

HHS Public Access

Author manuscript

J Occup Environ Hyg. Author manuscript; available in PMC 2022 September 09.

Published in final edited form as:

J Occup Environ Hyg. 2022 August; 19(8): 469-477. doi:10.1080/15459624.2022.2088772.

A field-portable colorimetric method for the measurement of peracetic acid vapors: a comparison of glass and plastic impingers

Angela L. Stastny,

Amos Doepke,

Robert P. Streicher

Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, Cincinnati, Ohio

Abstract

A method for measuring peracetic acid vapors in air using impinger sampling and field-portable colorimetric analysis is presented. The capture efficiency of aqueous media in glass and plastic impingers was evaluated when used for peracetic acid vapor sampling. Measurement of peracetic acid was done using an *N*,*N*-diethyl-p-phenylenediamine colorimetric method with a field-portable spectrometer. The linearity of the *N*,*N*-diethyl-p-phenylenediamine method was determined for peracetic acid both in solution and captured from vapor phase using glass or plastic impingers. The Limits of Detection for the glass and plastic impingers were 0.24 mg/m³ and 0.28 mg/m³, respectively, for a 15 L air sample. The Limits of Quantitation were 0.79 mg/m³ and 0.92 mg/m³ for the glass and plastic impingers, respectively. Both metrics were below the American Conference of Governmental Industrial Hygienists Threshold Limit Value Short-Term Exposure Limit of 1.24 mg/m³ (0.4 ppmv) during a 15-min period. The impinger sampling method presented herein allows for an easy-to-use and rapid in-field measurement that can be used for evaluating occupational exposure to peracetic acid.

Keywords

DPD; disinfectant; occupational exposure monitoring; PAA; peroxide; peroxyacetic acid

CONTACT Angela L. Stastny opf2@cdc.gov Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, Cincinnati, OH 45226, USA.

Author contributions

Angela L. Stastny's contributions to the work herein include project conceptualization, data curation, formal analysis, investigation, methodology, project administration, supervision, validation, visualization, and writing the original draft. Amos Doepke contributed to the conceptualization, methodology, project administration, procurement of resources, supervision, visualization, and writing and reviewing of this work. Robert P. Streicher contributed to the conceptualization, methodology, project administration, supervision, writing, and editing of this work.

Disclaimer

The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention. Mention of any company or product does not constitute endorsement by the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention. Study data are available upon request from the corresponding author.

Introduction

Peracetic acid (PAA) is a commonly used antimicrobial that has been used as a disinfectant in the food and beverage industries, healthcare settings, water treatment, and as a bleaching agent (Swern 1970; Baldry and French 1989; Pechacek et al. 2015; Luukkonen and Pehkonen 2017). PAA is more environmentally friendly than conventional chlorinecontaining disinfectants because it leaves behind no toxic residues (Pinkernell et al. 1994; Awad et al. 2000). However, PAA is irritating to mucous membranes of the respiratory tract, skin, and eyes. The effects of worker exposure to PAA vapors are a pertinent issue. There is a need to quantify air concentrations of PAA quickly and accurately in the workplace based on irritation of the upper respiratory tract and lacrimation occurring due to exposures as low as 15.6 mg/m³ (5 ppmv) within 3 min (Committee on Acute Exposure Guideline Levels; Committee on Toxicology; National Research Council 2010). A threshold for irritation to mucous membranes and eyes of 1.56 mg/m³ (0.5 ppmv) was derived from Fraser and Thorbinson (1986); this concentration of PAA vapor is not expected to cause discomfort (Fraser and Thorbinson 1986). The American Conference of Governmental Industrial Hygienists (ACGIH®) set a Threshold Limit Value (TLV®) for a Short-Term Exposure Limit (STEL) of 1.24 mg/m³ (0.4 ppmv) during a 15-min period (ACGIH 2014). A reliable and accurate method for PAA measurement is needed for quantifying exposures to PAA vapors at low concentrations in the workplace.

Several laboratory-based methods have been developed to detect PAA vapor. These include sampling methods using the reagents: 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid (ABTS) (Pinkernell et al. 1997; Effkemann et al. 2000; Wagner et al. 2002), 2-([3-{2-[4-Amino-2-(methylsulfanyl)phenyl]-1-diazenyl}phenyl]sulfonyl)-1-ethanol (ADS) (Effkemann and Karst 1998; Henneken et al. 2006), methyl *p*-tolyl sulfide (MTS) (Pinkernell et al. 1996; 1997; Effkemann et al. 1999; Hecht and Héry 2002; Pacenti et al. 2010; Nordling et al. 2017; Simmons 2019), and methyl *p*-tolyl sulfoxide (MTSO) (Hecht et al. 2004). These reagents have been placed in impingers, on sorbent tubes, on filters, or solid-phase microextraction fibers and analyzed by liquid chromatography, gas chromatography, or colorimetry. All these methods use laboratory instruments for analysis and can involve sample preparations that are not suited for making quick PAA vapor concentration measurements in the field. Electrochemical sensors used for real-time detection of PAA vapors are commercially available but have not been rigorously validated in the literature.

Acetic acid (AA) and hydrogen peroxide (HP) exist in equilibrium with PAA and water in solution:

$$CH_3CO_2H + H_2O_2 \rightleftharpoons CH_3CO_3H + H_2O$$

Accurate determination of PAA concentration without interferences from AA or HP is necessary for monitoring worker exposure. PAA detection methods can involve a two-piece sampling system, one to capture HP and one to capture PAA (Hecht et al. 2004; Simmons 2019).

In the work presented here, a field-portable photometric method using the N,N-diethyl-pphenylenediamine (DPD) reagent was used to quantify PAA in aqueous solutions. AA and HP are always present in equilibrium with PAA and do not interfere with the PAA concentration measurements using the DPD method (Howarth and Harvey 2010; Liu et al. 2014). The DPD method is based on PAA reacting with an excess of iodide, causing the iodide to be oxidized to iodine. The iodine reacts with DPD to produce a magenta-colored product that is measured by a photometer. DPD has been used for monitoring PAA in wastewater disinfecting treatments (Dell'Erba et al. 2004; Monarca et al. 2004; Crebelli et al. 2005; Monarca et al. 2005; Antonelli et al. 2006; Falsanisi et al. 2006; Rossi et al. 2007; Cavallini et al. 2013; Santoro et al. 2007; Domínguez-Henao et al. 2018). Proper and consistent timing from the sampling through the analysis steps are critical to making accurate determinations of PAA using the DPD method. PAA will degrade over time, so samples cannot be stored for later analysis. During the DPD analysis, the oxidation of iodide to iodine by PAA is a relatively fast reaction, while the oxidation of iodide due to HP is a slower process. This enables the analysis of PAA with the DPD solution before the HP has time to become an interference by significantly oxidizing the iodide. For consistent timing, the DPD analyses in this paper were completed within 5 min of sample collection.

The work herein demonstrates a method of measuring PAA vapors for occupational exposure monitoring using glass or plastic impingers that can offer low cost, easy to use, and rapid, in-field measurements. Additionally, this method is applicable for sensor evaluation and verification of generated atmospheres (Doepke et al. 2021). Also presented is a 3D printed impinger nozzle that offers an alternative to traditional glass impingers, which are undesirable for certain field uses, like food and pharmaceutical production sites.

Methods

Materials

All solutions were prepared using >18 M Ω -cm deionized water from an Evoqua water purification system. PAA solution (32% w/w of PAA, 40–45% w/w of acetic acid, and <6% w/w of H $_2$ O $_2$), HP solution (30.9% w/w), and Tygon tubing were supplied by Sigma Aldrich (St. Louis, MO). A V-2000 photometer and PAA and HP Vacu-vials instrumental test kits (K-7913, K-5543) were purchased from Chemetrics (Midland, VA). A DR1900 portable spectrophotometer and DPD total chlorine reagent powder pillows were purchased from HACH (Loveland, CO). 50-mL glass impingers were purchased from Ace Glass (Vineland, NJ). Plastic impingers were purchased from SKC Inc. (Eighty Four, PA). 25 mm Acrodisc syringe filters with 0.45 µm PTFE membranes were purchased from Pall Corporation (Port Washington, NY). The 3D printed nozzle was printed using an Objet Eden 260vs 3D printer with Veroclear and SUP 705 was used as support material. The support material was washed away before experimental use. Flow-limiting critical orifices (1 L/min) and Aerosol Adapter connectors were purchased from Millipore (St. Louis, MO). A Bios DryCal Defender flowmeter from Mesa Labs (Lakewood, CO) was used to measure the orifice flow rate.

Colorimetric determination of PAA concentration from stock solutions

The concentrations of the PAA solutions ($C_{Measured}$) were determined using the Chemetrics Vacu-vials ampoule and HACH PAA test kits according to the manufacturer's instructions. The photometers were zeroed according to the manufacturer's instructions. In the Chemetrics method, excess potassium iodide is added to the PAA solution where peracetic acid oxidizes the iodide to iodine. The iodine then oxidizes the DPD contained in an ampoule, forming a pink-colored Würster dye. The HACH method is similar to the Chemetrics method; a DPD total chlorine powder pillow is mixed with the PAA solution in a cuvette, producing a pink color. The intensity of the pink color is directly proportional to the peracetic acid concentration. The V2000 photometer from Chemetrics and DR1900 portable spectrophotometer from HACH were used to measure the absorbance of the pink color and automatically converted that to $C_{Measured}$. The Chemetrics method was used for all studies in this paper and the HACH method was used to verify the Chemetrics results. Impinger samples must be worked-up and colorimetric measurements made as soon as possible to avoid PAA degradation and HP interference. For HP measurements, based on Chemetrics' recommendations to avoid false positives from PAA, five drops of A-7900 Activator Solution were added to the sample before the K-5543 Vacu-vials and Chemetrics method 95 was used. The HP was also analyzed using HACH method 10290. The measurements were made within 5 min of dilution or impinger collection; within this time frame, the HP was found to be <6% of the product content, which was the stated Sigma Aldrich HP weight percent of the product (see online supplemental materials, Table S1). To determine if HP interference was a large contributor to error in PAA measurements, aliquots of 30.9% w/w HP were placed into a 0.5-L volumetric flask and diluted to the mark with deionized water. The solution was analyzed for PAA and HP using the Chemetrics methods (Supplemental Table S2). Based on these findings, HP had good recoveries (> 90%) and PAA was found to be near the LOD and below the LOQ (LOD/LOQ reported in results: Weight Percent of Stock Solutions and Linearity of Dilutions). Because HP was not a significant interference, filter cassettes were not required to remove HP before the impinger, as in the OSHA impinger method PV2321.

Various amounts of 32% PAA (V_{PAA}) ranging from 0.45 to 14.9 μ L were placed into a 0.25-L ($V_{Dilution}$) volumetric flask and diluted to the mark with deionized water. To make lower concentrations, aliquots of the aforementioned solutions were placed into 50-mL volumetric flasks and diluted to the mark with deionized water. The experimentally determined concentration of PAA ($C_{Experimental}$) was used to calculate the weight percent ($W_{\%}$) of PAA in solution, using the density of PAA solution (ρ_{PAA}) (Equations [1] and [2]). $C_{Measured}$ was determined for each V_{PAA} three times and an average weight percent ($W_{Average\%}$) for all experimental concentrations was determined and used to calculate the theoretical concentration ($C_{Calculated}$) (Equation [3]). The V2000 photometer has a maximum measurable concentration of 5 mg/L and the DR 1900 photometer has a measurement range with a maximum of 10 mg/L, thus sample solutions must be diluted below these concentrations before measurements are made.

$$C_{Experimental} = \frac{C_{Measured} \times V_{Dilution}}{V_{PAA}} \tag{1}$$

$$W_{\%} = \frac{C_{Experimental}}{\rho_{PAA}} \times 100 \tag{2}$$

$$C_{Calculated} = \frac{V_{PAA} \times \rho_{PAA} \times W_{Average\%}}{V_{Dilution} \times 100}$$
 (3)

Colorimetric determination of PAA collection efficiency as collected by glass impinger

For vapor spiking, an Acrodisc syringe filter was connected by Tygon tubing to the inlet of a glass impinger filled with 15 mL of deionized water. A tube length of 2.54 cm between the impinger and filter was used in all experiments unless noted. The impinger outlet was connected by Tygon tubing to a critical orifice and then a vacuum (Figure 1). Aliquots from 0.5–6.5 µL of PAA were pipetted onto the filter and air was pulled through the impinger at $1 \text{ L min}^{-1} \pm 8\%$ for 15 min. Full evaporation of the PAA aliquot was observed after 10 min of collection time. The impinger sample was diluted to 500 mL and the PAA concentration was measured by the Chemetrics method, according to the manufacturer's directions. Each measurement was taken in triplicate. From the linear regression, the LOD was calculated as 3 times the standard error of the slope and the LOQ as 10 times the standard error of the slope. No backup impinger was used for these studies because impinger collection efficiency was greater than 96.6% (Supplemental Table S3).

Colorimetric determination of PAA collection efficiency as collected by plastic impinger

Three plastic impingers were compared for use in the PAA collection. Two impingers were commercially available from SKC; one having a vertical inlet and horizontal outlet, and the second having both inlet and outlet ports vertical. The third plastic impinger had vertical inlet and outlet ports with a 3D printed nozzle to improve the collection of PAA vapor. The commercially available perfluoroalkoxy alkane (PFA) plastic impingers have a non-tapered, flat tip whereas a typical glass impinger has a nozzle (Figure 2). The taper of the tip increases the linear velocity of air, which improves the collection efficiency of the impinger for aerosols, and reduces the bubble size, which improves the collection efficiency of the impinger for vapors. Nozzles and ports for the plastic impingers were designed and 3D printed in-house (Supplemental Figure S1). The 3D-printed impinger was assembled from the printed ports and nozzle piece with an SKC impinger body and lid. The sampling procedure for the plastic impingers was the same as the procedure for the glass impinger described above. There was poor collection efficiency when using the plastic impinger with the horizontal port as received from the manufacturer (Supplemental Table S4). The impinger with the 3D printed nozzle had a collection efficiency greater than 95.8%, so no backup impinger was used for these studies (Supplemental Table S5).

Results

Weight percent of stock solution and linearity of dilutions

Stock PAA solution was diluted to concentrations ranging from 0.062 to 3.96 mg L⁻¹ for the Chemetrics method and 0.34 to 10 mg L⁻¹ for the HACH method. Each dilution was measured three times. The average weight percent ($W_{Average\%}$) \pm the standard deviation, calculated from Equations (1) to (3) for Chemetrics was 29.2% \pm 2.0% PAA by weight and 29.7% \pm 2.8% PAA by weight for HACH. For the calculations in the rest of the paper, a weight percent of 29.2% PAA was utilized.

To assess the linearity over the range of PAA concentrations, Equation (3) was used to determine the calculated PAA concentration. The experimental PAA concentrations versus the calculated PAA concentrations were plotted (Supplemental Figure 2, Supplemental Table S6), and a linear regression resulted in an R² of 0.9974 with a linear equation of:

$$C_{Measured} = (1.0517 \pm 0.00973) \times C_{Calculated} - 0.0336 \pm 0.0147$$
 (4)

From the linear regression, the LOD for the Chemetrics method was calculated as 3 times the standard error of the slope and the LOQ as 10 times the standard error of the slope. The LOD for a liquid sample of PAA was 0.038 mg/L and the LOQ was 0.13 mg/L using the Chemetrics test kit method.

Tube length from PAA vapor source to impinger

To determine if tube length affected the PAA recovery, four glass impingers were prepared (as in Figure 1) with tubing lengths of 2.54, 8.9, 27.9, and 68.6 cm between the filter and the impinger (Table 1). Vapor experiments were conducted by pipetting 5.5 μ L of 29% PAA solution onto the filter. After 15 min of airflow at 1 L min⁻¹, the impinger solution was diluted to 500 mL and the Chemetrics test kit was used to determine PAA concentration. The total recovery of PAA was between 94.8% and 97.3%.

Commercial plastic impinger modification

An impinger consists of two pieces, a lid with a vertical stem and connecting ports, and a vessel that holds the impinger collection liquid. Two versions of the SKC impingers are available; one has two ports coming from the top (vertical ports) and the other has one vertical port on the top and one on the side (horizontal port). Commercially available plastic impingers from SKC have a 0.64 cm diameter stem without any constriction of the diameter at the tip. A 3D-printed, tapered tip was designed to address this. An aliquot of 1.8-µL or 6.5-µL of 29% PAA solution was added to each filter and after 15 min of airflow at 1 L min⁻¹, the impinger solution was diluted to 500-mL and then the Chemetrics test kit was used to determine PAA concentration. Table 2 shows the differences in percent recovery between commercially purchased glass, plastic, and the 3D printed nozzle impingers. The glass and 3D printed nozzle had recoveries above 97%. The commercially available plastic impingers had lower recoveries, ranging from 69.1 to 84.3%.

Recovery of PAA vapors using glass or 3D printed plastic impingers

Since the results of experiments with the four impingers showed much better performance for the glass and 3D-printed plastic impingers, subsequent experiments over a wide concentration range were conducted using only the glass and 3D-printed plastic impingers (Tables 3 and 4). The recoveries for the glass impingers were at least 97.4% and the recoveries for the plastic impingers were at least 95.0% with the relative standard deviations below 5.8%. The LOD for the glass and plastic impingers were 0.24 mg/m³ (glass impinger) and 0.28 mg/m³ (plastic impinger) of PAA vapor for a 15 L air sample. These LODs correspond to impinger solution concentrations of 0.077 mg/L (aq) and 0.091 mg/L (aq) for the glass and plastic impingers, respectively. The LOQ for the glass and plastic impingers were 0.79 mg/m³ and 0.92 mg/m³, respectively, for a 15 L air sample, with corresponding values of 0.25 mg/L (aq) and 0.30 mg/L (aq).

Discussion

Measurements of working range standards were taken three times by Chemetrics using the methods described. From those measurements, the weight percent of stock PAA solution was measured as $29.2\% \pm 2.0\%$. Calculations for subsequent experiments were done using a 29.2% w/w PAA concentration of the stock solution.

Glass impingers are not always permitted in food and beverage facilities, so a plastic impinger could offer an alternative impinger option. For field use, plastic impingers are more desirable because they have a screw top that cannot be separated easily and there is less of a concern about breakage compared to glass impingers. The flat tip of the inlet tube from commercially available impingers produced larger bubbles than a typical glass impinger with a constriction at the tip. Smaller bubbles increase collection efficiency because there is more surface area per volume of gas and decreased distance for the diffusion of gas inside to the surface of the bubble. To decrease the bubble size and increase the diffusion of PAA molecules into the water, a 3D printed tapered tip for the plastic impinger was designed and tested. The commercially available plastic impingers with the flat tip were found to have significantly lower recoveries, in the range of 69% to 84%, but the 3D printed impinger and glass impingers had nearly 100% recoveries. Therefore, using the 3D printed plastic impinger with a constricted nozzle is recommended over the commercially available plastic impingers.

The effect of the length of air sampling tubing preceding the impinger on recovery of PAA was investigated. When sampling in the field, longer tubing may be used to sample near the breathing zone of a worker while using an impinger worn at the waist. The tube length was varied from 2.54 to 68.6 cm and PAA recoveries were 94 to 97%; the tube length did not appreciably affect the recovery of PAA in the impinger solution.

The Occupational Safety and Health Administration (OSHA) method PV2321 is an impinger method that uses the reaction of MTS with PAA to produce MTSO in acetonitrile/water, which is analyzed by GC-FID (Simmons 2019). This method involves connecting a filter cassette before the impinger to collect HP vapors. Data from OSHA showed that vapor spikes at various concentration levels, in a similar concentration range to those in

Table 3, had recovery efficiencies between 95.7% and 97.5% with coefficients of variation ranging from 0.6% to 10.8%, with the DPD method having recovery efficiencies > 97.4% and standard deviations ranging from 1.2% to 5.8%. The DPD method presented in this work, which uses only water, was found to have similar recoveries as the OSHA method which uses an in-solution reagent (MTS). Using the DPD methods presented herein, PAA vapors can be collected and analyzed within minutes, in the field, using low-cost supplies. In contrast, the OSHA method needs HP filter cassettes, an in-solution reagent (MTS) to react with the PAA, and a GC-FID instrument, which is usually found only in analytical labs, to analyze samples.

One of the largest influences on the accuracy and precision of this method might be the slow, but undesirable, reaction of HP with the iodide from the DPD method. HP is produced from the degradation of PAA but is also present in the 29% PAA solution. To prevent the reaction of HP with MTS in the impinger solution, the OSHA method adds a cassette filter at the front of the impinger to capture the HP. In the DPD method presented in this work, there is not an observed significant interference from HP if the samples were analyzed promptly. Solutions ranging from 0 to 5 mg L⁻¹ of HP were made and the concentration of PAA and HP was tested using the Chemetrics method (Supplemental Table S2). If HP reacted with the iodide from the DPD method, then a false positive result of PAA would be observed. But the measured concentration of PAA as interference from HP was near the LOD, below the LOQ, and <4.3% of the HP concentration present. Additional potential sources of error might include: the impinger collection efficiency and loss of PAA to the inner surfaces and tube of the sampling device. However, based on the high recoveries presented in this paper, there does not seem to be a large error in sampling with the impingers.

Going forward, this DPD impinger method can be compared to other sampling and analysis approaches. Recently, an atmosphere generation chamber was developed to produce constant PAA atmospheres at different concentrations, humidities, and temperatures (Doepke et al. 2021). The sampling column contains multiple ports so various sensors can be connected and tested against one another. The sampling column can allow for side-by-side comparisons of other PAA sampling methods with the DPD impinger method.

Conclusions

A method using glass or plastic impinger collection and colorimetry for measurement of PAA vapor in the field was developed. Tube length between the filter and the impinger does not significantly influence the recovery of PAA. The 3D printed plastic impinger gave near quantitative recovery of PAA, closely matching the performance of the glass impinger. By comparison, the commercial plastic impingers had recoveries in the range of 69% to 84%. When using the 3D printed plastic impinger, the LOD and LOQ for a 15-L air sample of PAA vapors was 0.28 mg/m³ and 0.92 mg/m³, respectively. Similarly, when using a glass impinger the LOD and LOQ of the method was 0.24 mg/m³ and 0.79 mg/m³, respectively, for a 15-L air sample. Use of plastic impingers with water may be more cost-effective and offer a safer alternative to glass impingers, which are not permitted in the food and pharmaceutical industries. The 3D printed impinger improves the collection efficiency compared to commercially available plastic impingers. The method presented

herein has a quick analysis time and measurements were quantifiable below the AGCIH TLV STEL. This method could be used in the field to monitor PAA as part of a measurement strategy to evaluate worker exposure.

Recommendations

When considering the concerns about PAA exposure, the work presented in this manuscript demonstrates a reliable sampling method for PAA vapors that could be useful for industrial hygienists. While other methods and sensors have been developed for PAA measurement, this is a robust and low-cost method, with a rapid on-site analysis, that can be used in the field for air concentrations below the AGCIH TLV STEL. This method also assessed a 3D printed impinger which increased recovery efficiency compared to commercially available plastic impingers and can be used for PAA measurement in places where glass impingers are not permitted.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

The authors gratefully acknowledge our collaborators within NIOSH. The authors thank Barbara M. Alexander and Kevin H. Dunn for helpful conversations and suggestions, Kevin Menchaca for assistance with printing the 3D printed impinger, and Peter B. Shaw for statistical analysis.

References

- ACGIH (American Conference of Governmental Industrial Hygienists). 2014. Annual TLVs® (Threshold Limit 4 Values) and BEIs® (Biological Exposure Indices) booklet. Cincinnati (OH): ACGIH Signature Publications.
- Antonelli M, Rossi S, Mezzanotte V, Nurizzo C. 2006. Secondary effluent disinfection: PAA long term efficiency. Environ Sci Technol. 40(15):4771–4775. doi:10.1021/es060273f [PubMed: 16913137]
- Awad MI, Harnoode C, Tokuda K, Ohsaka T. 2000. Simultaneous electroanalysis of peracetic acid and hydrogen peroxide using square-wave voltammetry. Electrochemistry. 68(11):895–897. doi:10.5796/electrochemistry.68.895
- Baldry MGC, French MS. 1989. Disinfection of sewage effluent with peracetic acid. Water Sci Technol. 21(3):203–206. doi:10.2166/wst.1989.0100
- Cavallini GS, De Campos SX, Beber De Souza J, De Sousa Vidal CM. 2013. Comparison of methodologies for determination of residual peracetic acid in wastewater disinfection. Int J Environ Anal Chem. 93(8):906–918. doi:10.1080/03067319.2012.702274
- Committee on Acute Exposure Guideline Levels; Committee on Toxicology; National Research Council. 2010. Acute exposure guideline levels for selected airborne chemicals. Vol. 8. US: National Academies Press. p. 328.
- Crebelli R, Conti L, Monarca S, Feretti D, Zerbini I, Zani C, Veschetti E, Cutilli D, Ottaviani M. 2005. Genotoxicity of the disinfection by-products resulting from peracetic acid- or hypochlorite-disinfected sewage wastewater. Water Res. 39(6):1105–1113. doi:10.1016/j.watres.2004.12.029 [PubMed: 15766965]
- Dell'Erba A, Falsanisi D, Liberti L, Notarnicola M, Santoro D. 2004. Disinfecting behaviour of peracetic acid for municipal wastewater reuse. Desalination. 168:435–442. doi:10.1016/j.desal.2004.07.028

Doepke A, Stastny AL, Streicher RP. 2021. Controlled generation of peracetic acid atmospheres for the evaluation of chemical samplers. Anal Methods. 13(34):3799–3805. doi:10.1039/D1AY00958C [PubMed: 34368829]

- Domínguez-Henao L, Turolla A, Monticelli D, Antonelli M. 2018. Assessment of a colorimetric method for the measurement of low concentrations of peracetic acid and hydrogen peroxide in water. Talanta. 183:209–215. doi:10.1016/j.talanta.2018.02.078 [PubMed: 29567166]
- Effkemann S, Brødsgaard S, Mortensen P, Linde SA, Karst U. 1999. Determination of gas phase peroxyacetic acid using pre-column derivatization with organic sulfide reagents and liquid chromatography. J Chromatogr A. 855(2):551–561. doi:10.1016/s0021-9673(99)00734-7 [PubMed: 10519092]
- Effkemann S, Brødsgaard S, Mortensen P, Linde SA, Karst U. 2000. Spectrophotometric and direct-reading methods for the analysis of gas phase peroxyacetic acid. Fresenius J Anal Chem. 366(4):361–364. doi:10.1007/s002160050073 [PubMed: 11220319]
- Effkemann S, Karst U. 1998. Reagent for the high-performance liquid chromatography determination of peroxycarboxylic acids. Analyst. 123(8):1761–1765. doi:10.1039/a801697f
- Falsanisi D, Gehr R, Santoro D, Dell'Erba A, Notarnicola M, Liberti L. 2006. Kinetics of PAA demand and its implications on disinfection of wastewaters. Water Qual Res J Can. 41(4):398–409. doi:10.2166/wqrj.2006.043
- Fraser JAL, Thorbinson A. 1986. Fogging trials with Tenneco Organics Limited (30th June, 1986) at collards farm. Warrington (UK): Solvay Interox.
- Hecht G, Héry M. 2002. Generation of controlled atmospheres for the determination of the irritant potency of peroxyacetic acid. Ann Occup Hyg. 46(1):89–96. doi:10.1093/annhyg/mef010 [PubMed: 12005137]
- Hecht G, Héry M, Hubert G, Subra I. 2004. Simultaneous sampling of peroxyacetic acid and hydrogen peroxide in workplace atmospheres. Ann Occup Hyg. 48(8):715–721. doi:10.1093/annhyg/meh067 [PubMed: 15509632]
- Henneken H, Assink L, de Wit J, Vogel M, Karst U. 2006. Passive sampling of airborne peroxyacetic acid. Anal Chem. 78(18):6547–6555. doi:10.1021/ac060668h [PubMed: 16970333]
- Howarth J, Harvey M. 2010. Method of analyzing low levels of peroxyacetic acid in water. US Patent No. 7,651,860 B2.
- Liu D, Steinberg C, Straus DL, Pedersen L, Meinelt T. 2014. Salinity, dissolved organic carbon and water hardness affect peracetic acid (PAA) degradation in aqueous solutions. Aquac Eng. 60:35–40. doi:10.1016/j.aquaeng.2014.03.006
- Luukkonen T, Pehkonen SO. 2017. Peracids in water treatment: a critical review. Crit Rev Environ Sci Technol. 47(1):1–39. doi:10.1080/10643389.2016.1272343
- Monarca S, Feretti D, Zani C, Rizzoni M, Casarella S, Gustavino B. 2005. Genotoxicity of drinking water disinfectants in plant bioassays. Environ Mol Mutagen. 46(2):96–103. doi:10.1002/ em.20137 [PubMed: 15880733]
- Monarca S, Zani C, Richardson AD, Thruston AD, Moretti M, Feretti D, Villarini M. 2004. A new approach to evaluating the toxicity and genotoxicity of disinfected drinking water. Water Res. 38(17):3809–3819. doi:10.6/j.watres.2004.07.003. [PubMed: 15350433]
- Nordling J, Kinsky OR, Osorio M, Pechacek N. 2017. Description and evaluation of a peracetic acid air sampling and analysis method. Toxicol Ind Health. 33(12):922–929. doi:10.1177/0748233717739165 [PubMed: 29137570]
- Pacenti M, Dugheri S, Boccalon P, Arcangeli G, Dolara P, Cupelli V. 2010. Air monitoring and assessment of occupational exposure to peracetic acid in a hospital environment. Ind Health. 48(2):217–221. doi:10.2486/indhealth.48.217 [PubMed: 20424354]
- Pechacek N, Osorio M, Caudill J, Peterson B. 2015. Evaluation of the toxicity data for peracetic acid in deriving occupational exposure limits: a minireview. Toxicol Lett. 233(1):45–57. doi:10.1016/j.toxlet.2014.12.014 [PubMed: 25542141]
- Pinkernell U, Effkemann S, Karst U. 1997. Simultaneous HPLC determination of peroxyacetic acid and hydrogen peroxide. Anal Chem. 69(17):3623–3627. doi:10.1021/ac9701750. [PubMed: 21639287]

Pinkernell U, Effkemann S, Nitzsche F, Karst U. 1996. Rapid high-performance liquid chromatographic method for the determination of peroxyacetic acid. J. Chromatogr. A. 730(1–2):203–208. doi:10.1016/0021-9673(95)01346-6

- Pinkernell U, Karst U, Cammann K. 1994. Determination of peroxyacetic acid using high-performance liquid chromatography with external calibration. Anal Chem. 66(15):2599–2602. doi:10.1021/ac00087a028
- Pinkernell U, Lüke H-J, Karst U. 1997. Selective photometric determination of peroxycarboxylic acids in presence of hydrogen peroxide. Analyst. 122(6):567–571. doi:10.1039/a700509a
- Rossi S, Antonelli M, Mezzanotte V, Nurizzo C. 2007. Peracetic acid disinfection: a feasible alternative to wastewater chlorination. Water Environ Res. 79(4):341–350. doi:10.2175/106143006x101953 [PubMed: 17489268]
- Santoro D, Gehr R, Bartrand TA, Liberti L, Notarnicola M, Dell'Erba A, Falsanisi D, Haas CN. 2007. Wastewater disinfection by peracetic acid: assessment of models for tracking residual measurements and inactivation. Water Environ Res. 79(7):775–787. doi:10.2175/106143007x156817. [PubMed: 17710922]
- Simmons M 2019. Peracetic acid. Salt Lake City (UT): Occupational Safety and Health Administration (US). Report No.: PV 2321.
- Swern DE. 1970. Organic peroxides. Vol. 1. New York (NY): John Wiley & Sons. p. 362.
- Wagner M, Brumelis D, Gehr R. 2002. Disinfection of wastewater by hydrogen peroxide or peracetic acid: development of procedures for measurement of residual disinfectant and application to a physicochemically treated municipal effluent. Water Environ Res. 74(1):33–50. doi:10.2175/106143002X139730 [PubMed: 11995865]

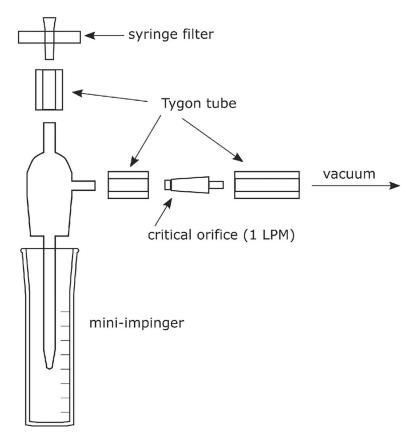


Figure 1. Diagram of impinger connection to syringe filter, critical orifice, and tubing.

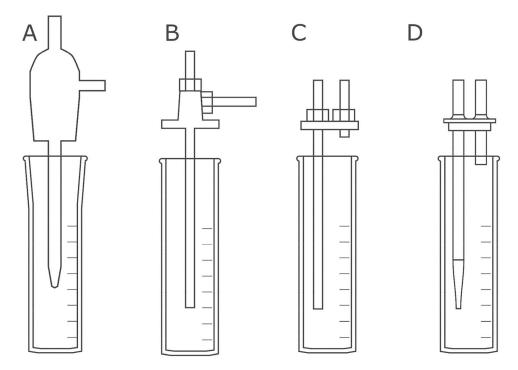


Figure 2. Illustrations of a (A) glass impinger, (B) plastic impinger with a vertical inlet and horizontal outlet, (C) plastic impinger with vertical inlet and outlet ports, and (D) 3D printed impinger.

Table 1.

PAA recovery of commercial glass impinger with varying tube-to-filter lengths (four measured replicates), V_{PAA} was 5.50 μ L (1.82 mg PAA) and diluted to 500-mL, $C_{Calculated}$ is 3.63 mg L⁻¹.

Tube-to-filter length, cm	PAA recovery
2.54	97.3% ±1.66%
8.9	95.5% ±0.72%
27.9	94.8% ±0.55%
68.6	94.9% ±0.96%

Table 2.

PAA recovery of a commercial glass impinger, plastic impingers with a vertical and horizontal port, and a 3D printed nozzle impinger (four measured replicates).

Impinger type	Recovery $C_{Calculated} = 1.19 \text{ mg L}^{-1}$	Recovery $C_{Calculated} = 4.29 \text{ mg L}^{-1}$
Glass	$103.8\% \pm 4.8\%$	100.4% ± 1.2%
Commercial Plastic Horizontal Port	$69.1\% \pm 2.0\%$	$84.3\% \pm 1.7\%$
Commercial Plastic Vertical Port	$72.6\% \pm 3.2\%$	$82.2\% \pm 2.5\%$
3D Printed Plastic Tip	$104.1\% \pm 4.3\%$	$97.0\% \pm 2.0\%$

When VPAA was 1.80 μ L (0.59 mg PAA) and diluted to 500-mL, $\mathit{CCalculated}$ was 1.19 mg L $^{-1}$. When VPAA was 6.50 μ L (2.15 mg PAA) and diluted to 500-mL, $\mathit{CCalculated}$ was 4.29 mg L $^{-1}$.

Stastny et al. Page 16

 Table 3.

 Recovery of PAA from glass impinger studies (three measured replicates).

xecovery	OI FAA IIC	om grass impinge	recovery of FAA from glass impinger studies (unee measured replicates).	a repiicates).
$V_{PAA}, \mu m L$	mg of PAA	$C_{Calculated}, \mathbf{mg} \mathbf{L}^{-1}$	VP1A, µL mg of PAA Ccalculated, mg L-1 Averaged CMeasured, mg L-1 PAA recovery	PAA recovery
0.50	0.17	0.33	0.37 ± 0.018	112.7% ± 5.5%
0.70	0.23	0.46	0.47 ± 0.015	$101.0\% \pm 3.3\%$
08.0	0.26	0.53	0.53 ± 0.031	$101.0\% \pm 5.8\%$
1.30	0.43	0.86	0.84 ± 0.025	$97.5\% \pm 2.9\%$
1.80	0.59	1.19	1.23 ± 0.058	$103.8\% \pm 4.8\%$
3.30	1.09	2.18	2.12 ± 0.052	$97.4\% \pm 2.4\%$
5.00	1.65	3.30	3.27 ± 0.061	$99.1\% \pm 1.9\%$
6.50	2.15	4.29	4.31 ± 0.053	$100.4\% \pm 1.2\%$

Stastny et al. Page 17

Table 4.

Recovery of PAA from 3D printed plastic impinger studies (three measured replicates).

kecovery	of PAA fro	om 3D printed pla	ecovery of PAA from 3D printed plastic impinger studies (three measured repl	hree measured r	iebl Je
V_{PAA} , $\mu ext{L}$	mg of PAA	$C_{Calculated}, \mathbf{mg} \mathbf{L}^{-1}$	VPAA, µL mg of PAA CCalculated mg L-1 Averaged CMassured mg L-1 PAA recovery	PAA recovery	
0.50	0.17	0.33	0.38 ± 0.011	$116.0\% \pm 3.2\%$	
0.70	0.23	0.46	0.44 ± 0.017	$96.1\% \pm 3.8\%$	
08.0	0.26	0.53	0.52 ± 0.025	$97.8\% \pm 4.7\%$	
1.00	0.33	99.0	0.64 ± 0.011	$96.7\% \pm 1.6\%$	
1.80	0.59	1.19	1.24 ± 0.052	$104.1\% \pm 4.3\%$	
3.30	1.09	2.18	2.24 ± 0.034	$103.0\% \pm 1.6\%$	
4.60	1.52	3.04	2.89 ± 0.038	$95.0\% \pm 1.2\%$	
6.50	2.15	4.29	4.16 ± 0.085	$97.0\% \pm 2.0\%$	