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Outgassing studies of materials for the TRT construction

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Abstract

We present a systematic outgassing study of 7 materials and 10 glues candidate to be used in the construction of the TRT. Each sample has been tested before and after irradiation up to a 50 Mrad dose. Some results on tests of the electrical and mechanical properties of the materials are also presented.

1 Introduction

Gaseous proportional chambers operated in a high-radiation environment can have their performance seriously compromised by *ageing* effects. By ageing it is intended a degradation with time of the performance of the chamber that can appear as one or more of the following effects: loss of gas gain, worsening of the energy resolution, discharges or sparking. The cause of this behaviour has to be ascribed to the complex chemical phenomena that take place in the avalanche (an environment similar to the one studied by plasma chemistry) leading to the creation of a variety of molecular species (oxides, polymers and others) that can be accumulated as deposits on the electrode surfaces.
The ageing processes that can appear in the chamber depend on the design choices: type of gas used, anode and cathode material and operating conditions. However, even if the initial design choices are correct, ageing processes can be initiated by contaminant substances already present in the gas mixture or appearing at a later stage as a consequence of natural or radiation-induced outgassing (at the LHC, the TRT will be exposed to a maximum dose of 10 Mrad) of all the detector materials in contact with the gas.
In the TRT, where very large charge is expected to be accumulated (about 6 C/cm) after 10 years of operation, ageing is an extremely important issue. Basic principles and recommendations for the material choice are described in[8-1]. After a first pre-selection of candidates, two more steps need to be performed:

1. Selection on the basis of the outgassing properties. Materials with no or low outgassing are likely candidates to be used in the construction of the TRT.

2. However,they still have to pass a further ageing test to be fully qualified for the detector production.

2 Experimental Set-up and Measurement Method

The set-up used for these tests is shown in Figure 2-1 and is, with minor changes, the

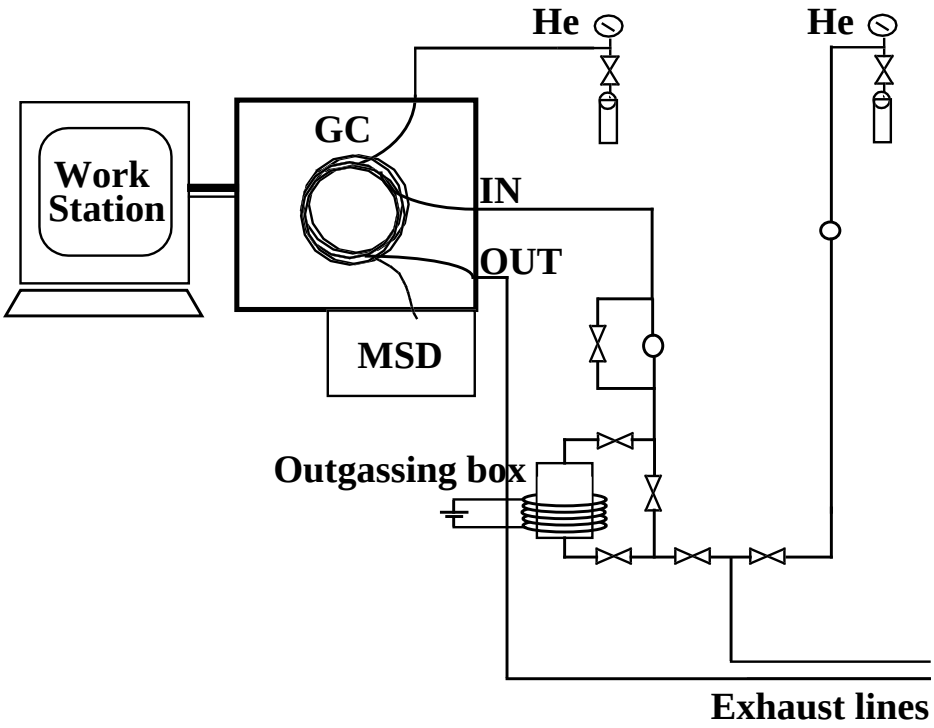

Figure 2-1 Schematic view of the set-up used for the outgassing test. Shown are the two Helium lines, Gas Chromatograph (GC), the Mass Selective Device (MSD) and the outgassing box where the samples are inserted. The outgassing box is surrounded by a resistive strip that allows to heat-up the samples.

one used along the years by the RD-10 Collaboration[8-2]. The sample under test is inserted in a cylindrical stainless steel box having a volume of about 200 cm^3. The box is then closed with a stainless steel cover and an aluminium O-ring (to avoid possible contamination from the outgassing of rubber joints). The temperature of the samples can be changed, if needed, by mean of a resistive tape folded around the box. A cleaning procedure of the box, which was repeated before any new measurement, consisted in flushing the box with more than 10 box volumes of pure helium. The box is then filled with helium and closed, by mean of two valves, in order to allow the possible outgassing from the sample to accumulate in the box. The absolute helium pressure in the box was about 2.5 atm. After some time, the output valve is opened and the gas is sent to a Gas Chromatograph for the analysis.

The Gas Chromatograph (GC) is an Hewlett-Packard[1] 5890 Series II device. It con-

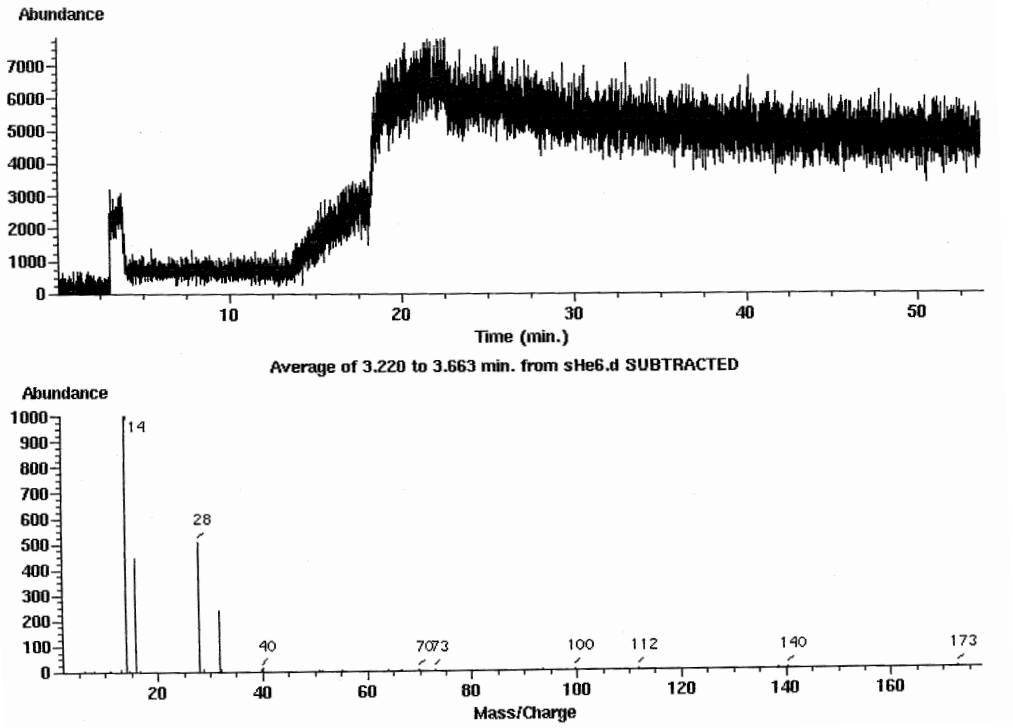

Figure 2-2 Total Ion Chromatogram (top plot) for pure Helium. The bottom plot show the M/Z spectrum for the small air peak detected at about 3.4 min retention time.

sists of a long capillary column through which a gaseous sample of the substance to be analysed is flown together with a carrier gas (high purity helium in our case). The column is contained in an oven, whose temperature can be programmed. The different substances composing the gas samples are separated according to their interaction properties with the capillary column material. Depending on the column length and diameter, as well as on the temperature programme, the carrier gas nature and flow rate, each separated substance is sent, with its specific retention time in the column, to the MSD, for identification.

The performance of the GC are determined mainly by the characteristics of the column. For these tests, a 50-m long Hewlett-Packard PLOT Fused Silica column has been used. This column is coated with a 5 μm-thick film of Alumina (Al_2O_3), treated with KCl, and has an inner diameter of 0.32 mm; the column is optimized for the separation of light hydrocarbons (in the range C_1-C_{10}). The GC output is connected, through an appropriate interface, to an Hewlett-Packard HP 5971A Mass Selective Device (MSD). The MSD contains a 70 eV electron gun that ionizes and fragments the molecules of the sample. The ions are then sorted by their M/Z ratio (mass-to-charge ratio) by mean of an electromagnetic filter and transformed in electric signals which are then sent to the computer. The standard computer output is the so-called Total Ion Chromatogram (TIC) which shows the total ion abundance as a function of the retention time in the column. Alternatively, it is possible to display, for a certain time interval, the ion abundance as a function of the M/Z ratio. This last

1. Hewlett-Packard Company, 3000 Hanover Street, Palo Alto, CA 94304-1185, USA.

spectrum allows to identify, with a certain probability, the substances contained in the gas by performing a match with a library containing the M/Z spectra of more than 50,000 chemical compounds.

The standard run used for all the measurements described in the following sections had the GC/MSD system programmed for a 53 min total running time, during which the column temperature changes from 70 °C (first 12 min) to 200 °C (after 22 min).

An example of the GC output is shown in Figure 2-2. The top plot shows the chromatogram for a sample of pure helium. The total ion abundance starts to increase at about 12 min as a consequence of the increase of the oven temperature and then reaches a plateau as soon as the temperature stabilizes at 200 °C. The small peak at about 3.4 min is due to an air leak in the system. This is confirmed in the bottom plot where the M/Z spectrum for this peak is shown: the peaks of molecular and atomic nitrogen (M/Z=14 and 28) and oxygen (M/Z=16 and 32) and their relative abundances are the clear indication of the presence of air.

The GC column, the settings of the GC/MSD system and the standard measurement procedures we used are the same as the ones used by the RD-10 Collaboration and are described in greater detail in[8-3].

3 MSD calibration

The MSD has been calibrated using a commercial calibration gas mixture that con-

Table 3-1 Composition of the calibration gas used in part-per-million (by volume). Also shown are the retention times (in minutes) and the areas (in arbitrary units) of the GC peaks and the resulting calibration factor.

Alkane	Quantity (ppm)	Time (min)	Area ($x10^6$)	Calibration factor (ppm $x10^{-5}$/area)
Methane (CH_4)	101	n/a	n/a	n/a
Ethan (C_2H_6)	101	n/a	n/a	n/a
Propane (C_3H_8)	98.3	5.263	3.027	3.247
n-Butane (C_4H_{10})	98.7	10.925	6.971	1.416
n-Penthane (C_5H_{12})	96.5	15.330	16.035	0.6018
n-Hexane (C_6H_{14})	91.3	17.877	28.412	0.3213

tains known quantities of six alkanes in helium.However, due to the nature of this mixture, only a partial calibration in a limited mass range has been possible. The composition of the mixture is summarized in Table 3-1: the accuracy of the determination is guaranteed by the supplier at the ±2% level. In the same table, the retention time at which the substance is detected in the GC and the corresponding area under the peak (expressed in arbitrary units) are also shown. The chromatogram from which these results have been obtained is shown in Figure 3-3. From this data is

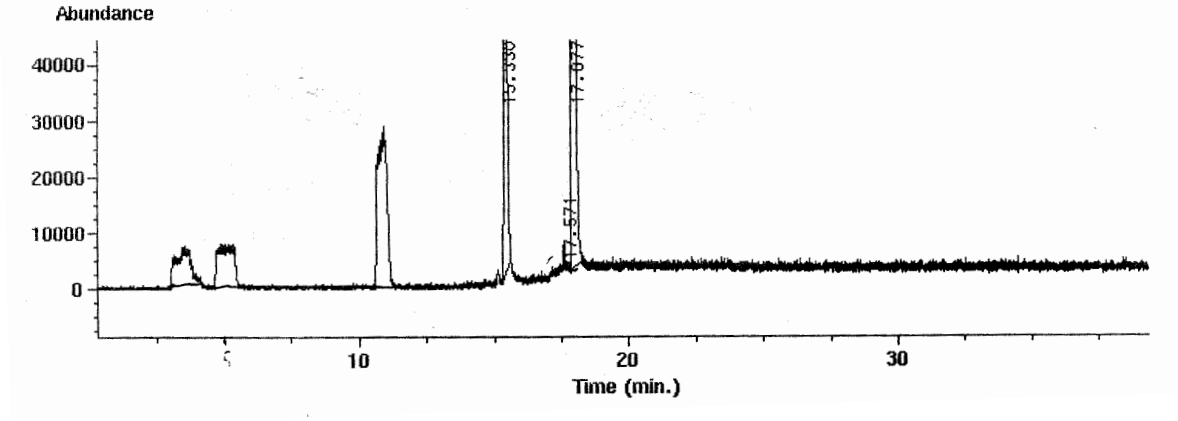

Figure 3-3 GC spectrum from the calibrated alkane mixture. From shorter to longer retention times one can see the air peak (with superimposed the unresolved methane and ethane peaks), the propane, the n-butane, the n-penthane and the n-hexane peaks.

straightforward to deduce a calibration factor translating the area under the peak into ppm unit. The calibration is a function of the retention time in the column. The table cells for methane and ethane are empty because these two peaks superimposed with the air leak peak in the GC and could not be resolved. As a consequence no calibration is available for retention times less than 5 min. The calibration factor (ppm/peak area) is reported in Table 3-1 and plotted as a function of the retention time in Figure 3-4. This kind of calibration can be effectively used for quantitative estimates only at retention times less than 18 min. Extrapolation to higher retention times is affected by large uncertainties not only because of the non-uniformity of the time scale (the GC column has different temperatures at different times) but also because the ionisation current in the MSD strongly depends on the numbers of ions produced, and so on the complexity of the gas molecules.

3.1 Sensitivity of the system

The sensitivity of the system has been estimated using the same mixture of 6 alkanes used to calibrate the GC. Observing with more accuracy the hexane peak in Figure 3-3, one can notice a tiny peak nearby, at smaller retention times (see Figure 3-5). This impurity could be identified as probably being an isomer of the hexane. The impurity peak is 0.52% smaller than the main peak and, due to the fact the hexane was present in a 91.3 ppm concentration, we deduce that the isomer peak is about 0.5 ppm. So, from the observation of this peak, one can estimate that the sensitivity of our apparatus is 0.5 ppm or better.

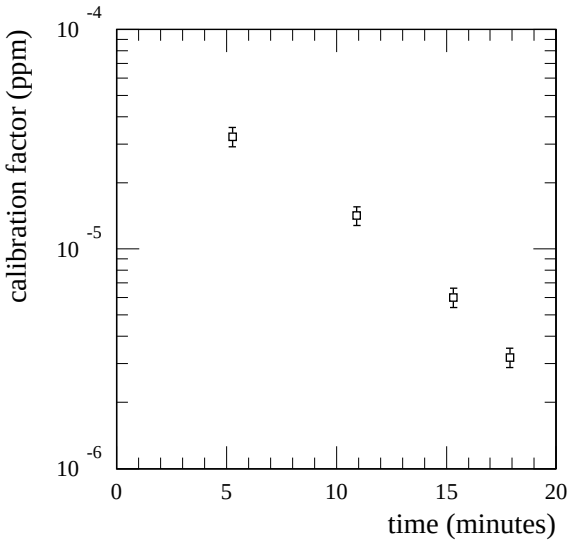

Figure 3-4 Calibration factor for the GC as a function of the retention time in the column.

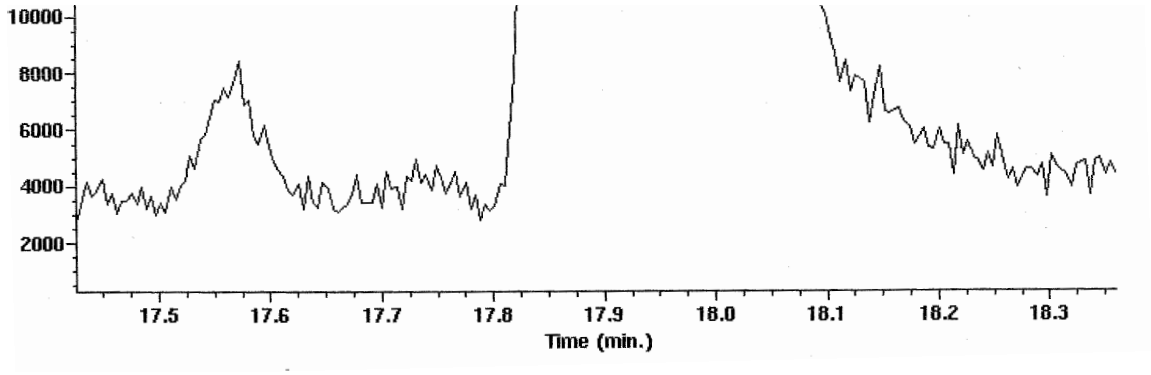

Figure 3-5 Blow-up of the region around the hexane peak. To the left of the main peak, at about 17.571 min retention time, a smaller peak due to one hexane isomer can be clearly seen.

4 Outgassing Studies

The results of the tests performed on all glues and construction materials, pre-selected in[8-1] as candidates, are summarized in this section. Two samples, before and after irradiation, have been tested for each of the substances. The irradiated samples have been exposed to a ^{60}Co source and have accumulated a dose of 50 Mrad at a 0.5 Mrad/h dose rate. This dose is about a factor of 5 higher than what is expected to be accumulated by the TRT after 10 years of operation at LHC which is, at maximum, about 10 Mrad[8-4]. Also the irradiation dose rate is much higher than what is expected once the TRT will be operated in ATLAS. Radiation-induced effects that may depend on the dose rare are partly compensated by the higher than expected dose at which the samples have been exposed. The irradiated samples were tested as soon as they were received from the irradiation facility: outgassing produced *during* the

exposure at the irradiation facility might have been lost.
Each of the samples was tested twice. In a first run the sample was kept about 15 hours in the box, filled with helium at room temperature (about 22 °C). Then, after the gas analysis, the box was flushed, filled with helium and closed again. With the help of the resistive tape around the box, the temperature of the sample was raised to 60 °C. After 6 hours, a new gas analysis was performed. Each measurement at both temperature settings was performed twice to check its reproducibility. The test at high temperature is done in order to speed-up the outgassing.
For some materials, in order to study the variation of the outgassing with time, the samples have been left in the box at 60 °C for one or more days. Moreover, some samples have been re-tested after some time in order to study the variation of the outgassing with the time since the sample was prepared (Section 4.3.6).

4.2 Construction Materials

The material tested and the results of the outgassing tests in various conditions are summarized inTable 4-2. In the second and third columns, the results of the tests for the non-irradiated samples at two different temperatures are reported. Columns

Table 4-2 Summary of outgassing results for all the materials tested: “No” means that no outgassing has been observed: “n/a” (not available) means that the irradiated sample was not available for that particular material.

Material	Non-irradiated		Irradiated		Comments
(temperature)	(22 °C)	(60 °C)	(22 °C)	(60 °C)	
Web circuit (G10 based, End-cap TRT)	No	No	No	No	Irradiated sample smells.
Ultem®	No	No	No	No	
Carbon fibre (RD6 wheel)	No	No	No	trace	
Carbon fibre (Perm)	No	No	n/a	n/a	
Tension plate (G10 based, Barrel TRT)	No	No	n/a	n/a	
HV plate G10 based, (Barrel TRT)	No	No	n/a	n/a	Non-irradiated sample smells.
Polycarbonate	No	No	No	No	

four and five report the results in the same conditions for the irradiates samples. In column six it is briefly summarized some additional observation done during the tests, that can be important for the final choice of the material. More details on the

test results are given below.

4.2.1 Web circuit

The *Web circuit* is a multi-layer printed circuit board with two flexible Kapton® layers sandwiched between two glass-fibre reinforced plastic boards[8-4]. It is used in the TRT end-cap wheels and the prototype tested here was produced in the Printed Circuit Shop of the EST/SM group at CERN. The samples consisted of two elements of size 55x34 mm^2 and 22x44 mm^2. No outgassing was observed during the standard set of measurement for both the non-irradiated and the irradiated samples. The irradiated sample exhibited a characteristic smell: however, no outgassing was observed during the set of measurements. This is probably due to the fact that the molecules outgassed are heavier than the ones to which our chromatograph is sensible.

4.2.2 Ultem®

Ultem® PEI (polyeterimide) resin from General Electric[1] is a plastic material candidate to be used for the production of the straw end-plug pieces. The material was available as 60 mm long cylinders with a 9 mm diameter. The non-irradiated (irradiated) sample tested consisted of three (two) of these elements. No outgassing was observed for this material.

4.2.3 Carbon Fibre (RD6 Wheel)

This sample of carbon fibre is the one used in the construction of the old RD6

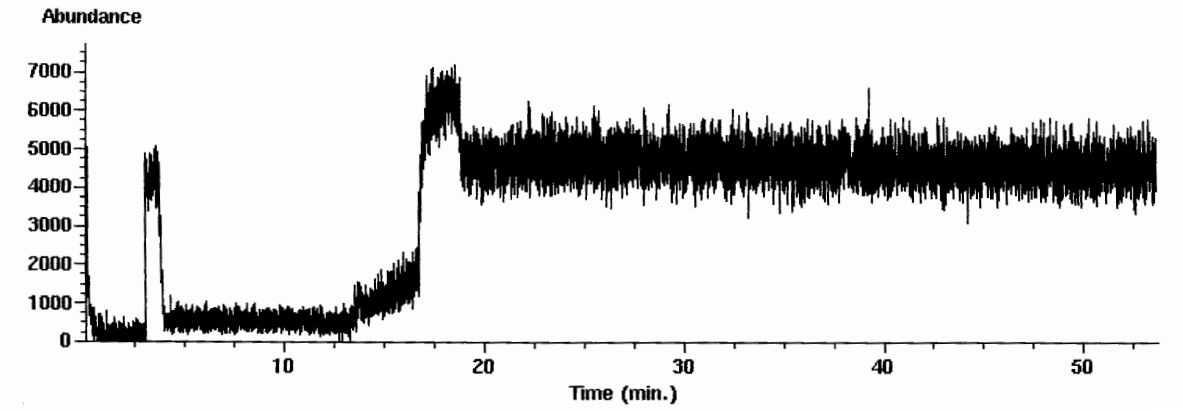

Figure 4-6 Chromatogram of an irradiated sample of carbon fibre, measured at 60 °C. A small outgassing peak can be seen at about 13.7 min retention time.

wheel[8-4]. The size of the non-irradiated sample was 29x95x3 mm^3 and the size of the irradiated one was 40x95x2 mm^3. Only a small trace of outgassing is observed for

1. General Electric Company, 3135 Easton Turnpike, Fairfield, CT 06431, USA.

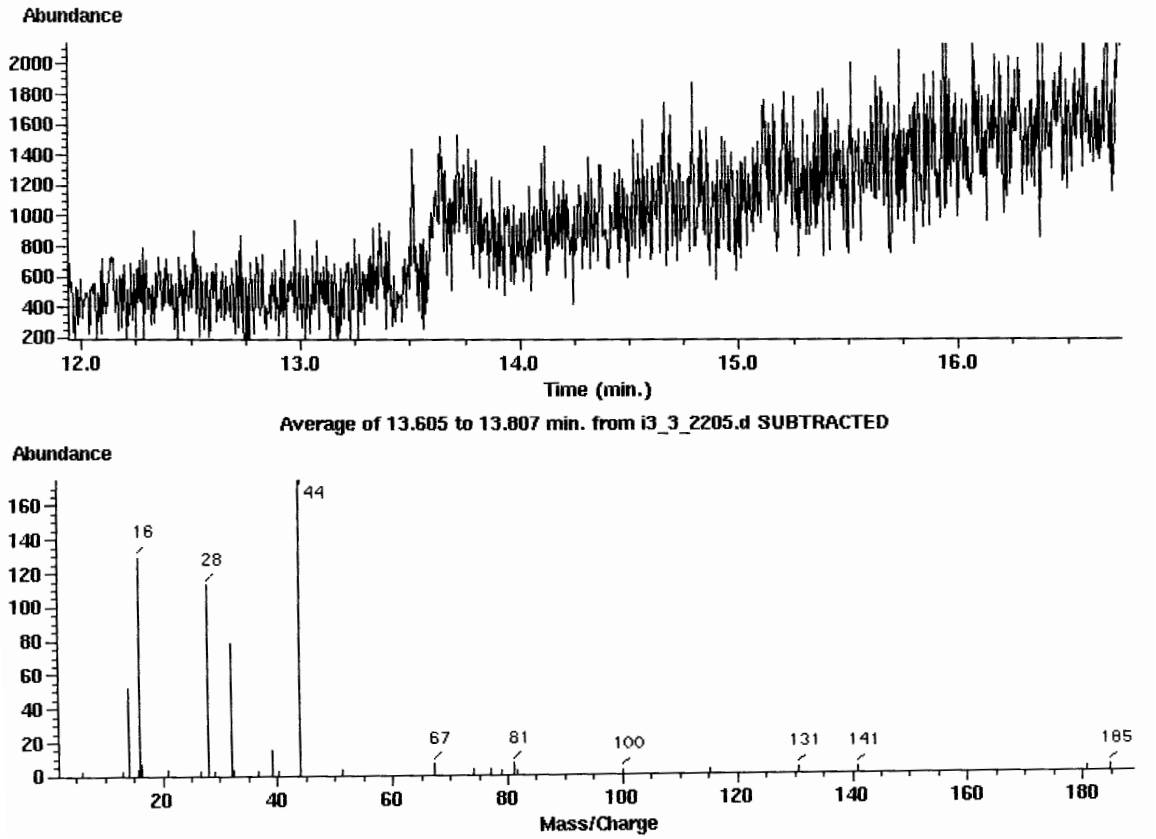

Figure 4-7 Blow-up of the peak observed for irradiated carbon fibre at 60 °C (top plot). The M/Z spectrum is not matched with any pattern contained in the system library (bottom plot).

the irradiated sample at 60 °C. The chromatogram for this sample is shown in Figure 4-6. Probably because of the small entity of this peak, the system is not able to identify the substance outgassed, from the its M/Z spectrum (see Figure 4-7). The peak has area of 76,921 and appears at retention time 13.718 min. By using the calibration factor determined in Section 3, we estimate this peak to be about 0.6 ppm.

4.2.4 Carbon Fibre (Perm)

The second sample of Carbon Fibre tested was produced at Perm (Russia) and it was available as five 32x32x2 mm^3 elements. Only non-irradiated samples were available. No outgassing has been observed, even at high temperature.

4.2.5 Tension Plate

The *Tension Plate* is a G10 based printed circuit board covered by varnish used in the construction of the barrel TRT[8-4]. The sample tested consisted of 8 pieces of approximate size 25x80x1.5 mm^3. Only non-irradiated samples were available. No outgassing has been observed.

4.2.6 HV Plate

The *HV plate* is a printed circuit board used in the construction of the barrel TRT[8-4]. The sample we tested consisted of a 13 mm thick G10 board. The sample was received as a single 103x103 mm^2 piece, so that it was necessary to cut it in smaller slices before the test. Eight 103x13x4 mm^3 elements were tested. The sample exhibited a clear and characteristic smell whose intensity was also found to depend on how the sample was cut, machined and treated (washed in alcohol, kept in air or flushed with dry nitrogen). However, despite several attempts, no outgassing signal was ever observed for this sample in the GC/MSD system. Irradiated samples were not available.

4.2.7 Polycarbonate

Polycarbonate is another plastic material candidate to be used for the production of the straw end-plug pieces. We tested 25 (23 for the irradiated sample) 1 cm long cylindrical elements with an average diameter of 4 mm and having a 1 mm diameter hole drilled in the center. No outgassing was observed for both the irradiated and non-irradiated samples.

4.3 Glues

The list of all the glues that have been tested and the results of our standard set of tests are summarized in Table 4-3. We divide all glues in three samples: *construction*, *sealing* and other (*conductive* and *instant*) glues[8-1]. Construction glues are required to provide the necessary rigidity in the TRT construction, the required gas tightness, low outgassing and also good electrical insulation properies. These glues should have large shear strength (above 10 MPa) and high viscosity. Among this category of glues we have tested: Araldit® AW 106, Araldit® AW 134 and Redux 420. The sealing glues are used in operations such as gluing the end-pieces to the straws and gluing the straws to the wheels. They are not required to have large shear strength but should have low viscosity and minimum outgassing because the surface in contact with the active gas mixture is large. Good electrical insulation properties is also an iussue. We have tested: Araldit® AW 103, Rutapox L20, Stycast 1266, Stycast part A and TRA-BOND 2115. Finally, we have also tested one conductive glue (TRA-DUCT 2902) and one instant glue (Pronto).

All samples were prepared according to the manufacturer's instruction as 20x80x3 mm^3 elements. For the non-irradiated case, the sample of glue we put in the outgassing box consisted of two of these elements, while for the measurements after irradiation we tested one and a half elements. This quantity of glues is well in excess of what will be really in contact with the gas mixture during standard TRT operations. More details on the test results are given below.

Table 4-3 Summary of outgassing results for all the glues tested: “No” means that no outgassing has been observed: “n/a” (not available) means that the irradiated sample was not available for that particular material. The comments in last column apply to both the irradiated and non-irradiated samples.

Glue	Non-irradiated		Irradiated		Comments
(temperature)	(22 °C)	(60 °C)	(22 °C)	(60 °C)	
Araldit® AW 106 (cured in air)	Large	Large	No	Large	Very good mechanical properties. Bad surface. Bad electrical properties.
Araldit® AW 106 (cured in N_2)	Large	Large	No	Large	(same comments as for AW 106 cured in air)
Araldit® AW 134	Large	Large	n/a	n/a	Bad surface. Very brittle. Bad electrical properties.
Redux 420	No	Small	No	No	Large shear strength. Electrical surface conductivity better than AW 106 (but not perfect)
Araldit® AW 103	No	No	No	No	Very good electrical and mechanical properties. Capillary effect not very good.
Rutapox L20 (cured at 60 °C)	No	No	No	No	Very good electrical and mechanical properties. Capillary effect very good. Too brittle if cured at 20 °C.
Stycast 1266	No	No	No	No	Wet surface at 60 °C. Not plastic.
Stycast A + Catalys 9	No	No	n/a	n/a	
TRA-BOND 2115	No	No	No	Small	Excellent capillary effect. Good mechanical and electrical properties.
TRA-DUCT 2902	No	No	No	No	Good conductivity.
Pronto	No	No	No	No	

4.3.1 Araldit® AW 106

The glue Araldit® AW 106 with hardner HV 935 U, from Ciba Geigy[1] is a glue well known for its high shear strength and its excellent radiation hardness. However this glue has also bad electrical properties (high surface conductivity: see Section 5) and it is not completely cured at the surface (needs to be cured at zero humidity).

1. Ciba Specialty Chemicals, Basel, Switzerland.

Two samples of this glue have been tested: one sample has been cured in air while a second one has been cured in a dry nitrogen atmosphere. The tests of this glue ac-

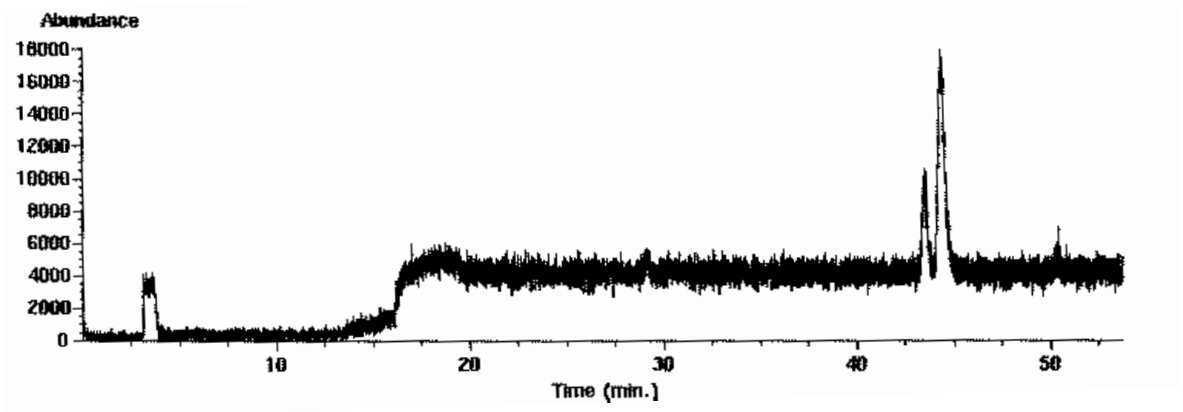

Figure 4-8 Chromatogram of non-irradiated AW 106 after 6 hours 60 °C.

cording to the procedure detailed at the beginning of this section have shown large

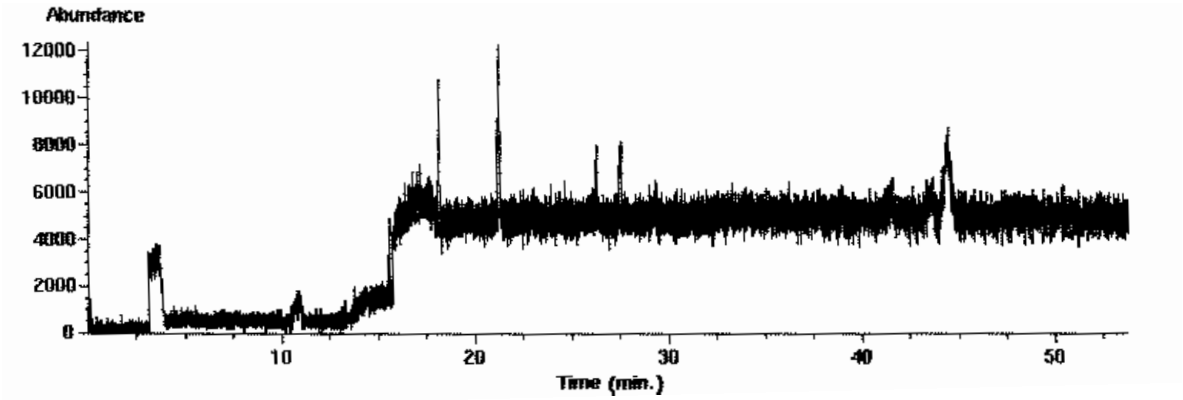

Figure 4-9 Chromatogram of irradiated AW 106 after 6 hours at 60 °C.

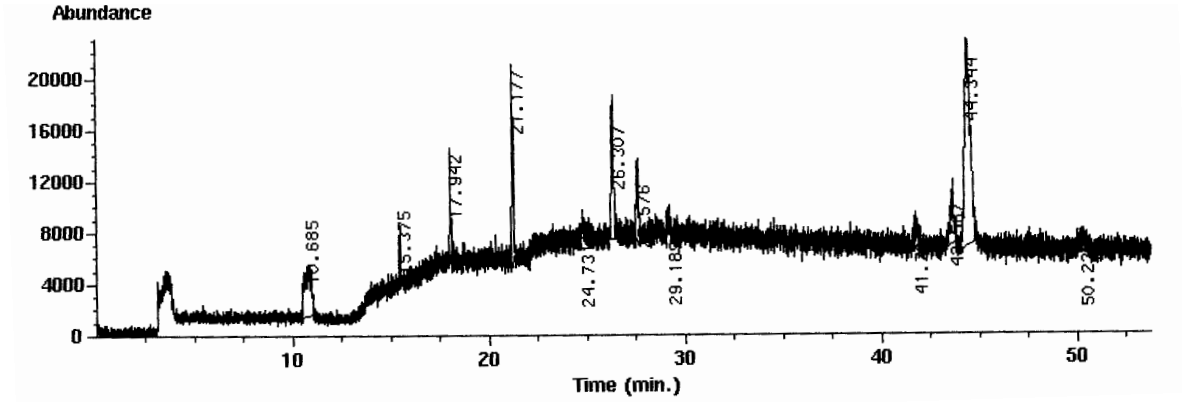

Figure 4-10 Chromatogram of irradiated AW 106 after 3 days at 60 °C.

outgassing for both the air and nitrogen-cured samples. The outgassing is already

present at ambient temperature but increases considerably at high temperature (see Figure 4-8). In fact, this glue has shown the largest outgassing among all the glues that we have tested. The outgassing was such that the system remained polluted and it was necessary to flush the box with helium at high temperature for about one day and to leave the GC column at 200 °C for few hours in order to recover clean operating conditions.
The irradiated AW 106 sample also showed outgassing when tested. However, by comparing the chromatogram before and after irradiation (Figures 4-8, 4-9 and 4-10), one can notice that the two large outgassing peaks observed before irradiation at retention times 44.431 and 50.560 min, become smaller in amplitude after irradiation and that new outgassing peaks from lighter compounds appear at shorter retention times. The list of all the peak observed and their amplitudes are summarized in Tables 4-4 and 4-5. The substances outgassed before irradiation are aromatic

Table 4-4 Details of outgassing peaks of Figure 4-8.

time (min)	area	substance
29.193	100,455	methyl-benzene
43.556	1,539,032	ethyl-benzene
44.431	3,675,860	1,2-dimethyl-benzene
50.560	186,437	1,3-dimethyl-benzene

Table 4-5 Details of outgassing peaks of Figure 4-10.

time (min)	area	substance
10.685	897,435	2,4-hexadiene-1-ol or butane
15.375	238,939	2-methyl-propanal or 2,2-dimethyl-oxirane
17.492	324,250	hexane
21.177	776,229	2,3,4-trimethyl-pentane or pentyl ester of Butanoic acid
24.731	173,733	oxybis-methane
26.306	855,770	2,2,3-trimethyl-butane
27.576	467,868	2-Pyrrolidinone
29.189	142,126	methyl-benzene or 1,3,5-cycloheptatriene
41.701	275,955	2,5-dimethyl-3-hexanone or 2-methyl-3-heptanone
43.567	710,829	ethyl-benzene or 1,2-dimethyl-benzene
44.344	3,233,855	xylene or 1,2-dimethyl-benzene (or 1,3-... or 1,4-...)
50.230	331,308	1,4-dimethyl-benzene or xylene

chemicals (containing the benzene C_6H_6 ring) while after irradiation also light alkanes appear. It might interesting to notice, as a double check of the performance of our system, that the peaks of Table 4-5 identified from their M/Z spectrum as butane and hexane (rows 1 and 3), appear at the same retention time in which they appeared in our alkane calibration mixture (see Table 3-1). This allows to estimate that the butane peak is about 13 ppm and the hexane peak is about 1 ppm. All the other peaks cannot be calibrated, because they appear at retention time which are outside the range of the calibration done in Section 3. The chromatogram of Figure 4-10 (3 days in the outgassing box at 60 °C) has been recorded about three months after the chromatogram of Figure 4-9 (6 hours in the outgassing box at 60 °C), during which the sample was kept at room temperature.

4.3.2 Redux 420

Redux 420A (hardner 420B) is an epoxy resin from Ciba Geigy. This glue has a large shear strength but its electrical properties (see Section 5) are not adequate to the use in the TRT.

In our standard tests both before and after irradiation, this glue has shown only small traces of outgassing before irradiation at high temperature. The chromatogram and the M/Z spectrum of the outgassing peak found for this glue are shown inFigure 4-11. The peak appears at 34.222 min retention time, has an area of 134,186

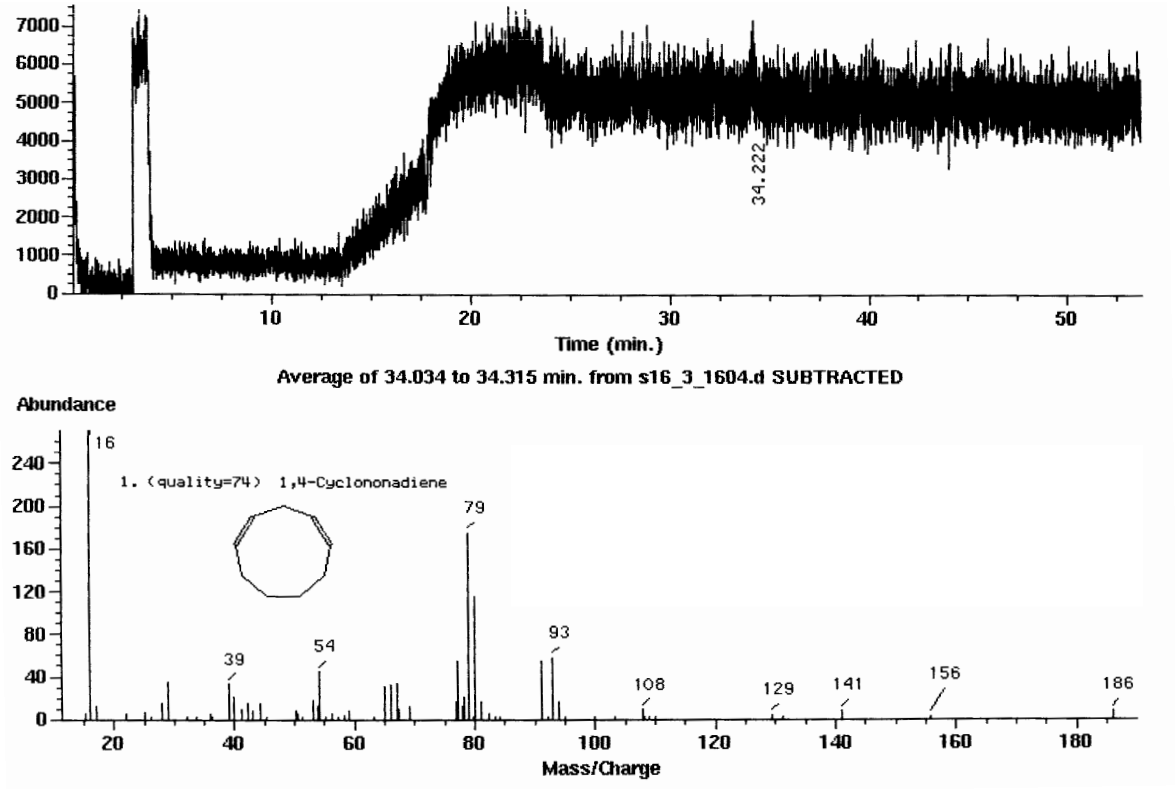

Figure 4-11 Chromatogram of Redux 420 (top). The M/Z of the small peak appearing at 34.222 min retention time is also shown (bottom).

and is identified as 1,4-cyclononadiene.

4.3.3 Araldit® AW 134

Araldit® AW 134 with hardner HY 991, from Ciba Geigy, is an epoxy resin which is expected to have properties similar to the Araldit® AW 106 already tested. As this glue, Araldit® AW 134 has also a bad surface and bad electrical properties. Moreover, this glue is very brittle.
Only non-irradiated samples where available for this kind of glue. The Chromato-

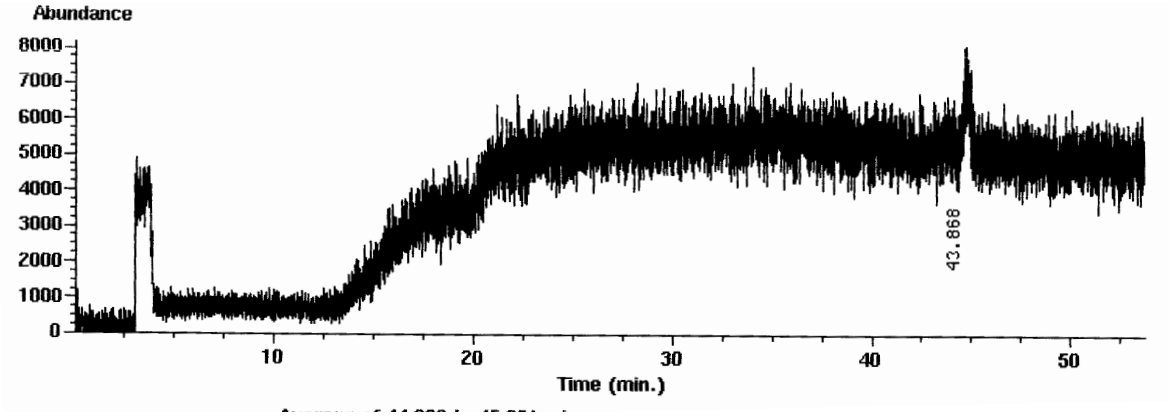

Figure 4-12 Chromatogram for AW 134 after 15 hours at ambient temperature. A small peak appears at about 43.888 min retention time.

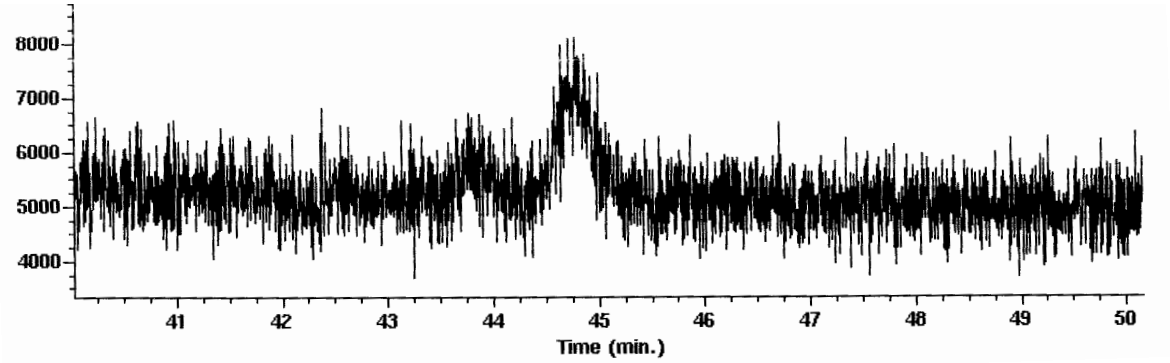

Figure 4-13 Blow-up around the region of the AW 134 peak at 43.888 min: a smaller peak can be seen at the left of the this peak.

gram of this glue at room temperature shows two small outgassing peaks (see Figures 4-12 and 4-13). These peaks considerably increase when the sample is heated up (see Figure 4-14) and another small peak appears at higher retention times. The kind of outgassing is very similar to what has been observed with Araldit® AW 106 and is summarized in Table 4-6 (for the measurement at high-temperature).

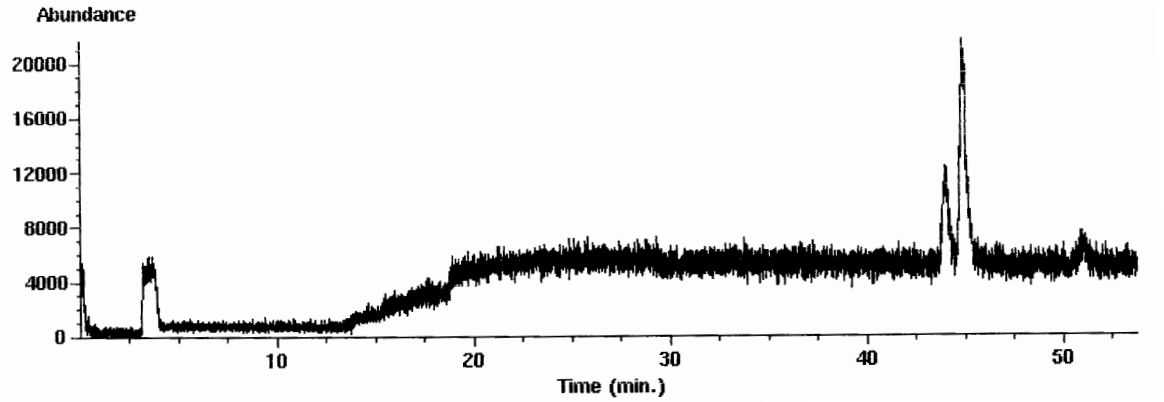

Figure 4-14 Chromatogram for AW 134 after 6 hours at 60 °C.

Table 4-6 Details of the three outgassing peaks observed for AW 134 kept 6 hours at 60 °C.

time (min)	area	substance
43.939	1,190,520	ethyl-benzene
44.806	3,644,889	xylene or 1,3-dimethyl-benzene
50.919	632,321	1,4-dimethyl-benzene

4.3.4 Araldit® AW 103

Araldit® AW 103 with hardner HY 956, from Ciba Geigy, has shown no outgassing during our standard set of tests. This glue has good electrical an mechanical properties (see Section 5): its capillary effect, however, is not perfect.

4.3.5 Rutapox L20

Rutapox L20 with hardener Rutadur SL, from Backelite, has been tested after curing at 60 °C. We decided to cure this glue at 60 °C instead of 20 °C, because if cured at room temperature this glue proved to be too brittle. For the rest this glue has both very good mechanical and electrical properties. The capillary effect is also very good. This glue has shown no outgassing during our standard set of tests.

4.3.6 TRA-BOND 2115

TRA-BOND 2115 (resin 2115A hardner 2115B) from TRA-CON[1] is a very low viscosity glue manifested good mechanical and electrical properties and also with an excellent capillary effect (see Section 5). However, it has to be mentioned, that producer specifies quite low volume resistance (~10^9 Ohm·cm) but in reality it has been found to be much larger (see Section 5).
This glue showed a small outgassing peak only for the irradiated sample kept at high

1. TRA-CON Inc., 45 Wiggins Ave, Bedford, MA 01730, USA.

temperature. The chromatogram for this sample (after 12 hours at 60 °C) is shown in Figure 4-15 and the M/Z spectrum is shown in Figure 4-16. The peak is identified as

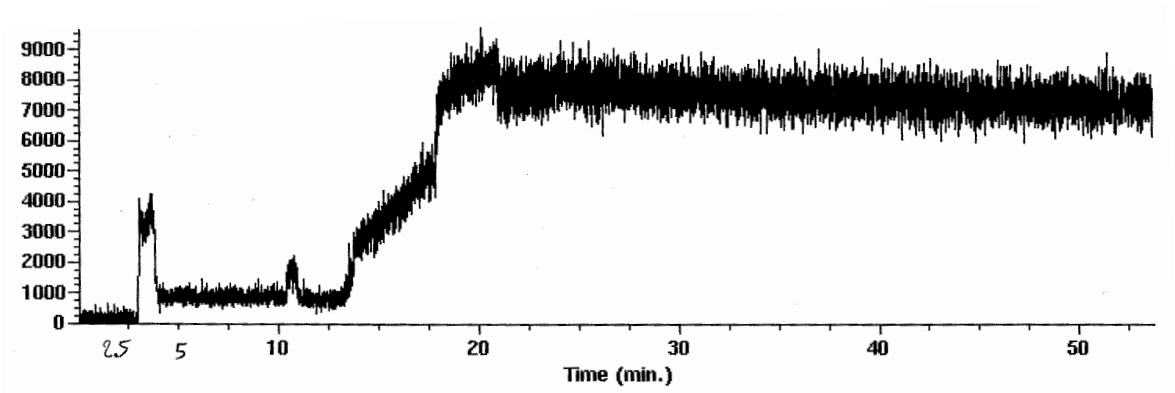

Figure 4-15 Chromatogram of TRA-BOND 2115 after 12 hours at 60 °C.

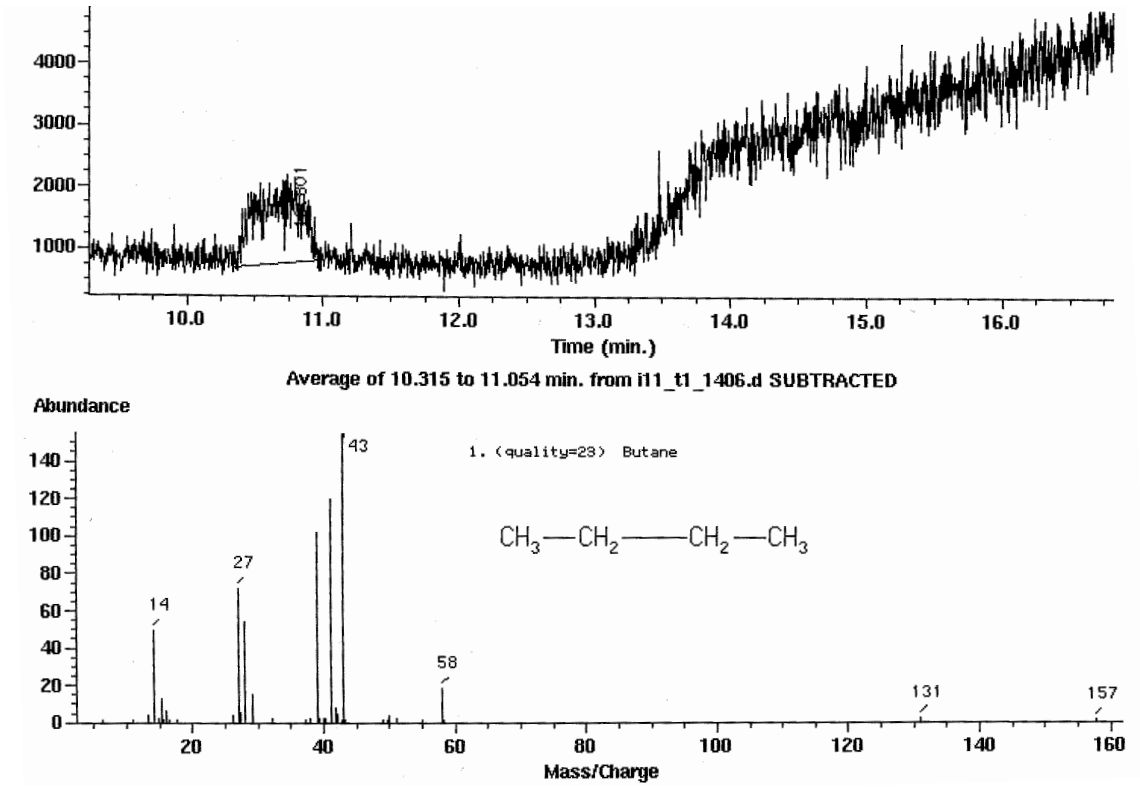

Figure 4-16 M/Z spectrum of TRA-BOND 2115 after 12 hours at 60 °C.

coming from butane. It appears at retention time of 10.801 min and has an area of 280,872. The retention time is consistent with the one at which we observed a butane peak with our calibration gas (see Section 3), and we can estimate that it corresponds to about 4 ppm.

Because of the its excellent mechanical and electrical properties, that makes it a good candidate for use in the TRT construction, this glue (irradiated sample) was studied in more detail. In Figure 4-17 we show the variation of the area of the butane peak as

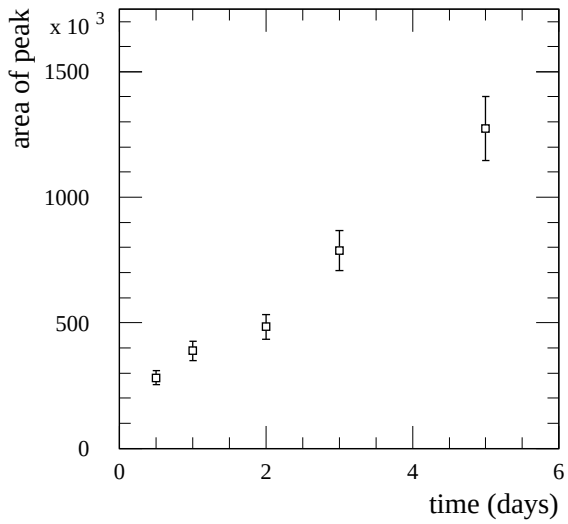

Figure 4-17 Outgassing from irradiated TRA-BOND at high temperature as a function of the number of days the sample has been left in the box. A total 20% uncertainty on each point is assumed.

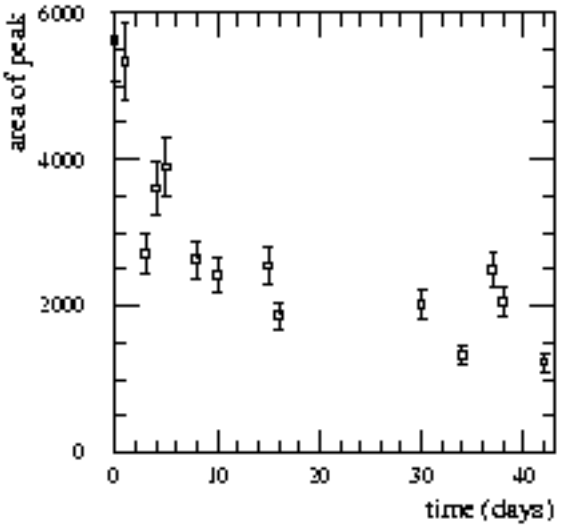

Figure 4-18 Outgassing from irradiated TRA-BOND left 24 hours in the box at high temperature as function of the time at which the measurement was taken. A total 20% uncertainty on each point is assumed.

a function of the number of days the sample has been left in the box at high temperature. A rough linear dependence can be observed. The measurement for irradiated TRA-BOND kept 24 hours in the box at high temperature was also repeated several times, in order to study the time-dependence of the outgassing. The results are shown in Figure 4-18: from this figure, one can see that after a small decrease of the outgassing in the first few days, then the outgassing seems to exhibit not much change with time.

4.3.7 Stycast 1266

Stycast 1266 from E&C[1] Specialty Polymers has shown a small trace of outgassing during the analysis of the non-irradiated sample at high temperature. This seemed to be confirmed by the clear presence of liquids on the surface on the heated sample. However, successive tests of this glue in the same conditions, while confirming that the surface of the glue becomes wet after heating at 60 °C, have not confirmed the presence of outgassing, probably because our GC/MSD system is not tuned to operate efficiently for heavy molecules. For what concern the mechanical point of view, we observed that this glue is not perfectly plastic (for instance, small cracks appeared in the glue, while cutting the sample).

4.3.8 Stycast part A

Stycast part A + Catalys 9 from E&C Specialty Polymers showed no outgassing during our measurements. Only non-irradiated samples were available.

1. Emerson & Cuming Inc., 77 Dragon Court, Woburn, MA 01888, USA.

4.3.9 Pronto

Pronto CA Instant Adhesive from 3M[1] has shown no outgassing during our standards set of tests. No surface and adhesive body degradation was observed after irradiation.

4.3.10 TRA-DUCT 2902

TRA-DUCT 2902 from TRA-CON has shown no outgassing during our standard set of tests. No remarkable change of conductivity was found after irradiation.

5 Mechanical and electrical properties of glues

In our studies for the selection of the glues for the TRT construction, particular attention was also Section 5paid to the mechanical and electrical behaviour of the glues before and after irradiation. Mechanical properties (shear strength and elongation at break) of Araldit® AW 106, Araldit® AW 103, Rutapox L20, and Stycast 1266 in a wide range of irradiation doses (up to 300 Mrad) were studied in[8-5][8-6][8-7]. Single lap shear test results show that ionising doses up to 50 Mrad do not degrade the mechanical performance of the adhesive joints.. This behaviour can be explained by the fact that failures of the shear strength tests occur at the interface and the interface cannot be strongly modified by irradiation: Van der Waals bounds are not as sensitive as covalent ones. On the contrary, the behaviour during the elongation at break test and the shear module depend on the adhesive. The value of the elongation at break is a very important parameter. Any force applied to the glue should not lead to a creation of cracks inside of the glue body. From that point of view, as already mentioned, Araldit® AW 134 and Rutapox L20 cured at room temperature are not good candidates to be used in TRT, because they have proven to be brittle. Elongation at break becomes larger after irradiation: for example, Araldit® AW 106 at a dose of 50 Mrad shows a factor of two increase in elongation at break with respect to the non-irradiated sample. The shear module normally has little increase up to doses of ~100 MRad then drops by factor ~2. These studies allow to conclude that up to 50 MRad there is no substantial degradation of the mechanical properties of the glues tested.

Dedicated measurements of the electrical properties of all the glues tested for outgassing before and after irradiation were performed. In particular, we measured the surface and volume conductivity for each sample. The surface conductivity was measured applying a 1600 V difference of potential between two parallel strips of conductive copper tape of 10 mm width glued to the sample surface; the two strips were separated by 1 mm. The volume conductivity was measured applying a 5 kV tension to two 10x10 mm^2 conductive copper tape electrodes fixed to both sides of a 2 mm thick glue sample: the electrodes were kept at a distance of about 3 mm from any side of the sample. Before testing, the surface of the glue was washed with alco-

1. 3M, 3M Center 304-01-01, St. Paul, MN 55144-1000, USA.

Table 5-7 Results of surface and volume current measurements for the samples of glues before and after irradiation. Each measurement has been repeated twice, in condition of low humidity (less than 50%) and high humidity (greater than 70%).

Glue	Dose (Mrad)	Surface current (nA)		Volume current (nA)	
(humidity)		(Low)	(High)	(Low)	(High)
Araldit® AW 106	0	15	250	4	70
(cured in air)	50	7	260	2	4
Araldit® AW 106	0	1	150	2	3
(cured in N_2)	50	2	150	2	3
Araldit® AW 134	0	680	n/a	6	n/a
	50	n/a	n/a	n/a	n/a
Redux 420	0	60	n/a	15	n/a
	50	170	n/a	6	n/a
Araldit® AW 103	0	5	20	2	4
	50	2	3	4	70
Rutapox L20	0	8	70	6	3
(cured at 60 °C)	50	1	2	2	4
Stycast 1266	0	2	n/a	5	n/a
	50	2	n/a	5	n/a
TRA-BOND 2115	0	1	10	5	6
	50	3	5	4	17

hol and then exposed to dry atmosphere.

The measurements were performed in dry atmosphere (humidity below 50%) and in conditions of high humidity (humidity above 70%): the results are shown in Table 5-7. The surface leak current results are expected to depend on the ambient humidity and, in fact, an increase by at least one order of magnitude for all glues is observed going from low to high humidity conditions. This dependence is not observed in the case of the volume conductivity, also proving that there is no large edge effect in these measurements.

From this table, one can see that all construction glues (Araldit® AW 106, Araldit® AW 134 and Redux 420) have at least by one order of magnitude larger leak current (particularly at high humidity) than the other glues tested. Without whashing with alcohole surface conductivity of all costruction glues was much higher.

In some cases, volume conductivity for these glues is also slightly larger. The exposure to a 50 Mrad ionising radiation does not change the electrical properties: in some cases, even an increases of the surface resistivity has been observed.

6 Conclusions and Recommendations

On the basis of studies described in this paper one can draw a set of recommendation for the choice of materials to be used in the construction of the TRT.

Glues. None of the construction glues tested had optimal performance both in term of outgassing and electrical properties. Some of them are not suitable even from the mechanical point of view (like Araldit® AW 134). As we said, the main parameters for the selection of the construction glue are large shear strength and quite high viscosity. Araldit® AW 103 has shear strength of more than 10 MPa but medium viscosity. It has very good electrical and mechanical properties and it has not shown any outgassing during our tests. The recommendation is to use this glue as construction one.

Among the sealing glues, the best candidate is TRA-BOND 2115. This glue has little outgassing (only at high temperature after irradiation) and excellent electrical and mechanical properties.

Materials. None of the materials we have tested has shown large outgassing, not even during the measurements at high temperature and after irradiation. Probably, materials containing G10 do have some outgassing (they exhibit a characteristic smell): however, the sensitivity of our test method does not seem enough to register it.

The outgassing studies that we have performed allow to narrow down the choice of materials to be used in the construction of the TRT. Direct ageing studies are necessary for the final validation of the materials.

7 Acknowledgements

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8 References

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