A review of measurement methods for peracetic acid (PAA)

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HIGHLIGHTS

• Physical and chemical properties and application of peracetic acid solution.
• Determination method of high concentration peracetic acid.
• Determination method of residual peracetic acid (low concentration).

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ABSTRACT

Peroxyacetic acid has been widely used in food, medical, and synthetic chemical fields for the past several decades. Recently, peroxyacetic acid has gradually become an effective alternative disinfectant in wastewater disinfection and has strong redox capacity for removing micro-pollutants from drinking water. However, commercial peroxyacetic acid solutions are primarily multi-component mixtures of peroxyacetic acid, acetic acid, hydrogen peroxide, and water. During the process of water treatment, peroxyacetic acid and hydrogen peroxide (H₂O₂) often coexist, which limits further investigation on the properties of peroxyacetic acid. Therefore, analytical methods need to achieve a certain level of selectivity, particularly when peroxyacetic acid and hydrogen peroxide coexist. This review summarizes the measurement and detection methods of peroxyacetic acid, comparing the principle, adaptability, and relative merits of these methods.

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1 The general applications of peracetic acid

Peracetic acid (CH₃CO₃H, PAA) has a high oxidation potential (1.81 V), a relatively high reactive oxygen content (21.1%), as well as simple and safe decomposition products (oxygen, water, acetic acid). Consequently, it has been the most widely used peroxy acid in the industry...
effects of different concentrations of PAA (1.5–4.5 mg/L) with chlorination with VigorOx® WWT II PAA disinfection technology (Peroxychem, 2017). They compared the disinfection performance of PAA to VigorOx® WWT II PAA disinfection, which showed high activity against viruses, even at the lowest concentration (1.5 mg/L). (Peroxychem, 2017b). The team also explored the disinfection mechanism of PAA (Peroxychem, 2017a) and examined the combined disinfection performance of PAA and ultraviolet (UV) (Peroxychem, 2016). They also concluded that the use of PAA minimized safety concerns and environmental problems, and reduced the toxicity of wastewater to aquatic organisms without producing harmful disinfection byproducts. Some studies have also shown that PAA is able to oxidize (micro)contaminants in wastewater (Virkutyte and Varma, 2014).

Prior to this, PAA has been widely used as a disinfectant in various industrial productions, such as the pharmaceutical industry, food industry, chemical industry, paper industry, and various water treatment systems (Crow, 1992; Kolyagin et al., 2010). Among these applications, PAA disinfection is the most widely used in the food industry, and is also used in fresh agricultural washing water, on-site cleaning processes, food processing equipment, and disinfectants in pasteurizers (Orth, 1998; Banach et al., 2015). In the field of health care, PAA has been used for endoscopic sterilization (Chenjiao et al., 2016) and hospital surface disinfection. PAA is also used as a raw material in fine chemical production (Kaur and Kishore, 2014).

Over the past few years, the global market of PAA has experienced tremendous boost due to its strong disinfection and oxidation properties. Looking forward, International Mining and Resources Conference (IMARC Group) expects the market to grow at a compound annual growth rate (CAGR) of 7.5% during 2020–2025 (IMARC, 2020). According to another report from Persistence Market Research (PMR), the ranking of the international PAA market is Europe, North America and Asia-Pacific; On the basis of individual nations, China and India are leading markets for PAA; In 2013, China occupied the largest market share in the regional consumption of PAA (PMR, 2015). The main global operators of PAA are Peroxychem, Solvay Chemicals, Kemira Chemicals, Christeys and Promox SPA, etc (Dugheri et al., 2018).

### 2 Chemical properties, synthesis and decomposition of peracetic acid

In terms of physical and chemical properties, PAA is a colorless liquid with a strong pungent odor. Its molecular structure is shown in Fig. 1 (Giguère and Olmos, 1952). In the family of peroxides, PAA is a monocarbonyl derivative of H₂O₂. The PAA molecule consists of two parts: an acetyl group and a peroxy group. The O-O bond length is 1.49 Å, and the bond angle of O-O-H is 100° (Giguère and Olmos, 1952). The structure of the acetyl group is identical to that in acetic acid (HAc). Both O and H in the peroxy group form stable intramolecular hydrogen bonds with the acyl oxygen in the acetyl group. PAA has both an acidic property with a pKₐ of 8.221 at 25°C (Koubek et al., 1963) and a peroxy property that can spontaneously decompose or explode under high concentrations, heating, mechanical stress, or exposure to catalytic amount of impurities (Klenk et al., 2000; Wang et al., 2015).

![Fig. 1 Peracetic acid structural formula (a) and chemical bonds and intramolecular hydrogen bond structures (b).](image)

The commercially available PAA is often a mixture of H₂O₂, HAc, and water (Kitis, 2004) with a PAA content of 5% to 25% (wt.%)(Leveneur et al., 2016). The most common preparation method for PAA is through perhydrolysis in which HAc and H₂O₂ are mixed under acid catalysis, as shown Eq. (1).

\[
\text{CH}_3\text{CO}_2\text{H} + \text{H}_2\text{O}_2 \rightarrow \text{CH}_3\text{CO}_3\text{H} + \text{H}_2\text{O} \quad (1)
\]
The reaction (Eq. (1)) is $2.10^{-2}$ (Yousefzadeh et al., 2014; Leveneur et al., 2016). A higher ion exchange resin can catalyze the formation of PAA including sulfuric acid, ascorbic acid, boric acid and acidic substances in the reactor, this analytical method is critical (Cristofari-Marquand et al., 2007). In quantitative measurement of trace amount PAA in aquatic bodies, the residual PAA concentration in the solution is important. Therefore, the titration process requires a quantitative reaction by using a substance that acts as an oxidizing agent (D’ans et al., 1969).

$$2\text{CH}_3\text{CO}_3\text{H}\rightarrow2\text{CH}_3\text{CO}_2\text{H} + \text{O}_2 \quad (2)$$

$$\text{CH}_3\text{CO}_3\text{H}\rightarrow\text{CH}_3\text{COO}^- + \text{HO}^+ \quad (3)$$

$$\text{CH}_3\text{CO}_3\text{H} + \text{H}_2\text{O}\rightarrow\text{CH}_3\text{CO}_2\text{H} + \text{H}_2\text{O}_2 \quad (4)$$

At room temperature (25°C), the equilibrium constant of reaction (Eq. (1)) is $2.10^{-2}$–2.91 mol/L. Various acids, including sulfuric acid, ascorbic acid, boric acid and acidic substances, can catalyze the decomposition of PAA to form oxygenated radical products with different redox potentials and lifetimes (Eq. (2)) (Koubek et al., 1963). PAA spontaneously decomposes in aqueous solution (Eq. (3)) to acetic acid and oxygen. Transition metal ions such as cobalt and manganese ions can catalyze the decomposition of PAA to form oxygenated radical products with different redox potentials and lifetimes (Eq. (3)) (Koubek et al., 1963). PAA can also be hydrolyzed to form H$_2$O$_2$ and HAc (e.g., Eq. (4)). In addition, the safety and stability of commercial PAA solutions can be enhanced by adding inert substances (water or inorganic salts, etc.) or stabilizers (alkyl metal polyphosphates, dipicolinic acid or quinoline derivatives) (Sennewald et al., 1969).

### 3 Analytical and measurement method of peracetic acid

Traditionally, the measurement of PAA concentration is a part of quality control of this commercial chemical. For instance, the national standard of the People's Republic of China GB/T19104-2008 “Peracetic acid solution” uses redox titration to simultaneously measure PAA and H$_2$O$_2$ (GB/T, 2008). The measured concentration of PAA is in the range of 5% (wt.%) to 15% (wt.%) with an accuracy of 83%–96%. In the newly developed fields of water treatment, the residual PAA concentration in the solution can be used to evaluate disinfection performance, wastewater toxicity, and related reaction kinetics. Therefore, the quantitative measurement of trace amount PAA in aquatic solution is critical (Cristofari-Marquand et al., 2007). In particular, the rapid decay of PAA in wastewater requires a reliable and accurate analytical method to estimate the actual PAA concentration and design relevant oxidation processes (Santoro et al., 2015). Additionally, since PAA coexists with H$_2$O$_2$ in the reactor, this analytical method must be highly selective to distinguish between PAA and H$_2$O$_2$ (Wagner et al., 2002; Flores et al., 2014). Iodine redox titration is recommended in US EPA Guidance Report EPA/600/R-14/332 (EPA, 2014) and Water Quality Standard 40 CFR 136 (EPA, 1984). The US has gradually promoted PAA disinfection, and the PAA residues allowed to be eliminated before the determination of PAA by redox titration. Therefore, we need to exclude the effects of H$_2$O$_2$ by using a substance that reacts specifically with H$_2$O$_2$.

For example, H$_2$O$_2$ is titrated with permanganate (D’ans et al., 2008
iodine method is significantly different from the other methods in the measurement of low-concentration PAA, while the cerimetry/iodometry method has obtained more satisfactory results. For concentrations between 0.5 and 10 mg/L, the difference in mean values is less than 5.2% of the actual value by using the cerimetry/iodometry method. The catalase/iodine method is more accurate for measuring the PAA concentration in the range of 1–5 mg/L. For PAA concentrations below 0.5 mg/L, none of the current redox titration methods are recommended, even when using the most developed and reliable method for redox titration.

The redox titration method is cumbersome in that the analyte is not suitable for long-term storage. All the reagents need to be freshly prepared and well-calibrated. Due to the measurement limit, this method is not recommended for the determination of residual low concentration PAA for wastewater treatment either.

3.2 NMR

Protons are present in H₂O₂, PAA, HAc, and water, while under different chemical environments. Therefore, proton NMR (¹H NMR) can be used to distinguish H₂O₂, PAA, and other peroxides in the presence of water and HAc. For instance, Stephenson and Bell (2005) developed the method to detect the formation and decomposition of H₂O₂ over solid surfaces by in situ NMR when studying oxygen-activated intermediates in heterogeneous catalytic systems. Similarly, liquid or solid NMR techniques can also be applied to determine PAA. Surprisingly, no related reports have been used to quantitatively measure PAA by using the NMR method. However, Ni and Kang (2007) used the ¹H NMR technique to confirm the formation of PAA during the peroxide bleaching of mechanical pulp.

The effect of in situ generated PAA on the increase in brightness during peroxide bleaching was discussed. This method used D₂O with 1% DSS (3-trimethylsilyl-1-propanesulfonic acid sodium salt) as the solvent. Taking the peak of D₂O as the reference peak, the experiment compared the difference in peak position under different PAA ratios. It was found that PAA peaked at 2.123 mg/L and HAc peaked at 2.073 mg/L, while the peak position of H₂O₂ was not mentioned.

The cost of NMR is relatively high and the testing process is cumbersome. It is necessary to refer to the standard matrix for quantitative testing. Therefore, the currently reported research does not use it as a rapid quantitative method for PAA, but only for qualitative measurement of PAA.

3.3 Chromatography

Chromatography is a method of separation and analysis. The method utilizes the selective partitioning of different substances in different phase states to elute with a mixture of the mobile phase relative to the stationary phase. Different substances in the mixture move along the stationary phase at different speeds, ultimately achieving the separation purpose. Most of the reported spectroscopic methods used direct detection of peroxides without prior derivatization. Despite the simple procedure, calibration is required using a PAA standard solution. Therefore, an auxiliary method, such as redox titration, must be used to confirm the reliability of the concentration of the PAA standard solution. In addition, the test results are often prone to problems such as highly acidic solutions or extremely high concentrations of H₂O₂. Since most samples require analysis after sampling, the direct separation method is not suitable for rapid determination.

At present, the derivatization technology has been widely used in chromatographic testing. The principle of the derivatization method is that the sample and the derivatizing reagent can rapidly and specifically react to form a derivatized product that can be quantitatively
analyzed by chromatographic methods. The amount of sample is indirectly determined by detecting the amount of the derivatized compound. Commonly used derivatization reagents include alkylation reagents, silylation reagents, acylation reagents, etc. Based on the different reactivity of organic sulfides reacting with PAA and H$_2$O$_2$, a derivatization method using organic sulfides is proposed and realized by gas/liquid phase chromatography (Eq. (10)). Specifically, organic sulfide R–S$_n$–R’$_1$ and peroxyacetic acid generate derivatized products R–S = O–R’ containing S = O bond, which can be detected by gas or liquid chromatography analysis.

$$R–S_n–R'_1 + PAA \rightarrow R–S = O–R'$$

Di Furia et al. (1984) proposed the selective oxidation of methyl p-tolyl sulfide (MTS) to the corresponding sulfoxide material (MTSO) to determine both PAA and H$_2$O$_2$, which were then separated by gas chromatography (Eq. (11)).

Separation using gas chromatography requires extraction of the aqueous sample using chloroform as an internal standard and decreases the accuracy of the test. Pinkernell et al. (1994) used liquid chromatography to analyze the reaction products between MTS and PAA. This method combines the advantages of directly measuring an aqueous solution of PAA and not requiring external standards. For the measurement of H$_2$O$_2$, triphenylphosphine (TPP) can be selected as a reagent for measuring H$_2$O$_2$ by HPLC (Eq. (12)) (Pinkernell et al., 1997).

$$2\text{CH}_3\text{COOOH} + \text{C}_6\text{H}_5\text{OH} \rightarrow \text{C}_17\text{H}_15\text{N}_3\text{O}_2 + 2\text{H}_2\text{O} + 2\text{CH}_3\text{COOH}$$

Binder and Menger (2000) proposed an improved colorimetric technique for measuring PAA in the presence of excess H$_2$O$_2$. Excess hydrogen peroxide is oxidized by HRP to reduce interference. PAA oxidizes 2,2-carbazole bis (3-ethylbenzothiazoline-6-sulfonate (ABTS)) and forms free radical cations (ABTS$^+$) which can be measured at 405 nm, as shown in Eq. (14).

$$\text{ABTS} + \text{H}_{13}\text{N}_3\text{O}_2 \stackrel{\text{HRP}}{\rightarrow} \text{C}_17\text{H}_15\text{N}_3\text{O}_2 + 2\text{H}_2\text{O} + \text{2CH}_3\text{COOH}$$

Gas and liquid chromatography techniques have high selectivity and low detection limits (about 0.1–10 mg/L), making them easily adaptable to different sampling conditions by diluting samples or reagents. Compared with the titration method, the method is characteristic of low detection limit and long storage time after derivatization (at least 1 week).

3.4 Colorimetric methods

Colorimetric methods can detect compounds with significant chromogenic groups, which can be formed by direct complexation, addition or related reaction of the sample by reagents. The formed compounds were quantitatively analyzed by the Lamber-Beer law with a UV-visible absorption technique. This method requires a fast and selective reaction between the reagent and the target compound in a specific concentration range. The colorimetric method generally only allows the measurement of compounds at low concentrations (0.1–10 mg/L), which is suitable for environmental analysis. Many enzyme-related biochemistry methods and specialized kits for biological sample detection have also been developed based on such processes (Zhu et al., 2014). For instance, Abcam corporation (USA) produced ab102500 H$_2$O$_2$ assay kit with OxiRed probe, which can be used for rapid determination of hydrogen peroxide in biological samples.

Similarly, PAA can also be measured using colorimetry. PAA has strong oxidizing properties and can undergo the Tringer (Zhang et al., 2000) reaction (in phosphate buffer solution) with phenol and 4-aminoantipyrine under the enzymatic catalysis. The reaction produces a red anthraquinone-based compound which has a maximum absorption wavelength at 505 nm. By measuring the absorbance at 505 nm and calculating the amount of the anthraquinone-base compound produced, the PAA content can be deduced (Eq. (13))

$$2\text{CH}_3\text{COOOH} + \text{C}_6\text{H}_5\text{OH} \rightarrow \text{C}_17\text{H}_15\text{N}_3\text{O}_2 + 2\text{H}_2\text{O} + \text{2CH}_3\text{COOH}$$

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Another coloring method is the measurement of PAA by reacting with DPD (N,N-diethyl-p-phenylenediamine). The detection method of DPD (N,N-diethyl-p-phenylenediamine) in wastewater was first introduced by Palin (1957). For many years, it has been the most commonly used method for determining free chlorine and total chlorine in water. Similar to the DPD chlorine oxidation
reaction, the primary oxidation product of the DPD-PAA oxidation reaction at a near-neutral pH is a semiquinone type cationic compound called Würster dye. This relatively stable free radical species exhibits a magenta color, the main reaction process is shown in Fig. 2.

![Fig. 2 DPD-PAA oxidation products.](image)

The absorption spectrum of Würster dyes is bimodal with peaks at 512 and 553 nm, which can be detected at both wavelengths (Buschini et al., 2004; Antonelli et al., 2006). At higher oxidant levels, an unstable colorless imine is primarily formed and causes the solution to "fade" significantly. In addition, both PAA and H₂O₂ have oxidizing power for potassium iodide. Therefore, this method cannot simultaneously measure PAA and H₂O₂ (see Eqs. (7) and (8)). In general, the interference of H₂O₂ can be removed by adding catalase before the start of the oxidation reaction (Cavallini et al., 2013).

3.5 Online detector and quick measurement devices

PeroxyChem recommends the use of a CHEMetric I 2020 unit analyzer (SAM) based on DPD-PAA oxidation to measure low concentrations (0.25–5 mg/L) of PAA (Fig. 3) (Chemetrics, 2019).

![Fig. 3 CHEMetric I2020 Unit Analyzer.](image)

The I-2020 SAM system claims an accuracy of ±20% at the low end of the detection range and ±0% at the detection limit. HydroQual HDR investigated the accuracy of the I-2020 SAM system, measured different concentrations of PAA solution, and analyzed the standard deviation of all measurements for each PAA concentration for the average measured PAA concentration. The maximum uncertainty in the SAM measurement is then determined by the boundary of the confidence interval set by the measured standard deviation. A regression analysis was performed to correlate the SAM measurement uncertainty with the average PAA concentration: for 1, 0.5 and 0.25 mg/L concentration of PAA solution, the measurement inaccuracy was ±3%, ±6%, and ±15%, respectively (Peroxychem, 2014).

Electrochemical potential analysis refers to an analytical method for measuring the content of a substance by measuring the potential of the electrode. The potential analysis uses a pointing electrode and a reference electrode to form a chemical battery with the test solution, and the electromotive force of the battery is measured under zero current conditions, and the analysis is performed accordingly. The activity or concentration of the substance can be determined by the measured electrode potential. A relatively simple potential analysis method has been reported in the literature for the simultaneous determination of PAA and H₂O₂. Similar to redox titration, this method is based on the instantaneous change in electrode potential during the reaction of PAA with iodide. The uncatalyzed reaction rate of PAA and iodide ion is 5 orders of magnitude higher than that of iodide ion and H₂O₂ (Awad et al., 2003). This method produces an electrical response in a very short time with a low detection limit (approximately 0.076 mg/L in the detection limit μmol/L range) (Awad and Ohsaka, 2003). PAA and H₂O₂ can be detected at the same time, because the response time of the potential change of PAA and H₂O₂ is quite different (a few seconds and a few minutes respectively). This method is suitable for PAA solutions containing a large amount of H₂O₂ (the highest concentration ratio of H₂O₂ and PAA is 165, as shown in Fig. 4).

![Fig. 4 Different concentrations of H₂O₂ (PAA concentration maintained at 0.041 mmol/L) were added to a 0.05 mol/L acetate buffer solution (pH 5.4) containing 10.0 mmol/L I⁻ and 0.010 mmol/L I₃⁻, indicating the potential change of the electrode. (a) 0.012 mmol/L, (b) 2.7 mmol/L, and (c) 6.8 mmol/L (H₂O₂), respectively (Awad and Ohsaka, 2003), Reprint with permission copyright by Clearance Center’s RightsLink® service.](image)
The method is simple and easy to operate and does not require secondary processing of the sample. In this process, pH may greatly affect the reaction rate of H₂O₂ and I⁻ while the reaction of PAA and I⁻ will not be interfered (in the range of pH 3–6). With the increase of pH, the reaction between H₂O₂ and I⁻ becomes slow. To reduce the total response time, selecting the appropriate pH can increase the selectivity of the potential analysis.

Online, real-time, continuous measurement of PAA can be achieved with commercially available in situ probes. The DULCOTEST® Sensors Peracetate Probe is a membrane-covered Ampere two-electrode detector (Fig. 5) for continuous measurement of PAA in wastewater (Prominent, 2011). The electrode uses gold as a cathode, a silver halide coated anode as a reference electrode, and PAA contained in the sample water diffuses through the membrane. The constant polarization voltage applied between the two electrodes causes an electrochemical reaction of PAA at the working electrode. The measured current is used as the main signal for detection (current measurement principle), which is proportional to the PAA concentration near the working electrode. The probe was calibrated using a CHEMetrics I-2020 Single Analyzer (SAM) to provide a reference PAA concentration (Che- metrics, 2019).

To provide greater intuition, we have summarized the methods described above in a table (Table 1).

### 4 Discussion & conclusions

PAA has been widely used as a disinfectant and oxidant in all aspects of production and life. The complex composition of commercial PAA and the specificity of PAA
increase the difficulty in the quantitative analysis of PAA. In this paper, based on the physical and chemical properties of PAA, different detection methods of PAA using different systems are comprehensively discussed, and the principle, adaptability, and relative merits of these methods are compared and analyzed.

Redox titration is the most basic method compared to various methods of determining PAA described above. However, the measurement results of this method are susceptible to interference from various factors (such as pH, analyte concentration, etc.). Moreover, the detection limit is higher than other methods, so that it is not suitable for the measurement of residual PAA solution, which is why it is preferred for a higher concentration PAA stock solution. For low concentrations of PAA solution, the available analytical methods are nuclear magnetic, chromatographic, potentiometric, and colorimetric techniques. These methods have low detection limits and are relatively easy to operate. However, due to the coexistence of PAA and H2O2 in water samples, especially at low concentrations, PAA is extremely unstable, so improving the selectivity of these methods has become the key to control analysis accuracy.

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