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# Why do we flush gas in gaseous detectors? S. Procureur<sup>a</sup>, D. Attié<sup>a</sup>, S. Bouteille<sup>b</sup>, D. Calvet<sup>a</sup>, X. Coppolani<sup>a</sup>, B. Gallois<sup>a</sup>, H. Gomez<sup>a</sup>, M. Kebbiri<sup>a</sup>, E. Le Courric<sup>a</sup>, P. Magnier<sup>a</sup>, I. Mandjavidze<sup>a</sup>, N. Sellami<sup>a</sup>, R. Veenhof<sup>c</sup> *<sup>a</sup>IRFU, CEA, Université Paris-Saclay, F-91191 Gif-sur-Yvette, France <sup>b</sup>Iris Instruments, 45100 Orléans, France <sup>c</sup>CERN, Geneva, Switzerland*

## 8 Abstract

The effects and the origin of the gas degradation in a gaseous detectorbased tracker are investigated. The study focused on the so-called T2K gas, which turned out to be highly sensitive to pollutants. In particular the  $H_2O$  and  $O_2$  concentrations were monitored online in different conditions to establish their influence on the gain of the detectors. This pollution was first mitigated by a recirculating and accelerating gas system with the use of a turbine and different absorbers. Further measurements revealed that this pollution originates from a continuous permeation process through the different materials of the gas circuit. In particular, polyurethane-based gas pipes or polyester materials largely increase the level of humidity. As a direct consequence of this work, the gas autonomy of the muon telescopes currently deployed inside the Khufu's pyramid have been dramatically improved.

9 Keywords: Micro Pattern Gaseous Detectors, MPGD, Micromegas, Gas

- <sup>10</sup> purity, Humidity, Muography, ScanPyramids
- <sup>11</sup> *PACS:* 29.40.Cs, 29.40.Gx

## 12 1. Introduction and context

Gaseous detectors, and in particular Micro-Pattern Gaseous Detectors (MPGDs) [1] have been widely used in particle physics for decades thanks to their outstanding performance and their relatively low price. In contrast with many other detectors, however, their interaction medium often needs to be permanently regenerated, and the gas is usually released in the atmosphere. The environmental cost of these emissions is significant for every

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research center in the world, not to mention the associated gas purchase 19 budget. Beyond fundamental physics, there are also applications where de-20 tectors are installed in nature, in public places or in confined areas, and for 21 which the delivery, storage, and usage of high pressure gas bottles may be 22 an issue. Such a situation recently occurred when our team deployed sev-23 eral muon telescopes to scrutinize the inside of the Great Pyramid of Khufu, 24 within the ScanPyramids project [2]. After the first campaigns in 2016 and 25 2017 where the instruments were placed outside of the pyramid, the tele-26 scopes could indeed be installed inside the highly touristic Grand Gallery in 27 2018, where ventilation is intrinsically limited. The need to minimize the gas 28 flushing became quite apparent, and encouraged us to study in laboratory 20 the possibilities to reduce it in a way compatible with the required budget, 30 transportability and robustness. 31

#### 32 2. Experimental setups and first measurements

## 33 2.1. Detectors

The detectors used in our muography activity are  $50 \times 50 \text{ cm}^2$  bulk Mi-34 cromegas [3, 4] called *MG2D*, equipped with a resistive strip layer [5] and 35 a 2D strip readout connected with genetic multiplexing [6]. The bulk and 36 the cathode are based on two, 3.2 mm thick PCBs and the resistive layer is 37 obtained by screen printing on a Kapton film. The gas tightness and the 38 drift gap are ensured by an Aluminum frame of 8 to 15 mm depending on the 39 detectors. For the ScanPyramids project, they are flushed with the non flam-40 able T2K gas (Ar-iC<sub>4</sub>H<sub>10</sub>-CF<sub>4</sub>, 95%-2%-3% in volume) from 5 or 20 L high 41 pressure premix bottles. Typically, from 4 to 16 such detectors are flushed 42 in series, for a total volume between 8 and 50 L depending on the drift gap 43 (from 8 to 15 mm). For the outside campaigns of 2016-2017, the flushing was 44 ensured by a manual flowmeter approximately set at  $1.2 \,\mathrm{L/h}$ , resulting in an 45 autonomy of about 3 weeks with a 5 L bottle filled at 120 bars. Starting from 46 the second campaign, the high voltages were automatically adjusted through 47 a feedback using the signal amplitudes recorded in each detector from the 48 online reconstructed muons [7]. This feedback allowed to keep the gain prac-49 tically constant and ensured data taking in stable conditions. It also showed 50 a very significant HV gradient between the detectors of a single telescope, 51 with a difference of several tens of Volts between the first and the last in 52 gas, see Fig. 1. After these observations, it was decided to set a dedicated 53 test bench in our Saclay laboratory for investigations in a well controlled 54

environment.



Figure 1: High voltage evolution of the first and last detectors in gas within a muon telescope during a ScanPyramids outdoor campaign using the online amplitude feedback. The fluctuations mostly originate from temperature variations (daily oscillations) but also from pressure changes. Two 5L bottles were used for these data, with a gas flow reduction at the end of the campaign to prolongate the data taking.

55 56

#### 57 2.2. The TomoMu test bench

The TomoMu cosmic test bench consists of 4 horizontal MG2D detectors 58 mounted on a vertical axis. It is operated with the same equipment as de-59 scribed in [8], except that control is now done by a Nuc mini-PC from Intel, 60 and our new custom-made high voltage power supply board also controls 61 two commercial digital flowmeters from Bronkhorst to set the input gas flow 62 and measure the output flow. The temperature, pressure and humidity of 63 the gas were continuously measured by Yocto-Meteo sensors from Yoctopuce 64 placed inside the gas circuit. They were initially embedded in small, 50 mL 65 polyester-based boxes which were later replaced by aluminum ones and com-66 plemented by a Luminox sensor from SST Sensing, as shown in Fig. 2. The 67 Luminox sensor provides measurements of the temperature, pressure, and 68 oxygen level. These two sensing devices were read out by an online moni-69 toring program running on the Nuc and resulting data files were processed 70



Figure 2: The aluminum probe box containing the Yocto-Meteo (T,P,H measurement) and the Luminox read by a Yocto-Serial  $(T,P,O_2)$ .

<sup>71</sup> offline using the ROOT framework.

## 72

## 73 2.3. First measurements

For the first measurements, the gas was flushed in series in the 4 detectors, initially at a flow of 4 L/h. The high voltages adjusted automatically every 5 minutes with the amplitude feedback based on the online muon reconstruction. After a few hours, the HV stabilized <sup>1</sup>, and the input flow was reduced to 0.4 L/h. The result of the HV evolution afterwards is shown in Fig. 3: as was observed before, a large voltage difference appeared, with a time scale evolution of several days.

## 81

## 82 3. Improvements of the gas circuit

The above measurements clearly point to an important degradation of 83 the gas mixture all along the circuit. A Fluent-based simulation at  $0.4 \,\mathrm{L/h}$ 84 revealed that the gas velocity can differ by a factor 50 within the active 85 volume of the Micromegas, with velocities down to  $0.7 \,\mathrm{m/day}$  in the vicinity 86 of the detector corners. The gas quality thus differs even within a detector, 87 leading to gain inhomogeneities and an efficient loss of a few %. As the 88 intrinsic gain inhomogeneities of the detectors are not taken into account 89 in the Fluent simulation, a quantitative comparison with real data is not 90 straightforward. However, a 5% decrease of the muon rate recorded by the 91

 $<sup>^1\</sup>mathrm{apart}$  from the fluctuations due to T,P variations affecting the gain



Figure 3: High voltage evolution (at constant gain) of 4 detectors flushed in series in the TomoMu setup. After a few hours at 4L/h, the gas flow is reduced down to 0.4L/h (at T=10 hours on the plot).

telescope has indeed been observed in correlation with a gas flow reduction from 1 to 0.4 L/h.

## 94 3.1. Recirculation

One way to reduce these inhomogeneities is to increase the gas flow in 95 the circuit by decorrelating it from the input gas flow. This can be achieved 96 using a recirculation system which accelerates the gas and reinjects it at 97 the entrance, as depicted in Fig. 4. In particular, it makes use of a turbine 98 developed within the HARPO project [9] for the reinjection. In this setup, the 90 gas velocity can therefore be maintained at a high value independently on the 100 input gas flow. Using the same setup and the same detectors as before, the 101 implementation of this modified circuit provides a good homogeneity between 102 the detectors, as shown in Fig. 5. In this case, the HV are essentially equal<sup>2</sup> 103 and close to the mean HV value from the initial setup, as the recirculation 104 simply mixes the gas without changing the pollution level. 105

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<sup>107</sup> We open here a parenthesis to briefly discuss the question of the gas flow in

<sup>&</sup>lt;sup>2</sup>up to small intrinsic differences due to the detector manufacturing



Figure 4: Standard (left) and recirculation (right) gas circuits.



Figure 5: High voltage evolution of 4 detectors flushed with the recirculation mode in the TomoMu setup using the amplitude feedback as before. The recirculation starts at T=2 hours. Both vertical and horizontal scales are identical to Fig. 3 for easier comparison.

such a circuit. Its actual value indeed cannot be accessed by inserting another
flowmeter, as such a device introduces some inertia which slows down the gas.
Instead, we made use of the Darcy-Weisbach equation relating the pressure

111 drop  $\Delta P$  in a circuit flushed with a given gas at a velocity v:

$$\Delta P = f \frac{L}{D} \frac{\rho v^2}{2},\tag{1}$$

where L is the pipe length, D its diameter,  $\rho$  the volumic mass of the gas and f is a dimensionless pressure drop coefficient. In our case, the flow is completely laminar <sup>3</sup>, providing the following expression for f:

$$f = \frac{64}{R_e},\tag{2}$$

<sup>115</sup> where the Reynolds number  $R_e$  writes:

$$R_e = \frac{\rho v D}{\mu},\tag{3}$$

and  $\mu$  is the gas dynamic viscosity. Introducing the gas flow F one finally gets:

$$\Delta P = \frac{128}{\pi} \frac{\mu LF}{D^4},\tag{4}$$

This relation was checked separately with a Bronkhorst flowmeter related to 118 2 Yocto-Meteo sensors measuring the pressure drop through a 31 m pipe of 119 3 mm inner diameter. As can be seen in the Table 1 the agreement is very 120 good, which allows in turn to get the flow in an open circuit containing the 121 turbine through the measurement of the pressure drop. This measurement 122 was performed for different values of the turbine power supply, and for two 123 different engines, yielding values up to 5 L/h (see Fig. 6), *i.e.* 12 times the 124 input gas flow. 125

F [L/h]	0.1	0.2	0.3	0.5	1.0	2.0	3.0	4.0	5.0
$\Delta P_{calc} [mbar]$	0.10	0.19	0.29	0.48	0.96	1.92	2.88	3.84	4.80
$\Delta P_{mes} [mbar]$	0.09	0.20	0.32	0.50	0.93	1.94	2.91	3.88	5.01

Table 1: Pressure drop calculated from Eq. 4 and measured, as a function of the input gas flow.

126

127

## <sup>128</sup> 3.2. Effects of humidity and oxygen absorbers

As stated in the previous section, the turbine only provides a better mixing of the gas in the circuit, but does not change the contamination level.

<sup>&</sup>lt;sup>3</sup>The Reynolds number numerically yields less than 10 times the gas flow in L/h.



Figure 6: Gas flow obtained from the pressure drop measurement using a turbine with different configurations of engines, as a function of the turbine supply voltage.

An easy way to reduce this contamination is to add specific absorbers in the 131 circuit, as can be seen in Fig. 7. In this measurement, the TomoMu setup was 132 supplied with a constant 0.3 L/h input gas flow, using the recirculation mode. 133 The high voltages were monitored, as well as environmental parameters, hu-134 midity and oxygen levels. As can be seen from the upper right of Fig. 7. 135 the atmospheric pressure underwent large variations during the first 2 days, 136 leading to similar variations of the high voltage (adjusted for constant gain). 137 When the pressure finally stabilized, a stainless steel capsule containing hu-138 midity absorbers was introduced. Within a few hours, and thanks to the fast 139 gas circulation from the turbine, the humidity dramatically decreased from 140 4.5 to  $1 \text{ g/m}^3$ . Because of the better gas purity, all the high voltages dropped 141 by a considerable amount, about 18 V. After one day, all the parameters were 142 stable, so the oxygen absorber was introduced in a second stainless steel cap-143 sule. It consists of small iron-based bags initially developed by ATCO for 144 food industry. As for the humidity, the oxygen concentration rapidly de-145 creased from 1.2 to 0.4%, and was accompanied by another 18 V drop of the 146 high voltages. The amplitude of the HV drops are not yet understood. A 147 Garfield simulation showed a 0.5% increase of the level of  $H_2O$  would require 148 to increase by less than 5V the HV of the detector to keep the gain con-149 stant. However, the required HV increase was measured to be about 18 V in 150

our test. Such a discrepancy is not understood, and in general the extreme
sensitivity observed with this specific gas mixture to temperature, pressure
and pollutants is a mystery.



Figure 7: Measurements of the mean high voltage of the four detectors (upper left), environmental parameters (temperature and pressure, upper right), humidity and oxygen contamination in the gas (bottom left) and input gas flow (bottom right) in the setup with the turbine. The humidity and oxygen absorbers are introduced 3.4 and 4.4 days respectively. See text for details.

154

To better emphasize the correlation between humidity and high voltages for 155 a constant gain, we analyzed cosmic ray data taken during August 2019. 156 During this time, all the conditions were particularly stable (constant atmo-157 spheric pressure within 7 mbar, constant temperature within  $1.2 \,^{\circ}$ C, constant 158 oxygen level in the gaz within 0.25%) except for the humidity in the gas which 159 slowly increased due to the saturation of humidity absorbers. Fig. 8 shows 160 the remarkable correlation between the HV and the humidity level, with a 161 slope close to  $4 V/(g/m^3)$ . 162

163

## <sup>164</sup> 3.3. Effects of preliminary pumping and heating

<sup>165</sup> Another usual way to lower the gas contamination is to force outgassing <sup>166</sup> by pumping and heating the gas inside [10]. We tested this method with



Figure 8: Correlation between the mean high voltage (at a constant gain) of a telescope and the humidity level in the gas.

the 50 mL, polyester boxes containing the Yocto-Meteo, after gluing the box 167 cover to ensure the sealing. Though the Yocto documentation stated that 168 the probes should not be used in vacuum, it turned out they survived all the 169 pumping cycles without any degradation. The box first underwent a short 170 pumping at 54 °C, then it was filled with pure Argon with an overpressure 171 of 50 mbars. The humidity evolution was then measured by the Yocto-172 Meteo. This operation was repeated several times, with 4, 2.5 and 2.5 days 173 of additional pumping/heating. Because of the glue, the heating temperature 174 could not exceed 60 °C. The primary pump used provided a modest vacuum 175 slightly below  $10^{-1}$  mbar. The humidity measurements are shown in Fig. 9, 176 and reveal as expected a progressive drying of the box, though 9 days seem 177 still not enough to evacuate completely the humidity. 178

179

All these improvements allowed for a stable operation of the TomoMu setup with an input gas flow below 0.2 L/h, *i.e.* 6 times smaller than during 2016 measurements. In spite of our efforts, it was however not possible to operate TomoMu in a stable mode without any gas input. Indeed, a further reduction of the input gas flow generated a new increase of the high voltages which this time was not correlated with the humidity and oxygen levels. It is likely this contamination is due to Nitrogen, but a mass spectrometer would be needed



Figure 9: Humidity measurements inside the sealed Rose, polyester box after successive cycles of pumping/heating and Argon filling.

187 to confirm this assumption.

## <sup>188</sup> 4. Origin of humidity and oxygen contaminations

We now turn to the question of the origin of the observed gas contamination. It can proceed *a priori* through only 2 mechanisms:

## • leakage

• outgassing

The effect of direct leakage can be minimized with special care on the gas 193 tightness, and also by setting an overpressure in the detectors. Many mea-194 surements were performed over the last 2 years on the gas tightness, and 195 values well below 1 mL/h per detector were routinely achieved. Still, resid-196 ual leakage can contaminate the gas even in the presence of an overpressure, 197 as the gas concentration inside and outside the circuit homogenized at the 198 interface. This phenomenon was evaluated with detectors with different leak-199 age values, and no significant effect was observed on the humidity and oxygen 200 level. 201

The outgassing can be separated in two processes: desadsorption and permeation. The first one refers to a contamination from the inner surface of the circuit elements, while the second corresponds to a migration of molecules in the volume of the materials. They fundamentally differ on their duration, as the former is limited in time and can be suppressed by initial pumping, while the latter is by nature a continuous process. We decided to focus on specific measurements with the probe boxes and with the gas pipes to determine the true origin of this contamination.

#### 210 4.1. Measurements on the probe boxes

The short term measurements shown in Fig. 9 could point to a pollu-211 tion from desadsorption only. However, a couple of polyester boxes was then 212 prepared with different, long pumping/heating cycles, filled with Argon and 213 left for 3 months with an overpressure to study their long term evolution. 214 With sufficiently long pumping, it was possible to obtain a humidity below 215 the detectable level of the Yocto-Meteo, *i.e.*  $0.01 \text{ g/m}^3$ , during several days. 216 However, the humidity reappeared after that time, and later increased at a 217 nearly constant rate, as shown in Fig. 10. This continuous increase is per-218 fectly compatible with a permeation from the outside, especially since the two 219 boxes experienced exactly the same increase, though outgassed during differ-220 ent times. The period with undetectable humidity level can be understood 221 as a complete drying of the box, followed by a progressive rehumidification 222 by the permeation. 223

A similar measurement was performed on oxygen, with a dramatic increase in only a few weeks with the polyester box, as shown in Fig. 11. A stainless steel box in the same conditions yields an oxygen contamination 100 times smaller. For this reason, the polyester boxes were then replaced in the gas circuit by aluminum and stainless steel ones.

## 229 4.2. Effects of gas pipes

Another element which can contribute to the mixture degradation are 230 the gas pipes. In practice a large number of different pipes can be used in 231 test installations. In our building, more than 10 were identified, and several 232 are often combined in a given setup. Flexible pipes are mostly used, and 233 in this case polyure than based ones (labelled PU or PUN) are frequently 234 recommended in research centers for their fire resistance. Other flexible 235 pipes include polyamide (PA12), Teflon-like materials - polytetrafluoroethy-236 lene (PTFE), fluoropolymer (FEP) - or aluminum. 237

To investigate their influence, we monitored their humidity and oxygen during Argon flushing, before and after an outgassing procedure. We first col-



Figure 10: Long term humidity monitoring inside two polyester, probe boxes after intensive outgassing.



Figure 11: Oxygen monitoring inside a polyester and a stainless steel probe boxes after outgassing.

lected 9 different pipes in the neighbouring laboratories (5 PU, 2 PUN, 2
PA12, differing only by their colours), and bought FEP, PTFE and aluminum
pieces. Each of them were tested with a 1 m long sample, first flushed one

by one during 15 minutes in a 4 L/h Argon flux, *i.e.* equivalent to a 80 times 243 renewal of the pipe inner volume. The pipes were then closed with gastight 244 valves, and left for 50 days in the lab. After that time, they were again 245 flushed during 10 minutes at the same Argon flux, and the humidity and 246 oxygen levels at the exit of the pipes were monitored. The measured humid-247 ity levels are shown in Fig. 12 (left) for the 6 different types (pipes of the 248 same material showed nearly identical behaviour). PU and PUN yield the 249 highest humidity values, and still suffered from high humidity after the 10 250 minutes flushing. PA12 was found better than PU, but still at a high level. 251 PTFE, FEP and aluminum were found to be very similar, and much better 252 than PU and PA12. The same operation was repeated after outgassing: each 253 pipe was placed in a vacuum chamber, heated and periodically flushed with 254 Argon. After less than a week, the chamber vacuum stabilized, suggesting 255 that the outgassing was over. Pipes were than again filled with Argon, left 256 for 30 days in the lab, and the same measurements were done, see Fig. 12 257 (right). Considering the pipes were at rest during only 30 days instead of 50 258 for the first tests, these results convincingly prove that the gas degradation 259 did not change significantly after the outgassing.



Figure 12: Humidity monitoring during a 4 L/h Argon flushing in 1 m long pipes of different nature, before (left) and after (right) the outgassing procedure. In the left (resp. right) case, pipes were left at rest during 50 (resp. 30) days, which explains the relative humidity difference between the 2 plots.

260

Oxygen levels were monitored in parallel both before and after outgassing, as shown in Fig. 13, with quite different conclusions. In this case the aluminum pipe is by far the best one, whereas FEP and PTFE seem to have a large permeability to Oxygen, even higher than PU, PUN and PA12.



Figure 13: Oxygen monitoring during a 4 L/h Argon flushing in 1 m long pipes of different nature, before (left) and after (right) the outgassing procedure. In the left (resp. right) case, pipes were left at rest during 50 (resp. 30) days.

265

All the previous measurements suggest that permeation effects are already 266 significant after only a few days in the pipes, a particularly rapid time scale 267 compared to typical operations of the instruments. As a final test to show 268 the rapidity of the migration process, we placed one of the PU pipes in a 269 sealed chamber, and connected its extremities to 2 gas holes in the walls 270 of the chamber. The inner and outer volumes of the pipe were thus sepa-271 rated, and the pipe was continuously flushed with a  $0.5 \,\mathrm{L/h}$  Argon flux. The 272 humidity level was monitored at its entrance and exit, and the difference 273 is shown in Fig. 14. Digital flowmeters were also plugged at the entrance 274 and exit, to check the absence of any direct leakage and to set an overpres-275 sure of 15 mbar in the pipe. During the first 3 days, the chamber was open, 276 and thus filled with air. Once the pipe humidity stabilized, the chamber 277 was sealed and flushed with a  $10 \,\mathrm{L/h}$  Argon flux. After only 3 hours, the 278 pipe humidity started to decrease dramatically, proving that the humidity 279 migration within the pipe is primarily determined by the water vapor par-280 tial pressure difference. After 2 days, the humidity difference stabilized very 281 close to zero, and the Argon atmosphere within the chamber was replaced 282 by a vacuum around 0.1 mbar. As expected, the humidity difference did not 283 change. After an additional day the chamber was re-filled with air at the end 284 of the measurement, and as can be seen the humidity difference re-increased 285 within 3 hours. The humidity difference in air provides a measurement of 286 the water contamination in the gas in the laboratory conditions  $(28^{\circ}C \text{ and})$ 287

a humidity around 10 mg/L) with this PU pipe, *i.e.*  $1150 \mu \text{g/h}$  per meter of pipe. Similar measurements with 50 m long PTFE and aluminum pipes showed permeation rates of respectively 20 and  $0.62 \mu \text{g/h}$  per meter of pipe. In the same conditions, the oxygen permeation yields  $90 \mu \text{L/h}$  per meter of pipe for PTFE, and less than  $1.4 \mu \text{L/h}$  per meter of pipe for aluminum. Last

<sup>293</sup> but not least, the oxygen permeation timescale was found to be about 30 times faster than for humidity.



Figure 14: Evolution of the humidity difference between both extremities of a 0.9 m PU pipe placed in a sealed chamber. The pipe is flushed continuously with Argon at 0.5 L/h with a 15 mbar overpressure. The sealed chamber is first open and filled with air. After 2.8 days (left vertical line), the sealed chamber is closed and rapidly flushed with Argon. After 2 more days (middle vertical line) the chamber is pumped down to 0.1 mbar. Finally the chamber is opened at the end of the measurement and re-filled with air (right vertical line).

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## <sup>296</sup> 5. Conclusion

The degradation of the gas mixture in gaseous detectors have been studied in some details, showing its large impact on the detector gain. The effects of humidity and oxygen have been quantified and turned out to have a primary and similar importance in this degradation. If this can be mitigated by the use of specific absorbers and by a faster homogeneization of the gas,

other contaminants seem to participate to the gas degradation at very low 302 flushing. This work also revealed the importance of the gas pipe material, 303 generating large differences of the contamination level. This contamination 304 could never be stopped by a preliminary outgassing procedure, and the mea-305 surements unambiguously show that a continuous permeation process is at 306 work. Polyurethane-based pipes are found to be the worst choice in terms 307 of humidity, though often recommended for their fire resistance capabilities. 308 FEP and PTFE are good candidates to lower humidity, but exhibit a larger 309 oxygen permeability. Not surprisingly, the aluminum pipe appears as the 310 best choice, of course at a cost of a lower flexibility. More studies are now 311 planned to measure the permeation rate of different gases in several materials 312 using a mass spectrometer, and to investigate anti-permeation coatings. 313

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