Materials and Methods

Instrumentation

SIFT-MS was done on a Voice 200 Ultra from Syft Technologies (Christchurch, New Zealand) fitted with a HPI hex inlet (20 mL/min). Calibration of the SIFT-MS to vapor phase acetone and MTBE was done by using gas tight syringes to measure 5 µL of neat acetone or MTBE, which was injected into Tedlar bags containing known volumes of air to make stock solutions of vapor. Aliquots of the vapor phase stock were then diluted into secondary Tedlar bags of air to achieve the desired concentrations of acetone and MTBE. The reagent-ion reaction rates and the product-ion ratios in the Syft analysis library were adjusted to align the calculated instrument response to the known concentrations of acetone and MTBE.

Experimental Setup

A Secador® Techni-dome® 360 Large Vacuum Desiccator from Bel-Art Products (Pequannock, NJ) was customized by adding a 30 cm tall cylinder the same diameter as the desiccator between the desiccator halves. Glove ports (20 cm dia.) in the cylinder enabled the desiccator to be used as a glove chamber with a volume of 131 liters. A fan was used to circulate air within the chamber. The SIFT-MS was connected to the chamber via a 4 inch (1/8 inch dia.) length of perfluoroalkoxy (PFA) tube.

Materials

The test solution, referred to as PGAB solution, contained 2 M propylene glycol (Sigma-Aldrich, St. Louis, MO), 0.33 M acetone (Fisher Co., Fair Lawn, NJ), 0.33 M methyl t-butyl ether (MTBE) (Baxter Health Care, Muskegon, MI) and 1.3 mM sodium fluorescein (Sigma-Aldrich) in deionized water.

Calibration of vapor phase acetone and methyl t-butyl ether

Gastight syringes were from the Hamilton Company (Reno, NV). A Cole-Parmer (Vernon Hills, IL) syringe pump was used to deliver the liquid and headspace aliquots. The SIFT-MS response to releases of liquid and headspace aliquots of PGAB solution was done inside the chamber to create calibration curves. Calibration of SIFT-MS response to volumes of liquid leaks was done by releasing liquid aliquots of PGAB solution delivered via gas-tight syringes and a syringe pump into the chamber where the resulting vapor phase concentrations were measured by SIFT-MS.

Calibration of headspace leaks was done by taking aliquots of headspace, from a 0.5 liter Tedlar bag containing 250 mL of PGAB solution and 250 mL of headspace, into a gas-tight syringe fitted with a closure valve. A 50-mL gastight syringe was used for the upper range of headspace volumes and a 1-mL syringe was used for the range of lower volumes. All experiments were equilibrated at room temperature of 21.5$\pm$0.5 °C.

Data Sets.

Data Set #1.

Figure 1 from Paper. Typical instrumental acetone and MTBE response to aliquots of PGAB solution. Aliquots of PGAB solution were released inside the glove Schamber described above. A gas-tight syringe (Hamilton Co.) and a syringe pump (Cole-Parmer) were used to measure and deliver the solution inside the glove chamber. Volumes of 0.5, 1, 10 and 20 µL of PGAB solution were released and the instrumental response was recorded. The data is presented as ppbV versus time. From this data set we determined mean response as a change in acetone or MTBE response from the instrument versus the aliquot volume. The standard error of the mean measurement was also determined as $s/\sqrt{n}$ where s is the standard deviation and n is the number of measurement points recorded.

Data Set #2.

Figure 2. From paper. Mean response and standard error from the release of aliquots of PGAB solution when released inside the glove chamber. The initial concentration of analyte before the release of an aliquot of PGAB solution minus the final concentration after the release of an aliquot was labeled as the change in acetone (ΔA) or MTBE (ΔB) concentration.

Data Set #3.

Figure 3 from paper. Mean instrumental response as change in MTBE from six replicates of the calibration curve for MTBE from liquid aliquots of PGAB solution.

Data Set #4.

Mean instrumental response as change in acetone from six replicates of the calibration curve for acetone from liquid aliquots of PGAB solution.

Data Set #5.

Mean instrumental response as change in MTBE from six replicates of the calibration curve for MTBE from headspace aliquots of PGAB solution. Headspace was taken from equilibrated headspace above PGAB solution. The volume of the liquid occupied half of the volume of the bag from which the headspace was taken.

Data Set #6.

Mean instrumental response as change in acetone from six replicates of the calibration curve for acetone from headspace aliquots of PGAB solution. Headspace was taken from equilibrated headspace above PGAB solution. The volume of the liquid occupied half of the volume of the bag from which the headspace was taken.

Data Set #7. Linear fit of calibration data from experimentally generated calibration curve data. Linear fit of high and low volume range data from releases of headspace and liquid aliquots from PGAB solution.

Data Set #8. Instrumental response of acetone and MTBE measurements versus time when using an air-cleaning type CSTD to transfer 45 mL of PGAB solution from vial one to vial two.

Data Set #9. Instrumental response of acetone and MTBE measurements versus time when using an barrier type CSTD to transfer 45 mL of PGAB solution from vial one to vial two.

Data Set #10. Instrumental response of acetone and MTBE measurements versus time when using an bladder-barrier type CSTD to transfer 45 mL of PGAB solution from vial one to vial two.

Data Set #11. Linear fit of calibration data from experimentally generated calibration curve data produced when testing CSTDs as in data sets 7,8 and 9. Linear fit of change in instrumental response to acetone and MTBE to volumes of headspace or liquid aliquots.