Feasibility of a selective epoxidation technique for use in quantification of peracetic acid in air samples collected on sorbent tubes

Dataset Number:

Introductory Information

The occupational exposure risk to peracetic acid (PAA), a common disinfectant and sterilant use in industrial settings like healthcare facilities and meat processing plants, is typically assessed through collection and analysis of atmospheric workplace samples. In this study, a new personal sampling technique was developed for determining the atmospheric concentration of PAA in the workplace. Samples of humidified air containing peracetic acid (concentration range: 25-2000 ppb) were collected on 350-mg XAD-7 solid sorbent tubes to adsorb the analyte. Peracetic acid was desorbed using anhydrous acetonitrile, after which it was treated with excess cyclohexene to generate the corresponding epoxide via the Prilezhaev reaction. The epoxide product was analyzed and quantified using gas chromatography-mass spectrometry, indirectly determining the amount of peracetic acid in the original air sample. The specificity for peracetic acid over other contaminants was assessed through a series of hydrogen peroxide (HP) and acetic acid (AA) spiking experiments, in which selectivity for PAA was demonstrated at several different concentrations of both HP and AA. The limit-of-detection (LOD) and the limit-of-quantitation (LOQ) for this method was determined to be 60 ppb and 198 ppb, respectively. Unlike many other methods, the sorbent tube negates the need for solvent in the sampling apparatus, adding to its ease of use. These features as well as inexpensive materials and the use of small, wearable vacuum pumps could allow for implementation of easier personal sampling in the workplace.

Methods Collection

1. Preparation of Peracetic Acid Solutions
    - Peracetic acid solutions were prepared using 32% peracetic acid in acetic acid (Sigma Aldrich) solution diluted to 1 mL with deionized H_2O (18 MΩ cm).
    - Concentration range: 0.041-3.27 M.

2. PAA Solution Experiments
    - PAA solutions were prepared as described above.
    - 1 mL of acetonitrile (ACN), 2 µL of PAA solution, and 100 µL cyclohexene were combined in a clean 7-mL clear glass vial.
    - After 24-hour reaction at room temperature, 1.5 mL of 0.178 mM 1-bromohexane in *n*-hexane solution was added to extract the epoxide product.

3. Control Solution Experiments
    - Control experiments were performed using cyclohexene oxide aliquots of the same concentration as the PAA samples. These were prepared using cyclohexene oxide (CHO) (98%, Sigma Aldrich) diluted to 1 mL in acetonitrile.
    - Five mL of acetonitrile, 2 µL of CHO solution, and 100 µL cyclohexene were combined in clean, 16-mL glass vials.
    - Samples were processed using same procedure as above after 24-hour reaction period, and data were compared to that of analogous PAA product samples (also prepared in 5 mL of acetonitrile).

4. Contaminant Reactivity Experiments
    - A 100-ppb PAA solution was prepared as described above.
    - Hydrogen peroxide solutions (1000 and 10000 ppb) were prepared using a 30% (w/w) hydrogen peroxide solution purchased from Sigma Aldrich and deionized H_2O (18 MΩ cm).
    - Acetic acid solutions (1000 and 10000 ppb) were prepared using glacial acetic (Sigma Aldrich) and deionized H_2O (18 MΩ cm).
    - 1 mL of ACN, varying quantities of PAA and HP/AA, and 100 µL cyclohexene were combined in a clean 7-mL glass vial.
    - Samples reacted for 24 hours, after which time they were processed as described above.

5. PAA Chamber Creation and Sorbent Tube Sampling
    - A Teflon chamber containing PAA-spiked air was prepared by introducing 2 µL of the prepared PAA solution of known concentration in 80 L of 50% relative humidity air.
    - Using 350-mg XAD-7 sorbent tubes (SKC Inc.) and SKC personal sampling pumps, the PAA-containing air was sampled for 4 hours at 250 mL/min.

6. Sample Processing
    - After sampling, the sorbent from the tube was treated with 1 mL ACN (Sigma Aldrich) and 100 µL cyclohexene (Sigma Aldrich) in a clean 7-mL clear glass vial.
    - After 24-hour reaction at room temperature,1.5 mL of 0.178 mM 1-bromohexane (Sigma Aldrich) in *n*-hexane (Sigma Aldrich) solution was added to the sorbent to extract epoxide product.

7. Vial Experiments Containing Sorbent for Calibration Curve
    - PAA solutions were prepared as described above.
    - The sorbent from a fresh 350-mg XAD-7 sorbent tube, 1 mL of ACN, and 2 µL of PAA solution of known concentration were combined in a clean 7-mL glass vial.
    - 100 µL cyclohexene was added to vial, and the sample was vortexed.
    - Following 24-hour reaction at room temperature, sample was processed as described above.

8. Fortified Sampler Experiments
    - PAA solutions were prepared as described above.
    - A Teflon chamber was filled with 80 L of clean air at 50% relative humidity.
    - A 350-mg XAD-7 sorbent tube was loaded with 2 µL of PAA solution of known concentration.
    - The spiked tube was attached to the sampling pump and to the Teflon chamber.
    - Clean air from the chamber was pulled across the spiked sorbent tube for 4 hours at 250 mL/min.
    - Sample was processed as described above.

9. Sorbent Capacity Experiments
    - A 2000-ppb PAA solution was prepared as described above.
    - 2 µL of the solution was added to the Teflon chamber, which was subsequently filled.

- A second sorbent tube, a 175-mg XAD-7 tube, was added behind the 350-mg XAD-7 tube in the sampling train.
- Sampling proceeded as described above.
- Each tube was processed separately as described above.

10. Sorbent Tube Storage Experiments
    - Chamber was prepared and sampled as described above.
    - Following sampling, sorbent tubes were sealed on both ends, wrapped in foil to prevent light exposure, and were stored in 1 of 2 ways:
        i. At room temperature (approx. 25˚C) for 72 hours.
        ii. At -20˚C for 72 hours.
    - Tube was processed as described above following storage, except 5 mL of acetonitrile were used instead of 1 mL.

11. Stability and Variable Sampling Time Experiments
    - Chambers were prepared as described above.
    - Two treatments conducted:
        i. PAA-spiked chamber was sampled at 250 mL/min of PAA-spiked gas for 4 hours, with the sorbent tube being replaced with a fresh tube at the end of each 1-hour period. Each of the 4 resulting sorbent tubes were processed separately as described above.
        ii. PAA-spiked chamber was sampled at 1 L/min for 15 minutes (total volume = 15 L). Samples processed as described above.

Citations

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