# Recovery of sevoflurane anesthetic gas using an organosilica membrane in conjunction with a scavenging system

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## 7 Abstract

8 Approximately 95% of the anesthetic gas administered to a patient is exhaled and ultimately released 9 to the atmosphere. Most anaesthetic gases have high global warming potential and so this approach 10 adds significantly to the global greenhouse gas footprint. In this work we develop a feasible means to 11 capture such an anaesthetic gas (sevoflurane) before it is released to the hospital scavenging system, so 12 that it is retained within the anaesthetic circuit. Sevoflurane is retained using a microporous 1,2-13 bis(triethoxysilyl)ethane (BTESE) membrane prepared by a sol-gel method. The use of a ceramic 14 membrane facilities sanitisation at high temperatures. A rapid thermal processing (RTP) technique is 15 employed to reduce production time and to create a looser organosilica network, resulting in higher gas 16 permeances, compared with the membrane synthesized from conventional thermal processing (CTP). 17 The RTP membrane shows a slight decline in gas permeance when used with a dry mixture of 18 CO<sub>2</sub>/N<sub>2</sub>/sevoflurane. This permeance falls again under 20% relative humidity feed conditions but the 19 CO<sub>2</sub>/sevoflurane selectivity increases. The membrane performance shows little variation when the 20 relative humidity is further increased. These promising results demonstrate that this microporous 21 BTESE membrane has great potential for the recovery of sevoflurane in an anaesthetic application.

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#### 27 Introduction

Volatile anesthetic gases such as desflurane, isoflurane and sevoflurane are widely used in clinical anesthesia.<sup>1</sup> However, less than 5 vol% of the patient's total inhaled dose is metabolized.<sup>2</sup> The exhaled anaesthetic is captured within the hospital scavenging system and ultimately exhausted to the atmosphere. These volatile anesthetic gases are expensive and have high greenhouse warming potentials (GWPs).<sup>1, 2</sup> There are tens of millions of general anaesthetic procedures every year worldwide and so retaining and recycling these anesthetic gases has significant environmental benefits.

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In modern anesthetic systems, the air stream is recirculated in a 'circle' breathing circuit to reduce consumption of both oxygen and the anesthetic (Figure 1 (a)). To prevent the accumulation of CO<sub>2</sub>, soda lime absorbs CO<sub>2</sub>. Alternative methods to remove expired CO<sub>2</sub> has attracted much research, including using new absorbents <sup>3</sup> or membrane separation. <sup>4-6</sup> As one example, 13X molecular sieves have been shown to be effective CO<sub>2</sub> absorbers, without the formation of harmful by-products. <sup>3</sup> Similarly, Wilfart et al. <sup>5</sup> recently studied a membrane-based device to remove CO<sub>2</sub> from anesthesia circuits using a sweep gas.

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The use of a  $CO_2$  removal device does not eliminate the need for a purge stream from the circuit to maintain pressure control, as fresh gas flow is required for  $O_2$  and volatile gas delivery. The hospital vacuum scavenging system is used for this purpose (Figure 1(a)). As 95% of the anesthetic gas is not metabolized, most is vented to the scavenging system, and thereafter the atmosphere. <sup>1</sup>

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50 Various absorbents such as activated carbon, <sup>7</sup> metal-organic-frameworks, <sup>8-10</sup> and zeolites <sup>11</sup> 51 have been studied to explore their potential to capture anesthetic gases. The use of such 52 adsorbents prevents atmospheric pollution by anaesthetic gases and thus adding to global 53 warming. Anaesthetic gas adsorption is also useful in situations where a scavenging system is 54 unavailable to remove the gases from the operating room (affecting operating theatre staff), such 55 as in veterinary clinics or less economically developed/remote regions. However, unless the 56 adsorbed anesthetic can be recovered thereafter, it does not reduce the consumption of these 57 gases and hence their cost. Recovery from the adsorbent can be energy intensive and there can 58 be regulatory concerns due to potential biological contamination.





Figure 1: Schematic of (a) a standard anesthetic breathing circuit, and (b) our proposed approach
where a membrane is used to retain anesthetics within the circuit.

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An alternative approach is to use a size selective membrane that can retain anesthetic gases in the breathing circuit, while allowing other gases (CO<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub>O) to enter the scavenging system (Figure 1(b)). The approach has a number of advantages. Firstly, the membrane can be run continuously while the adsorbents must be replaced or regenerated. Secondly, the gases can be *reused* directly by the patient during the operation, reducing the regulatory concerns associated with *recycling*. Further, the approach utilizes the vacuum provided in the scavenging system to provide a pressure driving force for separation. This avoids the need for a sweep gasused in other membrane approaches.

80 For the approach to be effective, a molecular sieving membrane is needed, as this can retain the 81 larger anesthetic gas molecules such as sevoflurane (molecular weight, MW 200 g/mol) while 82 allowing passage of smaller molecules such as nitrogen (MW 28), oxygen (MW 32) and CO<sub>2</sub> (MW 44). A solubility selective membrane will be less effective, as these gases are readily 83 84 condensable (sevoflurane has a critical temperature of 429 K) and so would be readily permeated 85 with other condensable gases such as  $CO_2$  (critical temperature 304.2K). An inorganic 86 membrane can provide the necessary molecular sieving capability and also has the advantage 87 that it is resistant to high temperatures and so can be periodically thermally disinfected with hot 88 water or steam, as commonly used for decontaminating other elements of the anesthetic machine. 89 It has already been reported that a DD3R zeolite membrane can effectively retain xenon (an alternate anaesthetic gas) in a CO<sub>2</sub>/xenon mixture. <sup>12</sup> The CO<sub>2</sub> permeance was 60 GPU and the 90 91 CO<sub>2</sub>/xenon selectivity was 67 for a wet CO<sub>2</sub>/xenon mixture.

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93 In this work, microporous organosilica membranes derived from 1,2-bis(triethoxysilyl)ethane 94 (BTESE) were synthesized by a sol-gel method followed by either a conventional thermal 95 processing (CTP), or a rapid thermal processing (RTP) step to explore BTESE's potential for 96 sevoflurane recovery. Although there are many reports on the gas separation performance of 97 microporous BTESE membranes, most focus on high temperature H<sub>2</sub>/CO<sub>2</sub> separation. <sup>13-15</sup> Moreover, a few reports have shown that BTESE membranes also have promising performance 98 for CO<sub>2</sub>/N<sub>2</sub> separation <sup>16</sup> and CO<sub>2</sub>/CH<sub>4</sub> separation. <sup>17</sup> Due to the presence of Si-C-C-Si groups, 99 100 BTESE membranes have a slightly larger pore size than conventional microporous silica membranes, <sup>13</sup> which may be beneficial to the transport of the light gases in the expired stream. 101 It is known that the effective pore size of silica membranes can be readily tailored by sol-gel 102 parameters, <sup>15, 18, 19</sup> the number of coatings and calcination parameters. <sup>20, 21</sup> In general, for a 103 certain sol solution, several coatings and a slow calcination process (i.e. ramping rate of 0.5-104 105 2 °C min<sup>-1</sup>) are needed to eliminate possible defects, which have a significant impact on the

final membrane performance. Recently, preparing inorganic membranes with an RTP treatment
 (e.g. ramping rate >100 °C min<sup>-1</sup>) has been shown to also give promising separation
 performance, <sup>22-25</sup> further reducing production time.

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Single gas permeation tests (CO<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub> and sevoflurane) were initially carried out for both CTP and RTP membranes at 30 °C. The RTP membrane, which showed better single gas separation performance was further assessed at 30 °C using a  $CO_2/N_2$ /sevoflurane mixture (9.4 vol%/84.6 vol%/6 vol%). Finally, the influence of water vapor on the mixed gas separation performance of the membrane was also investigated.

#### 115 Materials and Methods

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1,2-bis(triethoxysilyl)ethane (BTESE, 96%) was purchased from Sigma-Aldrich and was used 117 without further purification. The BTESE was added dropwise into a mixture of ethanol, water 118 119 and HCl under vigorous stirring for 30 mins at room temperature. The molar composition was BTESE :  $H_2O$  : HCl :  $C_2H_5OH$  was 1: 60 : 0.1 : 123. The resultant solution was kept at 4°C until 120 use. Tubular inorganic nanofiltration membranes composed of an a-alumina support and 121 TiO<sub>2</sub>/ZrO<sub>2</sub> top-layer (supplied by Atech Innovations, GMBH, Molecular cut-off: 5 kDa, ID: 122 6mm, length: 100 mm) were used as a membrane support to reduce BTESE membrane thickness 123 124 and to provide mechanical strength.

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The BTESE solution was coated on the inner side of the membrane support by a dip-coating method using a syringe pump with a withdrawal speed of 10 cm min<sup>-1</sup>. <sup>26</sup> For the CTP treatment, the membrane was dried at 200 °C for 30 mins with a heating/cooling rate of 1°C min<sup>-1</sup>. For the RTP treatment, an oven was preheated to 200 °C <sup>27</sup>. The membrane was put into the oven and kept for 30 mins, then it was removed using tongs directly, leading to a heating/cooling rate of >100 °C min<sup>-1</sup>. The coating/thermal treatment process was repeated three times to eliminate possible defects on the membrane layer. Both membranes were then kept in a desiccator for further membrane performance testing. Hereafter, they were named as the CTP and the RTP
membrane. For comparison, a commercial polydimethylsiloxane PDMS thin film composite
membrane (Sulzer Pervap 4060) was also evaluated.

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Initially, all membranes (*i.e.* PDMS, RTP and CTP membrane) were tested using a a constant volume and variable pressure apparatus as described elsewhere. <sup>28</sup> CO<sub>2</sub>, O<sub>2</sub> and N<sub>2</sub> permeances were tested at 100 kPa feed pressure (absolute) and 30 °C. Sevoflurane permeance was tested at 20.6 kPa (absolute) by evaporating sevoflurane liquid at 20 °C into a feed chamber that had previously been evacuated. The permeate side was under vacuum conditions before measurement and the permeance was calculated based on the pressure change on the permeate side.

145 The RTP membrane was further tested to evaluate its mixed gas separation performance and the influence of water vapor. Figure 2 shows the schematic diagram of the mixed gas permeation 146 apparatus. The membrane module was kept in an oven at 30 °C. For the dry gas mixture, a 10 % 147  $CO_2/N_2$  mixture was bubbled through a sevoflurane vessel with controlled temperature (-2 °C) 148 to reach a final feed composition of 9.4 %/84.6 %/6 % (CO<sub>2</sub>/N<sub>2</sub>/sevoflurane). CO<sub>2</sub>/N<sub>2</sub> was used 149 instead of CO<sub>2</sub>/O<sub>2</sub> as N<sub>2</sub> has a larger kinetic diameter (3.64 Å) than O<sub>2</sub> (3.46 Å). The sevoflurane 150 partial pressure was estimated from the supplier's product information<sup>29</sup> and the work by 151 Schenning et al. <sup>30</sup> and also confirmed through simulation in Aspen Plus<sup>(c)</sup>. For the wet gas 152 mixture, the above dry feed gas mixture was further bubbled through a water vapor generator to 153 introduce a certain amount of water vapor. The water vapor relative humidity (RH) on the feed 154 side was controlled by adjusting the temperature of the water vapor generator. The membrane 155 was tested at 20.1 %, 40.8 % and 55.4 % RH. The feed pressure was controlled at 120 kPa 156 absolute pressure and the feed flow rate was set to 3 L min<sup>-1</sup> to reduce the effect of concentration 157 polarization. For the dry gas mixture experiments, the permeate pressure was controlled at 20 158 159 kPa using a back pressure valve. For the wet gas mixture tests, Helium was used as a sweep gas 160 as the permeate flow rate was too low to maintain this permeate pressure. It is worth noting that such a sweep gas is not necessary for the full scale application as the membrane module wouldhave a much larger surface area.

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The water vapor relative humidity on the permeate side was measured by a humidity sensor (Vaisala HMT330 Series). An iced water cold trap was then used to condense this water vapor on the permeate side, with a downstream vacuum pump maintaining the permeate pressure at 20 kPa. Sevoflurane could not condense in this trap, as the partial pressure required in the permeate for condensation to occur (8kPa at 0°C) would be greater than the feed partial pressure of 7.2kPa generated at -2 °C

170 The sevoflurane composition on the permeate side was analyzed using a Perkin Elmer FTIR (Fourier Transform Infrared Spectrometer) equipped with a CycloneTM gas cell accessory with 171 a path length of 2 m.<sup>31</sup> Each spectrum was recorded in the range of 1000-4000 cm<sup>-1</sup> with a 172 resolution of 2 cm<sup>-1</sup> and an average of 8 scans was used. A background scan was conducted 173 under vacuum conditions before the measurement. An example of the sevoflurane spectrum is 174 shown in the Supporting Information (Figure S1). The FTIR was calibrated against a 175 sevoflurane/N<sub>2</sub> mixture reference (0.07 vol% at 1 bar) in a range of 0.5 to 0.2 bar. At least three 176 177 measurements were carried out at each pressure. The calibration curve was based on the peak area between 1450 and 1000 cm<sup>-1</sup> and is shown in Supporting Information, Figure S2. Results 178 from the FTIR analysis confirmed that sevoflurane had not condensed in the cold trap, as the 179 measured permeate partial pressure was more than two orders of magnitude below the saturation 180 partial pressure of 8 kPa. 181

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The permeate flow rate was measured by a mass flow meter (Aalborg) with the calibration confirmed using a bubble flowmeter. The CO<sub>2</sub> and N<sub>2</sub> composition on the permeate side was analyzed by an Agilent 480 Micro gas chromatography (Micro GC) equipped with a Micro-machined Thermal Conductivity Detector (μTCD) and a PoraPLOT U (PPU) column.

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- Each data point was recorded 0.5-1 hour after changing the experimental conditions to ensure
  equilibrium had been achieved. Each single gas and mixed gas experiment was repeated five
  times to allow error margins to be established.
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Figure 2: Schematic diagram of the mixed gas permeation apparatus. MFC= mass flow controller;
SVG= sevoflurane vapor generator; WVG = water vapor generator; FTIR = Fourier Transform
Infrared Spectrometer and MFI = mass flow indicator.

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#### 198 Results and discussion

Figure 3 shows the single gas permeances of  $CO_2$ ,  $O_2$ ,  $N_2$  and sevoflurane of both the CTP and the RTP membrane at 30 °C. Both membranes show typical molecular sieving behaviour, with the gas permeances in the order of penetrant kinetic diameter, indicating the membranes were defect-free. It is also clear that the RTP membrane has higher permeance for all tested gases than the CTP membrane. The  $CO_2$  permeances were 700 and 4200 GPU for the CTP and the RTP membrane, while they were 500 and 850 GPU for N<sub>2</sub>. The permeances of sevoflurane were 205 2.0 and 11 GPU, respectively. On the other hand, the  $CO_2$ ,  $N_2$  and sevoflurane permeances of 206 the PDMS membrane were 480, 65 and 550 GPU, respectively. The high permeance of 207 sevoflurane may be attributed to its high critical temperature and confirms that a solubility 208 selective material such as PDMS is not suitable for this separation.

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Figure 3: Single gas permeance of the CTP and RTP membrane as a function of kinetic diameter
(CO<sub>2</sub>:3.3 Å; O<sub>2</sub>: 3.46 Å; N<sub>2</sub>: 3.64 Å; critical diameter of 7.1 Å was used for sevoflurane <sup>11</sup>). Note: the
maximum experimental error is within 10% of each data point shown.

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215 Importantly, the  $CO_2$ /sevoflurane selectivity was 350 for the CTP membrane and 370 for the RTP membrane, indicating both membranes have similar molecular cut-off and can retain the 216 anesthetic gas. The  $CO_2/N_2$  selectivity was quite low for both membranes (*i.e.* 1.4 and 4.9 for 217 218 the CTP and the RTP membrane) compared with typical BTESE membrane performance reported in the literature. <sup>16, 17</sup> For instant, Ren et al. <sup>16</sup> has reported a CO<sub>2</sub> permeance of 2300 219 GPU with CO<sub>2</sub>/N<sub>2</sub> selectivity of 36 at 40 °C under 1 bar transmembrane pressure for a BTESE 220 membrane calcined at 300 °C. Yang et al. 17 demonstrated a BTESE membrane with CO2 221 permeance of 3900 GPU with CO<sub>2</sub>/N<sub>2</sub> selectivity of 24 at 20 °C by employing a two-step hot 222

223 method. The low  $CO_2/N_2$  selectivity may be attributed to the relatively low calcination coating temperature (i.e. 200 °C), leading to a lower degree of densification/condensation. <sup>20</sup> Further, 224 225 only three coatings were applied in our work, resulting in a broad pore size distribution. It is 226 beneficial to have low  $CO_2/N_2$  selectivity in the present case as we aim to only retain sevoflurane 227 in the feed side, with minimal transport resistance to other gases, in particular N<sub>2</sub>. A larger 228 membrane area is needed if  $N_2$  permeance is low, and this would be a limiting factor for the 229 membrane design. This means that the percolative pathways that control the gas selectivity 230 should be in the size range of N<sub>2</sub> and sevoflurane rather than in that of CO<sub>2</sub> and N<sub>2</sub>. It is worth 231 noting that the thermal processing time was much shorter for the RTP membrane than the CTP membrane (30 mins versus 340 mins for each coating), making the fabrication process more 232 233 productive. Hence, the RTP membrane was chosen for further investigation.



Figure 4: (a) gas permeances of the RTP membrane under mixed-gas conditions and (b) CO<sub>2</sub>/N<sub>2</sub> and
CO<sub>2</sub>/sevoflurane selectivity as a function of water humidity. Note: the maximum experimental error is
within 10% of each data point shown in Fig. 4(a).

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As the operation of an anesthetic machine is under humid conditions, it is important to understand the mixed-gas separation performance and the influence of water vapor on the membrane performance. Figure 4(a) shows gas permeances of the membrane under dry and wet mixed gas conditions. For the dry mixture, the CO<sub>2</sub>, N<sub>2</sub> and sevoflurane permeances were 3650, 810 and 9 GPU, respectively. All values were slightly lower than those of the single gas permeance data shown in Fig. 3, which might be due to competitive sorption or hindereddiffusion.

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In a humid gas stream, the water vapor has higher permeance than CO<sub>2</sub> as it has a smaller kinetic
diameter and thus higher diffusivity and probably also higher solubility in silica (Figure 3). The
permeance of all three other gases were significantly reduced by the presence of water vapor.
When the feed humidity was 20.1%, the H<sub>2</sub>O, CO<sub>2</sub>, N<sub>2</sub> and sevoflurane permeances were 5000,
950, 57 and 1.5 GPU, respectively.

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253 The permeances remained almost constant when the relative humidity was further increased to 254 40.8% and 55.3%. This means that the N<sub>2</sub> permeance was reduced to 7% of the initial value under dry conditions, while the sevoflurane permeance was reduced to 17%. The decrease of 255 256 gas permeance is understandable as the membrane material is hydrophilic and porous. Water vapor can adsorb on the silica surface via hydrogen bonding with silanol groups. <sup>16, 32, 33</sup> 257 Moreover, water vapor can form clusters thus likely blocking the transport pathways of other 258 gases, which has been reported in both inorganic and polymeric membranes.<sup>34-36</sup> For example, 259 Ren et al. <sup>16</sup> reported that CO<sub>2</sub> permeance of BTESE membrane was reduced by 99 % at 83 % 260 RH at 40 °C. It is not surprising that the reduction is more prominent than in our case (74 % 261 reduction) as the membrane has a smaller pore size evidenced by its higher  $CO_2/N_2$  selectivity. 262 Moreover, their membrane was calcined at 300 °C, hence fewer organic groups (more 263 hydrophilic groups) can be expected on the membrane surface. Interestingly, the more stable 264 CO<sub>2</sub> permeance in this work was similar to the trend for a BTESO (bis(triethoxysilyl)octane) 265 membrane<sup>16</sup>, which is expected to have a larger pore size and be less hydrophobic. These results 266 267 clearly demonstrate that water vapor has a significant impact on the separation performance of 268 the membrane, which should be taken into consideration during process design.

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The influence of water vapor on  $CO_2/N_2$  and  $CO_2$ /sevoflurane selectivity is shown in Figure 4(b). The  $CO_2/N_2$  selectivity was increased from 4.5 under dry mixed-gas condition to about 17 under

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humid conditions (*i.e.* 20.1%, 40.8% and 55.3% RH), while the  $CO_2$ /sevoflurane selectivity increased from 410 to above 600. The increase is mainly because water has less impact on the CO<sub>2</sub> permeance than N<sub>2</sub> and sevoflurane.

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276 As an example, if the gas in the breathing circuit was at 101 kPa and contained 4 vol% water 277 vapor, 2 vol%  $CO_2$  and 2 vol% of sevoflurane, the permeate pressure was 10 kPa and there was 278 complete mixing on both sides of the membrane, these wet gas permeance values would lead to 279 a permeate composition of approximately 26 vol% water vapor, 11 vol% CO<sub>2</sub> and 0.04 vol% 280 sevoflurane (assuming the O<sub>2</sub> permeance was identical to that of N<sub>2</sub>). The high concentrations 281 of CO<sub>2</sub> in the permeate can reduce the load on the CO<sub>2</sub> absorber in the circuit, allowing this to 282 be replaced less often, or for a smaller unit to be used. In itself this is of environmental benefit, 283 as the loaded absorber and the soda lime/calcium carbonate it contains is currently sent to 284 landfill. Similarly, the strong permeation of water vapor removes the excess produced by patient breathing to be removed. Conversely, the anesthetic is retained, reducing the volume that needs 285 286 to be added and thus reducing both cost and greenhouse gas emissions. Indeed, it was estimated that the emissions of volatile anaesthetic agents worldwide are equivalent to the CO<sub>2</sub> emissions 287 288 from 1 million cars in 2010<sup>37</sup>. Our results clearly demonstrate that membrane technology can minimize these emissions with minor costs. The use of a ceramic membrane is ideal within the 289 hospital environment as it can be readily sanitized or sterilized. Nevertheless, the membrane 290 needs to be tested in a real anesthetic circuit to evaluate its long term performance and its impact 291 on the operation of the anesthetic machine. This will require careful adjustment of the pressure 292 293 relief valves and pressure control valves within the anesthetic machine to ensure patient safety.

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### **300** Supporting Information

301 IR spectrum of sevoflurane (Figure S1); IR peak area of sevoflurane at 1450-1000 cm<sup>-1</sup> as a function of

302 gas cell pressure (Figure S2).

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