2.962 <u>+</u> 0.1 60	76.01
Rusty laminated steel	12.348 <u>+</u> 0.486	1.281 <u>+</u> 0.079	1.854 <u>+</u> 0.114	84.98
Aluminium	12.348 <u>+</u> 0.486	1.314 <u>+</u> 0.068	1.957 <u>+</u> 0.101	84.16
Glossy glass	12.348 <u>+</u> 0.486	0.791 <u>+</u> 0.032	0.866 <u>+</u> 0.035	92.99
PVC tiles	12.348 <u>+</u> 0.486	0.776 <u>+</u> 0.036	1.605 <u>+</u> 0.074	87.00
PVC Mipolan Esprit 500 GELSOR	12.348 <u>+</u> 0.486	1.534 <u>+</u> 0.098	2.711 <u>+</u> 0.173	78.04
PVC type Mipolan Mural Club N	12.348 <u>+</u> 0.486	1.272 <u>+</u> 0.101	2.349 <u>+</u> 0.186	80.98



Surface type	Real conventional activity [kBq]	Residual activity x f _{av} [kBq]	Residual activity determined at LSC [kBq]	DF _{AV} [%]
Stainless Steel 316L smooth surface	3.127 <u>+</u> 162	195 <u>+</u> 08	248 <u>+</u> 10	92.07
Stainless Steel 316L matte surface	3.127 <u>+</u> 162	221 <u>+</u> 11	308 <u>+</u> 15	90.16
Steel coated with antistatic paint	3.127 <u>+</u> 162	378 <u>+</u> 25	728 <u>+</u> 48	76.71
Rusty laminated steel	3.127 <u>+</u> 162	311 <u>+</u> 18	443 <u>+</u> 26	85.84
Aluminium	3.127 <u>+</u> 162	324 <u>+</u> 17	458 <u>+</u> 24	85.36
Glossy glass	3.127 <u>+</u> 162	59 <u>+</u> 03	62 <u>+</u> 4	98.01
PVC tiles	3.127 <u>+</u> 162	163 <u>+</u> 09	322 <u>+</u> 18	89.70
PVC Mipolan Esprit 500 GELSOR	3.127 <u>+</u> 162	421 <u>+</u> 28	707 <u>+</u> 47	77.39
PVC type Mipolan Mural Club N	3.127 <u>+</u> 162	373 <u>+</u> 26	653 <u>+</u> 45	79.10

Table 4. The average value of decontamination factor (DF_{av}) of DeconGel 1102 for analysed surfaces contaminated with tritiated oil

4.Conclusions

As it has resulted from prior experience, the decontamination methods for surfaces and equipment need to be improved. Subsequently, in order to lower the difficulty of maintenance, operation and other activities in the nuclear field, improvement of present decontamination protocols or techniques is necessary. This will also help with the possibility of reusing certain infrastructure and conforming to the waste management requirements. The opportunity to use the DeconGel 1108 (for radioactive decontamination) and 1102 (dedicated for the decontamination of oily and greasy surfaces) as decontaminant agents in the specific case of the radiochemical laboratories which operates with tritium sources was analysed. Both types of DeconGel were proved to be efficient in the decontamination process of different types of surfaces for specific contaminants type, the DF being in 85 and 99% range at one application for metallic and glass surfaces. For the steel coated with antistatic paint and MIPOLAN PVC surfaces, the obtained DF was lower (75-85%). These results can be explained by diffusion of tritiated compounds inside of the material surfaces with conversion in a quasi-fixed contamination. The DF obtained values are higher in comparison with the values obtained for the sampling factor by wiping. This suggests a greater efficiency of decontamination when using the DeconGel comparing to the classic decontamination methods by wet wiping. The decontamination efficiency of the DeconGel has been quantified as decontamination factor (DF), which represent the ratio between tritium activity incorporated in gels and total tritium activity deposited onto the analysed surface. Summing up, because the decontamination percent for both types of hydrogel were between 70% and 95% for any type of surface analysed, it can be concluded that the two types of DeconGel tested can be used as a decontamination and cleaning method within the tritium laboratories. The variation of the decontamination percent can be explained by the different characteristics of each type of surface (such as porosity).

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Rev. Chim., 71 (3), 2020, 269-281



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