Study of the outgassing rate of the NA62 Large-Angle Photon Veto system

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Abstract

We report on measurements of the outgassing rates of different parts of the Large-Angle Photon Veto detectors for the NA62 experiment. All of the measurements were performed at the facilities of the Servizio Vuoto of the Divisione Acceleratori at the Laboratori Nazionali di Frascati of the INFN. Many parts of the lead-glass detector modules were measured: wrapped lead-glass blocks, bare blocks, the original OPAL wrapping, and the PMT + mu-metal assemblies. The Tyvek® wrapping used to replace the original wrapping and the optical fibers for the monitoring system were also tested. A complete study of the vacuum properties of the ANTI-A1 vessel was performed at the Fantini factory. In all cases, the measured outgassing rates are compatible with the operation of the detector in high vacuum.

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1 Introduction

In order to be able to measure the very rare decay $K^+ \rightarrow \pi^+\nu\bar{\nu}$, the NA62 experiment must reject background from, e.g., $K^+ \rightarrow \pi^+\pi^0$ decays at the level of $10^{12}$. Kinematic cuts on the pion track provide a factor of $10^4$ and ensure 40 GeV of electromagnetic energy in the photon vetoes; this energy, carried by the $\pi^0$ decay photons, must then be detected with an inefficiency of $\sim 10^{-8}$. The NA62 large-angle photon veto system consists of 12 rings of $\sim 160$ to $\sim 250$ lead-glass blocks from the OPAL electromagnetic barrel calorimeter. The rings will be placed inside the existing NA48 “blue tube”, which provides a vacuum decay volume 120 m in length. For the large-angle photon vetoes, the maximum tolerable detection inefficiency for photons with energies as low as 200 MeV is $10^{-4}$. The interaction of the beam with residual gas in the decay region can produce a photon-free background to $K^+ \rightarrow \pi^+\nu\bar{\nu}$ that will become important if the vacuum is worse than a few $\times 10^{-6}$ mbar. For this reason, the large-angle veto system must be able to operate in a high-vacuum environment. To validate the technological choices made in the design of the veto system, a comprehensive series of outgassing measurements is mandatory.

2 Experimental equipment

The vacuum facility used for these studies is shown in Fig. 1. The test chamber is constructed of 316 stainless steel with an internal volume of $\sim 29$ liters (not including the small tubes for the measuring devices). In Fig. 1, the following equipment is shown:

1. Vacuum gauge system
2. Turbomolecular vacuum pump
3. System isolation valve
4. Residual gas analyzer
5. Main in/out flange

An Ionivac Transmitter ITR90 vacuum gauge was used to record pressure measurements. The combination of a hot-cathode ionization sensor after Bayard-Alpert and Pirani sensors permits vacuum pressure measurements on non-flammable gases and gas mixtures in the pressure range from $5 \cdot 10^{-10}$ to 1000 mbar.

The vacuum pump was a Varian TV301 Navigator 8919 turbomolecular pump. The TV301 Navigator is an integrated system for high and ultra-high vacuum applications, consisting of both the pump and its controller. The pumping action is provided by a high-speed turbine (max. 56000 rpm), driven by a high-performance, three-phase electric motor. The quoted base pressure, with the recommended fore pump, is $< 2 \cdot 10^{-10}$ mbar, while the nominal pumping speed for nitrogen is 250 l/s.

The vacuum valve was a VAT DN100 gate valve. The nominal leak rate for this type of valve is $< 1 \cdot 10^{-10}$ mbar $\cdot$ l $\cdot$ s$^{-1}$.

The ITR90 vacuum gauge was read out using an RS232 PC interface and LabView® software to allow pressure measurements at fixed time intervals.

3 Measurement of outgassing rates

To perform the outgassing rate measurements, two different techniques were simultaneously used. The redundant measurements allow the results to be cross-checked, providing a rough estimate of the accuracy obtained. We will use the following notation:

- $Q_S$: outgassing rate of the sample under investigation;
- $Q$: specific outgassing rate, i.e., the outgassing per $cm^2$ ($Q=Q_S/A$).
While the values of $Q_S$ for single detector elements are useful for estimating the outgassing of the veto system as a whole, the value of $Q$ for any given material is a property of that material, so that the measured values of $Q$ can be compared to the values in the literature.

### 3.1 Rate-of-rise technique

The basis of the rate-of-rise technique is the general flow equation:

$$ Q_{tot} = V \frac{dp}{dt} + Sp $$

derived for a typical vacuum chamber [1]. In this equation, $Q_{tot}$ is the total outgassing rate in the test chamber, $V$ is the volume of the test chamber, $dp/dt$ is the rate of pressure change in the chamber, $S$ is the pumping speed at exit from the chamber, and $p$ is the chamber pressure. In the application of the rate-of-rise method, the pumping speed $S$ is made zero by isolating the test chamber from the vacuum source. The resulting flow equation is:

$$ Q_{tot} = V \frac{dp}{dt} $$

As shown in Fig. 2 $Q_{tot}$ is composed of two parts: the test-sample outgassing rate ($Q_S$), which is the rate desired, and the empty-chamber outgassing rate ($Q_{EC}$). Once the empty-chamber outgassing rate has been estimated, its contribution can be subtracted
from the total observed outgassing rate \(Q_{\text{tot}}\) to obtain the test-sample outgassing rate \(Q_S\):

\[
Q_S = Q_{\text{tot}} - Q_{EC} = V \frac{dp}{dt} - Q_{EC}
\]  

(3)

Any process which results in a value of \(S\) different from zero violates the assumption underlying Eq. (3). Two such processes that can occur frequently during a rate-of-rise measurement are adsorption pumping and ionization-gauge pumping. Both adsorption and gauge pumping remove gas from the test chamber and result in a reduction of the observed outgassing rate. The volume in Eq. (3) represents the volume of expansion for the gas and corresponds to that of the empty chamber only when the sample being analysis is very small. In our tests, the volume of the lead-glass block is \(\approx 20\%\) of the total chamber volume, while the other samples have negligible volumes. Therefore, for the volume in Eq. (3) we use the effective volume \(V_{\text{eff}} = V_{EC} - V_S\):

\[
Q_S = V_{\text{eff}} \frac{dp}{dt} - Q_{EC} = (V_{EC} - V_S) \frac{dp}{dt} - Q_{EC}
\]  

(4)

### 3.2 Ultimate-pressure technique

The ultimate-pressure technique is also based on the general flow equation (Eq. 1). In the ultimate-pressure technique, the pressure is assumed to reach a certain, “ultimate” value and thereafter to remain constant. At this point, \(dp/dt = 0\) and Eq. (1) reduces to:

\[
Q_{\text{tot}} = S p_{\text{lim}}
\]

(5)

While it is quite easy to measure \(p_{\text{lim}}\), this is not the case for the pumping speed \(S\). In fact, the pumping speed is normally provided by the pump manufacturer with poor accuracy, and must be measured. To eliminate the uncertainty related to the pumping
speed, a diaphragm with a calibrated hole can be placed in between the pump and the vacuum chamber to reduce the pumping speed to the maximum flux thorough the hole. The conductance \( C \) of the hole connecting the pump to the vacuum chamber can be analytically calculated once the geometry of the system is known. For a circular hole of radius \( r \) we get:

\[
C = \frac{v_m \cdot A}{4} = \frac{RT}{2\pi M} \cdot \pi r^2
\]

where \( T \) is the temperature in K, \( R \) is the molar gas constant, and \( M \) is the molecular mass of the gas. Using an hole radius of 6.05 mm at room temperature (293.15K) we get, for common air, the following conductance:

\[
C_{\text{Hole}} \approx 13.3 \text{ l/s}
\]

After correcting for the presence of the pipe connecting the pump to the vacuum chamber, the effective pumping speed \( (S_{\text{eff}}) \) is:

\[
S_{\text{eff}} = C_{\text{Hole}} \cdot \frac{S_0}{S_0 + C_{\text{Hole}}} = 12.6 \text{ l/s}
\]

Using \( S_{\text{eff}} \), Eq. (5) becomes

\[
Q_{\text{tot}} = S_{\text{eff}} \cdot p_{\text{lim}} = C_{\text{Hole}} \cdot \frac{S_0}{S_0 + C_{\text{Hole}}} \cdot p_{\text{lim}}.
\]

As in the case of the rate-of-rise technique, the ultimate pressure of the empty chamber \( (p_{\text{EC}}) \) must also be subtracted to obtain the outgassing rate for the sample. Equation (9) becomes

\[
Q_S = Q_{\text{tot}} - Q_{\text{EC}} = S_{\text{eff}}(p_{\text{lim}} - p_{\text{EC}}).
\]

4 Outgassing measurement

All of the measurements presented were performed using the apparatus described in Sec. 2. The same procedure was used for each measurement. The sample was pumped until the pressure stabilized, and the ultimate pressure \( (p_{\text{lim}}) \) was recorded. The chamber was then isolated from the pump by closing the isolation valve, and the pressure was measured at fixed time intervals to allow the rate-of-rise measurement. Unfortunately, the system was not equipped with a temperature monitor system; therefore no correction to the measured pressures can be applied. In any event, all tests were performed in the same environment and roughly at midday, so that the temperature can safely be assumed to have been stable to within a few degrees. For all measurements we assume the temperature to be 293K and the precision of the pressure measurement to be ±15%. In addition, the dependence of the outgassing rate on the temperature of the sample may induce systematic differences in comparing different sample measurements. All errors quoted are to be considered as estimates of the accuracy achieved and not as precise determination of the experimental uncertainties.

4.1 Chamber leaks and outgassing rate

The vacuum chamber was examined for leaks using an ALCATEL TD 180 helium leak detector with a sensitivity of \( 5 \cdot 10^{-11} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1} \). The test program was started only after the leak detector indicated that all leaks present were negligible (i.e,
on the order of $10^{-10} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$). After 48 hours of pumping, the chamber reached an ultimate pressure of $7.8 \cdot 10^{-9} \text{mbar}$. This value is very near to the ultimate pressure of the vacuum pump, $5 \cdot 10^{-10} \text{mbar}$, once the presence of the diaphragm of known conductance is taken into account. Moreover, the residual-gas analyzer and vacuum gauge mounted on the chamber introduce a non-negligible amount of outgassing in these ultra-high-vacuum conditions. The measured outgassing rate for the empty chamber is $Q_{EC} = P_{lim} \cdot C_{Hole} = 1 \cdot 10^{-7} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$. This result should be considered as the total outgassing rate for the entire experimental setup, i.e., the sum of the rates for the vacuum gauge, the residual-gas analyzer, and the chamber itself.

### 4.2 Outgassing rates for lead-glass blocks

The first sample analyzed consisted of an entire lead-glass detector as shown in Fig. 4a. The detector was pumped for two weeks to reach the ultimate pressure. The outgassing rate was obtained using both the ultimate-pressure and the rate-of-rise methods. The value appearing in Tab. 1 is the average of the two results, while the uncertainty is their difference. The second test consisted of removing the wrapping from another crystal and pumping the unwrapped detector (Fig. 4b). The result in Table 1 was obtained after two weeks of pumping. The original OPAL wrapping removed from the crystal (Fig. 4c) and the PMT and mu-metal shield (Fig. 4d) were separately measured. After only one day of pumping, the results in Table 1 were obtained. Finally, a bare crystal with no PMT light guide that was never used in the OPAL experiment and never stored under the BA5 floor was tested.

The table shows that the contribution of the wrapping and the PMT to the total single block outgassing rate is small. A sizeable difference between the unwrapped detector and the bare crystal was observed. The difference might be explained by the fact that the two detectors were stored for the last ten years under very different conditions, rather than by the absence of the glue between the PMT, light guide, and crystal in the case of the crystal when tested alone. The origin of the outgassing observed was investigated using a residual-gas analyzer with a maximum mass value of 100 amu. In Fig. 3 the response of
Table 1: Outgassing results for block component.

<table>
<thead>
<tr>
<th>Component</th>
<th>Outgassing Rate $Q_S (mbar \cdot l \cdot s^{-1})$</th>
<th>Pumping Time (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full detector</td>
<td>$(2.0 \pm 1.3) \cdot 10^{-5}$</td>
<td>15</td>
</tr>
<tr>
<td>Unwrapped detector</td>
<td>$(1.9 \pm 1.2) \cdot 10^{-5}$</td>
<td>15</td>
</tr>
<tr>
<td>OPAL wrapping</td>
<td>$(0.9 \pm 0.5) \cdot 10^{-6}$</td>
<td>1</td>
</tr>
<tr>
<td>PMT + mu metal</td>
<td>$(2.0 \pm 1.6) \cdot 10^{-7}$</td>
<td>1</td>
</tr>
<tr>
<td>Bare crystal only</td>
<td>$(3.1 \pm 0.6) \cdot 10^{-6}$</td>
<td>15</td>
</tr>
</tbody>
</table>

4.3 Outgassing rate for 3M DP490 epoxy

A new gluing procedure has been adopted to strengthen the existing glue bonds between the crystals and the stainless-steel mounting flange, therefore eliminating the possibility for blocks to fall out during the life of the experiment. After the block is unwrapped, very thin stainless-steel strips (0.3 mm) will be glued onto the modules at the junction between the glass and the flange. The strips will be applied on all four sides. 3M DP490 epoxy will be used for the gluing. The contributions to the total outgassing rate for a single block were initially expected to be negligible. Nevertheless, a block with the glass-flange bond reinforced was pumped for two weeks and its outgassing rate was measured to be $Q_S = (0.9 \pm 0.6) \cdot 10^{-6} mbar \cdot l \cdot s^{-1}$. This value is somewhat lower than that for the bare crystal alone. Any sizable contribution to the outgassing rate from the epoxy can be excluded.
4.4 Outgassing rate for Tyvek® wrapping

As a part of the refurbishment process for the detectors, the original OPAL wrapping will be replaced by DuPont® Tyvek®. To understand the vacuum properties of this material, a sheet of 1 m$^2$ of DuPont® Tyvek® type 1560 B, equivalent to the amount necessary to wrap seven blocks, was pumped for five days. The outgassing rate was measured to be $Q_S = (1.4 \pm 0.3) \cdot 10^{-6} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$ for the 1 m$^2$ sheet, or $Q_S = (2.1 \pm 0.4) \cdot 10^{-7} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$ for a single block. This value is negligible compared to the total outgassing rate for a single block as listed in Table 1.

4.5 Test of the monitoring system outgassing

The monitoring system for the large-angle veto system will introduce into the vacuum region several meters of optical fiber to deliver LED light to each block. Two bundles of quartz fibers with Teflon sheathing, 400 and 200 \(\mu\)m in diameter, were pumped for 2 weeks. The outgassing rate was measured to be $Q_S = (1.0 \pm 0.2) \cdot 10^{-6} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$. In the final assembly, this amount of fiber will be used to illuminate \(\sim\) four blocks. The contribution to the outgassing rate per block will therefore be $(2.5 \pm 0.5) \cdot 10^{-7} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$. The total length of the measured fiber was \(\sim\)6 meters, so that the average outgassing rate per meter is $Q_{Sm} = (1.7 \pm 0.4) \cdot 10^{-7} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1} \text{m}^{-1}$. The light will be introduced into the vacuum region via a flange with a quartz optical feedthrough to which the fibers will be connected. A prototype flange has been tested, using a helium leak detector. The leak rate was lower than $1 \cdot 10^{-10} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$.

4.6 ANTI-A1 leaks and outgassing tests

The entire ANTI-A1 vacuum vessel was tested for vacuum leaks and outgassing at the Fantini Sud S.p.A. facility in Anagni (FR), Italy. All openings in the vessel were covered by blind flanges (i.e., with no feedthroughs). Figure 5 shows the vessel and the instrumentation used during the two tests. An ALCATEL TD 180 helium leak detector with a limit sensitivity $5 \cdot 10^{-11} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$ was used for the leak test (Fig. 5a 1)). The chamber was pumped to a pressure of $\sim 3 \cdot 10^{-6} \text{mbar}$, leading to a sensitivity of $\sim 1 \cdot 10^{-10} \text{mbar} \cdot \text{l} \cdot \text{s}^{-1}$ for the detector. A detailed test of the two flanges covering the open tube sections, the five service/ feedthrough flanges, the manhole, and all of the...
external welding points revealed no leaks. A limit of $<1 \cdot 10^{-10} mbar \cdot l \cdot s^{-1}$ can be set on the leak rate for the vessel.

The equipment described in Sec. 2 was mounted on the manhole flange, as shown in Fig. 5b), to measure the total outgassing rate for the vessel. The ultimate-pressure method was used, and the vessel was pumped for 96 hours. An ultimate pressure of $P_{lim} = (1.4 \pm 0.3) \cdot 10^{-6} mbar$ was reached, signifying an outgassing rate of $Q_S = (1.7 \pm 0.4) \cdot 10^{-5} mbar \cdot l \cdot s^{-1}$. The low value of the ultimate pressure demonstrates the high quality of the manufacturing—the vessel is free of virtual leaks. The total surface area of the vessel exposed to the vacuum is about $A = (1.82 \cdot 10^5) cm^2$; the specific outgassing rate of the vessel after cleaning can be computed to be $Q = (0.9 \pm 0.2) \cdot 10^{-10} mbar \cdot l \cdot s^{-1} cm^{-2}$. The value is in good agreement with values in the literature for the outgassing rate for unbaked Fe4340. In the final experimental configuration, the two yellow flanges will be removed. The outgassing rate for the vessel must be scaled to the reduced total surface of $A_{Eff} \sim (1.2 \cdot 10^5) cm^2$. The resulting total outgassing rate for the ANTI-A1 tube is: $Q_S = (1.1 \pm 0.2) \cdot 10^{-5} mbar \cdot l \cdot s^{-1}$.

5 Conclusion

The outgassing rates for individual parts of the lead-glass detectors have been measured. There is no evidence of significant contributions to the total rate from the wrapping, glue, or PMTs. The outgassing observed seems to arise mainly from water and air absorbed by the crystals. The difference between the outgassing rates for the first two blocks $O(10^{-5}) mbar \cdot l \cdot s^{-1}$ with respect to the bare crystal and newly reinforced block is significant $O(10^{-6}) mbar \cdot l \cdot s^{-1}$. One possible explanation involves the presence of the old wrapping, which is very tightly attached to the crystal and traps the humidity accumulated during storage in BA5. Unfortunately, in the test with the “unwrapped block”, the wrapping was removed only few minutes before the beginning of the test, so that the block didn’t have time to lose excess humidity. In the other cases, the blocks had been stored for many days and unwrapped in a low-humidity area.

A conservative estimate of the single block outgassing rate is $Q_{BLOCK} = (7 \pm 10) \cdot 10^{-6} mbar \cdot l \cdot s^{-1}$ after two weeks of pumping time.

The outgassing of the ANTI-A1 vessel can be compared with preliminary estimates obtained at CERN for the blue tube [3]. The blue tube shows a specific outgassing rate of $Q \sim 1.3 \cdot 10^{-9} mbar \cdot l \cdot s^{-1} cm^{-2}$ after 15 days of pumping time, while the ANTI-A1 vessel reached $Q \sim 1 \cdot 10^{-10} mbar \cdot l \cdot s^{-1} cm^{-2}$ after only 4 days. The difference is most probably due to the fact that the inside of the blue tube is painted.

As a final consideration, in Table 2, the estimated contributions to the overall outgassing rate from each of the components of the final ANTI-A1 prototype are listed. The estimates are obtained by scaling the measurements for each component to the amount to used in the entire assembly of ANTI-A1. For the outgassing of the blocks, both the worst case ($Q_S = (2 \cdot 10^{-5}) mbar \cdot l \cdot s^{-1}$) and the average ($Q_S = (1 \cdot 10^{-5}) mbar \cdot l \cdot s^{-1}$) outgassing rates are listed.

The table shows that the dominant contribution to the ANTI-A1 outgassing rate will come from the blocks. The contribution to the outgassing rate from the hardware and aluminum plates used to fix the blocks to the wall of the vessel is not included in the computation because it is considered to be small compared to the contribution from the blocks. The final outgassing of ANTI-A1 rate will be $(1 - 2) \cdot 10^{-3} mbar \cdot l \cdot s^{-1}$. A much more precise value of the total outgassing rate will be measured once ANTI-A1 has been fully assembled, averaging the outgassing rate of 160 blocks.
Table 2: Estimated outgassing rate of ANTI-A1 with 160 lead-glass (PbGl) blocks, after 15 pumping days.

A preliminary estimate of the outgassing rate for the entire large-angle veto system, consisting of 12 rings for a total of 2500 lead-glass blocks, is \((2 - 5) \times 10^{-2} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1}\). This value is very similar to that obtained at CERN for the entire blue tube[3], \((1 - 5) \times 10^{-2} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1}\).

References