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Organic Telluride Formation from Paint Solvents Under Gamma Irradiation

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Abstract — *The interactions between tellurium and organic material during a nuclear reactor accident are critical to source term estimations because of the possible formation of volatile species. Reactions taking place in the containment sump are of interest since these can lead to re-volatilization and increase the fission product source term. This paper presents results from experiments investigating the interaction of tellurium dioxide with three paint solvents—texanol ester, methyl isobutyl ketone, and toluene—under containment sump conditions. The experiments were performed by irradiating a mixed solution of tellurium dioxide and paint solvents at a dose rate of 4 kGy/h up to 300 kGy. The resulting samples were analyzed for tellurium concentration and speciation. Tellurium(IV) was found to reduce to metallic tellurium under irradiation when paint solvents were present. More importantly, several volatile organic tellurides were identified in the irradiated samples, which suggests that tellurium can form volatile species in sump conditions when in contact with dissolved paint solvents. This paper provides novel evidence of organic telluride formation in the sump and raises further interest in tellurium chemistry during a severe nuclear reactor accident.*

Keywords — *Severe accident, fission product, tellurium, source term.*

Note — *Some figures may be in color only in the electronic version.*

I. INTRODUCTION

In severe nuclear reactor accidents, tellurium can be considered one of the most significant radionuclides released. This is due to the high volatility and therefore high activities of tellurium isotopes released during an accident. The most relevant tellurium isotopes to nuclear accident conditions, ^{132}Te and ^{129m}Te , have half-lives long enough to cause potential dose to the public during the first few days to weeks from the accident. Tellurium was actually found to be one of the main contributors to

the internal dose of residents close to the Fukushima Daiichi power plant after the accident in March 2011 (Ref. 1). Another study on internal dose by ingestion of radio-tellurium in rice showed that tellurium should not be disregarded even in long-term dose evaluations.² In addition to its release, the decay of tellurium is also an important factor to consider. Many of the released tellurium isotopes decay to iodine, which is a significant nuclide due to causing increased risk of thyroid cancer.³ Previous research on tellurium behavior under severe nuclear accident conditions has focused on its release from the core,⁴ interactions with the cladding,⁵ and transport into the containment building.⁶ However, knowledge is limited when it comes to tellurium behavior inside the containment and especially the reactions in the sump. There are potential reactions leading to release by re-volatilization that need to be considered to accurately estimate the tellurium source term.

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Most of the severe accident chemistry research has been focused on the release behavior of iodine. However, tellurium has also been part of several severe accident research programs such as VERCORS (Ref. 7), PHEBUS (Ref. 8), and TETRA (Refs. 6 and 9). These programs have provided information on different aspects of the tellurium source term in the reactor coolant system (RCS) and in the containment. Of these programs, VERCORS focused on the release of fission products from the core and led to the characterization of fission products by their volatility. There, tellurium was grouped together with other volatile fission products such as iodine and cesium. The PHEBUS experiments focused on the phenomena related to fuel melting and release of fission products. Tellurium was part of the PHEBUS experiments related to the release from the core as well as the behavior in the containment. Last, the TETRA project focused on the transport of tellurium in the RCS and the interactions between tellurium with other fission products.

The main findings of these experimental programs and other individual studies have been on the volatility of tellurium, the interactions between tellurium with the zirconia in the cladding,^{5,10} the effect of humidity and atmosphere on the speciation of tellurium,⁶ the removal efficiency of the spray system toward tellurium from the containment atmosphere,⁹ and the dissolution and speciation of tellurium in the sump.¹¹ However, there remain uncertainties when it comes to possible further reactions taking place inside the containment. Of these reactions, the interactions between tellurium and organic material are of interest in this work.

II. BACKGROUND

Organic tellurium species have been proposed to form from interactions between elemental tellurium and organic material inside the containment building. Organic species in the sump originate from, e.g., paints, insulators, or resins.^{12–14} The significance of the organic tellurides is their relatively low boiling points compared to other tellurium species potentially present in the containment. The two main organic telluride species proposed to be of interest in severe accident conditions are dimethyl telluride (92°C) and diethyl telluride (138°C). Because of the relatively low boiling points, the organic tellurides could volatilize already in lower temperatures and escape from the sump into the containment atmosphere. Furthermore, this could increase the total amount of tellurium released during a severe nuclear accident. The

organic chemistry involving tellurium has not been extensively investigated in severe nuclear reactor conditions.

Although organic chemistry in a severe accident condition involving tellurium has not been explored, there is evidence of formation of volatile organic species under sump conditions, mainly involving iodine. Previous studies have shown the formation of different organic iodides and their re-volatilization from the sump. Organic iodides have been found to form from organic radicals originating from various paint constituents.^{15–17} The focus has mainly been on the formation of methyl iodide (CH₃I), but other forms of organic iodides have also been shown to form, such as ethyl, isopropyl, and butyl iodide,¹⁸ all of which are volatile species and can increase the iodine source term. Another fission product that could have interactions with paint during a nuclear accident is ruthenium. Although ruthenium can be in organometallic compounds, the main interactions with ruthenium and paint in severe accident conditions are either deposition on painted surfaces or possibly oxidation of organic molecules in the sump.^{19,20}

In addition to formation of organic iodides and ruthenium interactions, previous work related to fission products and paint solvents has also led to identification of the main ingredients in different types of paints and the dissolution of these in the aqueous phase in the containment. Wren et al. analyzed three different paint types—epoxy, vinyl, and polyurethane—and identified the main compounds dissolved in the aqueous phase in different temperatures.²¹ They found methyl isobutyl ketone (MIBK) to be the common compound in all the paints studied. In addition, they identified some aromatics and halogenated hydrocarbons in various amounts. Tietze et al. analyzed paint ingredients found in Swedish nuclear facilities and identified the main compounds that could potentially react with fission products in severe accident conditions.²² They investigated water-based epoxy paint (Teknopox Aqua VA), polyvinyl acetate-based emulsion paint, and oil-based alkyd paint. Consequently, the main paint constituents used in the previous studies related to the fission product source term have been MIBK (Refs. 15, 23, and 24); methyl ethyl ketone²⁵ (MEK); texanol ester; Stoddard solvent (white spirit)¹⁸; and aromatic solvents, e.g., xylene and toluene.¹⁶ The formation of organic iodides has been investigated with all these compounds. All except aromatic compounds have shown evidence of formation of different volatile organic iodides. This is likely due to the differences between the radiation stability of aromatic and aliphatic molecules. The radiation stability of toluene compared to the aliphatic molecules is due to the aromatic structure. The benzene ring in toluene

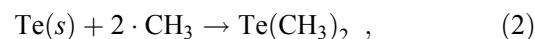
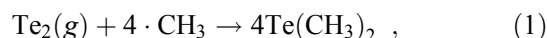
stabilizes the structure by efficient energy delocalization and consequently makes it more tolerant toward ionizing radiation. However, it is still important to include various paint solvents for experiments related to organic telluride formation. Although the chemistries of iodine and tellurium are very different, they can both form organic compounds and are released in significant amounts during a nuclear accident.

While organic tellurides have not been a focus in severe accident research, the formation of these species is not unknown, and some reactions can be presented.¹²⁻¹⁴ These reactions are based on studies from other disciplines, but they could provide information on organic telluride formation and behavior that could be of use in severe accident research. One of the main research areas involving organic tellurium species is biochemistry. Tellurium has been found to be biomethylated by bacteria to form dimethyl telluride from either tellurite(IV) or tellurate(VI) solutions.^{26,27} Another study relevant to organic tellurium chemistry is one by Miller and Winkler,²⁸ where elemental tellurium was used to trap methyl radicals. The result of this study was the formation of dimethyl telluride from solid tellurium and methyl radicals generated from acetone or dimethyl ether.²⁸ This is of interest as solid metallic tellurium may come into contact with organic radicals in accident conditions. Although, the microbial activity or radical removal studies as such are not relevant to severe accident conditions, the reaction mechanisms presented there provide insight about what types of reactions are possible to transform elemental tellurium or its oxyanions into a volatile organic species.

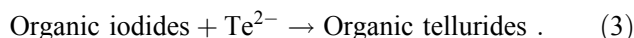
When it comes to organic telluride formation under irradiation, there are a few previous studies to consider. It has been found that organic tellurides could form from irradiated sodium telluride (Na_2Te) solution under a cover gas consisting of a mixture of methane and argon. The main volatile species was found to be dimethyl telluride.²⁹ In addition, there has been evidence of formation of volatile tellurium compounds under ultraviolet irradiation conditions from low molecular weight organic acids in aqueous solution.³⁰ In that study, a reaction mechanism was presented for selenium, but it can be expected to be comparable to tellurium since the chemistry of the two elements is analogous. In addition to the formation of volatile organic compounds, it was also mentioned that selenium(IV) can also be reduced to elemental selenium(0) by hydrogen and carboxyl radicals formed from the organic acids. Similar reactions can also be expected for tellurium.³¹

By combining results described in previous studies, we can present some reactions leading to organic telluride

formation. The suggested mechanisms include paths where tellurium oxyanion would first be reduced to elemental tellurium then further to telluride and then subsequently methylated. Another suggested mechanism would be the methylation of tellurite to form CH_3TeO_2 intermediate species that would then reduce to form dimethyl telluride. Both mechanisms are interesting for severe accident research since it has been proposed that tellurium can be present in the sump as either an oxyanion or elemental tellurium.¹¹ Some possible reaction mechanisms are presented as reactions (1), (2), and (3) (Refs. 13, 29, and 32, respectively):



and



Reactions (1) and (2) are presented as balanced reactions here, which differs from the original source.

This work provides information on the interactions between tellurium and various paint solvents in containment sump conditions as well as evidence for the relevance of organic tellurides. Moreover, the reactions and formation of organic tellurides were investigated under irradiation to provide a more realistic accident scenario. The results that were received increase the interest in tellurium behavior under severe accident conditions and highlight the need for further investigation of possible re-volatilization and even trapping of organic tellurides.

III. EXPERIMENTAL

III.A. Materials

All the reagents used in this study were analytical-grade chemicals used without further purification. Tellurium dioxide (TeO_2) (Sigma-Aldrich®, >99%) was used as the tellurium precursor in this study because of its possible presence in the containment sump. The alkaline borate [sump] solution (ABS) was prepared from 0.15 M of sodium hydroxide (EMPLURA®, 99%) and 0.23 M of boric acid (Merck, >99.8%) to milliQ water. The pH of the solution was approximately 9.3. The concentrations and pH mimic the conditions present in the containment

sump. Paint solvents were added to separate vials of the sump solution to the saturation limit. The paint solvents were texanol ester alcohol, 2,2,4-trimethyl-1,3-pentenediol monoisobutyrate (Sigma-Aldrich, 99%); MIBK (Janssen Chimica, 99.5%); and toluene (Fluka Analytical, >99.7%). All samples were done in triplicates for statistical significance.

III.B. Method

The samples were prepared by mixing a stock solution of 3 mM of TeO_2 dissolved in ABS and saturated paint solvent solution in ABS in a ratio of 1:1. The gas-to-liquid ratio in the samples was 1:1. The samples were irradiated in a Gammacell 220 ^{60}Co source (MDS Nordion, Atomic Energy of Canada Limited), giving a dose rate of 4 kGy/h for a period ranging from 1 to 3 days where the maximum dose delivered to the samples was around 300 kGy. The time was chosen in order to simulate a situation where possible later-stage releases would occur due to formation and re-volatilization of volatile species. In addition, we wanted to deliver higher doses to the samples to give significance to the species that potentially survive under irradiation before decomposing. After the samples were taken out from the gammacell, they were centrifuged for 5 min at 3500 rpm and filtered with a syringe filter (polypropylene, VWR®). Precipitation had formed in the irradiated samples, and this was prepared for analysis as described below. Reference samples were prepared in the same way, but they were not irradiated.

To determine the tellurium concentration, an inductively coupled plasma mass spectrometer (ICP-MS) (Thermo Scientific) was used. The filtered samples were diluted with 0.5 M HNO_3 (Suprapur®, Supelco), where 1 part per billion of holmium (High-Purity Standards) had been added as an internal standard due to its relatively inert chemical behavior. Tellurium standards for calibration were prepared by diluting from 10 parts per million of tellurium stock solution (High-Purity Standards).

Volatile species were analyzed using a headspace gas chromatography mass spectrometer (HS-GC-MS) (Thermo Scientific) (5%-Phenyl)-methylpolysiloxane column). Separate samples were prepared and irradiated directly in headspace vials (Genetec AB). This enabled not having to change vials between irradiation and analysis to guarantee a minimal loss of any potential volatile species. The gas-to-liquid ratio was kept the same as the samples prepared for ICP-MS analysis. The gas chromatography samples were not centrifuged nor filtered since there was no need to separate the precipitation from the

solution. Instead, the samples were analyzed directly after they had been taken out from the Gammacell 220.

A scanning electron microscope (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) (SEM-EDX) (Phenom ProX) was used to analyze the precipitation formed in the irradiated samples. A small amount of sample was placed on a glass slide and allowed to dry. This method was chosen due to the limited amount of solid material and the very fine particle size and adsorptive nature of the sample, which made it difficult to transfer the sample if dried in the original sample vial.

IV. RESULTS AND DISCUSSION

Both irradiated and reference samples were analyzed for tellurium concentration and speciation. Since no precipitation was formed in the reference samples, no SEM-EDX results are presented for these. Results presented herein aim to give an overall view of the reactions taking place under irradiation and the formation of volatile tellurides.

IV.A. Tellurium Concentration

The concentration of tellurium in the irradiated and reference samples was measured using ICP-MS. The concentration of the irradiated samples as a function of irradiation dose is presented in Fig. 1. As shown, the presence of paint solvents seems to decrease the concentration of tellurium in the irradiated samples. Texanol affected the tellurium concentration the most whereas toluene had the least effect. This is very likely due to the solubility of the solvent in water as well as the stability under irradiation. Toluene is known to have low solubility and high stability toward irradiation whereas texanol and MIBK are more soluble and susceptible to decompose under irradiation. Overall, the reduction of tellurium (IV) is likely due to water radiolysis products. The organic solvents tend to react with oxidizing radiolysis products whereas tellurium (IV) can react with either oxidizing or reducing radiolysis products due to its amphoteric nature. In the case of paint solvents, tellurium seems to react with the reducing radicals when the organic solvents scavenge the oxidizing ones. In addition, the decomposition of organic molecules produces hydrogen and carbon dioxide, both of which can facilitate tellurium reduction. The reference samples showed no change in the concentration of tellurium over time.

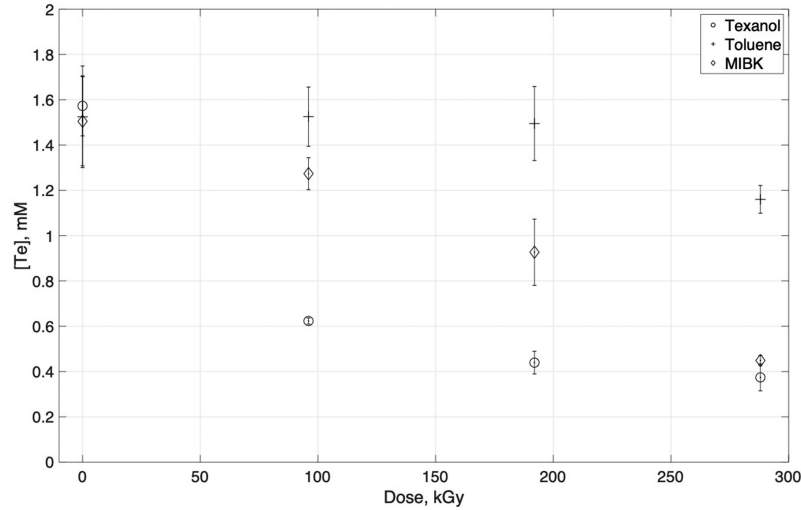


Fig. 1. Tellurium concentration as a function of radiation dose received. Uncertainties presented represent the standard deviation of the triplicate samples.

IV.B. Organic Telluride Analysis

The irradiated and reference samples were analyzed with a HS-GC-MS to identify any possible volatile tellurium-containing species. The paint solvents gave high signals in the spectra since the concentration of each solvent used was relatively high. However, the results show clear evidence of formation of organic tellurides. Different organic tellurium species were detected in samples containing texanol ester and MIBK. All mass spectra show the relative abundance of the mass-to-charge (m/z) signals that have been normalized to the highest-intensity base peak of each spectrum. Figure 2 presents the mass spectrum for a peak observed 4.26 min after injection to

the column for a sample containing texanol and TeO_2 after around 200-kGy irradiation. The spectrum has three m/z groups with distinctive isotope patterns at around 130, 174, and 216 m/z . The signals at around 130 m/z are Te^+ ions where the strongest signals come from 126, 128, and 130 m/z . These are the most abundant tellurium isotopes and therefore show the most distinctive peaks. The base peak with highest intensity is at 174 m/z , which corresponds to isopropyl telluride ($\text{C}_3\text{H}_8\text{Te}^+$), with corresponding peaks for the other two abundant tellurium isotopes at 170 and 172 m/z . Last, the molecular ion M^+ is found at 216 m/z with weaker signals at 212 and 214 m/z , which was identified as diisopropyl telluride $\{[(\text{CH}_3)_2\text{CH}]_2\text{-Te}^+\}$. A relatively high signal is found at

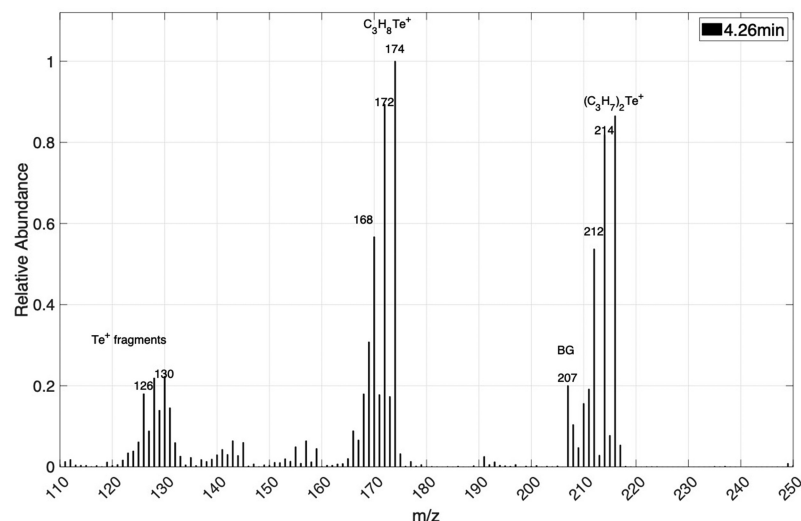


Fig. 2. Mass spectrum for the irradiated sample containing texanol and TeO_2 for a peak with retention time 4.26 min.

207 m/z, which is a background (BG) peak from the column itself.

Another peak in the irradiated texanol TeO₂ chromatogram was detected at 5.18 min. This had a similar mass spectrum pattern to the 4.26-min peak, but with slight differences (Fig. 2). The mass spectrum for this 5.18-min peak is shown in Fig. 3. The Te⁺ isotopic pattern is again found at around 130 m/z. The strongest signal for the 5.18-min peak is at 172 m/z, which corresponds to a species with a formula of C₃H₆Te. This does not correspond to isopropyl telluride. Instead, this fraction could be an allyl telluride with a C=C double bond. This assumption would also explain the signals at 212, 214, and 216 m/z, which would correspond to diallyl telluride. However, more accurate speciation analysis requires

further investigation. We can conclude by the isotopic patterns and the presence of Te⁺ fractions that the species is a form of organic telluride and the structure resembles that of diisopropyl telluride with slight, still to be confirmed, differences.

The peak with the longest retention time in the irradiated texanol TeO₂ sample was found at 7.72 min (Fig. 4). The mass spectrum for this peak shows evidence of a ditelluride, Te-Te, fraction at m/z of around 256. Moreover, the isopropyl telluride fractions are present at 170, 172, and 174 m/z in addition to the Te⁺ fractions at around 130 m/z.

The irradiated samples containing MIBK yielded different species of organic tellurides compared to those formed in the texanol samples. As in the samples containing texanol,

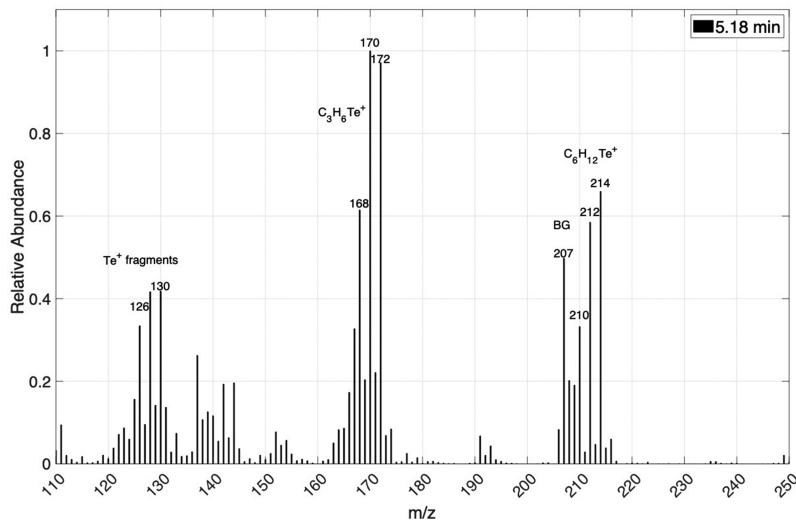


Fig. 3. Mass spectrum for the irradiated sample containing texanol and TeO₂ for a peak with retention time 5.18 min.

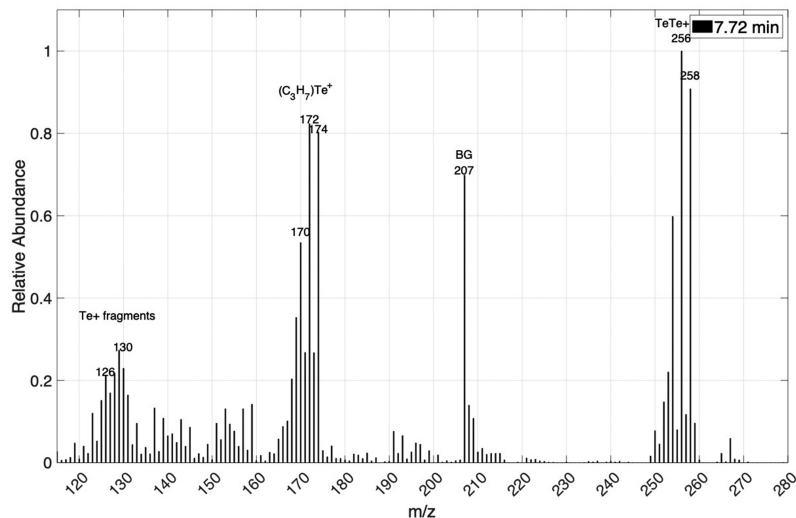


Fig. 4. Mass spectrum for the irradiated sample containing texanol and TeO₂ for a peak with retention time 7.72 min.

the irradiated MIBK samples had signals for tellurium fragments for m/z at around 126, 128, and 130 (Fig. 5). In addition, there were signals for m/z at around 144, which likely correspond to methyl telluride (CH_3Te). The mass ion M^+ was found at 202 m/z with weaker signals at 198 and 200 m/z . Compared to the methyl telluride signals, the mass difference between the signals is 57, which is a fragment that could correspond to C_4H_9 . Therefore, the tellurium species we propose for the mass ion is $\text{C}_4\text{H}_9\text{TeCH}_3$. A more thorough investigation of the exact fragments still needs to be done to accurately identify the species.

The samples containing toluene did not show any evidence of organic telluride formation nor did any of the reference samples.

IV.C. Precipitate Analysis

The precipitate from the irradiated samples was analyzed using SEM-EDX, and the results for the toluene, texanol, and MIBK samples are shown in Figs. 6, 7, and 8, respectively. The analysis confirmed that the precipitate that formed in all the samples contained tellurium. There were, however, slight differences in the tellurium speciation suggesting different behavior depending on the paint solvent.

Figure 6 shows the SEM and EDX analyses of the irradiated sample containing texanol ester. The EDX analysis identified strong signals for tellurium with very little or no oxygen, suggesting that the species was metallic tellurium. Other signals in the EDX spectrum also

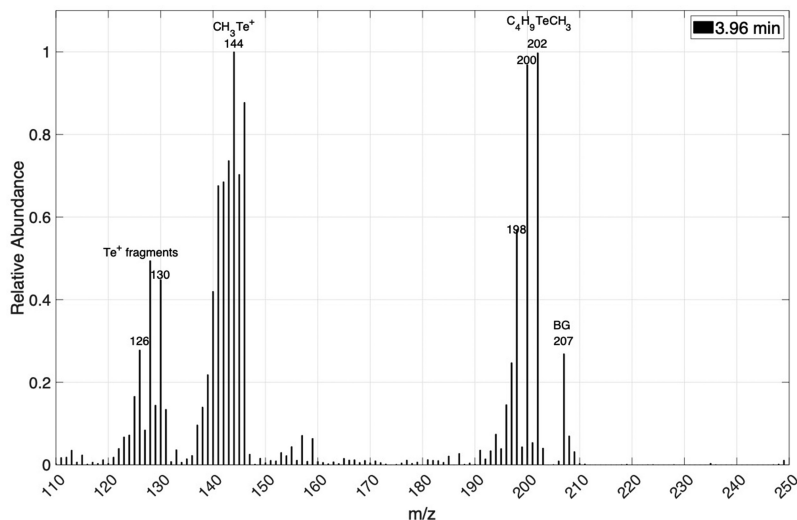


Fig. 5. Mass spectrum for the irradiated sample containing MIBK and TeO_2 for a peak with retention time 3.96 min.

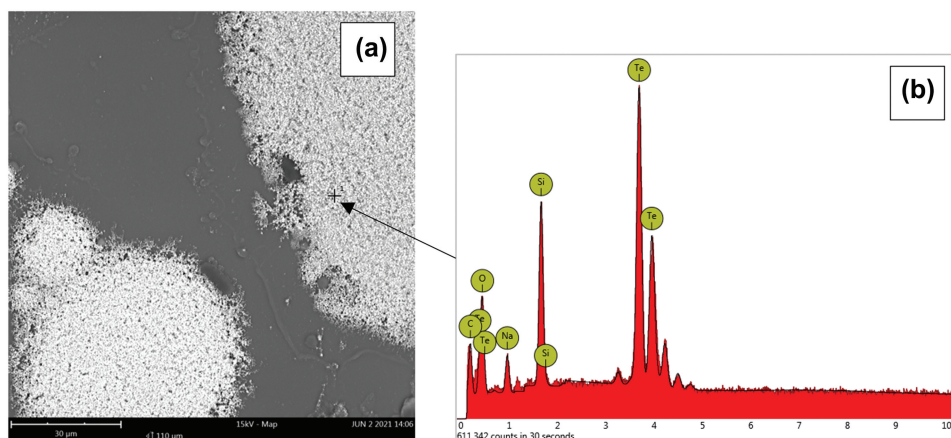


Fig. 6. Analyses from irradiated sample with TeO_2 and texanol ester: (a) SEM and (b) EDX.

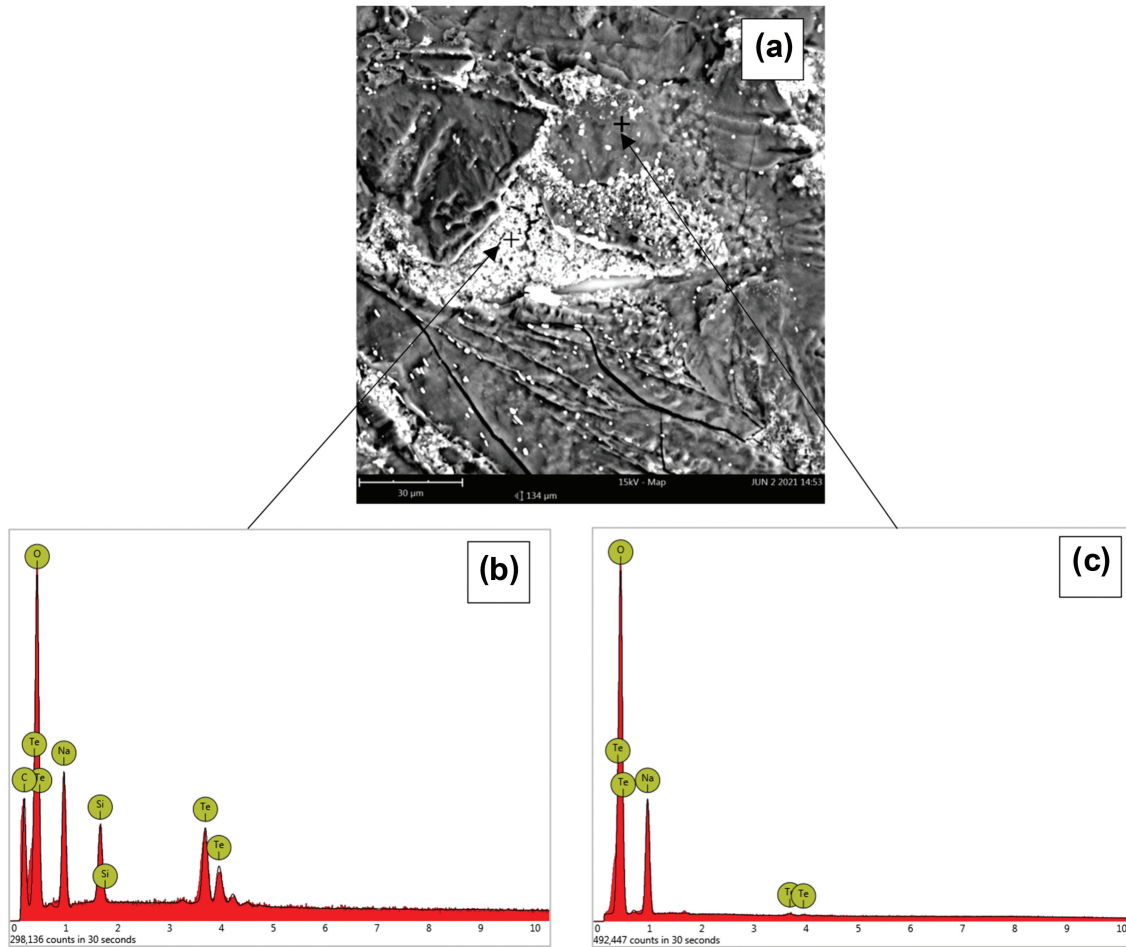


Fig. 7. Analyses from irradiated sample with TeO₂ and toluene: (a) SEM, (b) and (c) EDX.

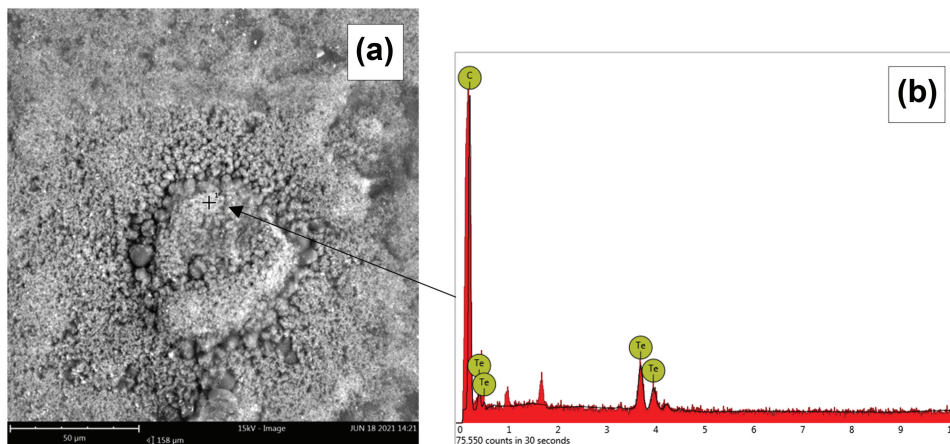


Fig. 8. Analyses from irradiated sample with TeO₂ and MIBK: (a) SEM and (b) EDX.

showed the presence of sodium and silica, which likely originate from the sodium hydroxide solution and borosilicate glass slide, respectively.

In the sample containing toluene (Fig. 7), the precipitation analysis also showed a species containing tellurium. The analysis with EDX gave stronger signals for oxygen

and weaker signals for tellurium than the sample with texanol. This probably results from incomplete reduction of TeO_2 to metallic tellurium due to weaker oxidizing radical scavenging properties of toluene compared to texanol. This in turn could be due to lower solubility of toluene in water or the higher radiation stability compared to texanol.

The SEM-EDX results for MIBK (Fig. 8) again show signals for tellurium and none for oxygen. However, there is a strong signal present for carbon, which most likely originates from MIBK droplets on the glass slide.

Overall, despite the different degrees of reduction with the three paint solvents, all the precipitates were identified as tellurium species. The differences are potentially due to several factors such as the solubility of the respective paint solvent and slight differences in the dose received, as well as the different radiolysis behavior of the solvents. However, we can conclude that all of the paint solvents react similarly by scavenging the oxidizing radicals and that in turn tellurium(IV) reacts with the reducing radiolysis product and reduces to metallic tellurium.

By combining the results presented herein, we can speculate on the interactions between tellurium and various paint solvents. There are several possible reactions for the formation of organic tellurides described in the literature, many of which could be applied to these results. Since only texanol and MIBK presented evidence of organic telluride formation, we can assume that solubility and radiation stability of the paint solvent play an important part. Toluene is known to be less soluble and relatively stable toward irradiation due to the aromatic structure whereas both texanol and MIBK are more soluble and decompose under irradiation. The reduction of tellurium (IV) to elemental tellurium seems to be the step needed for organic telluride formation. There was no evidence of any intermediate species containing tellurite and organic groups that would suggest a reaction mechanism of telluride formation directly from dissolved TeO_2 . It is also possible that the mechanism includes an intermediate ditelluride species since Te-Te signals were detected in one of the GC-MS spectra. Therefore, one of the proposed mechanisms is the formation of organic ditelluride, which decomposes to form an organic telluride. This mechanism also requires the reduction of tellurium (IV) to elemental tellurium. We were unfortunately unable to quantify the amount of organic tellurium species in the sample, and therefore, we cannot give mass balance estimations. However, it can be assumed that the amount of organic tellurides formed is very low and that the decrease in concentration under irradiation is mostly, if not entirely, due to the precipitation of tellurium. Future work will include more experiments focused on the mechanism, quantification, and stability of these organic tellurides.

V. CONCLUSIONS

The interactions between tellurium and organic molecules originating from paint solvents in the containment sump were investigated. The main interest was the potential formation of volatile organic tellurides. These organic tellurides, because of their high volatility, could increase the source term of tellurium via re-volatilization. The formation of volatile tellurium species in the sump has been suggested several times, but no research showing any relevant evidence has been presented until now. Our results suggest that the organic telluride formation in containment sump conditions is relevant, at least in the conditions presented in this work. The context in which these experiments were conducted aims to simulate the postaccident conditions where additional releases could occur as a result of re-volatilization from the sump. Results show that the formation of various organic species containing tellurium is plausible from dissolved paint solvent in the sump. The main species found was diisopropyl telluride, which was formed from TeO_2 and texanol under irradiation. Another volatile tellurium species was formed from decomposition of MIBK under irradiation. There was also evidence of dimethyl telluride formation in the samples containing MIBK. Toluene seemed to be the least affected by radiation, and no organic tellurides were formed in the solution containing toluene. Our results lay the foundations for future research involving organic tellurium species and validate the importance of further studying the formation, volatilization, and trapping of organic tellurides.

Disclosure Statement

No potential conflict of interest was reported by the author(s).

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