



## Determination of the filter capture efficiency for safe use of fixative solutions in the biological glove box of the International Space Station



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### HIGHLIGHTS

- In the International Space Station occasionally hazardous materials are used.
- An experimental setup was constructed to study a simulated spill scenario.
- Hazardous materials should be used in a glove box equipped with special filters.
- Aqueous solutions of formaldehyde, glutaraldehyde and ethanol can be used safely.
- Small spills are captured on a carbon filter but large spills lead to breakthrough.

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### ABSTRACT

For fixation of biological specimen in the International Space Station (ISS) aqueous solutions of formaldehyde, glutaraldehyde and ethanol are used in a biological glove box (BGB) containment. The air circulated through the BGB unit is filtered over a bank of filters consisting of a packed bed of activated bituminous coal-based granular carbon impregnated with sodium bromate. A procedure was developed to determine the performance of these filters in the case an unexpected spill would occur. In a simulated spill scenario under earth gravity conditions finely dispersed aerosols of the aforementioned aqueous solutions were drawn through the filter unit at a flow of 75 L/min. To determine the filter capture efficiency, glass fiber membrane filters were impregnated with 2,4-dinitrophenylhydrazine (DNPH) to capture formaldehyde and glutaraldehyde by chemical binding. The filters were analyzed for the DNPH-aldehyde complex by HPLC-UV. Ethanol was collected by adsorption on activated coal and subsequently analyzed by gas chromatography flame ionization detection (GC-FID). A challenge with 10.0 mL of test solution resulted in downstream air concentrations of  $0.295 \pm 0.094$  mg/m<sup>3</sup> for a solution of 3% of formaldehyde in water and  $0.018 \pm 0.015$  mg/m<sup>3</sup> for a solution containing 3.5% of glutaraldehyde in water. For a filter load of 5.0 mL of 70% ethanol in water, the downstream concentration was  $19.4 \pm 7.8$  mg/m<sup>3</sup>. This corresponds to filter capture efficiencies of 99.5% for formaldehyde, 99.9% for glutaraldehyde, and 97.5% for ethanol. The measured concentrations downstream of the filter unit all remained below the spacecraft maximum allowable concentrations (SMAC) for these substances. Breakthrough tests were performed at tenfold the volume of the simulated spill scenario. Complete breakthrough occurred at  $35 \pm 14$  min for formaldehyde and at  $15 \pm 2$  min for ethanol. For glutaraldehyde breakthrough was not observed within 75 min.

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**Abbreviations:** BGB, biological glove box; CRM, certified reference material; DNPH, 2,4-dinitrophenylhydrazine; GAC, granular activated carbon; GC-FID, gas chromatograph equipped with a flame ionization detector; HOMO, highest occupied molecular orbital; HPLC-UV, high performance liquid chromatograph equipped with UV detector; ISS, International Space Station; LOD, limit of detection; LOQ, limit of quantification; SMAC, spacecraft maximum allowable concentration.

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## 1. Introduction

The use of hazardous materials in confined spaces represents a major risk in workplaces. A particular critical situation may be the potential consequences of exposure to toxic agents of crew members working in the confinement of an aircraft or spacecraft interior (Todd et al., 1994; Belford and Rayman, 1996; Rayman, 2002). The release and build-up of toxic substances may lead to reduced alertness, reduced performance (Patterson and Rayman, 1996) on long-duration manned space flights, also long-term adverse health effects may occur. Guidelines for chemical air quality are required. In addition, chemical spills are potential sources of inhalation risk (Todd et al., 1994). During the Space Shuttle program the crew collected air samples if they smelled an odor related to a detected event or an undetected source of contamination (James et al., 1994). The frequency of a minor air contamination incident was estimated to be once per month and over 30 missions 10 minor incidents occurred (James et al., 1994). In this program one incident was classified as of 'moderate toxicological concern'. This incident involved overheating of the motor of a refrigerator/freezer. Post-flight analysis of contaminated air that was collected by the crew using evacuated canister revealed that the noxious odor detected by the crew was predominantly caused by the release of formaldehyde and ammonia. The concern in this incident was related to the high toxicity of formaldehyde and later in the Space Shuttle program efforts were made to reduce the concentration of this gas to levels ranging from 0.04 to 0.08 mg/m<sup>3</sup> (James et al., 1994). This incident clearly illustrates the necessity to carefully prepare the planned use of formaldehyde and other hazardous substances.

In spacecraft systems are in place to continuously monitor and control trace contaminants. Air contaminant hardware such as sensors and the modeling software was developed for an early detection of problems with air quality in a spacecraft (Narayan and Ramirez, 1998). Such real-time monitoring systems, including gas chromatography mass spectrometry-based detectors, are also operational in the current International Space Station (ISS) program (Jones and Kliss, 2010). Most chemical contaminants in the ISS modules are originating from off gassing of equipment and materials and also from leaks but studies indicate that the ISS air quality complies with the adopted standards (Pakhomova et al., 2006).

In April 2014 a research project was carried out in the Biolab facility of the Columbus module in ISS to study the influence of microgravity on the growth direction of plant roots under different amounts of acceleration force. Lentil seedling roots were used as a model to study the mechanisms by which the plant responds to a change in the direction of the gravity field. Such knowledge is required for future long-range space missions where plants can become part of a life support system, based on the principle of bio-regeneration of resources. This research project, known as the 'Threshold Acceleration for Gravisensing-2' or 'Gravi-2' experiment (see [http://www.nasa.gov/mission\\_pages/station/research/experiments/2.html](http://www.nasa.gov/mission_pages/station/research/experiments/2.html) for details), required improvements of the biological glove box (BGB) unit to allow the safe use of chemicals for fixation of the tested plant roots. The biological specimen were transported back to earth for further research on May 18th, 2014 (European Space Agency, 2014). The solutions needed for chemical fixation of these samples contain formaldehyde (3.0 w%), glutaraldehyde (3.5 w%) or ethanol (70% by volume). These chemical substances represent a potential health risk for the astronauts if these substances would escape from the BGB containment. Table 1 provides an overview of the known health effects following short-term exposure to relative high concentrations and the potential health risk of long-term exposure at a much lower concentration. As shown in Fig. 1 this unit offers a work volume of 35 liters and contains illumination and video equipment. It has removable doors and a large top face viewing window and two glove ports in the front. It provides two levels of containment: the closed structure and also under-pressure (see [http://bradford-space.com/productline/glovebox\\_family/biological\\_glovebox](http://bradford-space.com/productline/glovebox_family/biological_glovebox) for more information). As shown in Fig. 2 the BGB-unit has a system for treatment and reuse of air, involving a bank of eight filter cartridges that contain different absorption materials for filtration of particles and gas-phase components (see Fig. S1 for technical details). The BGB contains a two levels of containment enclosed environment (work volume) which operates at a negative pressure of 500 Pa relative to ISS cabin pressure (which is around 1013 hPa). The internal airflow is 300 L/min resulting in a flow of 75 L/min per filter. As a result of the negative pressure all entrapped air will only pass through the filters when leaving the work volume, see Fig. 2.

Carbon is much used to control air concentrations of VOC in confined spaces but there is limited information on the performance of such carbon filters in real life use (Yao et al., 2009). The early papers on the use of granular activated coal (GAC) were published several decades ago, mostly in the field of industrial hygiene (Nelson and Harder, 1974, 1976; Nelson et al., 1976). Because of the difficulty of predicting adsorption of polar and reactive organic vapors such as alcohols and aldehydes to GAC (Wu et al., 2003; Abiko et al., 2010b) we designed a bench-top experimental set up for laboratory testing of the filter capture efficiency using a specific spill scenario relevant to the type of use of aqueous solutions of fixatives in the BGB.

The first objective of this study was to determine the capture efficiency of the carbon cartridge in the BGB filter unit when challenged with aqueous solutions of formaldehyde, glutaraldehyde or ethanol dissolved in pure water. In the downstream air the concentration of these substances should not exceed the respective 24-hour time-weighted average spacecraft maximum allowable concentration (SMAC) values for these substances (National Research Council, 2008). A second objective was to determine the time to breakthrough for formaldehyde, glutaraldehyde or ethanol.

This study describes a laboratory approach to quantify GAC filter capture efficiencies and breakthrough times involving polar chemical substances in a high humidity chemical spill setting. Currently available models do not support predicted with sufficient precision to allow decision-making regarding the health and safety of humans.

## 2. Materials and methods

### 2.1. Materials

The carbon originally used in the BGB filter cartridges consists of granular bituminous coal-based activated carbon (type Formasorb, 12 × 40 mesh, mean particle diameter 1 mm), purchased from Chemviron Carbon (Ashton-in-Makerfield, United Kingdom). To improve the adsorption properties for aldehydes this particular coal received an additional treatment with sodium bromate performed by Chemviron Carbon. For the determination of aldehydes 2,4-dinitrophenylhydrazine (DNPH) reagent, phosphoric acid (85%) and polymerized formaldehyde powder (paraformaldehyde, purity 99.8%, obtained from Merck Chemical, Amsterdam Zuid-Oost, The Netherlands), formaldehyde-2,4-DNPH (purity 99.9%) and glutaraldehyde-2,4-DNPH (purity 99%) were obtained from Sigma-Aldrich Chemie (Zwijndrecht, The Netherlands). A certified reference material (CRM) for the determination of formaldehyde-2,4-DNPH from glass fiber membrane filters

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