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## Current investigations on tritiated dust and their impact on tokamak safety

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### Abstract

During ITER operation, due to plasma wall interaction, particles/dust will be created with sizes ranging from nanometer to tens of micron. The dust properties especially their ability to be covered by a thin oxide electrostatic insulating layer and their surface topology deeply affect their tritium inventory. Consequently, physico-chemical properties specific to tritiated tungsten particles and consequence on particles behavior in the facility and environment must be carefully assessed. For size relevant tungsten particles, the measured tritium inventory is ~10 GBq/g. However, it varies with the particle specific surface area (SSA). Due to tritium beta decay and to the oxide-insulating layer, dust exhibits a positive electrostatic self-charging. For a 5  $\mu\text{m}$  particle in diameter with a 10 GBq/g tritium inventory, self-charging rate could lead to  $5.5 \cdot 10^4$  elementary electric charges per day. These electrostatic properties could change the adhesion of dust on walls. In case of a single particle, the adhesion will be reinforced due to image and dielectric forces. However, if the tritiated particle is part of an aggregate, the adhesion remains unknown. Due to the limited free path of the  $\beta$  emission in material, the tritium inventory carried by airborne particles cannot be measured in real time by conventional continuous radioactive aerosols monitors (CAM's) and a new measurement strategy is needed for atmospheric survey in workplace and exhaust of facility. Toxicity studies dealing with exposure to untritiated/tritiated tungsten particles of 100 nm have been undertaken. It was shown that these particles are rapidly dissolved in biologic media. Finally, after collection, dust must be confined to avoid their spreading into the environment. Different technical solutions are presented in the paper.

## 1. INTRODUCTION

During operation of the International Thermonuclear Experimental Reactor (ITER), plasma interacts with the plasma facing components (PFCs) through various physical processes. These give birth to particles from nanometer to tens of microns in size that are called dust grains in the fusion community. Depending on the plasma wall interaction processes at play, different types of dust will be created, from almost spherical particles induced by high heat flux deposition on metal surfaces (unipolar arcs, ELMs, disruptions) to fractal ones created by accretion in the edge of high density/long pulse plasma discharges. The metallic dust properties, especially their ability to be covered by an electrostatic insulating oxide layer and their surface topology, deeply affect their tritium inventory, their chemical reactivity and their capability to become airborne and to be released out of the tokamak vacuum vessel.

This paper discusses dust properties linked with safety and radioprotection open issues. These aspects are synthetically presented in Fig. 1.

First, experimental results on dust tritium inventory obtained for W material are recalled. Extrapolations from deuterium trapping in massive beryllium samples are then established.

Second, the consequences of the natural dust charging induced by tritium radioactive decay on dust adhesion and on aerosol creation in case of Loss Of Vacuum Accident (LOVA) are investigated. The behaviour of an isolated particle is compared to a dust aggregate in terms of dust adhesion/suspension.

The limited free path of the  $\beta$  electron in materials used in fusion devices has major consequences on the aerosol tritium inventory measurement in real time. This is addressed in the third part of this paper.

When an aerosol is created, workers can be contaminated by inhalation of tritiated particles. Our team has been involved for years now on the evaluation of the *in-vitro* cyto and genotoxicity of tritiated and untritiated W particles on lung cells. Summarized results are presented in the fourth part of this paper. Finally, we will address the dust waste management open issues and the solution that we decided to promote.

## 2. DUST TRITIUM INVENTORY

Study of tritium trapping in W dust material has been initiated by our team in 2011 [1]. It has been shown that the dust tritium inventory is two to three orders of magnitude higher than in massive material. It ranges from 1 to 20 GBq/g depending on the Specific Surface Area (SSA) of the particles. The higher the SSA, the larger the measured tritium inventory. When extrapolated to 1000 kg of W dust (corresponding to the dust safety limit) only ~50 g of tritium is trapped in such powdery material [2]. That is well below the tritium safety limit in vessel of ITER (1000 g).

No comparable studies exist for beryllium (Be) powder. As a consequence, estimation of tritium (T) content per kg of Be powder in the ITER vacuum vessel requires extrapolation of existing studies conducted on deuterium (D) trapping in Be massive materials [3, 4]. One can thus estimate the tritium content in the ITER vacuum vessel per kilogram of beryllium powder. The D/Be atomic ratio varies from 0.01 up to 0.1 depending on the deuterium loading conditions and on the beryllium target temperature. By assuming that the T/Be ratio is comparable to the D/Be one, the tritium limit in the vacuum vessel is reached for 600 kg of Be dust for T+D/Be=0.01 and for 60 kg for T+D/Be=0.1. This last value corresponds to a rather strong tritium activity of ~6 TBq/g.

These preliminary estimations emphasize the necessity to collect and analyze particles produced during the ITER lifespan. Moreover, the collection of dust produced in current operating machines is

necessary in order to prepare the ITER operation, but not sufficient. The operating domain of ITER will be different from current tokamaks giving birth to dust with rather different properties. In dusty plasmas [5], the erosion of a W target in high density and low temperature plasma leads to the creation of large quantities of fractal dust with high SSA. These conditions are not observed in current tokamaks but would be reached in detached ITER plasma regime. As a consequence, extrapolation from current tokamak dust collection could be unrepresentative.

Finally, it has to be noticed that if some experiments are undertaken to study tritium inventory in tungsten dust, no comparable effort is put on beryllium powder. Such lack of experimental results is detrimental for ITER safety prediction.

### 3. TRITIATED DUST ELECTROSTATIC PROPERTIES

#### 3.1. Electrostatic properties of tungsten metallic dust

Metallic tungsten dust particles are covered by a thin oxide layer of 4-6 nm that induces insulating electrostatic properties. The experimental set-up used by Peillon *et al.* [6] to study dust suspension induced by an applied electric field consists of two circular stainless steel electrodes that are arranged in parallel and separated by a 4 mm gap. The top electrode is hooked to a high voltage source whereas the bottom electrode, which includes a central extraction orifice, is grounded. A monolayer circular deposit of particles, 4 cm in diameter, is spread out on the top electrode. An increasing electric field (E) from 0 up to 27.5 kV/cm is then applied by steps of 2.5 kV/cm in ambient atmospheric conditions. For each applied E, the detached particles are pumped out and transported by a radial airflow through the central extraction hole connected to an optical particle counter (OPC) that measures the size-resolved particle number concentration. A schematic representation of this set-up is depicted in Fig. 2.

The results obtained for different types of powder (silver, tungsten and aluminum oxide  $\text{Al}_2\text{O}_3$ ) are displayed in Fig. 3 where the cumulative fractions of particles detached and counted by the OPC are plotted with respect to the electric field strength. The data are normalized thanks to the total amount of particles detached by the maximum electric field achievable in the experimental set-up (*i.e.* 27.5 kV/cm).

This experiment reveals that the presence of a thin oxide layer on the tungsten particle surfaces alters their electrical conductivity by shifting their properties from conductive (silver behavior) to insulating (dielectric) material (aluminum oxide behavior). Consequently, tungsten particles will experience an accumulation of electric charges due to tritium  $\beta$ -decay even when deposited on grounded metallic surfaces. The next section presents the evaluation of the particle self-charging rate according to the tritium  $\beta$ -decay. One should notice that this work provides data for electrostatic charge build-up, which is a necessary input to assess a final charge equilibrium state taking also account for the neutralization processes due to the dielectric properties of the materials in contact or ion fixation. These neutralization processes are not studied in the framework of this paper.

#### 3.2. Tritium decay and consequence on dust electrostatic self-charging

Tritium is a radioactive isotope of Hydrogen. Its half-life is 12.3 years. During the tritium decay, a  $\beta$  emission is observed (mean energy of 5.7 keV) along with  $^3\text{He}^+$  production.

For the mean energy of 5.7 keV, the  $\beta$  maximum path ( $L_m$ ) can be calculated [7] and gives  $L_m = 43$  nm in the case of W and 450 nm for Be. The  $L_m$  limited value has several consequences. First, for large particles, not all the  $\beta$  emission escapes from the material. For W, only an external layer of

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2  
3 ~100 nm contributes to the resulting positive dust charging. As an example, taking into account the  
4 full spectrum energy of  $\beta$  decays of tritium, the probability of electron escape from a 100 nm  
5 thickness layer is 20 %. It has to be pointed here that the insulating properties of the metallic dust  
6 demonstrated in paragraph 3.1 will insure a quasi-permanent positive charge in the particles.  
7

8  
9 Full Monte Carlo calculations [8] of the self-charging rate of W particles with 10 GBq/g tritium  
10 inventory have been performed. The charging rate is presented in Fig. 4 as a function of the particles  
11 diameter.  
12

13 The continuous line represents the electrical self-charging rate of a particle where the tritium is  
14 homogeneously distributed in the material while the dashed one concerns a particle where the  
15 tritium is distributed in a surface layer having a thickness of 100 nm. As expected by the boundary  
16 conditions applied, the two self-charging rates differentiate (discrepancy above 10 %) when the  
17 particle diameter reaches 400 nm.  
18

19 For example, a single tungsten particle of 5  $\mu\text{m}$  in diameter with homogeneous distribution of  
20 tritium will exhibit a self-charging rate of  $5.5 \times 10^4$  positive charges per day.  
21  
22

23 Nevertheless, tritium is only a part of radionuclides to take into account in radioactive dust issues in  
24 fusion tokamak and self-charging processes. Other radionuclides are present in fusion tokamak  
25 coming from activation by neutron fluxes. Usually the calculations and study made on the activation  
26 due to neutron fluxes in the framework of fusion machine deal with dose rate and are mainly  
27 focused on activation products that are gamma emitters.  
28

29 It should be noticed that gamma decay and high radiation level does not lead to particles self-  
30 charging rate like one for beta or alpha decay. The case of gamma ray with high dose rate effect up-  
31 to 7 kGy/h on self-charging was investigated by Gensdarmes *et al.* [9, 10]. Self-charging due to  
32 gamma decay is a very weak process due to the poor probability of photon interactions  
33 (photoelectric absorption or Compton scattering) with particles. This mainly comes from the small  
34 length path available for photon interactions in micron size particles.  
35  
36

37 Let the case of beta emitters radionuclide produced by neutron activation be examined. There are  
38 few papers in the literature dealing with quantitative assessment of dust contamination for neutron  
39 activation scenario related to the ITER operational condition. Nevertheless, the paper by Saito *et al.*  
40 [11] reports input data to assess self-charging rate due to activation products in comparison to  
41 Tritium. In particular, the authors mention a list of radionuclides induced by neutron activation and  
42 their specific activity (Bq/g) in a comparable manner than to the specific activity value we determine  
43 for tritium in tungsten particles.  
44  
45

46 The main beta emitter radionuclides to be considered [11] for self-charging are two isotopes of  
47 tungsten:  $^{187}\text{W}$  and  $^{185}\text{W}$ . The other beta emitter radionuclides of the list, Ta and Re, have low  
48 specific activities that are one to three orders of magnitude below tungsten isotopes and they have  
49 very short half-life. The  $^{60}\text{Co}$ , have a medium half-life but a specific activity four to five orders of  
50 magnitude below those of tungsten isotopes.  
51

52 The specific activity values are  $10^{11}$  Bq/g for  $^{187}\text{W}$  is (half-life 23.7 hours) and  $3.7 \cdot 10^{10}$  Bq/g for  $^{185}\text{W}$   
53 (half-life 75 days). Besides, specific activity value for  $^3\text{H}$  in tungsten particle is up to  $10^{10}$  Bq/g  
54 (Grisolia *et al.*, [2]) while its half-life is 12.3 years. Consequently, according to these data, on short  
55 time period (< some days), the radionuclide  $^{187}\text{W}$  will dominate electrostatic self-charging; for  
56 intermediate term (some days to some months) tungsten isotopes  $^{185}\text{W}$  and  $^{187}\text{W}$  may contribute in  
57 equal part like tritium to self-charging. However, on long term period (> some months) the tritium  
58 beta decay will dominate electrostatic self-charging.  
59  
60

1  
2  
3 It is obvious that this conclusion depends on the neutron activation scenario. The data reported in  
4 [11] for activation products comes from the ITER Preliminary Safety Report.  
5

6 All these considerations shows that beta decay of tritium appears as a key component of  
7 electrostatic properties of dust as soon as trapped activity relies on such high level as  $10^{10}$  Bq/g.  
8  
9

#### 10 11 12 4. CONSEQUENCES ON THE DUST ADHESION

##### 13 14 **4.1. Adhesion of an uncharged particle**

15  
16 The adhesion of an uncharged particle relies mainly on the van der Waals force (for the sizes of  
17 particles considered, the gravity is negligible). The adhesion force depends on different parameters  
18 such as the Hamaker constant of materials into contact, the distance of closest approach between  
19 the materials, the roughness of the contacting surfaces, which are difficult to estimate.

20 Fortunately, adhesion forces can be directly assessed using the Atomic Force Microscope (AFM)  
21 technique. Details on the technique and experimental procedure can be found in [12]. Adhesion  
22 force measurements have been realized with a Multimode 8 (Bruker) AFM in PeakForce Quantitative  
23 Nano-Mechanical mode (PF-QNM) in environmental conditions. The measurements were realized  
24 between tungsten spherical particles with different sizes glued onto tip-less CP-FM (Colloidal Probe  
25 for Force Modulation) cantilevers and three different tungsten surfaces with various roughnesses.  
26 Samples were cleaned by successive ultrasonic baths of acetone and ethanol and dried before being  
27 mounted in the AFM. The results obtained are summarized in Fig. 5 where the W/W adhesion force  
28 is plotted as a function of the particle radius for different root mean square (rms) surface roughness.  
29  
30

31 As it can be seen in Fig. 5, the adhesion force decreases with the rms roughness of the substrate and  
32 becomes quasi-independent on the particle size for high values of roughness i.e.  $\approx 700$  nm.  
33 Moreover, the adhesion force on a relevant W tokamak surface (rms  $\sim 712$  nm) is two orders of  
34 magnitude lower than on a smooth mirror polished tungsten surface.  
35  
36

##### 37 38 **4.2. Adhesion of a tritiated particle**

39  
40 As we have seen, metallic particles (W or Be) are covered by an insulating oxide layer. When such a  
41 particle is charged, an image force appears as presented schematically in Fig. 6 for a W dust covered  
42 by a  $\text{WO}_3$  oxide layer.  
43

44 This electrostatic image force will reinforce the van der Waals force and the overall adhesion  
45 process. As an example and considering that the oxide layer acts as a perfect insulator, it will take  
46  $\sim 14$  hours to get an image force equivalent to the van der Waals one i.e.  $\sim 10$  nN for a particle of 5  
47  $\mu\text{m}$  of diameter and 10 GBq/g of homogeneous tritium inventory.  
48  
49

50 However, in a tokamak like ITER, dust will fall towards the bottom of the machine and pile up. In that  
51 case, the consequences of self-charging could be rather different. Indeed, at the top of a pile,  
52 particles could undergo an electric force from positively charged particles located below them. This  
53 situation can strongly affect the particles adhesion which can induce an easiest particles/aggregate  
54 suspension in case of a LOVA and jeopardize the prediction of aerosol creation.  
55  
56

57 Our team has started studies on the adhesion of isolated tritiated particles. Comparable studies  
58 must now take place for piled-up particles in order to get reliable information on this open issue.  
59  
60

## 5. MEASUREMENT OF TRITIUM CARRIED BY AEROSOLS

As already mentioned, due to the low  $\beta$  path in the material only a small part of the tritium activity can be measured out of a tritiated particle. Fig. 7 represents Monte Carlo calculations of electron escape probability for spherical tritiated tungsten particle, taking into account the full energy spectrum of tritium  $\beta$  decays. For micrometer spherical tungsten particles, these results show that only the  $\beta$ -decay of tritium located in a surface layer of  $\sim 100$  nm can lead to an electron escape probability from the particle above 20 % and thus be detected. As a consequence, a real time tritium measurement by an ionization chamber as recommended by the Standard IEC 60761-5 [13] will lead to an underestimated value of the aerosol tritium inventory.

Usually, at the laboratory scale, the total tritium inventory in dust is measured by full dissolution of the particles followed by liquid scintillation counting. This is a reliable diagnostic but this procedure cannot be applied in real time. Moreover, this method fails to distinguish tritium carried by particles from the gaseous form in a simple way.

This leads to another open issue dealing with atmospheric survey of workplace area and exhaust that must be addressed carefully: to develop a methodology/diagnostic able to accurately measure the total tritium content in tritiated aerosol.

## 6. TOXICITY STUDIES

In case of LOVA, an aerosol can be created. To prevent contamination to the environment and/or to people supervising the cleaning operations, High Efficiency Particulate Air (HEPA) filters are used. However, these filters have a lower retention capability for particles with aerodynamic diameters in the 100–300 nm range [14, 15].

We therefore studied the consequences of inhaling small particles that can reach the human inner lung. As we have seen above, on long term period ( $>$  some months) the tritium beta emission will dominate and the toxicity studies we undertaken were focused on tritiated particles.

Different topics were thus addressed such as the behaviour of particles and embedded tritium in biologic media or the *in vitro* toxicity of these tritiated particles.

The work was focused on 100-200 nm tritiated and non-tritiated W particles. From Fig 8, we can state that those particles were rapidly dissolved in different biologic media as saline solution, TRIS buffer and cell culture medium "lung solution". TRIS solution was used to prepare particles stock solution for *in vitro* studies.

A full dissolution was obtained after 2 to 60 days of exposure to 100-200 nm W particles depending on the dust type [16, 17]. In addition, a full dissolution of large W particles (up to 7  $\mu$ m of diameter) was also observed.

The consequence of the dissolution of the particles is the release of tritium into the solution. Tritium, once solubilized, will be removed quickly. Indeed, after human exposure, tritium in tritiated water form is eliminated very quickly from the body (half-life of 10 days) [18].

Toxicity of untritiated and tritiated tungsten particles has been studied on a 3D *in vitro* cell model of the human airway epithelium grown on air-liquid-interface, MucilAir<sup>®</sup>. This model is characterized by similar morphology and functions of the normal human airway epithelium and by mucus secretion and cilia beating [19]. Epithelia were exposed for 24 h to tungsten nanoparticles (tritiated or not) or tritiated water, as a control. Thanks to the long shelf life of this model, cytotoxicity was studied immediately after treatment and in a kinetic mode up to 1 month after cell exposure to assess the reversibility of any toxic effect. Acute and long-term toxicities were monitored by several endpoints: 1) epithelial integrity, 2) cellular metabolic activity, 3) pro-inflammatory response, 4) mucociliary clearance, and 5) morphological modifications. Transmission Electronic Microscopy observations,



Inductively Coupled Plasma Mass Spectrometry measurements and liquid scintillation were performed to determine tungsten and tritium human airway epithelium lung absorption as well as intracellular accumulation. In parallel, their potential toxicity was assessed on human-derived immortalized bronchial cells (BEAS-2B cells). Untritiated and tritiated tungsten particles exerted different degrees of cytotoxicity depending on the presence/absence of hydrogen on their surface. Differences were observed also in their genotoxic potential investigated by performing the centromeric cytokinesis-block micronucleus cytome test (CBMN-cyt) and the alkaline comet assay. Furthermore, DNA methylation analysis was also assessed to determine epigenetic effects on BEAS-2B cells.

These observations are the first steps towards establishing future potential guidelines for LOVA and workplace safety. However, further studies are needed on W particles as well as on Be particles.

## 7. TRITIATED DUST AND WASTE MANAGEMENT

Tritiated dust will be collected in rather large quantities (hundreds of kilograms) during the ITER life. After vacuuming the ITER in vessel, they will be temporarily stored in canister.

From a safety point of view, avoiding tritiated dust spreading into the environment is a major concern. As a consequence, the storage in canister is not appropriated as a permanent storage due to its weakness. Moreover, it is needed to define the best route for ITER tritiated dust to mitigate the risk as close as possible to the source. As a consequence, it is recommended to limit the recovery or handling of dust from the canister to avoid spreading.

In this framework, after deep analysis of all the possible technical possibilities, we propose to study two solutions: (i) immobilization in a hydraulic binder and (ii) dissolution followed by immobilization in a hydraulic binder. Both solutions will mitigate the risk of spreading before grouting and prevent from any reconditioning. However, the second one requires addition of a chemical and the associated risk has to be managed in a dedicated cell.

One of the main problem that is expected is the strong reactivity of metallic powder (W, Be) when introduced in cement due to the high pH of the solution. This leads to hydrogen emission that must be controlled. Experiments will start in fall 2018 at CEA to study both aforementioned solutions.

## 8. CONCLUSION

During the operation of a tokamak like ITER, dust will be produced by plasma wall interaction processes. These particles will have a large set of properties due to their diverse morphologies including particles of high chemical reactivity. The dust that have been collected in current tokamaks have certain properties that cannot be definitively extrapolated to ITER. Indeed, the current tokamak operation cannot yet represent the ITER operating regime. As an example, ITER will rely on quasi detached divertor plasmas with a high density and low plasma temperature characteristics for all the shot duration. In these conditions, it has been shown in dusty plasma laboratory studies [20] that relatively small particles (diameter < 1  $\mu\text{m}$ ) of large SSA and consequently large chemical reactivity are created in quantity. This will probably be also the case during ITER operation. It will be thus mandatory to collect dust produced during the ITER life in order to refine our knowledge on their physicochemical properties.

The dust tritium inventory in ITER-relevant particles has been studied in the case of W showing that the tritium inventory varies with the SSA of the particles. The measured tritium content is  $\sim 10$  GBq/g for W micrometer-sized particles. Extrapolation from existing results on deuterium trapping in massive Be leads to huge dust tritium inventory greater than 6 TBq/g. It is obvious that studies on tritium trapping in ITER relevant Be dust are urgently needed to confirm or deny this tendency.

Dust are covered by an oxide layer that is an electric insulator. Due to tritium decay this will lead to the build-up of positive charges in the particles. In case of a W particle of 5  $\mu\text{m}$  diameter with a tritium inventory of 10 GBq/g, the resulting charge is more than  $5.5 \cdot 10^4$  positive charges per day.

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2  
3 In case of an isolated particle deposited on the vessel wall, self-electrical charging will increase the  
4 dust adhesion force making aerosol creation more difficult. However, in the case of aggregates of  
5 tritiated particles, the adhesion remains unknown.

6 Due to limited low path of the  $\beta$  emission in metallic particles, the measurement of the tritium  
7 aerosol inventory in real time remains unachieved. This points the necessity to perform R&D to cope  
8 with this open issue.

9  
10 In case of LOVA and the creation of a tritiated aerosol, particles can escape the vacuum vessel and  
11 get through the HEPA filters. We have undertaken some toxicity studies that concern inhalation of  
12 tritiated and untritiated W particles of 100-200 nm. We have shown that these particles are rapidly  
13 dissolved in biologic media. All these studies will give useful information to define potential  
14 guidelines in case of LOVA and for occupational safety.

15 Finally, it has been shown that the waste management of tritiated dust needs special methodology  
16 in order to avoid any spread into the environment. This requires the development of a dust  
17 immobilization technique. After deep analysis of all the existing solutions, CEA has proposed two  
18 solutions e.g. immobilization in hydraulic binder or dissolution of dust followed by immobilization in  
19 hydraulic binder. These two solutions are currently under studies at CEA.  
20  
21

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26 tungsten compounds by neutron fluxes.  
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## FIGURES

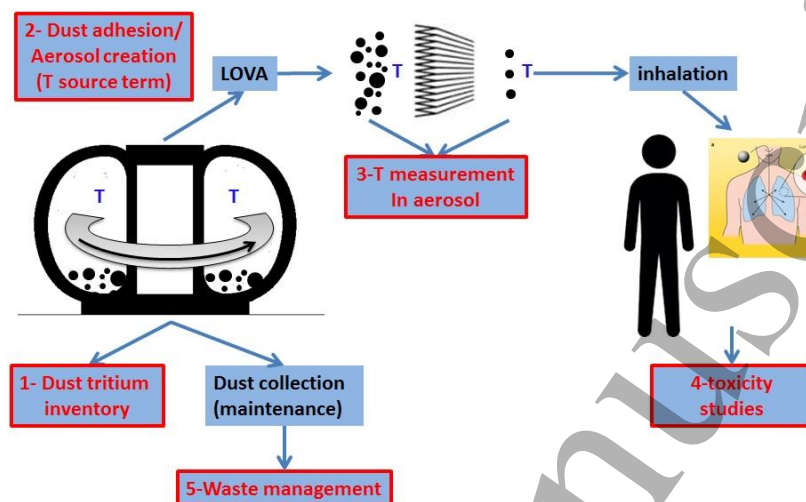


FIG. 1. Synthetic presentation of the topics covered in the paper.

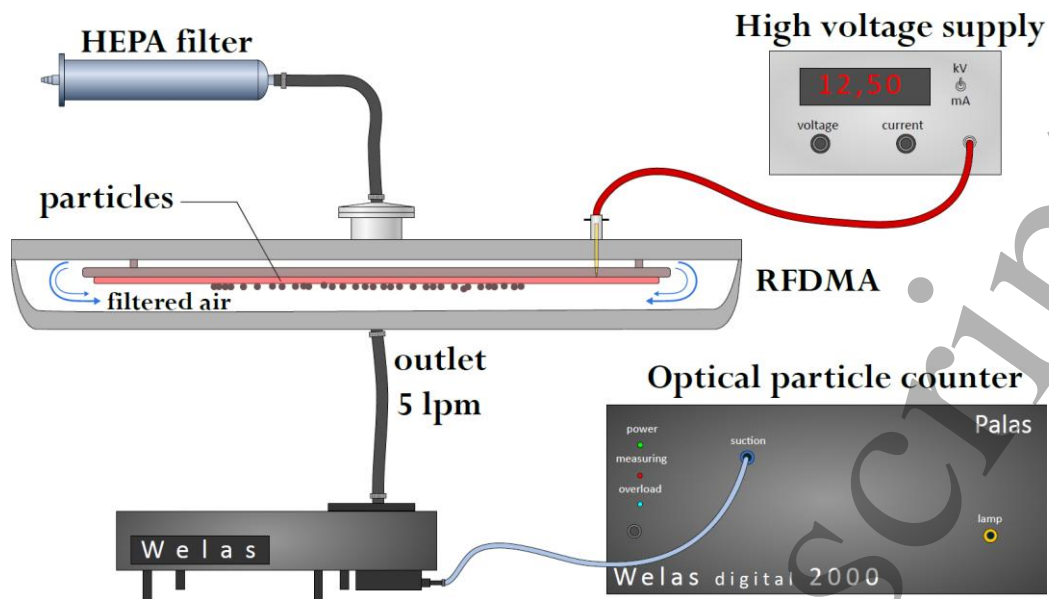


FIG. 2 Schematic representation of the experimental set-up used in [6]. RFDMA accounts for Radial Flow Differential Mobility Analyzer.

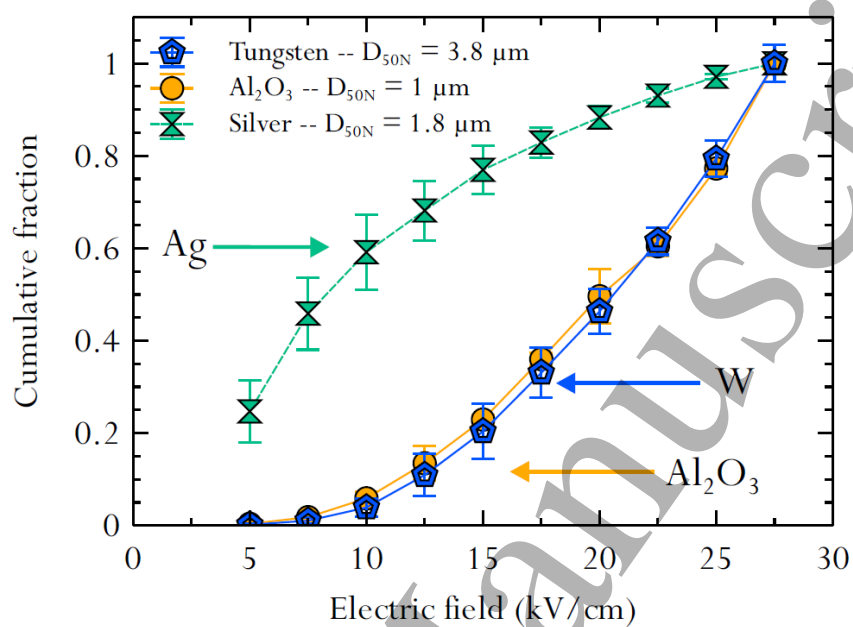


FIG. 3. Normalized cumulative number fraction of particles (W,  $\text{Al}_2\text{O}_3$  and Ag) experiencing increasing electric field strengths.

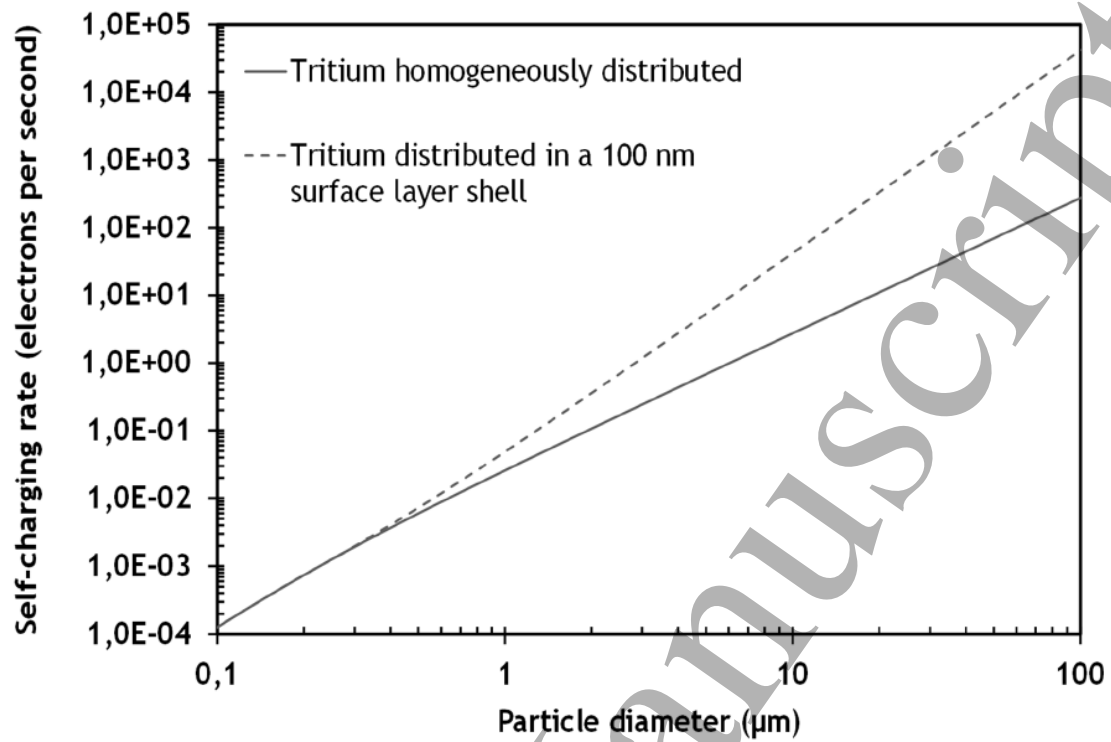


FIG. 4. Calculation of the electrical self-charging rate of tungsten particles considering a tritium inventory of 10 GBq/g.

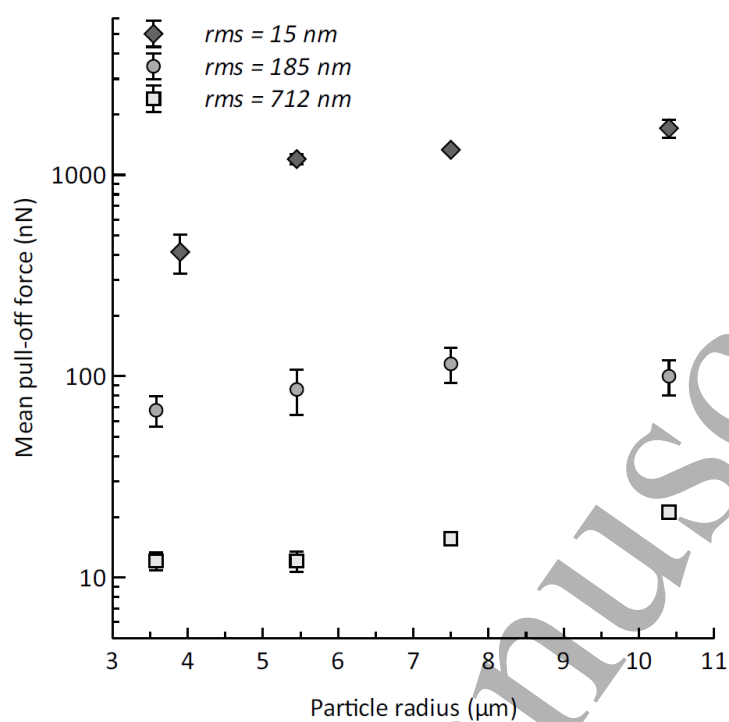


FIG. 5. Adhesion forces obtained by AFM for W spherical particles of different radius in contact with W surfaces of variable roughness.



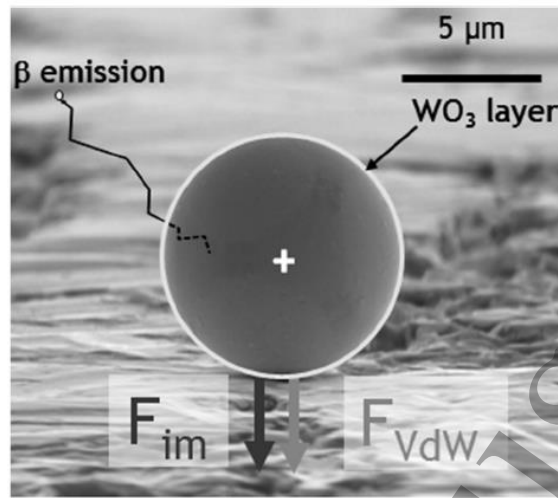
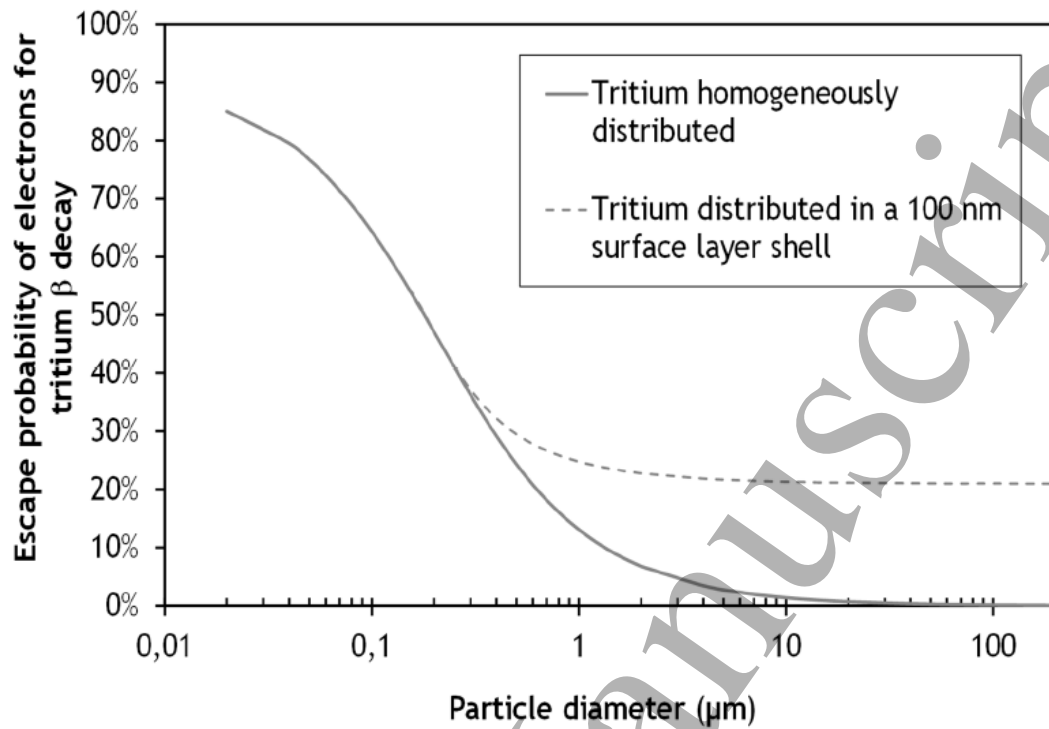


FIG. 6. Schematic of balance of forces in the case of a W spherical tritiated particle lying on a W surface



. FIG. 7. Calculation of the escape probability of  $\beta$  electrons from W tritiated particles versus the size of the particles for the 2 considered configurations.

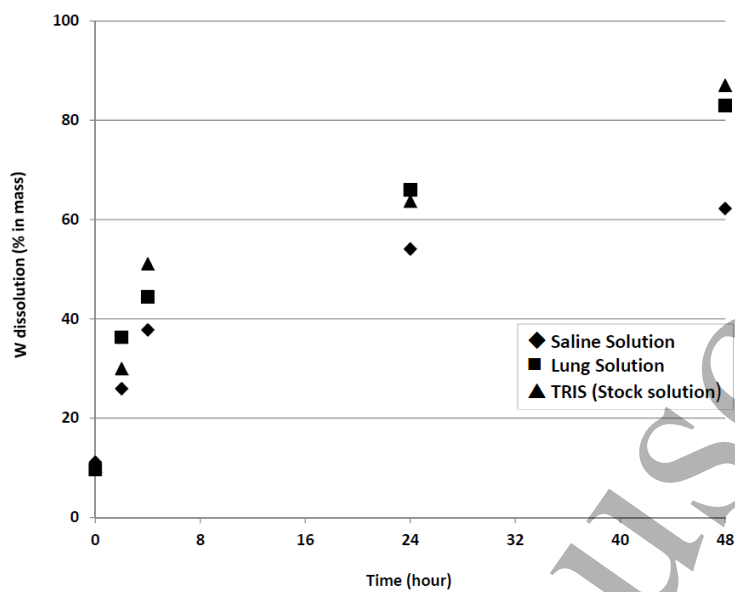


FIG. 8. Dissolution of 100 nm tungsten particles in different biologic media solutions.