Outgassing of glue in the Outer Tracker of LHCb; measurements with a quadrupole mass spectrometer

Maarten Buffing

Bachelor Project Report
April - June 2009

July 2009

Vrije Universiteit Amsterdam
Faculty of Sciences
Department of Physics and Astronomy
Abstract

Four different glues have been investigated in an outgassing measurement setup with a quadrupole mass spectrometer (QMS). One type of these glues (AY 103-1) was used in the construction of the Outer Tracker in the LHCb experiment. This detector shows an unexpected ageing effect, which can be traced back to irradiation of glue components in the detector gas. Recently, a heat treatment of all the detector modules at 40°C during approximately two weeks has been applied to reduce this ageing. With the QMS clear differences between the mass spectra have been observed for the different glues. These spectra did not change drastically by a similar heat treatment in our setup. One poisonous component in the older AY 103 was clearly recognised in its spectrum with help of the NIST 08 Mass Spectral Library, but the unknown replacement material in the glue AY 103-1 could not be identified.
1 Introduction

At CERN the LHC proton collider has been built. The LHCb experiment is one of the experiments associated with the LHC and is used to study CP violation. An unexpected ageing of the Outer Tracker (OT) in the LHCb experiment is observed, which can be traced back to irradiation of glue components in the detector gas. The reduced signal response associated with the ageing makes the LHCb detector less efficient over time. For this reason it is important to understand the ageing and find ways to prevent or undo the ageing.

A preventive method against ageing is heat treatment of all the installed OT detector modules to 40 degrees Celsius during about two weeks per modules cluster. In this report the effect of this method on the outgassing of glue will be analysed. By using a quadrupole mass spectrometer (QMS), the composition of the glue outgassing in vacuum can be studied collecting mass spectra. The setup containing the QMS can be heated to 40 degrees to create similar circumstances for the used glue samples as in the heated detector modules. Comparing the glue mass spectra with mass spectra from an external database of known glue compounds makes it possible to look for possible components in the glue outgassing. For this report four different glues, Araldite AY 103, Araldite AY 103-1, Araldite AW 106 and Trabond 2115, were analysed using the QMS. Araldite AY 103-1 is the glue that causes the ageing.

Section 2 gives a very short introduction of the physics of the LHCb experiment. This section also describes the detector in which ageing occurs. In section 3 the ageing in the LHCb detector will be discussed, as well as the glues that are used during the outgassing tests. Also previous outgassing tests of these glues will be mentioned. Details of the outgassing measurement setup will be given in section 4. In section 5 the results will be presented and in section 6 the results will be discussed.
2 The LHCb experiment

2.1 CP violation

Before describing the LHCb detector, a very short introduction of the physics of the LHCb experiment will be given [1, 2]. In nature multiple symmetries can be observed that lead to conservation laws in physics. Among these are charge conjugation and parity invariance. Charge conjugation means that a system behaves the same if all particles are interchanged for their antiparticles and vice versa. Parity invariance means that a system is symmetric under mirror reflection and a spin vector changes from left-handed to right-handed. It was believed that these symmetries are valid symmetries. However these symmetries are broken for weak processes. For example, only left-handed neutrinos exist in nature and all antineutrinos are right-handed. If C and P would have been valid symmetries half the neutrinos would have been right-handed. It appears that CP symmetry is more valid, as in the example of neutrinos was the case.

Another example of CP are $B_{d,s}^0$-mesons. A $B_{d,s}^0$-meson is composed of an anti b-quark and a d- or s-quark. As a result of CP symmetry conservation in weak processes it is possible to change a $B_{d,s}^0$-meson into an anti $B_{d,s}^0$-meson and vice versa. The dominant Feynman diagrams corresponding to this process can be seen in figure 1.

![Figure 1: The dominant Feynman diagrams related to the $B_{d,s}^0 \rightarrow \bar{B}_{d,s}^0$ process.](image)

It appears that CP can be violated too. This implicates that an anti $B^0$-meson is more likely to change in a $B^0$-meson than a $B^0$-meson changes into his antiparticle. The first measurements of CP violation were not observed in $B$-mesons, but in the decay of neutral kaons. In $B$-mesons the CP violation in decays can be relatively large. For example: if started with equal amount of $\bar{B}^0$ and $\bar{B}^0$-mesons, the decay $B^0 \rightarrow K^+ + \pi^-$ is 12% more common than $\bar{B}^0 \rightarrow K^- + \pi^+$ [3]. For the decay of neutral kaons CP violation causes an asymmetry in the observed decays of only a factor 1/500 compared to the amount of decays expected without CP violation.

2.2 The LHCb detector

The purpose of the LHCb experiment is not only to study CP in general, but also to study sources of CP violation beyond the Standard Model and other rare phenomena in $B$ decays. A picture of the LHCb detector can be seen in figure 2. The LHCb experiment is part of the LHC proton collider in which two proton beams collide with an energy per proton of 7 TeV. Such proton-proton collisions will take place every 25 ns. During these collisions $b\bar{b}$ quark-antiquark pairs are formed. These quarks will then hadronise independently. The luminosity of the bundles will be set to create just one $b\bar{b}$ pair per collision. At high energies the hadronisation are mostly produced in the same forward or backward cone. For this reason the LHCb detector is designed with an angular coverage of 10-250 mrad in the vertical direction and 10-300 mrad in the horizontal direction [4]. A more profound explanation of all components visible in figure 2 can be found in reference [4].

One detector in the LHCb experiment is the Outer Tracker (OT). In figure 2 the OT is labelled with $T_1$, $T_2$ and $T_3$. A schematic picture of the OT can be seen in figure 3. Only the green part is the OT, the purple part is the Inner Tracker. The OT is a straw tube detector used for the tracking of charged particles and the measurement of their momentum from their bending in the field of the magnet.
Figure 2: The LHCb detector [4].

Figure 3: On the left the outer tracker (green, light) [4] and on the right a cross section of a straw tube [5].

A cross section of a straw tube can be seen in figure 3. A straw tube (with a diameter of 4.9 mm), contains a gold-plated tungsten wire with a diameter of 25.4 µm which functions as an anode. The wall of the straw tube consists of a wound foil that is made of three layers. The inner part of the foil (the cathode) consists of 40 µm conductive carbon doped polyimide. A gas mixture of Ar (70 %) and CO₂ (30 %) is flushed through the straw tubes and a voltage of 1550 V is applied. Whenever a charged particle (created during the proton-proton collisions) enters a straw tube an ionisation track is formed. Electrons will then drift towards the anode wire as a result of the applied voltage. These electrons will create a current in the wire that can be measured due to the charge amplification close to the wire.

The OT contains 96 short (S) and 168 full (F) modules. An F-module has a length of 4850 mm and contains in total 256 straw tubes, while an S-module is half the length of an F-module and contains 128 straw tubes. In total over 55 thousand straw tubes are present in the OT.
3 Ageing and glue

3.1 Ageing

Ageing of detectors is the decline in performance when they are irradiated. It appears that the Outer Tracker faces this problem. It is determined that the ageing is caused by the glue AY 103-1 which is used to construct the OT modules and to fix the straw tubes to the light weight support panels [6]. Figure 4 shows the effect of this ageing.

![Figure 4](image)

Figure 4: On the left the response ratios of a part of an irradiated outer tracker module. At the coordinates (391, 32) a 2 mCi $^{90}$Sr source was present for 20 hours while ArCO$_2$ gas mixtures flow from right to left. On the right the amount of ageing as a function of the radiation intensity [6].

In figure 4 on the left the response ratio of an irradiated outer tracker module can be seen. A 2 mCi $^{90}$Sr source was present 15 cm above the module at the coordinates (391, 32) for 20 hours. The current in the wires was measured before and after radiation for every location on the OT-module using a scan with a collimated line source. The current after the radiation is divided by the current before the radiation. Basically figure 4 shows the decrease in performance for every position on the module, because it is a graph of $I_{after}/I_{before}$. If the irradiation should have had no effect, every point on the module would have had the value one.

As can be seen a decrease in performance is visible near the irradiation location. No ageing is observed directly after the gas has passed the area of highest radiation. The ageing does not occur in the areas of the highest irradiated intensity. In figure 4 on the right the percentage gain loss (decrease in performance) as a function of radiation intensity is shown. It can be seen that there is a peak in the graph corresponding to a radiation intensity in which the damage is maximal. A further increase in intensity results in less damage, which makes the ageing of the OT a special kind of ageing. A more profound analysis of this ageing can be found in reference [6].

In the not irradiated part of the module the ratio $I_{after}/I_{before}$ is between the 0.8 and 1.0. This does not implicate that these parts of the module have a decreased signal response as a result of ageing. The signal response is also related to the atmospheric pressure. If the atmospheric pressure decreases by 2% the signal response increases by 10%. On irradiated wires carbon deposits have been found using Energy-Dispersive X-ray spectroscopy (EDX) [6], this might be relevant for studies of the outgassing of AY 103-1.

A few methods against ageing have been presented so far [4]. All three methods are performed while the LHCb detector is not irradiated.
1. Heating the detector modules to a temperature of 40 °C while being flushed for approximately two weeks. This has the purpose of increasing the outgassing of glue components. These components are then removed from the detector as a result of flushing before it can condense on other parts of the outer tracker module where it might cause ageing.

2. Flushing of the detector with an ArCO₂ mixture.

3. A high voltage training, in which a high voltage (higher then the usual applied voltage) of 1900 V is applied to the wires and cleans the wires. This high voltage training can be used to partially undo the effects of ageing.

3.2 The glue in the Outer Tracker

As mentioned above the glue AY 103-1 is used in the OT. The glue is combined with the hardener HY 991 and glass bubbles are added to enhance the viscosity. These components are mixed in the proportions AY 103-1 / HY 991 / glass bubbles = 100 / 40 / 5.5. In a single F-module approximately 500 g of glue is used. Approximately half this amount is used to glue an S-module. Most of the glue is used to glue the side walls of the modules. Also glue is used to glue the strawtubes to the modules. It is believed that all glue can in time diffuse into the straw tubes.

For tests of outgassing properties of this glue, a total of four different glues are used. AY 103, AY 103-1, AW 106 and Trabond 2115 (see table 1). For the AW 106 samples in this experiment the hardener HV 953 U is used in the proportions AW 106 / HV 953 U = 100/80. In order to get an understanding of the glue outgassing it is important to know the components of the glue. From the safety sheets supplied by the manufacturer a number of glue components is known. This information is summarised in table 4 in section 5.2. The CAS numbers in this table are unique numerical identifiers that represent a chemical substance [7]. With the aid of this numbers it is easier to find information corresponding to the chemical substance. The molecular weight in table 4 does not represent the average weight in unified atomic mass units u of the molecule, but rather the mass calculated by using the most abundant isotope for each atom in the molecule.

In table 4 it can be seen that all glues, regardless of whether they cause ageing, are composed mostly of bisphenol A epoxy resin. This could indicate that bisphenol A epoxy is not responsible for the ageing. In section A more attention is given to bisphenol A epoxy resin. This conclusion is not entirely certain, because bisphenol A might chemically react differently with the other components in the glues. Also AY 103-1 has from all four glues the most unknown components (15 - 25 %).

3.3 The choice of glues

The abstract of an article on ageing and materials states: "Materials that might me used for assembling gaseous detectors and associated gas systems are catalogued according to their outgassing rate...". Among these materials are some glues [8].

First the amount of outgassing was tested using a method developed by NASA. In this method a sample (in this case glue) is preconditioned at 25 °C and 50 % relative humidity for 24 hours. After this the sample is heated to 125 °C for 24 hours. The sample is weighed before and after the outgassing so the mass loss can be determined. A collector is present to collect escaping volatiles. Also this collector is weighed before and after the 24 hours of heating. From these measurements the Total Mass Loss (TML) and CVCM are determined. The TML is given as the percentage mass loss of the sample. The CVCM is the mass that is collected by the collector, expressed as a percentage of the original mass of the sample. According to this method a sample is called not outgassing whenever TML < 1 % and CVCM < 0.1 %. Results for three different glue samples as stated in this article are given in table 1. According to this method Araldite AY 103 is a low-outgassing glue. However, Araldite AY 103 is out of production and Araldite AY 103-1 is used instead in the OT. The glue Araldite AW 106 is
interesting, because it has a higher outgassing and it is known that it has a bad effect on detectors, the ageing [8]. The article also states that some pollutants of Araldite AW 106 were identified using a mass spectrometer. These molecules are butane, hexane, trimethylpentane and trimethylbutane. Trabond 2115 is also recommended, like Araldite AY 103, and a module produced with Trabond 2115 does not show the same ageing effect as one with Araldite AY 103-1.

Table 1: The glues used in this project and the outgassing results of these glues by Capeans.

<table>
<thead>
<tr>
<th>Epoxy</th>
<th>Outgas</th>
<th>Effect in detector</th>
</tr>
</thead>
<tbody>
<tr>
<td>Araldite AY 103</td>
<td>NO</td>
<td>NO</td>
</tr>
<tr>
<td>(Hardener HY 991)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trabond 2115</td>
<td>NO</td>
<td>NO</td>
</tr>
<tr>
<td>Araldite AW 106</td>
<td>YES</td>
<td>YES</td>
</tr>
<tr>
<td>(Hardener HV 935 U)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Araldite AY 103-1[^]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Hardener HY 991)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

[^] Not tested by Capeans because this glue was not produced at the time.
4 Outgassing setup

4.1 The vacuum setup

A schematic of the setup used to study the outgassing of glue can be seen in figure 5. It consists of two vacuum chambers, from which the upper chamber contains a Balzers QME 200 quadrupole mass spectrometer (QMS) [9]. The two vacuum chambers are separated by a valve and the lower vacuum chamber contains an opening that can be used to insert a sample. This sample can be positioned in a (metal) basket. As a result of the valve, a sample can be brought into the setup without venting the upper chamber. This implicates that the upper vacuum chamber contains relatively few polluting gasses. For venting the lower chamber nitrogen gas is used.

Whenever the lower chamber is pumped down the valve can be opened. A magnet (not shown in figure 5) is present in the setup to transport the basket with the sample from the lower chamber to the upper chamber. However in this experiment the sample is kept in the lower chamber with the valve open (the pirani pressure gauge in the upper chamber contains an incandescent filament that would heat a sample if it would be placed in the upper chamber there due to its proximity).

Both vacuum chambers have their own vacuum pumps. These pumps consist of a three stage diaphragm pump [10] and a turbomolecular pump [11]. The diaphragm pump creates a fore vacuum pressure for the turbomolecular pump of approximately 1 mbar. The turbomolecular pump then creates a high vacuum of less than \(10^{-8}\) mbar if no sample is present. These pumps each have a pumping speed of 60 l/s. However this value is not corrected for several effects that limit the pumping speed. Calculated values of the effective pumping speed for nitrogen can be found in table 2. The calculation of these numbers and an explanation of the limitations can be found in appendix B.

The vacuum chambers can also be heated externally. This heating is normally used to clean the vacuum chambers. A high temperature will accelerate outgassing processes, thereby depleting the vacuum wall of gasses [12]. The maximum temperature that can currently be reached with the used setup is...
Table 2: Pumping speed in the vacuum setup for different situations for nitrogen at 40 °C.

<table>
<thead>
<tr>
<th>Status valve</th>
<th>Opened</th>
<th>Opened</th>
<th>Closed</th>
<th>Closed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Position of the sample (chamber)</td>
<td>Upper</td>
<td>Lower</td>
<td>Upper</td>
<td>Lower</td>
</tr>
<tr>
<td>Pumping speed $S$ (l/s)</td>
<td>120</td>
<td>120</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Effective pumping speed $S_{eff}$ (l/s)</td>
<td>69</td>
<td>70</td>
<td>35</td>
<td>40</td>
</tr>
</tbody>
</table>

53 °C. For this experiment the heating was also used as a way to create similar circumstances for the glue samples as in the heated modules in the outer tracker, by heating the vacuum setup to a temperature of 40 °C.

### 4.2 The quadrupole mass spectrometer

A quadrupole mass spectrometer (QMS) is used to analyse the outgassing of a sample in the vacuum setup. The QMS consists basically of three parts; an ion generating part, an analysis part and a detection part. A schematic picture of a QMS can be seen in figure 6.

![Figure 6: A Quadrupole Mass Spectrometer](image)

The ions are generated by bombarding the gasses in the vacuum chamber with electrons with an energy of approximately 70 eV. Most ions will be single ionised and have a charge +e, but double ionised compounds are also possible. After the ionisation, the (charged) ions will be accelerated toward the analysis part of the spectrometer, which is an electric quadrupole field. The quadrupole consists of four metal bars. Voltage is pairwise applied to these bars with both an AC- and DC-component. The AC-
component has an amplitude $U_0$ and a frequency $\omega$. As a result of these voltages an electric quadrupole field is present between the bars. For a given DC-component and frequency, only ions with a specific mass to charge ratio ($m/z$) will have a stable trajectory through the electric field in the quadrupole. These ions are called resonant ions [13]. Non-resonant ions (the ions with an other $m/z$) have an unstable path and will either collide with one of the bars or are shot out of the quadrupole. By varying the DC-component and the frequency of the alternating voltage ions with a different $m/z$ are allowed to pass the quadrupole. By doing so, a mass spectrum can be obtained which contains the amount of ions created by the ion source as a function of their mass to charge ratio. The highest $m/z$ that can be measured with this QMS is 200.

As detector of the ions a faraday cup or for higher sensitivity a channeltron is used. A schematic picture of a channeltron can be seen in figure 7. A channeltron is a resistive hollow tube functioning as a continuous dynode structure. Ions that made it through the quadrupole will collide with the wall of this channeltron. This collision will free one or more electrons. A voltage, applied over the channeltron, accelerates the electrons further down the tube: successive collisions of these electrons with the wall will create more secondary electrons. The applied voltage can be varied. So basically a channeltron functions like an electron multiplier and delivers a current. If more ions of a specific mass to charge ratio are present a higher current will be measured.

It is important to notice that with the QMS ions are measured that were made by bombarding the (neutral) gases in the setup. It is possible that such a molecule brakes apart during the ionisation and other peaks appear for the molecular fragments.
5 Results

5.1 Mass spectra

Mass spectra of the four glues can be seen in the figures 8 - 11. The spectra in these figures all have the same scale and are already corrected for the background spectrum (the spectrum that is visible when no sample is present in the setup). On the horizontal axis of the spectra the mass to charge ratio \( m/z \) is given in units of unified atomic mass units u over the charge in e. These four mass spectra were taken after 2 to 4 days outgassing in vacuum at room temperature (25 °C). AY 103-1 is used in the OT, AW 106 is a glue that is known to cause ageing and Trabond 2115 is a glue that does not cause ageing. Also AY 103 is included to compare with AY 103-1.

Some qualitative remarks on these spectra can be made. First of all the mass spectra of all four glues are more or less the same for the low mass part \( (m/z < 50) \) and are different for the middle mass part \( (50 < m/z < 100) \) and high mass part of the spectrum \( (m/z > 100) \). It also follows that most outgassing appears to be low mass components and in particular water. Water has a molecular mass of 18 and contributes to the mass to charge ratios 16, 17 and 18 in the mass spectrum with the highest peak at a mass to charge ratio of 18. Quantitative values for the amount of water in the outgassing can be found in table 3. The amount of water is defined as the partial pressure of water vapour relative to the total pressure. The error in these values is approximately 10 %. A calculation of these values can be found in appendix C.

<table>
<thead>
<tr>
<th>Glue</th>
<th>Outgassing of ( \text{H}_2\text{O} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>AY 103-1</td>
<td>68 %</td>
</tr>
<tr>
<td>AW 106</td>
<td>73 %</td>
</tr>
<tr>
<td>Trabond 2115</td>
<td>65 %</td>
</tr>
</tbody>
</table>

AY 103-1 has a lot of high mass peaks, with a difference of approximately 14 u between each group of peaks. This might indicate hydrocarbon chains. It is known that Araldite AW 106 causes ageing, but high mass peaks are less abundant for AW 106. Trabond high mass peaks are approximately 10 times less abundant than the high mass peaks in the AY 103-1 spectrum. For AY 103 it can be noted that the spectrum is very different from the AY 103-1 sample. This is striking, because the ingredients for AY 103 and AY 103-1 are for approximately 80 % the same and AY 103-1 was sold as the replacement of AY 103 that was taken out of production.

Mass spectra have been taken of glue samples before, during and after a heating process. The mass spectra shown in the figures 12 - 17 are corrected for background by subtraction of mass spectra taken for an empty vacuum setup. During the heating process the setup was heated to a temperature of 40 °C. AY 103-1 was heated for 12 days, Trabond 2115 for 7 days and AW 106 for 4 days.

The AY 103-1 spectra can be seen in the figures 12 and 13. Apart from the peak at a mass to charge ratio of 44, the shape of the spectrum remains the same. This indicates that the heating does not change the chemical structure of the glue in such a way that the outgassing changes. From these figures it can be seen that the heating increases the outgassing and that the amount of outgassing after the heating is lower than before. Previous analyses using this vacuum setup [15] indicates that a decrease in outgassing over time also occurs if the sample is not heated. Due to different experimental circumstances it was not possible to make a quantitative comparison. It can be concluded that the amount of outgassing at 40 °C is larger than at 25 °C without significantly change in the shape of the mass spectra, so heating the sample only seems to accelerate the outgassing.

The spectra of Trabond 2115 can be seen in the figures 14 and 15. It is noted that the peak in the mass spectrum at 185 seems to be the same before and after heating. This is also the case for AW 106.
The origin of this peak is unknown. Apart from this peak the shape of the trabond mass spectrum does not change at all during and after the heating procedure.

For Araldite AW 106 the shape of the mass spectrum remains the same before and after a period of outgassing in vacuum at room temperature, except for the peaks at 91 and 106. This can be seen in figure 17. It implicates that some component of the glue keeps outgassing at the original outgassing rate while the outgassing of other components decreases a factor of two after the heat treatment. A comparison with mass spectra of components will be carried out in section 5.2, but no molecule could be linked to these mass peaks.
Figure 8: mass spectrum of Araldite AY 103-1

Figure 9: mass spectrum of Araldite AW 106

Figure 10: mass spectrum of Trabond 2115

Figure 11: mass spectrum of Araldite AY 103
Figure 12: Mass spectra of AY 103-1 at room temperature (blue and dashed) and at 40 degrees (red).

Figure 13: Mass spectra of AY 103-1 before (blue and dashed) and after (red) heating.

Figure 14: Mass spectra of Trabond 2115 at room temperature (blue and dashed) and at 40 degrees (red).
Figure 15: Mass spectra of Trabond 2115 before (blue and dashed) and after (red) heating.

Figure 16: Mass spectra of AW 106 at 27 degrees (blue and dashed) and at 40 degrees (red).

Figure 17: Mass spectra of AW 106 before (blue and dashed) and after (red) three days of vacuum at room temperature.
5.2 Identifying compounds in mass spectra

The mass spectra of the glues contain many peaks and cannot be completely solved. Reference mass spectra can be found in an external database. An external database was needed because the setup database contains only 72 mass spectra. For this reason a comparison mass spectrum has been made using both databases for butane in order to compare the databases with each other. This can be seen in figure 18.

Figure 18: Butane mass spectrum according to the setup database (blue and left) and the NIST 08 Mass Spectral Library (red and right).

The butane mass spectra in figure 18 only slightly differ. This implicates that the mass spectra of the NIST 08 Spectral Library can be used in addition to the mass spectra in the setup database, although some uncertainties regarding this (which will be discussed later) are still present.

A difference can be made between low mass components (for example water) and high mass components (for example the components in table 4. Reference spectra of this low mass components (molecules with a mass of less than 50 u) can be used to look for similarities with the Araldite and Trabond spectra. From this water and hydrogen (with a mass of 2 u) can easily be identified due to their huge present. When solving the low mass part of the spectrum, other components such as ethane and propane could be identified. A previous experiment analysed the low mass part of the AY 103-1 spectrum. Solving a spectrum like the AY 103-1 spectrum just for the low mass part is very tricky. Some ions with a low mass to charge ratio could be building blocks of larger molecules that were broken apart during the ionisation.

Figure 19: Mass spectrum of dibutyl phthalate

\[\text{Relative intensity} \]

\[\text{m/z} \]

---

\[\text{For this the NIST 08 Mass Spectral Library was used }[16].\]
Mass spectra of the known components given in table 4 and section A can be found in the NIST 08 Spectral Library. As example of such a spectrum the spectrum of dibutyl phthalate (DBP) can be seen in figure 19. DBP is a molecule with a molecular weight of 278 u. The chemical structure can be seen in figure 19. This molecule consists of a phthalate group containing two butyl groups. When irradiated with electrons, circular structures are more stable than chains. For a circular group two bonds have to be broken to split the molecule in multiple fragments while in chains it is sufficient to break just a single bond in order to accomplish this. It appears that in a QMS almost all DBP molecules break apart and form an ion with a mass to charge ratio of 149. This also means that despite the finite mass range of the used QMS (0 - 200 u) it is possible to use these spectra to look for the presence of heavier molecules with molecular masses above the 200 u.

If DBP is present in the outgassing of a specific glue, a peak should be visible in the mass spectrum for a m/z of 149. For example in the spectrum of A 103 this peak is observed (see figure 11). This match is notable, because the peak in the spectrum of A Y 103 at a mass to charge ratio of 149 is much larger then the other peaks in the high mass part (m/z > 100) of the A Y 103 spectrum, as can be seen in figure 11. It is not completely certain that this peak is caused by DBP, because the peak can be a result of an other molecule. In the mass spectra of the other three glues no peak is visible for DBP, so the amount of DBP present in the outgassing of these glues is less then the detection limit. This qualitative approach can be used for all the known components of the glue using the mass spectra from the NIST 08 Spectral Library.

The mass spectrum of DBP is relatively simple, the peak at 149 is much larger than the other peaks. For other glue compounds the mass spectrum contains more of such high peaks. The highest peaks in the mass spectrum for each components of the glues, the hardener known from the safety sheet and some components that are present in the preparation of the bisphenol A epoxy resin can be found in table 5. A number is added to this m/z values. A ’1’ means it is the highest peak, a ’2’ means it is the second highest peak etcetera.

The mass peaks in table 5 are used to analyse the glue spectra (visible in the figures 8 - 11) for similarities. The analysis consists of checking whether the highest peaks as given in table 5 are present in the glue mass spectra. In table 6 the results of this analysis can be seen. YES means that the highest peaks in the component mass spectra are consistent with the glue mass spectra, NO means that they are not. The compounds in table 6 are sorted on molecular weight. The glue mass spectra have a lot of peaks in the low and middle mass part of the spectrum. For this reason a YES for α-epichlorohydrin, (butoxymethyl)oxirane and triethylenetetramine implicates a relatively large chance that these components are not present in the outgassing. Due to the small amount of peaks in the high mass part of the A Y 103 spectrum the YES for DBP in A Y 103 more likely implicates that DBP is really present in the outgassing.

Most surprisingly is that apart from dibutyl phthalate no high mass glue compound was compatible with the glue mass spectra. By checking the mass spectra for the most abundant ions as given in table 5, bisphenol A diglycidyl ether is compatible with the mass spectrum of Trabond 2115. A more profound analysis using all peaks in the bisphenol A diglycidyl ether mass spectrum revealed that bisphenol A diglycidyl ether is not compatible with the Trabond 2115 mass spectrum. It is though surprisingly that the bisphenol A diglycidyl ether mass spectrum is not compatible with the glue mass spectra, because all glue consists of approximately 80 % of bisphenol A epoxy. This epoxy contains a repetitive unit. In the analysis only the molecule with zero repetitive units (n = 0) was used. It is possible that other results are obtained when the glue mass spectra are compared to the mass spectra of bisphenol epoxy molecules with more repetitive units.
Table 4: Compounds of glues and hardeners as listed on the safety sheets.

<table>
<thead>
<tr>
<th>Glue/hardener</th>
<th>Compound</th>
<th>CAS-nr.</th>
<th>Concentration</th>
<th>Mol. weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>A Y 103</td>
<td>Bisphenol A epoxy resin</td>
<td>25068-38-6</td>
<td>75 - 85 %</td>
<td>&lt;700</td>
</tr>
<tr>
<td></td>
<td>Dibutyl phthalate (DBP)</td>
<td>84-74-2</td>
<td>15 - 21 %</td>
<td>278</td>
</tr>
<tr>
<td>A Y 103-1</td>
<td>Bisphenol A epoxy resin</td>
<td>25068-38-6</td>
<td>75 - 85 %</td>
<td></td>
</tr>
<tr>
<td></td>
<td>DPB free</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AW 106</td>
<td>Bisphenol A epoxy resin</td>
<td>25068-38-6</td>
<td>70 - 82 %</td>
<td>&lt;700</td>
</tr>
<tr>
<td></td>
<td>Bisphenol F epoxy resin</td>
<td>9003-36-5</td>
<td>5 - 10 %</td>
<td>700 - 1100</td>
</tr>
<tr>
<td></td>
<td>Bisphenol A epoxy resin</td>
<td>25068-38-6</td>
<td>1 - 5 %</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>Silicium dioxide</td>
<td>7631-86-9</td>
<td>1 - 5 %</td>
<td></td>
</tr>
<tr>
<td>Trabond 2115</td>
<td>Bisphenol A epoxy resin</td>
<td>25068-38-6</td>
<td>80 - 100 %</td>
<td>130</td>
</tr>
<tr>
<td></td>
<td>Oxirane, (butoxymethyl)-</td>
<td>2426-08-6</td>
<td>up to 20 %</td>
<td></td>
</tr>
<tr>
<td>HY 991 hardener</td>
<td>Triethylenetetramine</td>
<td>112-24-3</td>
<td>2 - 7 %</td>
<td>146</td>
</tr>
<tr>
<td></td>
<td>Phenol, 2,4,6-tris[(dimethylamino)methyl]-phenol</td>
<td>90-72-2</td>
<td>5 - 11 %</td>
<td>265</td>
</tr>
</tbody>
</table>

Table 5: Expected mass peaks for outgassing of glue components

<table>
<thead>
<tr>
<th>Compound</th>
<th>Highest peaks in the mass spectrum</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Epichlorohydrin[^i]</td>
<td>31[^2], 49[^4], 57[^1]</td>
</tr>
<tr>
<td>Oxirane, (butoxymethyl)-</td>
<td>29[^1], 31[^3], 41[^4], 55[^5], 57[^2], 58[^6]</td>
</tr>
<tr>
<td>Triethylenetetramine</td>
<td>30[^4], 44[^1], 56[^3], 73[^2], 99[^5]</td>
</tr>
<tr>
<td>Bisphenol A[^i]</td>
<td>91[^3], 119[^2], 213[^1]</td>
</tr>
<tr>
<td>2,4,6-tris[(dimethylamino)methyl]-phenol</td>
<td>42[^8], 44[^2], 45[^6], 58[^4], 133[^7], 162[^7], 176[^1], 177[^5], 220[^3]</td>
</tr>
<tr>
<td>Dibutyl phthalate</td>
<td>149[^1]</td>
</tr>
<tr>
<td>Bisphenol A diglycidyl ether[^i]</td>
<td>29[^4], 31[^6], 57[^5], 119[^7], 165[^9], 269[^8], 325[^1], 326[^2], 340[^4]</td>
</tr>
</tbody>
</table>

Table 6: Presence of the compounds listed on the safety sheets in the outgassing of the glues

<table>
<thead>
<tr>
<th>Compound</th>
<th>AY 103</th>
<th>AY 103-1</th>
<th>AW 106</th>
<th>Trabond 2115</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Epichlorohydrin[^i]</td>
<td>YES</td>
<td>YES</td>
<td>YES</td>
<td>YES</td>
</tr>
<tr>
<td>Oxirane, (butoxymethyl)-</td>
<td>YES</td>
<td>YES</td>
<td>YES</td>
<td>YES</td>
</tr>
<tr>
<td>Triethylenetetramine</td>
<td>YES</td>
<td>YES</td>
<td>YES</td>
<td>NO</td>
</tr>
<tr>
<td>Bisphenol A[^i]</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
</tr>
<tr>
<td>2,4,6-tris[(dimethylamino)methyl]-phenol</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
</tr>
<tr>
<td>Dibutyl phthalate</td>
<td>YES</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
</tr>
<tr>
<td>Bisphenol A diglycidyl ether[^i]</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
<td>NO</td>
</tr>
</tbody>
</table>

[^i]: CAS numbers and information of these components can be found in appendix A.
5.3 Weight changes

It is not only important to know the composition of the outgassing, also the mass loss is relevant. For this reason glue samples of all four different glues are weighed during periods up to two months. These measurements can be seen in figure 20. For every different type of glue between three and six samples were available and the data points in figure 20 are averages of multiple samples.

Figure 20: Percentage weight change as a function of time for glue samples under atmospheric conditions. 'New' samples were made a few days before these measurements started while 'old' samples were already a few months old.

The glue measurements have different error bars. This is related to the mass of the used glue samples. The average mass of the glue samples varied between 0.24 g for AY 103 and 1.19 g for AW 106. Lighter glue samples have a larger relative uncertainty, because the samples are measured with the same absolute uncertainty. Also not all glues have data points over the same period of time. This is related to the fact that some glue samples were made at a later stage of the project.

Under atmospheric circumstances, the weight of all glue samples increased. To verify that this mass increase is not caused by a drift of the balance electronics also two empty samples, consisting of only the supporting aluminium disks, were measured. The largest weight change measured for this empty samples (with masses 6.1648 g and 6.1787 g) during the two month measurement series was 0.7 mg. For Araldite AY 103-1, used in the OT, both new and old samples were measured. As can be seen in figure 20 the new AY 103-1 samples increase more in weight than the old AY 103-1 samples. The weight increase of new AY 103-1 is flattening out after a month. These observations indicate that the large weight increase is a start-up effect. It can further be seen that the difference between AY 103-1 and AW 106 samples is small. The glue AW 106 was chosen as a bad glue with a high outgassing rate (see table 1).

As a result of the mass increase due to the absorption of most probably water, it is not clear how much mass loss of glue components we have. Individual samples of AY 103, AY 103-1, AW 106 and Trabond 2115 have been analysed in the vacuum setup. The results of this can be seen in table 7. In the vacuum setup it was not possible for the samples to absorb gasses. The samples did not stay in the

\(^2\)The glue samples were kept in a cleanroom with a temperature between the 19 and 21 °C and a humidity between the 56 % and 64 %.  

21
vacuum under the same circumstances. However it is possible to make some qualitatively remarks from these data.

First of all, the mass loss of AY 103 is less than one percent over a period of 17 days. This is not in contradiction with the results in the article of Capeans. Further the mass loss of AY 103-1 at 40 degrees over a period of two weeks (about the same period as the modules are heated at CERN) is less than for the other glues. The AW 106 has as expected a higher weight loss than AY 103-1; a higher weight decrease is observed after a shorter period of outgassing. From the two results of AW 106 it is also clear that a higher temperature corresponds to a higher weight loss. This weight loss is still lower than one percent; the one percent in the paper of reference [8] was achieved with a temperature of 125 °C. Most surprisingly trabond has a higher outgassing compared to AY 103-1. Trabond 2115 does not cause ageing.

![Table 7: Weight change of several glues in vacuum](image)

<table>
<thead>
<tr>
<th>Epoxy</th>
<th>Weight change</th>
<th>T (°C)</th>
<th>Duration (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Araldite AY 103</td>
<td>−0.83±0.03 %</td>
<td>25</td>
<td>17</td>
</tr>
<tr>
<td>Araldite AY 103-1</td>
<td>−0.35±0.01 %</td>
<td>40</td>
<td>14</td>
</tr>
<tr>
<td>Araldite AW 106</td>
<td>−0.36±0.01 %</td>
<td>25</td>
<td>4</td>
</tr>
<tr>
<td>Araldite AW 106</td>
<td>−0.71±0.01 %</td>
<td>40</td>
<td>4</td>
</tr>
<tr>
<td>Trabond 2115</td>
<td>−0.90±0.02 %</td>
<td>40</td>
<td>10</td>
</tr>
</tbody>
</table>

[i] The first 2 days at a temperature of 25 °C.
[j] The first 3 days at a temperature of 25 °C.

Also weight measurements of the samples that have been in the vacuum setup were performed. The results of this for AY 103-1 and Trabond 2115 can be seen in figure 21.

![Figure 21: Percentage weight change as a function of time for AY 103-1 and Trabond 2115](image)

It can be seen that after the evacuation the samples have a relative large increase in weight compared
to samples that have not been in vacuum. The increase in weight is most likely caused by absorbing water that is available in the air. The other candidates are nitrogen, oxygen and CO$_2$ due to their percentage presence in air. In section 5.1 it is shown that most outgassing in vacuum is water. Therefore it is most likely that the increase in weight of the glue samples is caused by the absorption of water.

6 Discussion

The analyses of mass spectra obtained with the outgassing measurement setup could not solve the glue mass spectra. In order to improve the understanding of glue outgassing and identifying the outgassing molecules, a number of analyses/methods can be used.

1. AY 103-1 and HY 991 are mixed in a specific proportion prescribed by the manufacturer (the ratio AY 103-1/HY 991 is 100/40). It might be possible that a small deviation from the ideal proportion causes one of the components to be left over after the curing. This can be analysed by making AY 103-1/HY 991 samples with slightly different proportions and look whether the mass spectrum is different. A not ideal AY 103-1/HY 991 proportion might cause more outgassing of more different components.

2. Reference spectra from an external database are used. It might be possible that the used QMS gives slightly different mass spectra for these components than the mass spectra used from this database. A reason for this might be that more (or less) electrons are used in the ion generating part of the spectrum. In this case the ions generated by a single ionisation have a larger (or smaller) chance of breaking apart in smaller fragments. This will probably not change the conclusions in section 5.2 in where only the highest peaks in the spectrum were used. It can help solving the spectra in a more quantitative way if all components are known.

3. More experiments (other then outgassing tests using a QMS) should be performed that could give more clues on the possible composition of the glues. With this information, combined with a reference database of mass spectra, it might be possible to reveal possible molecules related to the high mass components in the AY 103-1 mass spectrum.

4. The mass spectra of AY 103 and AY 103-1 are different in the high mass region. AY 103-1 is known to cause ageing while AY 103 has no bad effect in detectors according to reference [8]. The glue composition of AY 103 and AY 103-1 is for approximately 80% the same, so this difference is most likely caused by the remaining 20% of the glue composition. For AY 103-1 this 20% of the composition is unknown. Therefore it could be useful to concentrate in further experiments on the unknown component(s) in AY 103-1. By comparing the mass spectra of these unknown component(s) with the AY 103-1 mass spectrum a better understanding of the glue outgassing could be achieved.

7 Conclusions

Heating glue samples to a temperature of 40 °C accelerates the outgassing, but does not drastically change the shape of the mass spectra. It also appears that the mass spectrum of Araldite AY 103-1 outgassing has more high mass components than the other glues (AY 103 and AW 106 and Trabond 2115). Mass spectra from a database must be used to look for the possible presence of components in glue mass spectra. An analysis of the glue-mass spectra of AY 103-1 did not reveal possible compounds that can be related to these high mass peaks. It can further be concluded that all four glues have a strong outgassing of water in vacuum and probably absorb water from the air.
References


[9] O. Postma, *Vacuum outgassing measurements for the HERMES vertex detector*, Nikhef, MT-99-03 (September 3, 1999);
Nikhef engineering department (contact B. Munneke), *Ontgassingstank; UHV gedeelte*, blueprint, drawing number 00001 (10-4-1996).


Bisphenol A epoxy resin

Bisphenol A epoxy resin is formed from epichlorohydrin[3] and bisphenol A[4][17]. For the preparation an excess of epichlorohydrin over bisphenol A is used in the presence of sodium hydroxide, which can be seen in figure 22. This excess of epichlorohydrin is removed from the system after the preparation of the glue. Firstly diglycidyl ether of bisphenol A epoxy[5] (called DGEBA) is formed. The reaction can be seen in figure 22.

As can be seen in figure 23 polymerisation of the diglycidyl ether of bisphenol A results in longer chains. This increases the viscosity of the glue. For this a catalyst is required. Among the catalysts that accelerate epoxy curing reactions are polyamines, molecules containing multiple nitrogen atoms. Both known components of HY 991 are such polyamines.

---

B Effective pumping speed of the pumps

Both turbomolecular pumps should operate at a pumping speed of 60 l/s. As a result of several limitations in both the pump itself and the vacuum setup the effective pumping speed is lower. The two most important contributions come from

1. The pumps are less effective for low-mass gas molecules.
2. Tubes and openings in the vacuum setup will create a sort of 'resistance' for the pump.

A turbomolecular pump contains a turbine with rotor disks which rotate about the thermal velocity of the molecules. However, the thermal velocity of light molecules like hydrogen is larger than for molecules like nitrogen. As a result of this the chance that a hydrogen molecule will diffuse back into the vacuum chambers after it has passed a rotor disk of the pump is larger than the chance a nitrogen molecule will do the same. This implicates the pump is less effective for light molecules. In the manual delivered by the manufacturer [11] the volume flow rate for hydrogen, helium and nitrogen is given. These values can be seen in figure 24.

![Figure 24: Mass dependance of the pumping speed of the turbomolecular pump.](image)

To obtain an estimate for the volume flow rate (or pumping speed) $S$ of molecules with an other mass, these data points have been fitted to the equation

$$S = S_0 \left( 1 - \frac{a}{m} \right).$$

Equation (1) satisfies the behaviour of the pump in the low mass region (a decrease in volume flow rate for lower masses) and in the high mass region (the volume flow rate as function of the mass is almost a constant for high masses). By setting the volume flow rate for high mass molecules at $S = 60$ l/s, the obtained value for $a$ is $0.61 \pm 0.02$.

In the used vacuum setup tubes and valves are present. A pressure difference $\Delta p$ between the ends of a tube exists. Given the amount of gas flowing through a tube $Q$, the conductance $C$ is defined as the proportionality constant between the pressure difference and $Q$:

$$Q = C \Delta p.$$  (2)

The conductance reduces the effective pumping speed of the pumps. For a tube of length $l$ and with a diameter $d$, the conductance is given by

---

26
\[ C = \frac{1}{6} \sqrt{\frac{2\pi RT}{m^3}}. \]  

(3)

In this equation \( T(K) \) represents the temperature, \( m(u) \) the mass of the molecules and \( R \) is the gas constant. The conductance for an opening with a diameter \( d_0 \) and a chamber diameter \( d_b \) is given by

\[ C = \frac{1}{8} \sqrt{\frac{2\pi RT}{m}} \frac{d_b^2 d_0^2}{d_b^2 - d_0^2}. \]  

(4)

Conductances in series, for example two tubes in a row, are added as

\[ \frac{1}{C} = \sum_{i=1}^{N} \frac{1}{C_i}, \]  

(5)

and parallel conductances are added as

\[ C = \sum_{i=1}^{N} C_i. \]  

(6)

From these conductances the effective pumping speed in case the valve is closed can be calculated with the aid of equation

\[ \frac{1}{S_{\text{eff}}} = \frac{1}{S} + \sum_{i=1}^{N} \frac{1}{C_i}. \]  

(7)

In case the valve is open and a sample is present in one of the vacuum chambers, effectively two pumps are pumping. Also the tube between the two vacuum chambers adds an additional conductance to the system. So the effective pumping speed in this case is calculated with the equation

\[ S_{\text{eff}} = \left( \frac{1}{S_1} + \sum_{i=1}^{N} \frac{1}{C_{1i}} \right)^{-1} + \left( \frac{1}{S_2} + \sum_{j=1}^{M} \frac{1}{C_{2j}} \right)^{-1}. \]  

(8)

For the physical dimensions of the setup data from the setup blueprints [9] and from the ‘Vacuum Valves 2000’ book [18] were used. In equation [8] the indices 1 and 2 stand for the first and second pump of the system. The results of the calculations for a nitrogen gas can be found in table [2] in section [4.1].

For future usage of the setup for other samples, the effective pumping speed as a function of the mass \( m \) in unified atomic mass units and temperature \( T \) in kelvins can be seen in table [8] in section [5.1]. The parameter \( a \) has the value 0.61±0.02. The equations in this table can be used to calculate to effective pumping speed for a gas with an arbitrary mass and temperature.

**Table 8: Pumping speed in the vacuum setup for a gas with an arbitrary mass and temperature.**

<table>
<thead>
<tr>
<th>Status valve</th>
<th>Position sample</th>
<th>Effective pumping speed (m³/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Opened</td>
<td>Upper chamber</td>
<td>( \left( \frac{50}{3} \frac{m}{m-a} + 38.44 \sqrt{\frac{mT}{m}} \right)^{-1} ) + ( \left( \frac{50}{3} \frac{m}{m-a} + 42.64 \sqrt{\frac{mT}{m}} \right)^{-1} )</td>
</tr>
<tr>
<td>Opened</td>
<td>Lower chamber</td>
<td>( \left( \frac{50}{3} \frac{m}{m-a} + 27.19 \sqrt{\frac{mT}{m}} \right)^{-1} ) + ( \left( \frac{50}{3} \frac{m}{m-a} + 53.88 \sqrt{\frac{mT}{m}} \right)^{-1} )</td>
</tr>
<tr>
<td>Closed</td>
<td>Upper chamber</td>
<td>( \left( \frac{50}{3} \frac{m}{m-a} + 38.44 \sqrt{\frac{mT}{m}} \right)^{-1} )</td>
</tr>
<tr>
<td>Closed</td>
<td>Lower chamber</td>
<td>( \left( \frac{50}{3} \frac{m}{m-a} + 27.19 \sqrt{\frac{mT}{m}} \right)^{-1} )</td>
</tr>
</tbody>
</table>
C  Partial pressure of water

An analysis has been performed to determine the amount of water in the glue outgassing. For this analysis mass spectra corresponding to empty vacuum chambers (also called background spectra) were taken for different gas pressures. An example of such a spectrum can be seen in figure 25.

The 'height' of the peaks in the mass spectrum represents the abundance of ions and we assume that the current given by the channeltron is linear to the number of ions that enter the channeltron. For this reason a twice as large peak implicates a twice as large abundance of the ion (when corrected for the offset). The mass spectra of the components that are thought to be present due to outgassing of the walls can be used to solve this spectrum. The scale is logarithmic, so although not every single peak is identified, around 95 % of the spectrum can be solved. For example: the three high peaks with a mass of charge ratio of 16, 17 and 18 are characteristic for water and the peaks around mass 28 are caused by ethane. The peak at 2 can only be caused by hydrogen. Other peaks are caused by gases like nitrogen, oxygen en CO₂. The mass spectra from databases give the relative abundances of each peak, with the highest peak set to 100. Molecules like nitrogen, hydrogen and oxygen have one dominant peak. For this reason the relative abundances of these molecules can be calculated by scaling the mass spectra of the components in the gas mixture with a factor \( c_i \) so they fit the background mass spectra. The index \( i \) is just a number to distinguish the different molecules and has no chemical meaning.

For molecules like water it is slightly more complicated to measure the relative abundance. Not only the peak at a mass to charge ratio of 18 corresponds to water, the peaks at a mass to charge ratio of 16 and 17 also correspond to water molecules. This implicates that the amount of water molecules is 1.78 times the amount of water that corresponds to the peak at a mass to charge ratio of 18. Other molecules, like ethane, have the same problem. For this reason an additional parameter \( q_i \) is introduced that compensates for this. The relative abundance of molecules is than calculated by scaling the mass spectra of the individual components in the gas mixture with a factor \( c_i q_i \).

Define \( P_w \) as the partial pressure of water. The partial pressure of water is the contribution of the factor \( c_i q_i \) for water, divided by the contributions of all molecules in the gas mixture and multiplied by the total pressure. The mathematical formulation of this is

\[
P_w = \frac{c_w q_w}{\sum_{i=1}^{N} c_i q_i} P.
\]

For the measurement of the total pressure, an ionisation pressure gauge is used. Some molecules are
easier to ionise than other molecules. This implicates that the measured pressure \( P_{\text{det}} \) is dependant on the composition of the gas mixture in the setup and is not necessarily the same as the actual pressure \( P_{\text{gas}} \). The used pressure gauge measures the \( \text{N}_2 \) equivalent pressure. If an other gas is present in the setup, the actual pressure of the gas can be calculated from the measured pressure using coefficients \( g_{\text{gas}} \) that compensate for the difference in ionisation. The relation between the measured and actual pressure is

\[
P_{\text{gas}} = P_{\text{det}} g_{\text{gas}}. \tag{10}
\]

For calculating the partial pressure of water, this effect has to be taken into account. This requires that the composition of the outgassing and the coefficients \( g_{\text{gas}} \) to compensate for this are known. The coefficients for the high mass components in the glue outgassing and the precise composition of the glue outgassing are unknown, so an approximation has to be used. It will be justified that equation 9 is a good approximation.

Table 9: Coefficients to compensate for the different ionisation of different molecules in the pressure gauge [19].

<table>
<thead>
<tr>
<th>Gas</th>
<th>Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{H}_2 )</td>
<td>2.17</td>
</tr>
<tr>
<td>( \text{N}_2 )</td>
<td>1.00</td>
</tr>
<tr>
<td>Air</td>
<td>1.00</td>
</tr>
<tr>
<td>( \text{O}_2 )</td>
<td>0.990</td>
</tr>
<tr>
<td>CO</td>
<td>0.952</td>
</tr>
<tr>
<td>( \text{H}_2\text{O} )</td>
<td>0.893</td>
</tr>
<tr>
<td>( \text{CO}_2 )</td>
<td>0.704</td>
</tr>
</tbody>
</table>

From table 9 it can be seen that the correction factor of water is close to one. Also it is from qualitative arguments already known that most glue outgassing is water (see section 5.1). Suppose that water really is the dominant outgassing component. For correctly calculating the partial pressure of water, the coefficients \( g_1 \) of water and all the other components in the glue outgassing are required. These coefficients are also required to calculate the actual total pressure from the measured total pressure. In this case the actual partial pressure of water is given by

\[
P_w = \frac{g_w c_w q_w}{\sum_{i=1}^{N} g_i c_i q_i} P_{\text{gas}}. \tag{11}
\]

The total pressure is largely a result of water vapour (due to the observation that most outgassing is water). If the amount of water in the outgassing is expressed as the percentage of the total pressure, the partial pressure of water vapour has to be divided by the total pressure in the setup. If this is done, the correction factors largely cancel, because for the partial pressure the correction factor for water is used and for the total pressure a large part of the correction is a result of water. This implicates that if the correction factors in table 9 are all neglected by setting them to 1, the error in the calculated percentage water will be relatively small.

The percentage water in the glue outgassing will be calculated by neglecting the effect that the pressure gauge has not the same sensitivity for all molecules. Using this approximation implicates an error. The largest individual contribution to this error is caused by the hydrogen in the setup, which is approximately 25 times less abundant than water. Hydrogen has a correction factor of 2.5. As a result of hydrogen alone the real partial pressure of water will be 3% lower than the partial pressure as calculated with the equation 9. Apart from water and hydrogen, most outgassing glue components are other low mass components, with a correction factor of approximately 1. The coefficients for the high mass components are not known. In table 9 it can be seen that the coefficients decrease if the amount of atoms in a molecule increases. If this tendency continues for high mass gasses (\( m/z > 100 \)) the error in the
partial pressure of water can not be higher than 20%, because this deviation from the calculated value implicates that no high mass components are present in the glue outgassing at all. By using an estimated uncertainty of a factor of two in the amount of high mass components, the error in the partial pressure of water, calculated by ignoring the coefficients, is estimated to be 10 % or less.

The partial pressure of water is calculated for several background mass spectra at different pressures, as can be seen in figure 26. The ion current on the horizontal axis is the ion current of the channeltron for a mass to charge ratio of 18.

![Figure 26: The partial pressure of water as a function of the channeltron current for the mass to charge ratio of 18 at a channeltron bias of 1000 V.](image)

A linear function has been fitted to the data in the range of $4 \cdot 10^{-11}$ to $17.5 \cdot 10^{-11}$ A. This results in the relation

$$P_w = a + bI_c,$$

with $a = 152 \pm 41$ mbar and $b = 377 \pm 4$ mbarA$^{-1}$. With the aid of this calibration, it is possible to calculate the partial pressure of water other mass spectra, by using the channeltron current at a mass to charge ratio of 18. It should also be noted that apart from water no other molecule has a significant ion fragment in the mass spectrum for a mass to charge ratio of 18. Due to the large presence of water (as can be seen in the mass spectra in section 5.1), the contribution of other molecules to the ion current for a mass to charge ratio of 18 is negligible small. From the partial pressure of water, the molecular percentage water $R$ in the outgassing is obtained as

$$R_w = \frac{P_w}{P_t} \cdot 100\%.$$

Numerical values for this can be found in table 10. In this table the total pressure of the gas in the setup can be found as well. These pressures are already corrected for the background pressure.

<table>
<thead>
<tr>
<th>Glue</th>
<th>$R_w$</th>
<th>Total pressure (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AY 103-1</td>
<td>68 %</td>
<td>$5.63 \cdot 10^{-8}$</td>
</tr>
<tr>
<td>AW 106</td>
<td>73 %</td>
<td>$7.55 \cdot 10^{-8}$</td>
</tr>
<tr>
<td>Trabond 2115</td>
<td>65 %</td>
<td>$4.31 \cdot 10^{-8}$</td>
</tr>
</tbody>
</table>