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Formation of Organic Tellurides and Organic Iodides Together Under Gamma Radiation

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Abstract — Tellurium is a volatile fission product and a considerable danger in case of a nuclear accident. It has a branch of organic chemistry that is a relatively unexplored part of its accident chemistry. Irradiation of an aqueous solution containing tellurium and organics can give rise to organic tellurides, which are highly volatile species and therefore represent a potential component of the tellurium source term. Iodine behaves similarly and has a well-established interaction with organic species in accidents. In this paper, the formation of organic iodides and tellurides is investigated using various organic species for precursors: acetaldehyde, methyl isobutyl ketone, Texanol ester, and solid flakes of paint representative of what is used in nuclear containments. The results imply that, while both organic tellurides and iodides can form, the presence of iodide serves to suppress the formation of tellurides. Considering this, it appears unlikely that organic tellurides are a practical concern in nuclear accidents, with the possible exception of very early in the accident sequence, when the fission yields of the fuel result in much more tellurium being present than iodine.

Keywords — Nuclear accident, accident chemistry, tellurium, organic iodide.

I. INTRODUCTION

A severe nuclear accident often entails the release of radioactive substances, posing a threat to human health. In terms of total activity released in past nuclear accidents, the elements iodine and tellurium are some of the most important ones [1,2] in terms of dose released and impact to humans. They are more volatile than most other fission products [1,3] and may therefore spread and contaminate areas far from the accident itself.

Systems are put in place at the nuclear power plants to counteract the releases of these elements (and others). Such countermeasures include the emergency core cooling

system to keep the fuel and the fission products inside it intact. Other measures include the containment spray system, which cools the nuclear containment and washes aerosols from the containment atmosphere into the aqueous sump at the bottom of the containment. In the sump, which is typically kept alkaline (pH 9.3 [4]), the iodine and tellurium will both (in principle) dissolve, preventing releases.

However, this approach is complicated by the presence of organic species in the sump. These organics may come from paint, electrical cables, lubricating oils in machinery, rubber seals, and other sources. By themselves, these organics are not a radio hazard; however, their thermal and radiolytic degradation may form reactive organic species. These, in turn, may form organic tellurides [5–7] and/or organic iodides [8–12], which are volatile.

In this paper, a simulated sump solution with both tellurium and iodine and some organic species is exposed to ⁶⁰Co gamma radiation to determine whether there is a preference for the formation of organic tellurides or organic iodides. This is a novel type of study, as the

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impact of iodine on the formation of organic tellurides has never been studied before. The organics considered are methyl isobutyl ketone (MIBK), acetaldehyde, 2,2,4-trimethyl-1,3-pentanediol diisobutyrate (Texanol), and finally cured epoxy paint. The paint experiments will use both fresh paint (cured for 2 days) and older paint (cured for approximately 2 years). The structural formulas of these species can be seen in Fig. 1.

Both MIBK and Texanol have been studied in relation to organic tellurides before [5]. In this study, it was found that organic tellurides could be formed from conditions comparable to those in the nuclear sump. Several organic tellurides were detected, including (among others) $C_4H_9TeCH_3$. Several fragments of ditellurides (with the general formula R-Te-Te-R') were also detected.

Texanol [12] and this exact paint [13] have also been studied related to organic iodides in the past. However, as tellurium and iodide are radiologically related, they will always be present together in a real accident. In terms of their fission yields, about 480 g of tellurium (of all isotopes) will be present per ton of heavy metal of spent nuclear pressurized water reactor fuel (at discharge), which can be compared to 241 g of iodine [14]. However, this is the initial situation and does not account for the decay of tellurium into iodine. During the Chernobyl accident, the tellurium isotope responsible for the most activity was ^{132}Te [15] (with a half-life of about 3.2 days). It decays through β^- decay into ^{132}I (with a half-life of about 2.3 h). However, the quintessential iodine isotope is ^{131}I (with a half-life of just over 8 days). These decay times mean that initially there is more tellurium present than iodine in terms of weight (and number of moles), but as tellurium decays more rapidly, iodine will (in principle) become the more common element. A scenario with only tellurium and no iodine is not possible due to the

formation of iodine from tellurium. A scenario with more tellurium than iodine could be considered. In practice, their relative abundance in the reactor sump also will depend on their relative releases from the fuel and the portion of the elements present in the sump as opposed to in the containment gas phase or the primary circuit.

In the Phébus FP1 experiment, a bundle of spent nuclear fuel was heated in order to simulate an accident. In this experiment, 87% of the iodine and 83% of the tellurium present in the fuel bundle were released [16]. It cannot be ruled out that the relative abundance of tellurium and iodine in the reactor sump will vary quite significantly as the accident progresses.

In principle, assuming comparable releases from the fuel, the different fission yields, and considering only ^{132}Te and ^{131}I , the system will be dominated by tellurium for, at the longest, about 6 days (this is without considering the decay of tellurium into iodine). After that, the tellurium will have decayed to such a degree that iodine will take precedence.

I.A. GCMS Analysis of Organic Tellurides and Iodides

A gas chromatograph–mass spectrometer (GCMS) measures the mass-charge ratio (m/z ratio) of any species that hits the detector. Due to the difference in the number of naturally occurring isotopes of iodine and tellurium, these elements will have different appearances in the GCMS spectrum. An iodine ion ($^{127}I^+$) will give a signal of 127 m/z (the mass is 127 u, and the charge is +1, giving a ratio of $127/1 = 127$). Tellurium-containing species, however, will give a series of signals since natural tellurium incorporates several isotopes, which gives several m/z ratios. Examples of natural tellurium isotopes are ^{126}Te , ^{128}Te , and ^{130}Te .

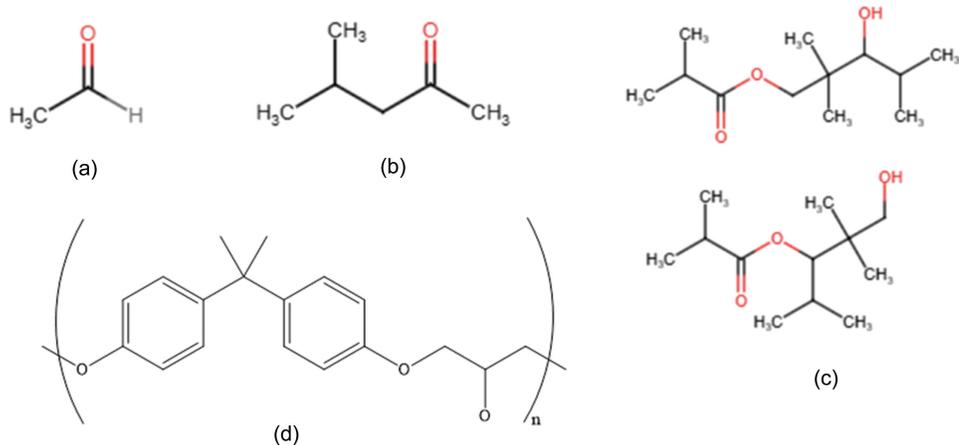
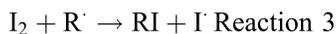
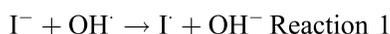


Fig. 1. Structural formulas of the organic species considered in this paper: (a) the structure of acetaldehyde, (b) the structure of MIBK, (c) the two isomers that make up the Texanol solution, and (d) the monomer unit of the epoxy polymer used for the paint.

Chemically, tellurium and iodine behave very differently. The organic chemistry of iodine, like the other halogens, is dominated by compounds in which the halogen atom forms a single covalent bond. Hypervalent iodine compounds do exist, but as these are involatile oxidants, we have chosen to ignore them. The heavier chalcogens (sulfur, selenium, and tellurium) are well known to form stable organochalcogen compounds where the chalcogen is bonded to two, three, four, or even more atoms. For example, sulfur can form dimethyl sulfide (Me_2S), dimethyl sulfoxide (Me_2SO), and dimethyl sulfone (Me_2SO_2). While the dimethyl chalcogens are volatile compounds (boiling points: Me_2S 35°C, Me_2Se 55°C, and Me_2Te 82°C), the hypervalent oxygenated dimethyl sulfides have high boiling points (Me_2SO 190°C and Me_2SO_2 238°C) and are very soluble in water. It was decided that we should concentrate on organic tellurium compounds that have two coordinate tellurium atoms, such as dimethyl tellurium.

The pH of the sump water is important, as iodine in water will form hydrogen iodide and oxyacids of iodine. In alkaline media, the iodine will be converted into iodide and iodate. Thus, unless the water is acidic, very little elemental iodine will be present. Elemental iodine is well known to trap organic radicals [17].

pH is considered a major factor for the formation of organic iodides from irradiation of aqueous solutions [8,18], as the proposed mechanism to form organic iodides requires the oxidation from I^- to I_2 , as shown in reactions 1 to 3.

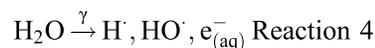


At alkaline conditions, I_2 is likely to dissociate to I^- , which reduces the formation of organic iodides.

Tellurium, just like its lighter homologues (oxygen, sulfur, and selenium), can form bonds with carbon, giving rise to organotellurium chemistry. In this context, the behavior of tellurium is not entirely unlike that of oxygen in that each tellurium atom will most often form two C-Te bonds. It will not, however, form double bonds with carbon. This means that the simplest organic tellurides are species such as dimethyl telluride or ethylmethyl telluride.

I.B. Radiation Chemistry

The concentration of water in a pure water sample is about 55.6 M. Therefore, irradiation of any water sample will be dominated by the effects of radiation on the water itself, rather than on the solute(s). Gamma irradiation of water has been studied thoroughly, and a generalized reaction is shown in reaction 4.



The radicals formed due to irradiation of water are extremely reactive and will quickly react with nearby species.

For this paper, irradiation of water samples with fission products and organics in the millimolar range with ^{60}Co gamma radiation is of interest.

The organic substances of interest for this study are all mainly aliphatic hydrocarbons with a few additional functional groups: acetaldehyde (an aldehyde), MIBK (a ketone), Texanol (an ester), and the cured epoxy paint (an aromatic ether). The concentration (50 mM) is, however, too low for direct radiolysis of the organic species to be expected to have a pronounced effect. The effects on the organic species, therefore, are expected to be due to interactions on the organic species by the radiolysis products.

For the reactions listed in Ref. [19], it can be said that the most common reaction for both HO^\cdot and H^\cdot with aliphatic hydrocarbons is the abstraction of one hydrogen to form either H_2 or H_2O and turning the original organic into a radical (ion). The location of the unpaired electron on the organic skeleton, however, depends on the structure. For solvated electrons, abstraction of hydrogen appears less common. Instead, most reactions seem to feature ionization of the organic compound, which may react further with surrounding water.

The radicals formed from the organic species will have different stabilities, which, in turn, will affect the reactivity of the radicals. For aliphatic hydrocarbons, radicals are stabilized by hyperconjugation (meaning that, for example, a methyl radical is less stable than a primary radical, which is less stable than a secondary radical, etc.).

For acetaldehyde, the formation of the methyl radical is expected. When water is irradiated, solvated electrons and a radical cation (H_2O^+) are formed. The lowest electron binding energy in acetone is 9.708 eV while that of acetaldehyde is 10.231 eV [20]. As the lowest electron binding energy in water is 12.61 eV [21], it can be reasoned that when an aqueous solution of aldehyde or

ketone is irradiated, an electron can be transferred from the carbonyl compound to the photoionized water.

It is well known that ketones and aldehydes, when irradiated with light of a 230- to 330-nm wavelength, can undergo an (n, π^*) transition. The excited molecule may then undergo cleavage [22,23] (this is referred to as a Norrish type 1 process) and form a carbon-centered radical together with an acyl radical.

As the C=O bond is much stronger (has a higher bond dissociation energy) than the C-C bond, MIBK may result in methyl radicals as well. However, from a radical stability standpoint, the isopropyl radical or isobutyl radical should be more likely. The Texanol has a lot of options [12] but noticeably includes isopropyl radicals. For all of these radicals, they can of course react with each other, creating even more possibilities.

I.C. Mitigation of Organic Iodides

Organic iodides are a well-known form of iodine in nuclear accidents [16], and filtered containment venting systems (FCVSs) are in place to prevent their releases. During an accident, the containment pressure is expected to increase, which may threaten the containment integrity. FCVSs are in place to allow for venting of the containment if the pressure increases to an unacceptable level, while still retaining gaseous radioactive species, such as organic iodides [24]. There are several types of such filters, including, among others, zeolites, sand bed filters, activated carbon, metals, and several forms of silica [24], all of which have their advantages and disadvantages for the retention of organic iodides. However, the efficiency of these filters for organic tellurides is almost completely untested. Some filters wholly or partially work by forming silver iodide from (organic) iodide that passes the filter [25]. This mechanism likely does not work with tellurium.

Activated carbon filters work well on organic iodides [26–28], as they can retain dimethyl selenide [29]. However, for organic iodides, efficiency decreases with age and humidity. How these factors influence organic tellurides is unknown.

II. METHODS AND MATERIALS

The sump solution was made from 0.1 M NaOH (98% pure) and 0.15 M B(OH)₃ (>99.5%) in Milli-Q water (this is termed “ABS”; alkaline borate solution). The pH of this solution was about 9, as is expected [4]. Fission products or one of the organic species were added to the ABS. See Tables 1 and 2 for the compositions.

For each experiment, 5 mL of a solution of ABS and an organic species (MIBK, >99.5%; Texanol, >99%; or acetaldehyde, ≥99.5%) were mixed with 5 mL of an ABS product with one or both fission products added (TeO₂, ≥99% and/or NaI, ≥99.5%) in a 20-mL headspace vial. The vials were then placed in a Gammacell 220. The dose rate was about 46.7 Gy/min, and the irradiation time for each sample was 48 h, giving approximately 134.5 kGy to the vials in total.

Three vials of each combination were prepared at the same time and from the same stock solution and were irradiated together. After irradiation, the vials were transferred to a headspace GCMS (Shimadzu GCMS-QP2020 NX, 2.5-mL gas-tight headspace syringe, headspace glass liner, zb-5 ms column) and measured on the same day. Blanks were also prepared, containing the same organic species but no fission products, and were irradiated alongside the samples, also in triplicate.

The GCMS analysis method was the same for all samples. The sample was heated for 1 min in a 60°C heating chamber and then injected into the column (splitless injection) with a 65°C syringe. The column was initially 40°C for 2 min. The temperature in the column was then increased by 10°C per minute up to 120°C. Finally, the column was held at 120°C for 2 min. The

TABLE 1
Organics Used Throughout the Study

Organic Species	Concentration/ Mass
MIBK	0.1 M
Acetaldehyde	0.1M
Texanol	Solubility limit
Solid epoxy paint (2 days old)	0.05 g
Solid epoxy paint (2 years old)	0.05 g

All species were dissolved in the ABS except for the epoxy paints, which remained as flakes.

TABLE 2
Fission Product Solutions

Fission Product Species	Concentration
TeO ₂	3 mM
NaI	3 mM
TeO ₂ + NaI	3 mM + 3 mM

ion source was at 200°C, the mass spectrometer (MS) interface was at 220°C, and the gas chromatograph (GC) inlet was at 250°C. The column flow was 1.5 mL/min, and the mass chromatography scan was between 50 and 300 m/z (scan speed 909, event time 0.3 s).

For two samples, epoxy paint used to paint the containment in a Swedish nuclear power plant (Teknopox Aqua VA) was used as the source of the organics. One set of samples used freshly cured paint (the epoxy started to cure 2 days before irradiation started), and another set used paint that was cured more than 2 years ago (886 days since it started curing). During this time, it was kept inside at room temperature. The complete experimental matrix is described in Table 3.

After the irradiation and the GCMS measurement, aliquots were collected from each sample containing tellurium in order to determine the amount of tellurium left in solution (as a significant amount of tellurium precipitated during the irradiation). These samples were filtered through 4.5- μm syringe filters and diluted 2000 times in 0.5 M HNO_3 spiked with 1 ppb holmium as an internal standard. The filtered and diluted samples were

measured with inductively coupled plasma mass spectroscopy (ICP-MS).

Iodine was not analyzed this way, as it does not precipitate.

III. RESULTS AND DISCUSSION

In all cases, the most prevalent signals in the head-space GCMS spectra are due to organic species. These differ depending on the starting material. Also, all samples were made in triplicate, and all the peaks discussed in the following sections could be confirmed in all three spectra of the same sample (though some were more or less clear in some cases). In general, the overall spectrum does not change much upon addition of tellurium or iodine, as the signals of organic tellurides and iodides are very faint compared to the signals that come from the organic species or the (stable) fragments formed after radiolysis. This is because of the relatively small amounts of organic tellurides and iodides that are formed compared to the other organics present. For this reason, the blank spectra are not included.

All spectra presented will have a blue and a pink spectrum overlaid, both with a 100 \times signal magnification. The blue spectrum is the signal measure of the m/z ratio of exactly 130. Since this is the most common tellurium isotope, any organic tellurides measured will have a signal of 130 to accompany it. Similarly, the pink spectrum measures the m/z ratio of 127. As this is the m/z ratio of the only natural iodine isotope, any organic iodides will entail this signal.

III.A. Acetaldehyde

Upon irradiating a system containing tellurium and acetaldehyde, the spectrum seen in Fig. 2 is obtained.

The blue overlay has three distinct signals. The m/z distribution from the peak at 3.5 min (peak 2 in Fig. 2) is presented in Fig. 3.

The distribution of m/z ratios shows three clear clusters of peaks. This is typical of organic tellurides, where the clusters all correspond to organic species incorporating different tellurium isotopes. The relevant information about the relevant peaks can be found in Table 4.

Organic tellurides are seen at the time stamps 2.47, 3.505, and 4.925 min (though the last one is only seen in two of the three spectra, and with a comparatively weak signal). Still, these signals all display the behavior seen in Fig. 3, with multiple m/z ratios comprising the signals. The three most prominent signals differ by a mass of 2

TABLE 3
Experimental Matrix

Organic Species	Fission Product(s)	Amount of Organic Species
MIBK	Tellurium oxide	50 mM
MIBK	Potassium Iodide	50 mM
MIBK	Both	50 mM
Texanol	Tellurium oxide	Solubility limit
Texanol	Potassium Iodide	Solubility limit
Texanol	Both	Solubility limit
Acetaldehyde	Tellurium oxide	50 mM
Acetaldehyde	Potassium Iodide	50 mM
Acetaldehyde	Both	50 mM
Old paint	Tellurium oxide	0.05 g
Old paint	Potassium Iodide	0.05 g
Old paint	Both	0.05 g
Fresh paint	Tellurium oxide	0.05 g
Fresh paint	Potassium Iodide	0.05 g
Fresh paint	Both	0.05 g
MIBK blank	—	50 mM
Texanol blank	—	50 mM
Acetaldehyde blank	—	50 mM
Old paint blank	—	0.05 g
Fresh paint blank	—	0.05 g

All experiments got the same dose and dose rate: 134.5 kGy and 46.7 Gy/min, respectively. The fission product solution was of the same concentration in every experiment, i.e., 1.5 mM for each fission product. All experiments were done in triplicate.

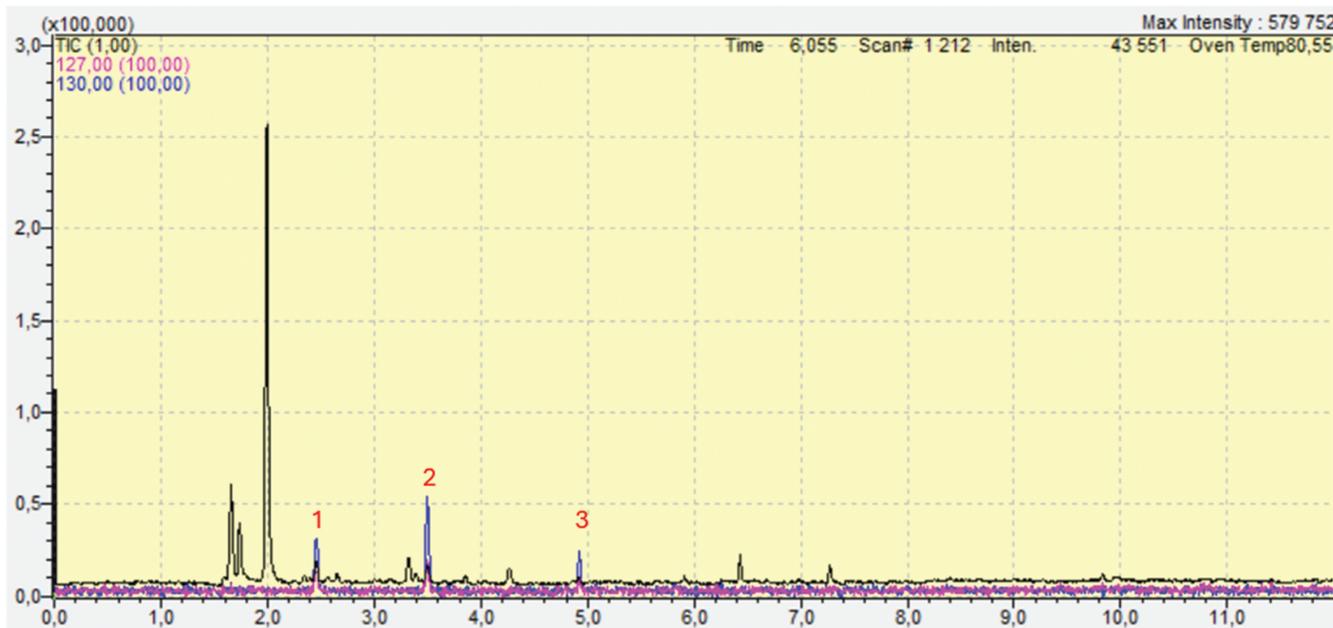


Fig. 2. GCMS spectrum of the acetaldehyde solution with 1.5 mM TeO_2 added. The blue spectrum indicates an m/z ratio of 130 (signal increased 100 \times for visibility), which is the m/z ratio of a tellurium isotope that should always accompany the formation of organic tellurides. There are three distinct peaks where tellurium appears.

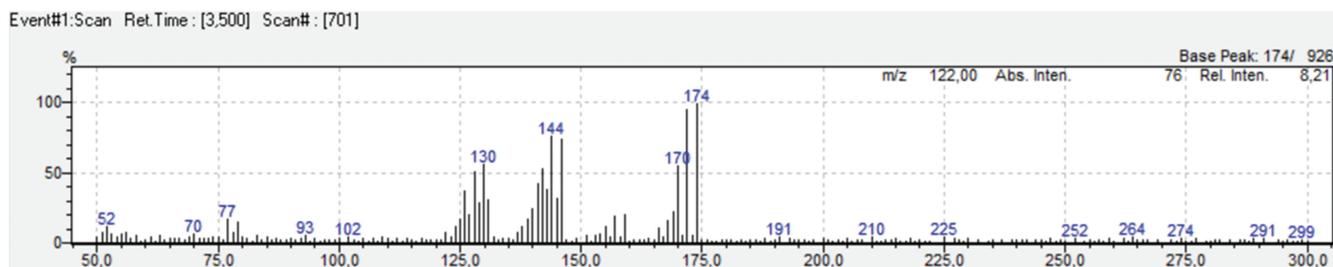


Fig. 3. m/z measurement of the peak at time 3.5 min (2 in Fig. 2), displaying the characteristic peaks of organic tellurides. The different masses within each group correspond to different tellurium isotopes.

(e.g., 170, 172, 174). This is because natural tellurium contains several isotopes, with the most common ones being 126, 128, and 130 u. Thus, any species incorporating tellurium will show a series of m/z ratios with a difference of 2. Thus, these signals correspond to organic tellurides. The specific signals 126, 128, and 130 are also seen alongside all the organic tellurides, and correspond to tellurium fragments.

The organic tellurides seen in the spectrum have the m/z ratios of 141/143/145, 142/144/146, 156/158/160, 170/172/174, and 184/186/188. While certain determination is not possible off of a GCMS spectrum alone, the signals can all be assigned potential species.

Signal 1 in Fig. 3 shows the masses 141/143/145 and 156/158/160. The latter trio corresponds to dimethyl telluride ($\text{CH}_3\text{-Te-CH}_3$). Furthermore, the mass spectrum

looks similar to what should be expected for this species [30]. This spectrum also contains the lighter signals, which can be interpreted as fragments of dimethyl telluride ($\text{CH}_3\text{-Te}^+$).

It is known that when acetone is irradiated, carbon monoxide, hydrogen, methane, and ethane are formed. The addition of radical traps (iodine or diphenyl picrylhydrazyl) only slightly reduces the G values for carbon monoxide and ethane, but the hydrogen (H_2) yield increases while the methane yield decreases. Thus, it can be assumed that the radiation causes the acetaldehyde to fragment into methyl radicals, hydrogen atoms, and carbon monoxide [31].

Similarly, for signal 2 in Fig. 3, the m/z ratios displayed are 142/144/146 and 170/172/174. The heavier signal corresponds to methyl-ethyl-telluride ($\text{C}_2\text{H}_5\text{-Te-CH}_3$). The

TABLE 4

The Organic Telluride Signals Determined in the Spectra for the Acetaldehyde Solution with Tellurium Added

Spectrum Peak (number in Fig. 3)	Signal (m/z)	Relative Signal Intensity (%)	Signal Time (min)
1	160/158/156	100	2.470
	145/143/141	93	
	130/128/126	25	
2	174/172/170	100	3.505
	146/144/142	81	
3 ^a	130/128/126	58	4.925
	188/186/184	100	
	160/158/156	62	
	130/128/126	84	

^aOnly seen in two of the three spectra.

lighter signal is less certain. If it corresponds to the same species as signal 4 in Fig. 3, then it is consistently off by one hydrogen atom. This can potentially be explained through the mechanism presented by Van Amsterdam et al. [32] for the case of *n*-propyl 2,6-dimethylphenyl thioether fragments forming a molecular ion. This was rationalized as being due to the sulfur-centered radical cation of *n*-propyl 2,6-dimethylphenyl thioether abstracting a hydrogen atom

from the beta carbon (2 carbon) in the propyl chain. The sulfur-carbon bond in the new carbon-centered radical then cleaved, thus creating a molecule of propylene. We suggest that the Te-ethyl compounds are able to undergo an analogous fragmentation reaction, thus forming radical cations such as MeTeH⁺.

Finally, signal 3 in Fig. 3 displays the m/z ratios of 156/158/160 and 184/186/188. This pattern can be interpreted in the same manner as the one for signal 4. The heavier triplet corresponds to diethyl telluride (C₂H₅-Te-C₂H₅), whereas the lighter triplet corresponds to a fragment that has lost one ethyl group (C₂H₅-Te⁺). This explanation does not account for the fact that the lighter triplet is made up of even numbers, however.

The formation of diethyl telluride and dimethyl telluride implies the formation of ethyl/ethyl radicals from acetaldehyde. Methyl radicals can be expected by cleaving the only C-C bond available, and ethane radicals would form from the combination of two methyl radicals or through removal of the oxygen (see also Sec. IV.A).

A representative spectrum for when only iodine is added (no tellurium present) can be found in Fig. 4. No significant signal is present with the m/z ratio of 130, implying that no organic tellurides are formed, as expected. The m/z ratio of 127 is very apparent, however, around 2 min into the experiment. Two signals are seen, though one is much weaker than the other.

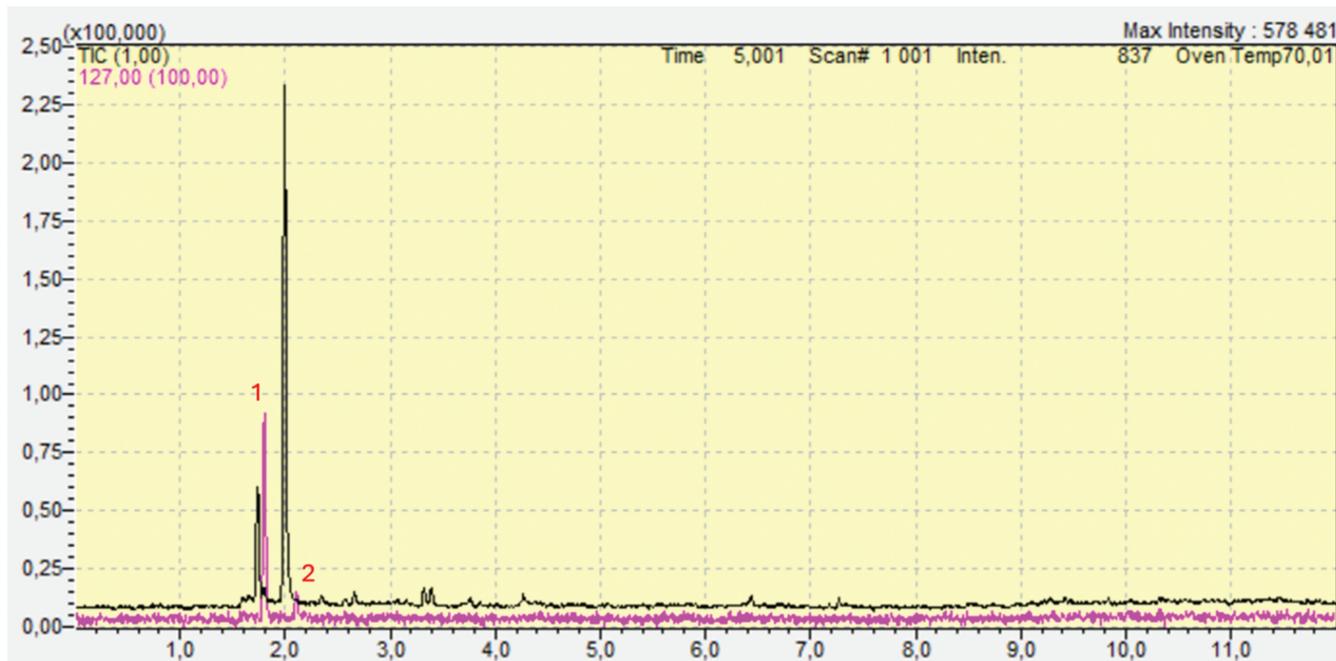


Fig. 4. GCMS spectrum of the acetaldehyde solution with 1.5 mM NaI added. The pink spectrum indicates an m/z ratio of 127 (signal increased 100× for visibility), which is the m/z ratio of an iodine ion that should always accompany the formation of organic iodides.

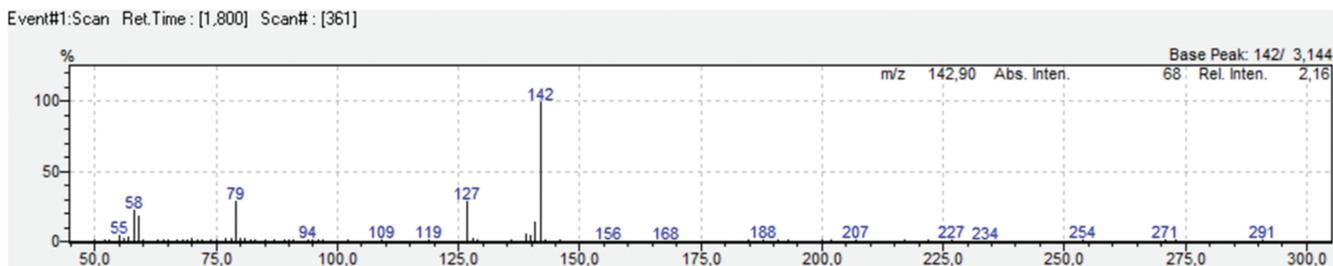


Fig. 5. m/z measurement of the peak at time 1.8 min (1 in Fig. 4). The strongest signals correspond to iodine ions (127) and methyl iodide (142).

The mass m/z distribution at signal 1 in Fig. 4 can be found in Fig. 5. The spectrum contains m/z ratios of both 127 and 142. This implies the formation of methyl iodide, which would have the m/z ratio of 142. The fact that the 127 m/z ratio appears alongside it makes it very likely that methyl iodide ($\text{CH}_3\text{-I}$) has formed. Signal 2 in Fig. 4, while weaker, looks similar, with the most prevalent signal having an m/z ratio of 156, corresponding to ethyl iodide ($\text{CH}_3\text{-CH}_2\text{-I}$).

When both tellurium and iodine of equal concentration are present and irradiated in a solution of acetaldehyde, the formation of organic tellurides appears to be suppressed. An example of such a spectrum can be seen in Fig. 6.

The m/z distributions at the time stamps 3.5 min (where organic tellurides were seen) and 1.8 min (where organic iodides were seen) can be seen in Figs. 7 and 8, respectively. Evidence of organic iodides (methyl iodide) is clear in Fig. 8 from the signal with m/z ratio of 142, and the accompanying an m/z ratio of 127. Organic tellurides, on the other hand, cannot be determined from Fig. 7, implying that formation of organic iodides suppresses the formation of organic tellurides. See also Sec. IV.B.

III.B. Methyl Isobutyl Ketone

Methyl isobutyl ketone was investigated in the same manner as acetaldehyde. It does generate several different

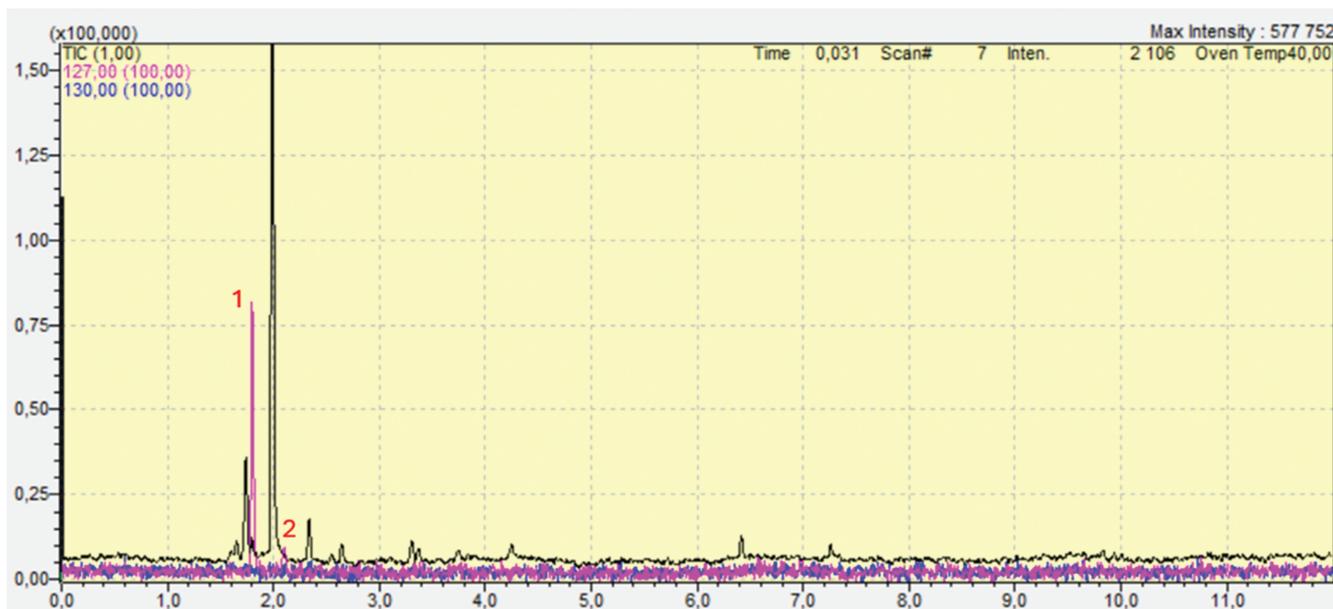


Fig. 6. GCMS spectrum of the acetaldehyde solution with 1.5 mM NaI and 1.5 mM TeO_2 added. The pink spectrum indicates an m/z ratio of 127 (signal increased 100 \times for visibility), which is the m/z ratio of an iodine ion that should always accompany the formation of organic iodides. The blue spectrum indicates an m/z ratio of 130 (signal increased 100 \times for visibility), which is the m/z ratio of one of the most common Te ions. Note that no clear signal for Te is visible.

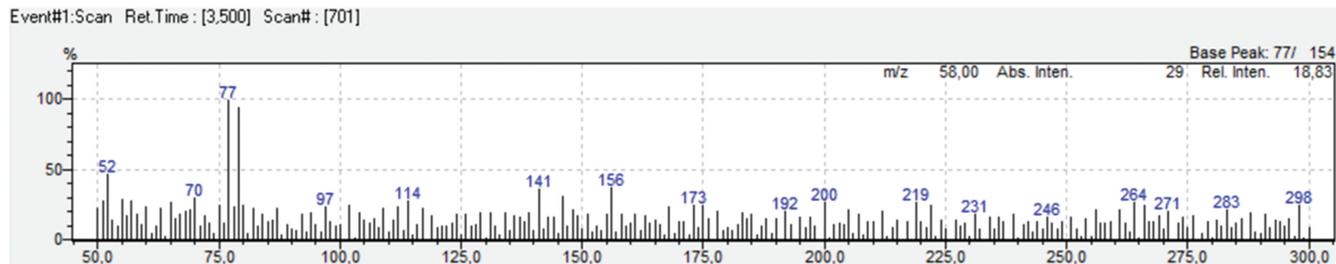


Fig. 7. m/z measurement at time 3.5 min in Fig. 6. No evidence of organic tellurides can be determined, despite the time stamp matching that of the organic tellurides when tellurium was present in solution without iodine.

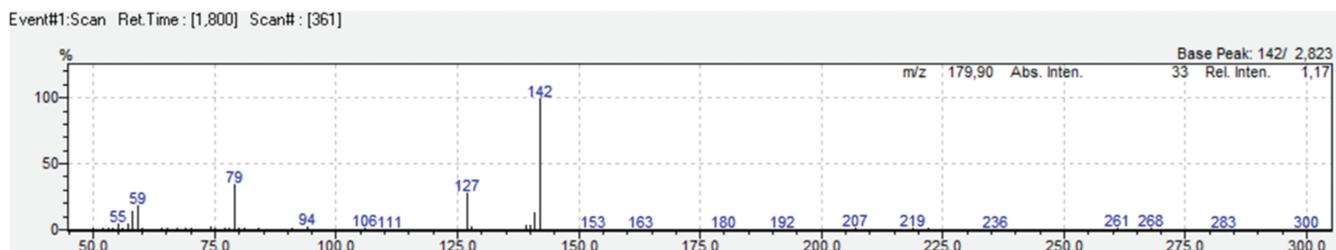


Fig. 8. m/z measurement at time 1.8 min (1 in Fig. 6). The strongest signals correspond to iodine ions (127) and methyl iodide (142), just as in the case with only iodine present in the solution.

fragments, however, resulting in a spectrum with considerably more peaks than acetaldehyde. Some of these have the m/z ratio weight of 130 or 127. However, upon comparison with the blank spectra, the ones corresponding to organic tellurides or iodides can still be determined.

The spectrum from a sample with MIBK and tellurium can be seen in Fig. 9.

Organic tellurides can be confirmed with certainty 2.43 and 6.16 min into the experiment (signals 1 and 3 in Fig. 9, respectively). Signal 2 also displays the characteristics of organic tellurides but is very weak. An example of the m/z distribution for signal 3 in Fig. 9 can be seen in Fig. 10.

The masses detected in signals 1, 2, and 3 are all listed in Table 5.

Signal 1 in Table 5 likely corresponds to dimethyl telluride. It has the same m/z ratio and arrives at almost the same time as signal 1 in Fig. 2, so the same logic applies. Signal 2 displays the same configuration of m/z ratios as signal 3 in Fig. 2. However, there is a difference in the measurement time between them, implying they are different species. Given that MIBK is a larger molecule (with potentially larger radical fragments forming due to the irradiation) than acetaldehyde, one possibility is that the species seen in the case of MIBK is methyl-butyl telluride ($\text{CH}_3\text{-Te-C}_3\text{H}_7$). This would have the same m/z ratio as diethyl telluride but behave differently in the

column, explaining the different times for their arrival and measurement.

Finally, signal 3 in Table 5 is an interesting signal. The heaviest triplet should contain in total five carbon atoms (so the telluride has the summation formula $\text{TeC}_5\text{H}_{12}$). However, by looking at Fig. 10, it is very clear that the organic telluride breaks up into Te-CH_3^+ much more often than any other fragment. This ought to imply that the species from which this fragment forms should contain the Te-CH_3 , specifically, which would make the only feasible compound methyl-propyl telluride ($\text{CH}_3\text{-Te-C}_4\text{H}_9$). Looking at the structure of MIBK, an appealing connection to make is that the propyl fragment may derive from breaking the bond between the carbonyl group and the aliphatic group. The organic telluride would then be methyl-isobutyl-telluride (depicted in Fig. 11).

The addition of iodine in place of tellurium gives a spectrum like the one depicted in Fig. 12. No noticeable signals for m/z ratio 130 can be seen, but four new signals for 127 have appeared early on in the spectrum.

Signals 1 and 2 in Fig. 12 have the same time stamp and m/z ratio distribution as the two signals seen for acetaldehyde. They correspond to methyl iodide and ethyl iodide, respectively. Signal 2 is very weak and barely

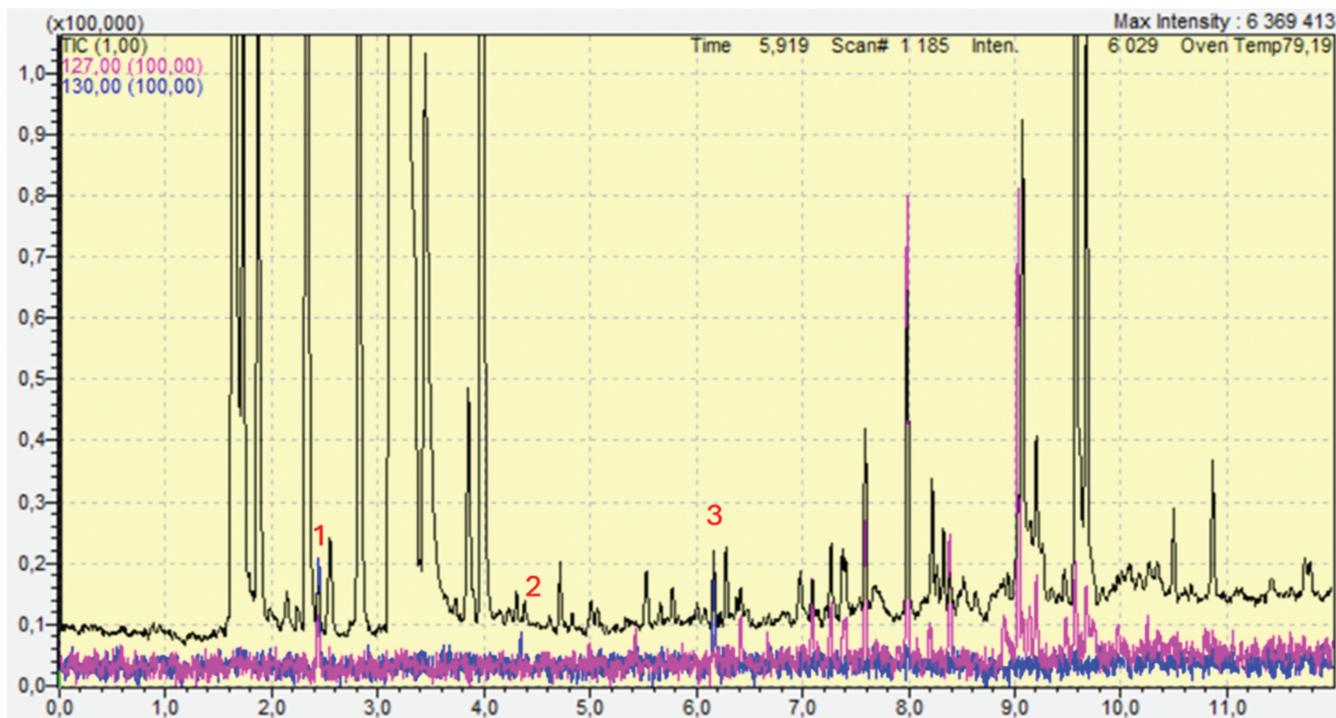


Fig. 9. GCMS spectrum of a sample containing 50 mM MIBK and 1.5 mM TeO₂ in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100× magnification) signals of the m/z ratio of 127 (pink) or 130 (blue).

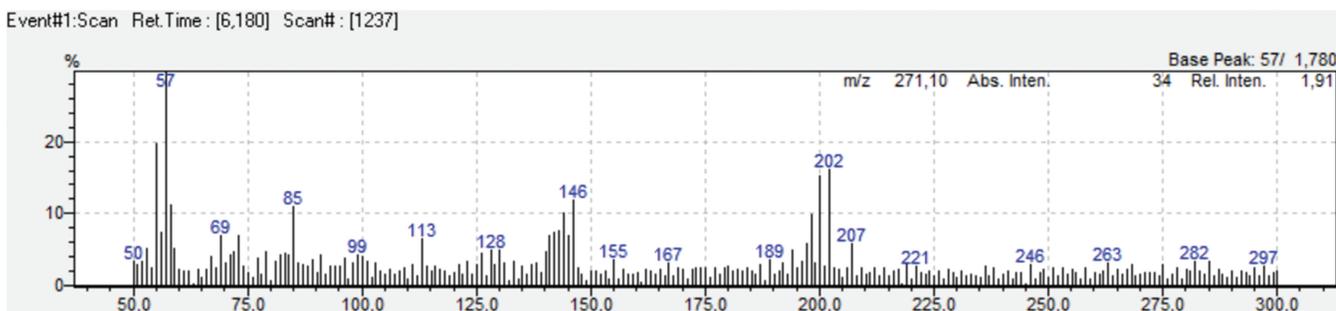


Fig. 10. m/z distribution 6.16 min into the experiment with ABS containing 50 mM MIBK and 1.5 mM TeO₂. Organic tellurides are evident at ratios around 146 and 200. Note that the strong signal at m/z ratio 57 makes the organic telluride signals have a low relative intensity.

visible in the other two samples of the same system. A sample diagram for signal 3 is presented in Fig. 13.

The sharp signal on the m/z ratio of 127 implies iodine fragments, and 170 matches the m/z ratio of propyl iodide. As perhaps could be expected, signal 4 in Fig. 12 displays a prominent m/z ratio of 184, corresponding to butyl iodide.

If both iodine and tellurium are present together in a solution of 50 mM MIBK and irradiated to 125 kGy, the spectrum depicted in Fig. 14 is observed.

Most signals are recognized from the previous investigations into tellurides and iodides. However, the fact that they are both seen at the same spectrum does confirm

that the presence of either does not by itself prevent the formation of the other. This becomes very clear by looking at the m/z ratio distribution of signal 2 in Fig. 14, where both organic tellurides and organic iodides can be seen reaching the mass spectrometer at the same time. This distribution can be seen in Fig. 15.

III.C. 2,2,4-Trimethyl-1,3-Pentanediol Diisobutyrate (Texanol)

Texanol was investigated in the same manner as the previous compounds. Of the three experiments performed

TABLE 5

The Organic Telluride Signals Determined in the Spectra for the MIBK Solution with Tellurium Added

Spectrum Peak (number in Fig. 9)	Signal (m/z)	Relative Signal Intensity (%)	Signal Time (min)
1	130/128/126	23	2.45
	145/143/141	98	
	160/158/156	100	
2 ^a	130/128/126	29	4.35
	146/144/142	43	
	188/186/184	45	
3	130/128/126	5	6.15
	146/144/142	12	
	202/200/198	20	

^aConsistently weak but detectable in all three spectra.

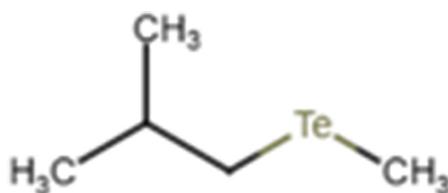


Fig. 11. 2-Methyl-1-methyltellanylpropene, the species tentatively assigned to the heaviest organic telluride detected upon irradiating the MIBK solutions.

on this system, organic tellurides are only detected in one of them, implying that this system may form organic tellurides but only with difficulty. The reason for this discrepancy is not clear. The spectrum where organic tellurides could be discerned can be found in Fig. 16. For the sake of clarity, the blue overlay is presented in the cut-in image.

In total, six peaks are detected with the m/z ratio of 130. However, peaks 2 and 6 do not display the clustered peaks that indicate organic tellurides. Instead, these signals are from organic compounds that happen to have an m/z ratio of 130. Furthermore, no conclusions can be drawn from peak 5, as the noise from other signals hides the signals from any organic tellurides. It is thus not possible to determine whether or not organic tellurides are present at this peak. The m/z distribution of the remaining peaks can be found in Table 6.

The organic tellurides formed in this system are heavy compared to those of the previously studied systems. Most likely this is because the source of organic is also the heaviest. This may be (part of) the reason only one sample of three yielded any organic tellurides with Texanol as the organic source.

Signal 1 is identical, in terms of both m/z ratio (s) and time of measure, to signal 2 in Table 5 and should therefore be the same species (probably methyl-butyl telluride).

Signals 3 and 4 both display the same m/z ratio (s) but arrive at different times, meaning that they are different species. The m/z ratio distribution for signal 3 is presented in Fig. 17.

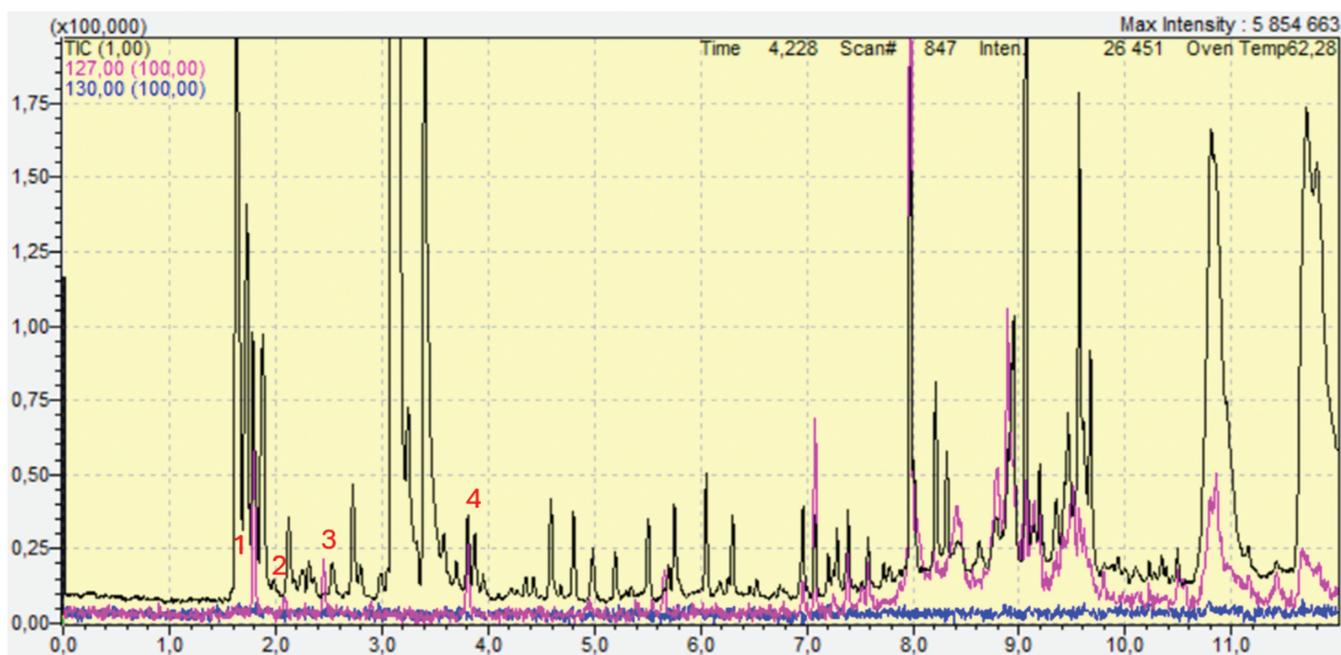


Fig. 12. GCMS spectrum of a sample containing 50 mM MIBK and 1.5 mM NaI in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100 \times magnification) signals of the m/z ratio of 127 (pink) or 130 (blue).

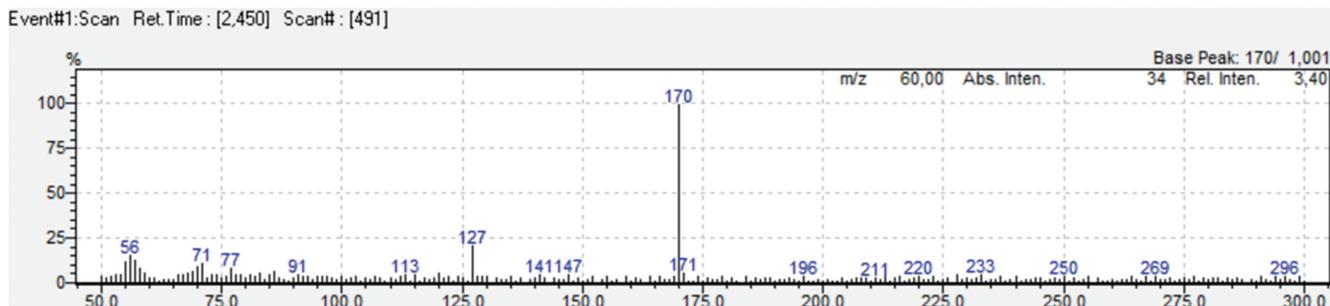


Fig. 13. m/z distribution 2.45 min into the experiment with ABS containing 50 mM MIBK and 1.5 mM NaI. The 170 signal together with the 127 signal implies the formation of propyl iodide.

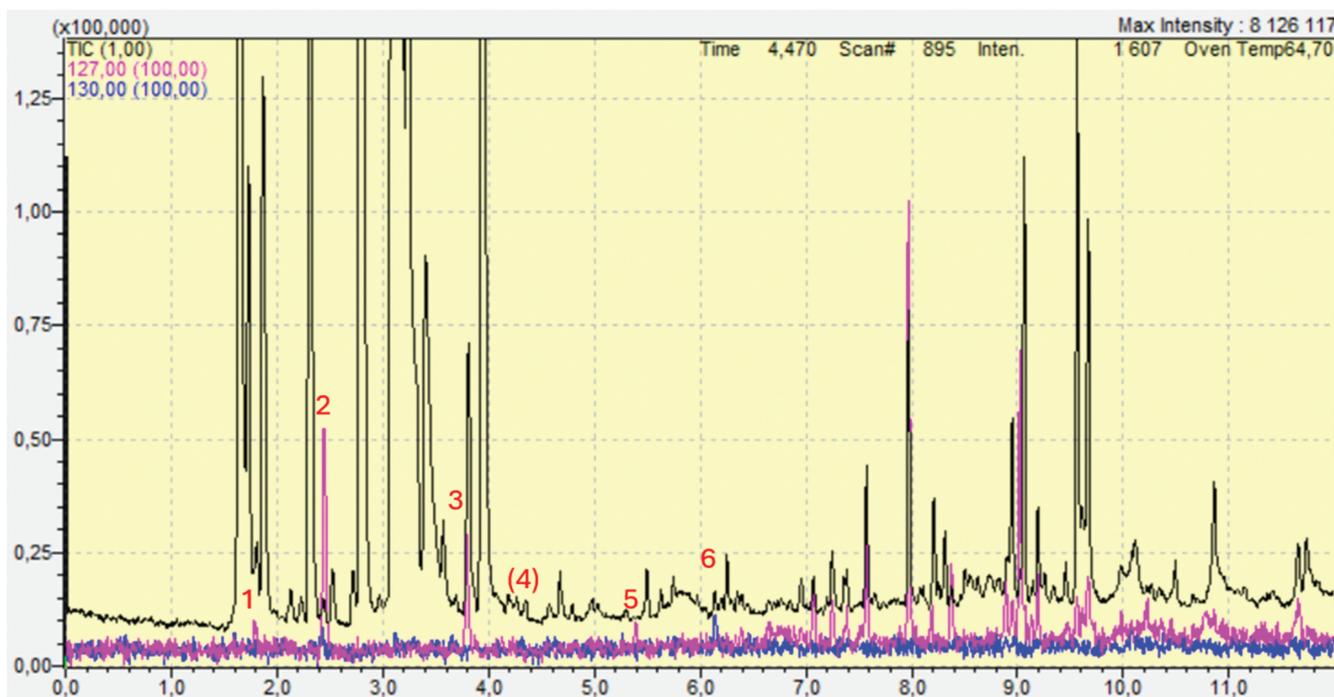


Fig. 14. GCMS spectrum of a sample containing 50 mM MIBK and 1.5 mM NaI and 1.5 mM TeO_2 in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100 \times magnification) signals of the m/z ratio of 127 (pink) or 130 (blue). The marked points 1, 3, and 5 only refer to the pink overlay; 4 and 6 only refer to the blue; and point 2 has both a blue and pink signal simultaneously. Point 4 is extremely weak and only recognizable as organic tellurides thanks to the time stamp lining up with what has been seen in Fig. 9.

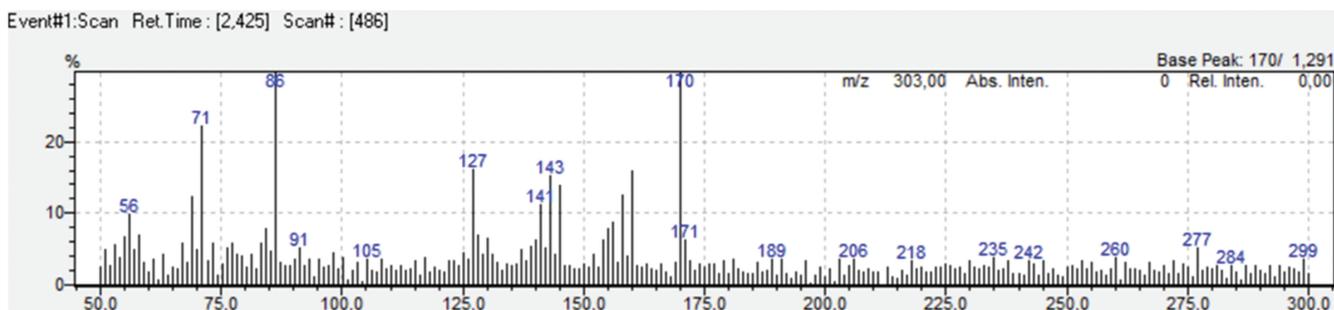


Fig. 15. m/z distribution 2.4 min into the experiment with an ABS solution containing 50 mM MIBK and 1.5 mM TeO_2 and 1.5 mM NaI. Both organic tellurides and iodides are evident.

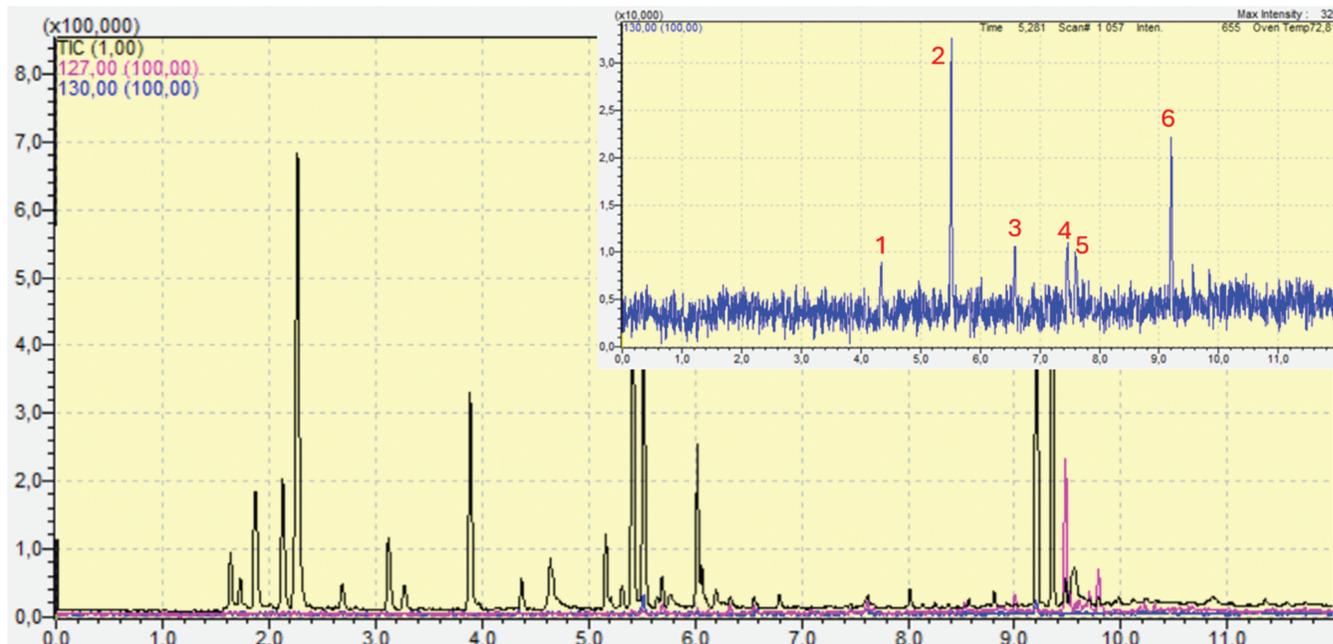


Fig. 16. GCMS spectrum of a sample containing 50 mM Texanol and 1.5 mM TeO_2 in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100 \times magnification) signals of the m/z ratio of 127 (pink) or 130 (blue). Several signals are detected that could indicate organic tellurides.

TABLE 6

The Organic Telluride Signals Determined in the Spectra for the Texanol Solution with Tellurium Added

Spectrum Peak (number in Fig. 9)	Signal (m/z)	Relative Signal Intensity (%)	Signal Time (min)
1	188/186/184	55	4.35
	146/144/141	56	
	130/128/126	38	
3	216/214/212	89	6.59
	174/172/170	80	
	130/128/126	19	
4	216/214/212	46	7.47
	174/172/170	55	
	130/128/126	20	

The total m/z ratio for the heavier signal corresponds to a summation formula of $\text{TeC}_6\text{H}_{14}$. Furthermore, the lighter fragments are also the same for both signals (with an m/z ratio of 174/172/170). This implies that both compounds predominantly form a fragment with the summation formula TeC_3H_7 , as this fragment corresponds to the lighter weights. This, in turn, means that both species should be on the form $(\text{H}_7\text{C}_3\text{-Te-C}_3\text{H}_7)$, with the only difference being one or both organic branches.

Conclusively determining the organic branches is not possible from these spectra alone.

Texanol under radiation has been investigated before [12], and one of the weakest carbon-carbon bonds in the molecule is noted to be the bond next to the double-bonded oxygen. When an aqueous solution of Texanol ester is irradiated, it could undergo a similar process to generate isopropyl radicals as a result of an α -cleavage reaction. Breaking of this bond would form a 2-propanol radical ($\text{H}_3\text{C-C}^*\text{H-CH}_3$), which could then give rise to an “isopropyl telluride group.” Another possibility is the addition of one “acetaldehyde” to a tellurium molecule ($\text{Te-CH}_2\text{-CH=O}$). Finally, there is of course the possibility of the “normal” straight propyl group. Any combination of these would match the m/z ratio(s) seen.

When iodine is added in place of tellurium, a typical spectrum as shown in Fig. 18 can be seen. Only four signals correspond to organic iodides. Of these, two are much clearer than the other two. Signals 1, 2, and 3 have the same m/z ratio distributions of 142, 156, and 170, respectively, as have been seen for the other organic species, meaning these correspond to methyl iodide, ethyl iodide, and propyl iodide (or isopropyl iodide), respectively.

Signal 4 is new, however, and has the same m/z ratio as signal 3; i.e., the m/z ratio of the organic iodide that accompanies the 127 signal is 170, implying another form or

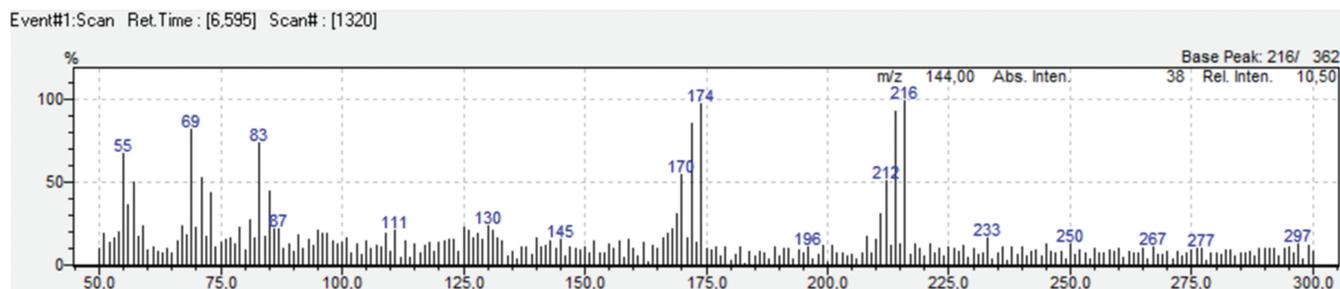


Fig. 17. Organic tellurides determined in the Texanol sample.

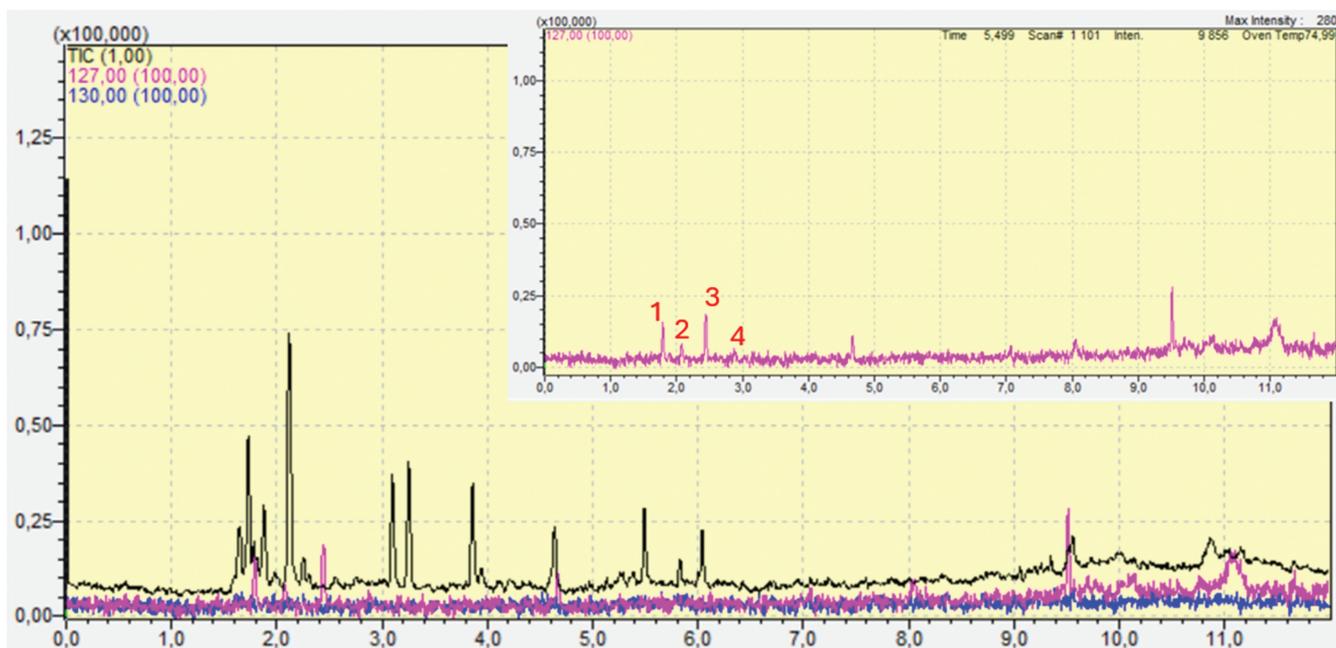


Fig. 18. GCMS spectrum of a sample containing 50 mM Texanol and 1.5 mM NaI in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100× magnification) signals of the m/z ratio of 127 (pink) or 130 (blue). The cut-in picture shows only the pink overlay. Four signals (marked 1–4) correspond to organic iodides.

propyl iodide. In light of the discussion of the different tellurides, isopropyl iodide appears to be a candidate.

If iodine and tellurium are added together, a representative spectrum as shown in Fig. 19 can be seen, with the spectrum for the m/z ratios 127 and 130 as the cut-in picture.

Organic iodides can be seen yet again, corresponding to methyl iodide (signal 1) and propyl iodides (signals 2 and 3). All of these signals appear in all three experiments. Ethyl iodide cannot be clearly discerned from any of these measurements, however.

For organic tellurides, no clear signals can be seen; however, by looking at the times when they are known to eluate, the signal at 6.59 min does appear even with iodine present in two of the three experiments. The signal is very weak, however, with a relative intensity of 10% or

less. An example of the m/z distribution at this time is displayed in Fig. 20.

III.D. Irradiation of Fresh and Old Epoxy Paint

Immediately after the radiation is complete, it is obvious that these samples behave differently compared to those using liquid organics. Both samples using paint as the organic source have failed to reduce the TeO_2 into Te metal. The paint flakes do not appear to have changed in any discernible way.

Upon measurement with the GCMS, no organic iodides or tellurides can be confirmed in any of the samples. Overall, the spectra for these samples show very little organic material overall. It does not appear likely that cured

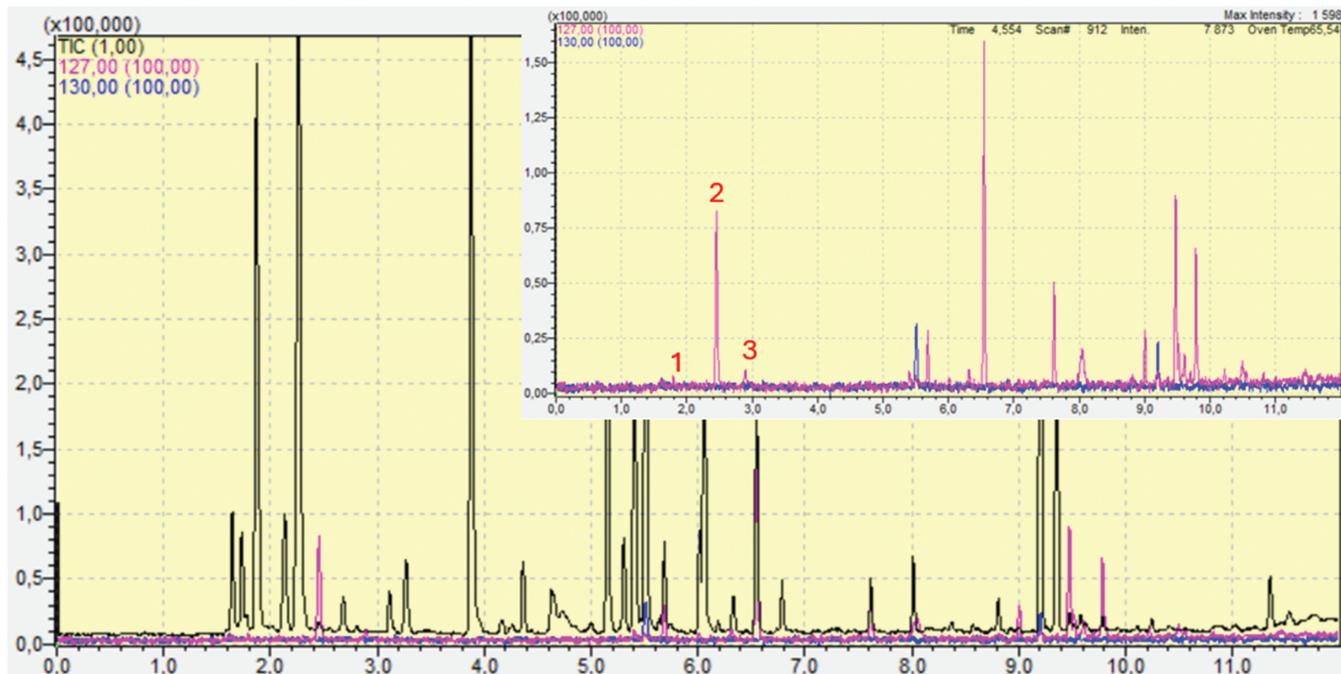


Fig. 19. GCMS spectrum of a sample containing 50 mM Texanol and 1.5 mM NaI and 1.5 mM TeO₂ in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100× magnification) signals of the m/z ratio of 127 (pink) or 130 (blue). The marked points 1, 2, and 3 indicate the three discernible peaks for organic iodides.

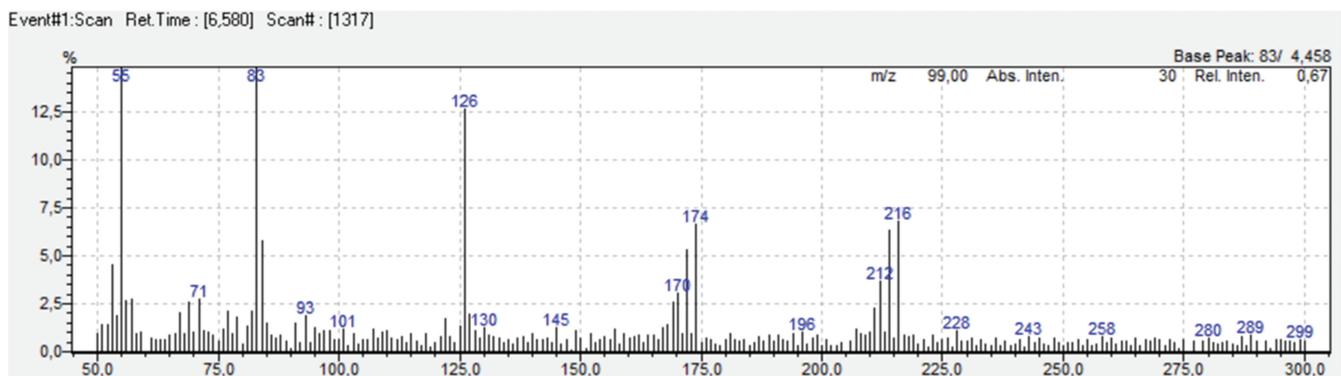


Fig. 20. Organic tellurides seen when 50 mM Texanol in ABS is irradiated with 1.5 mM TeO₂ and 1.5 mM NaI. The elution time is 6.58 min.

epoxy paint can serve as a viable precursor to organic iodides and/or tellurides, even under heavy radiation.

An example of a spectrum from the freshly cured epoxy paint with both tellurium and iodine present can be found in Fig. 21.

III.E. ICP-MS of the Liquid Phase After Irradiation

After the GCMS analysis was complete, aliquots were collected for each sample containing tellurium. These were analyzed with ICP-MS to determine the amount of tellurium still in solution after the

irradiation. The samples were diluted 2000 times for the measurement. The results of this measurement (corrected for the blank) are presented in Table 7. The concentration in millimolars is calculated based on the average concentration for each of the three samples of the same type and adjusted for the portion of ¹³⁰Te present in natural tellurium (34.1%).

The standard deviations are very large for most of these measurements, implying a significant difference between different samples in each triplicate. The reason for this is unknown. Furthermore, the concentration is only ¹³⁰Te, which makes up 34.1% of the total tellurium. It can be

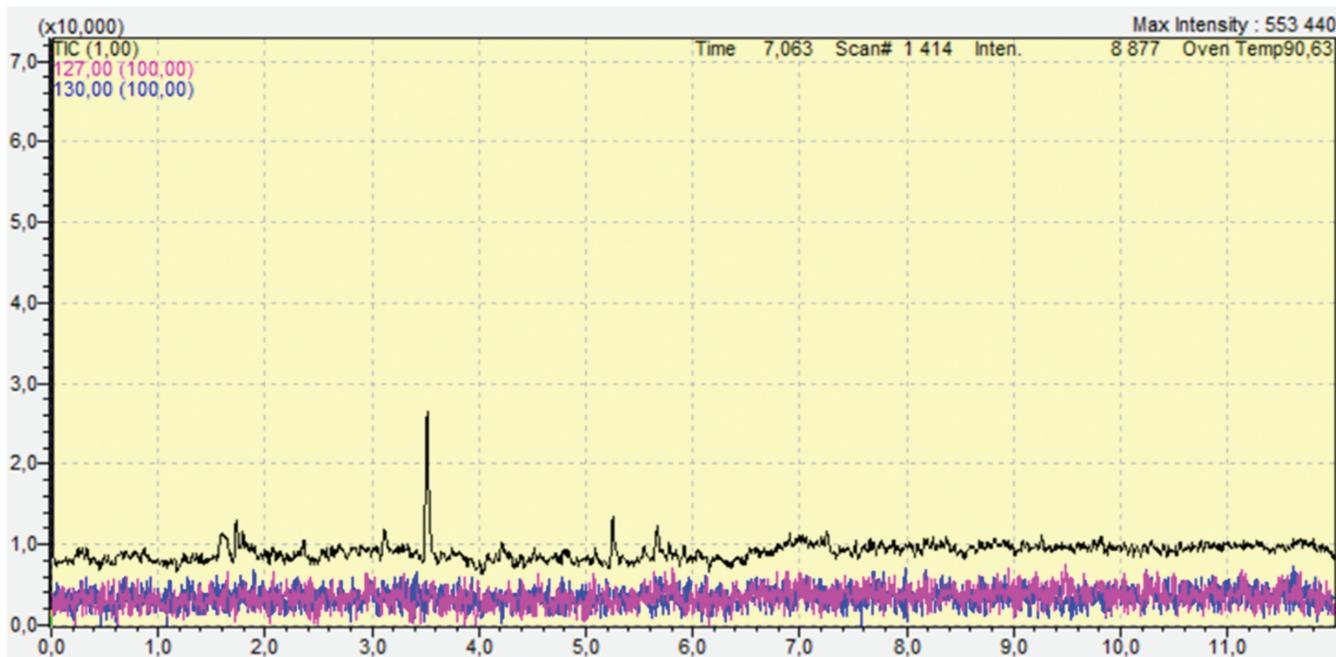


Fig. 21. GCMS spectrum of a sample containing freshly cured epoxy paint and 1.5 mM NaI and 1.5 mM TeO₂ in ABS after 125 kGy irradiation. The pink and blue overlays indicate (with 100× magnification) signals of the m/z ratio of 127 (pink) or 130 (blue). Only limited signals are seen in this spectrum.

TABLE 7

Results of the ICP-MS Measurements

Sample	Measured ¹³⁰ Te Concentration (ppb)	Standard Deviation (%)	Average Te Concentration (mM)
Acetaldehyde, Te	11.88	3.09	0.54
Acetaldehyde, Te+I	9.94	8.05	0.45
MIBK, Te	21.93	27.46	1.01
MIBK, Te+I	25.14	29.30	1.16
Texanol, Te	15.38	53.59	0.71
Texanol, Te+I	16.50	34.63	0.76
Fresh epoxy paint, Te	25.69	16.78	1.18
Fresh epoxy paint, Te+I	51.50	56.00	2.37
Old epoxy paint, Te	24.78	3.43	1.14
Old epoxy paint, Te+I	22.57	8.04	1.04

assumed that both the paint samples still retain all the 3 mM of the initial tellurium in solution. This also lines up with the fact that no tellurium could be seen in these samples with the naked eye, which was not the case for the other samples.

All the Texanol, acetaldehyde, and MIBK samples did have visible tellurium in their vials after the irradiation. In light of the concentrations presented in Table 7, most likely the MIBK solution was the least efficient at converting TeO₂ into Te metal (very little Te seems to have been reduced by this system), whereas the addition of Texanol and acetaldehyde

made for similar conversions. However, due to the large uncertainties, it is not possible to say for certain.

III.F. Potential Reaction Mechanism

pH was determined to still be alkaline in the samples after irradiation. This does go against the idea that the alkalinity by itself prevents the formation of organic iodides even under radiation by inhibiting the oxidation of I⁻ to I₂. Several forms of organic iodides could be detected in these experiments.

Of the species investigated in this study, all but the paint samples did form organic tellurides and iodides. The paint samples were also the only ones where tellurium failed to be reduced from Te^{4+} to tellurium metal. It is assumed that these two circumstances are connected. It is known that the presence of certain organic species can affect the radical species formed due to gamma radiation. Isopropanol, for example, is a known scavenger of both HO^\cdot and H^\cdot , making the system more reductive by converting the oxidizing HO^\cdot into reductive species [33].

The investigated systems do not contain isopropanol, so this mechanism is not directly applicable. However, given the decrease in tellurium concentration in solution for the systems containing MIBK, acetaldehyde, and Texanol, a similar reaction is expected to occur. It is also possible that oxidation or reduction of the organic species in a first step enables the formation of a scavenger in a later step.

All organic tellurides observed in this series of experiments have been of the form $\text{R-Te-R}'$, where R and R' can be the same organic group. It is known that aldehydes and ketones can react with single electron reductants (such as magnesium amalgam) to form radical anions, which can then dimerize in the pinacol reaction [34]. In the pinacol reaction, the radical-radical reactions occur on the surface of the metal.

If the radical anions are formed in a homogenous system with low (local) concentration of radicals, like under irradiation, then protonation of the radical anion can occur to form resonance stabilized radicals (such as the 1-hydroxyethyl radical from the reduction of acetaldehyde). If such a radical reacts with the surface of a particle of tellurium metal, it would give an alkyl bound to the tellurium surface.

An $\text{S}_{\text{N}}2$ -like process could then occur, in which another tellurium atom on the surface displaces the oxygen from the ligand to form a bridging carbene ligand. If a hydrogen atom could be chemically attached to the tellurium surface and be able to be transferred to the carbene carbon, then an alkyl

ligand would be formed. This process is schematically depicted in Fig. 22. If this happens twice and the tellurium atom is detached from the surface, it gives an organic telluride.

A similar concept was demonstrated by Paneth [35] in the gas phase, where hydrogen or nitrogen gas containing methyl radicals from the pyrolysis of tetramethyl lead can remove thin coatings of lead and zinc from glass tubing by forming tetramethyl lead or dimethyl zinc. We reason exposure of tellurium metal to radicals can form dialkyl tellurides [36].

Comparing the results of this study to the only previous study of organic tellurides in a comparable setting [5], a noticeable difference is that ditellurides never formed in this study, whereas they did appear in the previous study. This effect cannot be due to suppression from iodine, as ditellurides failed to appear even in the samples without iodine. The most prevalent difference between these two studies is the total dose and the dose rate. The dose rate in this study was 46.7 Gy/min, for a total dose of 134.5 kGy to each sample. The previous study had a dose rate of 4 kGy/h (66 Gy/min) and an irradiation time between 1 and 3 days, making the total dose between approximately 100 and 300 kGy. If this is the reason for the formation of ditellurides, it would imply that the reactions in which they are formed are dependent on a high dose. One possibility would be that ditellurides are formed by the reaction of two radical organic telluride radicals, as schematically described in reaction 5.



III.G. Suggested Mechanism for the Suppression of Organic Tellurides

Assuming that the reaction mechanism presented in Fig. 22 is true for organic tellurides and that a similar scheme (as detailed in Reactions 1, 2, and 3) holds true

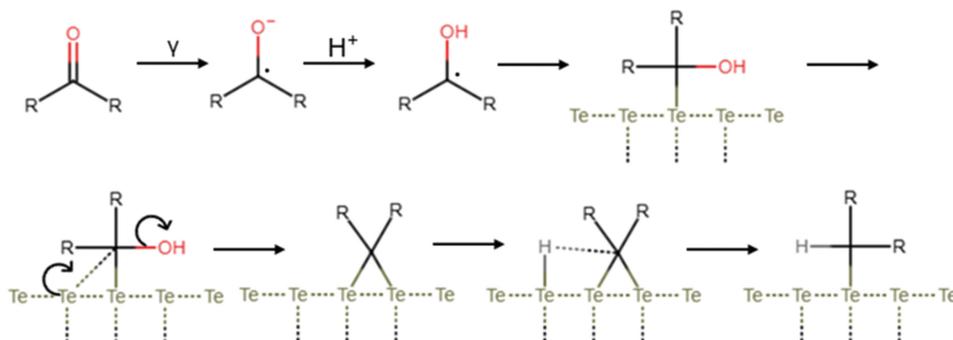


Fig. 22. Suggested reaction mechanism for the formation of organic tellurides.

for iodine, the mechanism behind the suppression of organic tellurides by iodine can be discussed.

An immediate observation is that all organic tellurides have been of the form R-Te-R', i.e., two organic constituents bound to a tellurium atom. All iodides have been of the form I-R, with only one organic constituent. As seen in Fig. 22, the proposed reaction mechanism requires two organic groups to bind to the same tellurium atom on a surface. The formation of organic tellurides is therefore dependent on the stability of the intermediate step where only one organic group is bound to the tellurium. If the reverse reaction happens, it lowers the rate of formation of any organic telluride. This would not be relevant to organic iodides, as they are formed without the intermediate. Naturally, a reaction that requires two organic radicals also has a further dependency on the diffusion of organic radicals to the active surface compared to a reaction that only requires one radical.

The proposed reaction mechanism also has another implication: organic tellurides require a surface of tellurium metal to form. As the tellurium takes some time to be reduced to metallic particles, organic tellurides cannot start forming immediately but are instead delayed. Also, the tellurium gradually builds up particles, which decreases the total amount of tellurium available for reaction (since a small number of larger particles has a smaller total surface area than a large number of small particles). Iodine, however, is present as I₂ and has a very large "surface area" in the sump where reaction with organic radicals is possible.

It should be noted that organic iodides are more volatile than tellurides and therefore more likely to be detected by headspace GCMS. Dimethyl telluride (82°C) has a higher boiling point than methyl iodide (42°C), and diethyl telluride (137°C) has a higher boiling point than ethyl iodide (72°C). However, this does not explain the suppression effect, as evidenced by the experiments with acetaldehyde. For those experiments, organic tellurides could be detected when tellurium was present and iodine was not. However, when both were present together, no tellurides at all were detected. If the low signals of organic tellurides were only due to low volatility, there should not be a difference in whether or not iodide was present in the system. However, the difference in volatility is a factor that must be considered for headspace GCMS.

IV. CONCLUSIONS

This paper is the first to study the formation of organic tellurides and iodides at the same time under gamma radiation and the first to point out the suppressive effect that iodine has on the formation of organic

tellurides. Furthermore, an overall reaction mechanism for the formation of organic tellurides under radiation has been presented, and its relationship to the suppressing effect of organic iodides has been discussed.

Both organic tellurides and iodides can be consistently formed in alkaline aqueous solutions under gamma radiation from a variety of different organic molecules. In the context of this work, however, a few limitations for their formation can be suggested. The paint experiments did not yield either organic tellurides or iodides, implying that the leaching of organics from such paint, both freshly cured and old, does not by itself give enough organic species to form either or, indeed, very little can be detected in the GCMS samples for all these experiments. If the leaching process is as slow as it appears, then some other source of organic species is likely necessary to form organic tellurides and iodides in a real accident. Such sources could include leaching or pyrolysis/combustion from cables, gas-kets, lubricating oils, etc.

Since about twice as much tellurium is formed compared to iodine, and the releases from the fuel for both elements are comparable, tellurium can be expected to dominate in the sump in the earliest part of the accident. However, due to its rather fast decay, iodine is expected to become the dominant species with time. In all the experiments where organic tellurides could form, organic iodides also formed when possible. In the case of every species tested aside from the cured paints, organic iodide formation served to completely or significantly suppress the formation of organic tellurides. Thus, organic tellurides should only be relevant while tellurium is the dominant species in the sump, meaning the first days after the accident, if at all. Whether or not this increases or decreases the danger to the public depends on the capacity for the safety measures of the nuclear power plant to cope with organic tellurides, something that is largely unknown. Some of the mechanisms used for FCVSs are expected to not work against organic tellurides (silver-based filters), whereas some are expected to work (activated carbon). Further studies are needed to fully determine the ramification of organic tellurides.

The reason for the suppressive effect of iodine cannot be determined with certainty but may stem from the fact that organic tellurides require two organic radicals to react with a solid tellurium surface, which is slower than the reaction of a single organic radical reacting with an I₂ molecule. It may also be related to the intermediate telluride, where one organic radical has bound to the tellurium surface, is not stable, and can revert, which then would further reduce the rate of formation of organic tellurides. Finally, the active surface area of solid tellurium particles would be expected to be lower than the "area" presented by dissolved I₂.

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Author Contributions

CRedit: **Fredrik Börjesson Sandén:** Conceptualization, Formal analysis, Investigation, Methodology, Project administration, Visualization, Writing – original draft, Writing – review & editing; **Mark R. St.-J Foreman:** Supervision, Writing – review & editing; **Per-Olof Bergh:** Methodology, Resources, Software; **Christian Ekberg:** Funding acquisition, Supervision, Writing – review & editing.

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