**EFFECTS OF γ -IRRADIATION ON THE ELECTRON LIFETIME AND
POSITIVE ION MOBILITY IN TETRAMETHYLSILANE AND
TETRAMETHYLPENTANE**

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Abstract

Two possible room-temperature liquids suitable for calorimetry, tetramethylsilane (TMS) and (2,2,4,4) tetramethylpentane (TMP) were irradiated with γ -radiation of a ^{60}Co source up to doses of 1 Mrad and 10 Mrad, respectively. Electron lifetime and mobility of positive ions were measured as a function of radiation dose at two different dose rates. The liquid samples were kept in sealed ionization chambers made of stainless steel and ceramic. A decrease of electron lifetime with increasing dose was observed, which is attributed to the formation of compounds with double bonds. In TMP the positive ion mobility changed with irradiation, probably owing to the formation of high-molecular-weight compounds.

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1. INTRODUCTION

Liquid ionization chambers are widely used in high-energy physics as detectors in sampling calorimeters. The UA1 Collaboration at CERN has constructed and successfully tested such a calorimeter using tetramethylpentane (TMP) as active medium and depleted uranium plates as radiator [1]. The construction was based on sealed liquid ionization chambers intercalated with uranium plates. It was shown that the 'sealed box' concept was viable, since boxes sealed off more than four years ago kept functioning without loss of signal [2]. In the operational environment of the UA1 calorimeter, the total absorbed radiation dose in the liquid was estimated to be negligible during the expected running time. With the advancement of accelerator technology and the appearance of high-luminosity machines such as the Large Hadron Collider (LHC) or the Superconducting Super Collider (SSC), more attention has to be paid to the question of radiation damage of the liquid ionization chambers. In the experimental areas of the future colliders, radiation doses of 1 to 10 Mrad per year are expected for a pseudorapidity interval $|\eta| \leq 2.5$ [3]. Such a high exposure to ionizing radiation may introduce additional requirements on the properties of liquids and on the construction of ionization chambers. It therefore seems imperative to investigate the effect of high radiation doses on liquid ionization chambers. Whilst the original liquid used in the UA1 calorimeter was TMP, preference was given to tetramethylsilane (TMS) because of its high electron mobility [4]. This makes TMS especially suitable for operation in the conditions of the high collision rates of the future colliders. The most important quantity characterizing the state of the ionization chamber is the electron lifetime of the liquid.

Here, we wish to report on the loss of electron lifetime with accumulated γ -radiation dose in test chambers filled with ultrapure TMP and TMS. In addition, the effect of radiation on the mobility of the positive charge carriers was investigated. Purification and filling of the chambers and measurement of the initial electron lifetime were carried out at CERN. Irradiation with ^{60}Co γ -radiation and measurement of the lifetime as a function of accumulated dose, as well as the determination of the ion mobilities, were done at the Hahn-Meitner-Institut in Berlin.

2. RADIATION DAMAGE

Radiation energy absorbed by the liquid is converted into ionization and excitation. The energy for the formation of one electron/positive ion pair is approximately 25 eV. Since the ionization potential is close to 10 eV [5], the difference between these two energies is found as excitation. In liquid, most electron/positive ion pairs are generated in close spatial correlation and they undergo geminate recombination after a short half lifetime, which is of the order of several picoseconds. The recombination energy is converted into excitation as well. The chemical result of these excitation processes is the breakage of chemical bonds and

the formation of molecular fragments and radicals. For the liquids considered in this article, important bond energies are compiled in table 1 [6].

It is obvious that the C–Si and the Si–Si bonds are weaker than the C–C bond. The main results of bond breakage is the formation of radicals ($R\cdot$), which are chemically reactive species. They disappear either by reaction with molecules of the liquid (I) or with impurities present in the liquid (II) or by reaction with each other (III),

The balance of radical production and disappearance can be written as,

$$\frac{d[R\cdot]}{dt} = k_g D_r - k_s [R\cdot] - k_r [R\cdot]^2 \quad (1)$$

Here, k_g , k_s , and k_r are the rate constants for generation, attachment, and recombination, respectively; D_r denotes the dose rate and $[R\cdot]$ is the radical concentration. The first term on the right-hand side is the generation term. The second term describes the disappearance of the radicals by a first-order reaction [see (I) and (II)] whilst the third term describes the process of radical recombination [see (III)], a second-order reaction. Low dose rates favour first-order reactions whilst high dose rates favour second-order reactions. This argument may be extended even to the explanation of the effect of the linear energy transfer of the radiation (LET). The spatial deposition by low LET radiation is equivalent locally to a low dose rate whilst high LET radiation produces locally a high concentration of radicals. The yield of a particular product is a function of the absorbed dose and of the LET.

Radiolysis of organic liquids has been investigated quite extensively [7]. Usually many products are formed and it is possible only to detect the most prominent ones. Experiments are usually carried out with sealed ampoules with a vapour space above the liquid. After the irradiation, the vapour phase is pumped off and its composition is analysed in a gas chromatograph or a mass spectrometer. Substances dissolved in the liquid are identified by gas chromatography, whereas insoluble substances are characterized by elemental analysis. The specific yield is obtained from data which show proportionality of the amount of product and absorbed dose. The yield is expressed as the number of product species formed per 100 eV of absorbed energy. As the irradiation proceeds the products formed initially may re-enter the radiolytic reactions, and deviations from this proportionality occur. The main radiolytic products of interest in the present context are gases, compounds containing double bonds, and high-molecular-weight compounds. The exhalation of hydrogen or methane may lead to an increase of pressure in the ionization chamber owing to the limited solubility of the gases in the organic liquids. Compounds with double bonds represent electron traps of moderate efficiency. Given a high enough concentration, there will

be a reduction of the electron lifetime. High-molecular-weight compounds (P) may capture the positive charge, either by charge transfer (IV) or by attachment (V). The result is a reduction of the positive carrier mobility.

Furthermore, high-molecular-weight compounds may become insoluble and form deposits on electrodes and insulators, thus changing the electrical properties of the chamber.

3. ELECTRON LIFETIME AND ION MOBILITY

The most important quantity which determines the operational capability of the chamber is the electron lifetime τ . The lifetime must be much greater than the electron drift-time in order to obtain the total charge produced at a given field strength by an ionizing particle in the chamber. The electron lifetime τ is limited by the presence of electron-attaching impurities of concentration [S] in the liquid,

$$\tau = \frac{1}{k_s [S]} , \quad (2)$$

where k_s is the rate constant for the attachment process. The reaction constant is related to the electron absorption cross-section by the following relation:

$$k_s = A \int_0^\infty \sigma(E) F(E) dE , \quad (3)$$

where the term $F(E)$ describes the Maxwell distribution of the electrons in the liquid:

$$F(E) = \frac{2}{\sqrt{\pi} kT} \sqrt{\frac{E}{kT}} \exp\left(-\frac{E}{kT}\right) . \quad (4)$$

The lifetime is measured by monitoring the decrease in time of the initial number of electrons N_0 which was produced by the passage of a cosmic-ray particle traversing the chamber parallel to the electric field strength. The electron current $I_{el}(t)$, due to the electrons drifting parallel to the externally applied electric field E , as a function of time, is given as

$$I_{el}(t) = I_{el}(0) \left(1 - \frac{t}{t_d}\right) \exp\left(-\frac{t}{\tau}\right) \quad (5)$$

and

$$I_{el}(0) = \frac{N_0 e_0}{t_d} , \quad (6)$$

where e_0 is the charge of the electron and t_d is obtained from the following equation:

$$t_d = \frac{d}{\mu_{el} E} . \quad (7)$$

Here, d denotes the electrode separation and μ_{el} the electron mobility. The term $\exp(-t/\tau)$ describes the attachment of electrons due to scavengers. Instead of cosmic-ray particles crossing the gap of the ionization chamber, hence ionizing the gap uniformly, a flash of high-energy X-rays can be utilized. It produces a homogeneous concentration of electrons $n_{el}(0)$ in a volume V between the plates. The initial current due to ionization $I_{el}(0)$ is given as

$$I_{el}(0) = \frac{n_{el}(0) V e_0}{t_d} . \quad (8)$$

For cases of $\tau \gg t_d$, eq. (5) is compared with the measured data by plotting

$$\ln \left[\frac{I_{el}(t)}{I_{el}(0)} \left/ \left(1 - \frac{t}{t_d} \right) \right. \right] \text{ against } t \quad (9)$$

in order to obtain τ . For cases where $\tau \ll t_d$,

$$\ln \left[\frac{I_{el}(t)}{I_{el}(0)} \right] \text{ against } t \quad (10)$$

is plotted.

The mobility of the positive ions μ_{ion} is obtained in analogy with eq. (7) as

$$t_d = \frac{d}{\mu_{ion} E} . \quad (11)$$

4. IRRADIATION TESTS

A schematic drawing of the test cells used for the irradiation tests is shown in fig. 1. They are made entirely of stainless steel. The centre electrode is held in place by ceramic spacers and is connected electrically through a ceramic feedthrough. Special care was taken during the manufacturing of the cells in order to ensure that no contamination of the inner part of the cell could occur. Before assembly, all parts of the cell were individually cleaned according to a modified standard procedure at CERN for ultrahigh vacuum parts [8]. The final treatment consisted in rinsing all parts, including the ceramics, with ultrahigh purity water ($\rho = 18.2 \text{ M}\Omega \text{ cm}$). In order to avoid oxidation during welding the stainless-steel parts of the cell were laser-welded under a He blanket.

After welding, each cell was filled several times with acetone p.A followed by several fillings of ethanol p.A. Finally, ultrapure water was continuously rinsed through each cell using a specially prepared flange with a long bent stainless-steel tube as inlet in order to reach the inside of the cell. The outlet tube, also made of stainless steel, was used to monitor the

specific resistance of the return water. The flushing with water was stopped when the resistance of the return water again reached 18.2 MΩ cm. Drying of the cell was carried out in an oven with high-purity argon flowing through the cell. The return argon flow was again monitored and the drying procedure was stopped when the residual water concentration in the argon was less than 2 ppm. The cells were evacuated, leak-tested, and baked out at 350 °C during 48 h. During the cool-down the cells were kept at 150 °C for 24 h before the heaters were switched off. As soon as room temperature was reached a further leak test was performed at a level of 2×10^{-10} mbar l / s.

Both liquids used in this test were produced in special custom-designed processes^{*)}. Both samples were further purified by two passages through the existing UA1 purifier equipped with newly activated filters [2,9]. The test cells were filled using a liquid column of ~ 1.5 m standing over the entrance port which is equivalent to a hydrostatic pressure of ~ 100 mbar. In this way complete filling and absence of vapour space in the cell was assured. The electron lifetime immediately after filling was measured using cosmic-rays as the source of ionization. The results obtained for TMS and TMP were 0.7 ms and 1.0 ms, respectively.

Four test cells—two filled with TMS and two filled with TMP—were sent to the Hahn-Meitner-Institut in Berlin. After arrival the lifetime was immediately remeasured and found to be in agreement with the measurements performed at CERN. The set-up and electrical circuit for the lifetime and drift-time measurements in Berlin are shown in fig. 2. A short pulse of 15 MeV bremsstrahlung produced by electrons from a linear accelerator impinging on a lead target, was used to ionize the liquid uniformly in the test cell. The photon beam was collimated at the centre of the cell by a lead tube of 10 mm inner and 50 mm outer diameter and a length of 100 mm. In this way the cell was only ionized in the region where the electric field was not distorted by edge effects. Cell and collimator were housed in a Faraday cage to remove pick-up noise from the accelerator. Positive voltage was applied to the centre electrode. The electron-current signal was decoupled from the supply voltage by a capacitor C. Under these conditions, the electron current is described by eqs. (5) and (8). Care has to be taken that the initial electron density $n_{el}(0)$ is small enough so that the recombination time t_r , given as

$$t_r = \frac{1}{k_r n_{el}(0)} , \quad (12)$$

is much longer than the lifetime and the drift-time; k_r is the recombination rate constant. This condition could be fulfilled by attenuating the photon beam by adding lead bricks in front of the cell. In this way the ionization density in the cell was kept such that the recombination

^{*)} The TMS was made by Wacker Chemie GmbH, Burghausen, Germany. The TMP was supplied by Wiley Organics, Columbus, Ohio, USA.

time was much longer than the electron lifetime owing to scavenger molecules present before or after irradiation. As long as $\tau > t_d$, the signal was recorded directly, i.e. without a preamplifier. At short lifetimes, $\tau < t_d$, the signals were preamplified by a fast, battery-operated, amplifier with 100 Ω input impedance and 50 Ω output impedance. The current traces were recorded on a storage scope.

Measurement of the positive ion mobility was carried out with the same set-up. Here, the current signal was either fed directly into the input of the 7A22 plug-in or it was preamplified by an electrometer amplifier.

Irradiation of the test cells with γ -rays was performed in a cave-type ^{60}Co source having an activity of 21.3 kCi. Dose rates of 150 krad/h and 9 krad/h were chosen by mounting the test cells at different distances from the centre of the cobalt source. The cells were placed at distances of 120 cm and 20 cm from the centre of the source; hence for each dose rate one separate cell was employed. Figure 3 shows the variation of the dose of the 21.3 kCi ^{60}Co source in Berlin with the distance from the centre. The absolute dose rate was measured by means of the Fricke solution [10]. After each partial irradiation dose the lifetime of the irradiated sample was measured using the described set-up. In order to check on possible recombination phenomena, for some of the points the lifetime was remeasured after 24 h, in some cases even after 4 weeks. No difference was found. Some irradiations were carried out in the presence of an electric field strength of 1 kV/cm. No influence was found on the lifetime.

5. RESULTS AND DISCUSSION

The collected electron charge produced by the passage of a cosmic-ray particle is plotted as a function of time for the test cells immediately after filling at CERN (figs. 4a and b). The lifetime was obtained by fitting the data to a function which is the integral of eq. (5). Figures 5a and b show the data of the electron current as a function of time plotted using eq. (9). The electron lifetime is read directly from the time axis, where the plotted electron current reaches the value $1/e$. In order to determine the electron lifetime in an as unbiased a manner as possible, the transit time t_d [see eq. (7)] was measured prior to the electron lifetime by increasing the electric field such that the term $\exp(-t/\tau)$ in eq. (5) became negligible. In this way changes of the electron mobility, which could have impaired the lifetime measurement, were excluded. Data of the electron current as a function of time obtained after delivery of a partial dose are plotted according to eqs. (9) or (10), and the results are shown in figs. 6a and b. The variation of the electron lifetime as a function of the absorbed dose for the two liquids investigated and for the two dose rates employed is shown in fig. 7. No influence of the dose rate on the decrease of lifetime is detected. The electron lifetime decreased for both liquids as

$$\tau = AD_r^{-m}, \quad (13)$$

with $m = 1.0 \pm 0.1$ in the case of TMP and $m = 1.3 \pm 0.1$ in the case of TMS. From eq. (2) it can be concluded that in the case of TMP the concentration [S] of the electron-attaching impurity is formed proportionally to the absorbed dose. In the case of TMS the concentration [S] increases with $D_r^{1.3}$, which may be an indication of some secondary reactions.

The yield of a radiolytic process is expressed by its G value, the number of species formed or decomposed per 100 eV of absorbed energy. The decomposition yield of the molecules of liquid hydrocarbons is around $G \approx 10$ [7]. The dose of 10 Mrad corresponds to 6.25×10^{20} eV/g or 4.5×10^{20} eV/cm³. The total number of molecules decomposed is then calculated to be 4.5×10^{19} cm⁻³. This is to be compared with the number density of 6.5×10^{21} cm⁻³. At 10 Mrad approximately 0.7% of the liquid has been decomposed.

At least two pathways for the formation of electron-attaching impurities are possible. First, both liquids are comprised of molecules with saturated bonds. It was found that radiolysis of such liquids leads to the formation of unsaturated compounds, i.e. molecules containing double bonds. Early studies on electron mobilities have shown that the presence of these compounds limits the lifetime of the electrons, although the reaction rate constants k_s are only of moderate magnitude when compared with electronegative compounds. For this reason, treatment of the samples with concentrated sulfuric acid was always included in the purification cycle in order to extract unsaturated compounds [11]. Secondly, the inner stainless- steel surfaces of the test cell are covered with molecular layers of oxygen and water. Reaction of these molecules with radiolytically-produced radicals may form electron-attaching compounds which are transported into the bulk of the liquid. TMS and TMP samples which received the maximum dose underwent tests to prove the presence of unsaturated compounds. Extraction with concentrated sulfuric acid was performed. A slightly brownish coloration of the sulfuric acid, after it had been several hours in contact with the irradiated liquid, demonstrated qualitatively the presence of unsaturated compounds. The G value for the formation of compounds with double bonds is of the order of 0.1 [7]. At 10 Mrad, this corresponds to a concentration of 4.5×10^{17} cm⁻³ in TMP. Using the measured lifetime of 180 ns at 10 Mrad, a reaction rate constant $k_s = 1.23 \times 10^{-11 \pm 1}$ cm³/s or $7.38 \times 10^{9 \pm 1}$ l/(mole s) is estimated from eq. (2). This value seems to be of the correct order of magnitude. Unsaturated compounds can be removed from the liquid by the usual purification procedure used at CERN [2,9].

Figure 8 shows the results of the TMP irradiation together with the data obtained by Holroyd [12]. His samples exhibit an initial lifetime approximately one order of magnitude smaller than the ultrapure samples used in this test. Small radiation doses left the lifetime unaffected, or led to a slight increase probably due to the radiolytical conversion of electronegative impurities initially present in less reactive compounds. For doses greater than

50 krad, one of his samples showed the same dependence on dose as our sample, whereas the lifetime in the second sample decreased much slower with dose. At present, it can only be speculated that this different behaviour reflects the presence of various kinds of impurities in the starting material. These impurities interacting with radiolytical products could lower the electron mobility such that the determined lifetime appears longer.

In addition to the measurement of electron lifetime the positive ion mobility was measured in the ultrapure samples. The ion mobilities of TMS and TMP have been determined before and after irradiation in order to obtain information on the size of the drifting molecules. Irradiation caused a decrease of the mobility in the case of TMP by roughly a factor of 8, indicating that large high-molecular-weight compounds are formed whilst the value for TMS was left unchanged. A plot of the drift time as a function of $1/E$ for positive ions in TMP is shown in fig. 9. The positive ion mobilities obtained in the two liquids are compiled in table 2.

It seems that process (III) leads to the formation of higher-molecular-weight compounds, which then react with positive species produced by ionization of the TMP molecule according to reactions (IV) or (V). The result is an ion of bigger size and mass exhibiting a lower mobility. No rate constants are available for the processes (IV) or (V). Charge transfer from positive ions of cyclohexane or n-heptane to various hydrocarbon solutes has been studied by pulse radiolysis, and maximum values of 10^{10} l/(mole s) were reported [13]. Charge transfer from parent positive ions to dimer or polymer ions reduces the overall mobility of the positive species. By drift, the polymer ion is transported to the cathode where it is deposited. The result is an electrically insulating layer. Such effects have been observed with partial discharges in n-hexane [14]. The formation of high-molecular-weight compounds may influence the electric properties of the ionization chamber. In addition, this effect is detrimental to the fast clearing of the gap from positive charges. Further deposition on insulator surfaces may lead to changes in the dielectric strength and hence to high-voltage problems.

Similar effects have been observed at CERN during the construction and tests of the uranium/TMP calorimeter, where chemical bonds have been broken by high-voltage breakdown in the liquid.

TMS does not show a change of the positive ion mobility. The reason for this behaviour may be found in the fact that the parent positive ion is highly unstable. In neopentane, the hydrocarbon equivalent of TMS, the parent positive ion, converts within 1 ps into a tertiary butyl ion. For TMS we would expect the following conversion to take place:

The trimethylsilyl positive ion is stable and does not transfer its charge to other hydrocarbon or silane molecules present in TMS.

6. CONCLUSIONS

The fast decrease of electron lifetime due to radiation damage makes the use of TMS as the sensitive medium of a calorimeter placed in an environment of high radiation dose precarious and imposes some limitations on the possible design of a calorimeter. The 'sealed box' concept does not seem to guarantee a sufficiently long operational period of the ionization chambers, whereas in 'swimming pool' type calorimeters an additional difficulty may arise owing to the pressure increase inside the calorimeter, since both phases, gas and liquid, are present.

Efficient on-line purification systems tailored to remove unsaturated compounds will have to be constructed to remove radiolytical products with the same speed as they are produced. Typical purification cycles as used for instance in the UA1 purifier, which was a batch-type purifier, would take too long. New purifiers capable of operating continuously will have to be developed in order to cope with the large amounts of liquid necessary for a LHC calorimeter. In the case of TMP an additional difficulty is the removal of the polymerized molecular chains which are created during irradiation. A TMP calorimeter probably has to be rinsed several times until all polymers are removed. Since they have a tendency to stick to the cathodes, their removal seems essential because they can become the source of high-voltage problems.

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Table 1
Average bond energies

Bond	(kcal/mole)	(eV)
C-C	83	3.6
C-H	99	4.3
CH ₃ -M	80-90	3.5-3.9
Si-Si	42	1.83
C-Si	69	3.0

Table 2
Ion mobility (cm²/V s) before and after irradiation

Liquid	Dose		
	0 Mrad	1 Mrad	10 Mrad
TMS	9×10^{-4}	9×10^{-4}	-
TMP	4.3×10^{-4}	-	5.1×10^{-5}

List of Figures

Fig. 1 Schematic drawing of the test cell.

Fig. 2 (a) Experimental set-up at the Linac for lifetime and mobility measurements; and (b) measurement circuit.

Fig. 3 Dependence of the dose rate on the distance from the centre of the ^{60}Co source at the Hahn-Meitner-Institut, Berlin.

Fig. 4 Cosmic-ray data for the freshly filled cells. The data points (+) are fitted using the integral of eq. (5) with τ as free parameter. (a) TMS and (b) TMP.

Fig. 5 Decay of the electron current in time for TMS and TMP cells after arrival at the Hahn-Meitner-Institut and before irradiation. (a) TMS and (b) TMP.

Fig. 6 Decay of the electron current in time for TMP after irradiation with (a) 150 krad and (b) 3381 krad.

Fig. 7 Variation of the electron lifetime as a function of the absorbed dose; black dots and diamonds are for TMP at two different dose rates; shaded squares are for TMP with an electric field of 1 kV/cm. Open circles and open squares are for TMS at two different dose rates.

Fig. 8 Variation of the electron lifetime as a function of the absorbed dose; comparison of our data (black points) with the data of Holroyd (open symbols) [12].

Fig. 9 Evaluation of the ion mobility of TMP before irradiation (open squares) and after an absorbed dose of 10 Mrad (black squares) using eq. (11).

Figure 1

Figure 2

Figure 3

Figure 4 a

Figure 4 b

Figure 5 a

Figure 5 b

Figure 6 a

Figure 6 b

Figure 7

Figure 8

Figure 9