Analytical Method for Peracetic acids

Photometric determination of low concentrated peracetic acids with ABTS

GENERAL INFORMATION ABOUT THE METHOD

The photometric determination of the peracetic acid content is preferably used for low concentrated peracetic acid products (e.g. PERACLEAN® 0.1 or PERACLEAN® 0.4).

ABTS is oxidized selectively by peracetic acid to an intensely green radical cation. The extinction measured with a spectrophotometer is linear with the peracetic acid concentration in a range of 0.2 – 10 mg/l . The absorption spectrum allows measuring the extinction at different wavelengths. The preferred wavelength in this description is 405nm. For the measurement, a commercial spectrophotometer with a 405nm filter is required.

EQUIPMENT

- analytical balance
- spectrophotometer with filter 405 nm
- 1cm cuvettes
- volumetric flasks 10ml, 100ml, 200ml, 500ml and 1000ml
- microliter pipettes 500µl, 1000µl, 1500µl, 2000µl, microliter tips
- pipettes, 10ml

REAGENTS

- peracetic acid solution (testing material)
- 2,2-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS) solution c (ABTS) = 1 g/l
- Acetic acid solution p.a. c(CH3COOH) = 1 mol/l
- Potassium iodid solution p.a. c(KI) = 100 mg/l
- PERACLEAN ® 15 (for preparation of a calibration solution)
- high purity water (osmosis and ion exchange treated drinking water

SPECIAL SAFETY INSTRUCTIONS

All reagents and chemicals must be handled according to the health and safety regulations. Refer to the safety data sheets.

SPECIAL PROCEDURE INSTRUCTIONS

Danger of decomposition by contact with incompatible materials, contaminants, metals, alkalis, reducing agents.

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PROCEDURE

Calculation of the conversion factor from extinction to concentration (Calibration):

Single-point calibration (quick method)

First, a predilution of PERACLEAN ® 15 is made by pipetting exactly 10 ml of PERACLEAN ® 15 in a 500ml volumetric flask and filling it up with high purity water. Immediately after preparing the predilution, the peracetic acid content *C1* is determined. The determination is carried out by titration. The titration takes place in 2 steps, in which first the hydrogen peroxide content is determined, directly followed by the peracetic acid determination. The hydrogen peroxide content is no longer required in the further course of the calibration. The peracetic acid content *C1* is calculated in mg/l. The complete calculation formula can be found in the description of the titration method. If the analysis is carried out according to this method, 10ml of the predilution must be used as sample volume for the two-step titration (note the point LITERATURE at the end of this document!).

For preparing a calibration solution, pipette 1.5ml (1500µl) of the predilution into a 500ml volumetric flask and fill up the flask with high purity water. To determine the calibration factor, this solution is used to carry out the photometric measurement described on the following page. Pay close attention to the work steps sequences! The measured value obtained is the extinction *E1*.

With the titrated peracetic acid content *C1* [mg/l] of the predilution and the measured extinction *E1* of the calibration solution the conversion factor *F* (PAA) is calculated as follows:

$$\textbf{Conversion factor } \boldsymbol{F}\ \textbf{(PAA)} = \frac{\boldsymbol{C1}\ \textbf{[mg/l]}}{\boldsymbol{E1} * \textbf{333.33}}$$

The complete procedure should be repeated at least 3 times. Any dilution must be carefully prepared. The Titration or the photometric measurement must be carried out immediately after preparing the dilution.

The conversion factor must be determined for each photometer used.

Multipoint calibration (creation of a calibration curve)

The multipoint calibration is the appropriate procedure if the entire measuring range from 0.2 - 10 mg/l peracetic acid should be used.

To perform a multipoint calibration, the calibration solution already prepared for the single-point calibration can be used. This solution has to be further diluted several times (e.g. 1:3 and 1:2 and 1:1 and 2:1 and 3:1). Immediately afterwards, the photometric measurement described on the following page has to be carried out of each dilution. With the measured extinction and the calculated peracetic acid content of each dilution a calibration curve can be created and the conversion factor for the entire measuring range can be calculated.

The complete procedure should be repeated at least 2 times. Every dilution should be prepared carefully and be measured immediately after preparing.

Calibration curve and conversion factor must be determined separately for each photometer used

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Photometric determination of peracetic acid content with ABTS:

Dilution of the present peracetic acid sample

When determining the peracetic acid content of PERACLEAN® products using the ABTS method, it is necessary to dilute the product samples in order to reach the measuring range of the method.

The dilution should have a peracetic acid concentration of preferably 2 - 10 mg/l, then the measured extinctions will be in a range of approx. 0.2 - 0.8. Make sure that the extinctions are always <1.

Basically, the method can be used to measure peracetic acid contents of 0.2 - 10 mg / l.

To prepare the dilution, weigh a defined amount of the sample on an analytical balance and transfer it quantitatively into an appropriate volumetric flask. Then fill up the flask with high purity water to the mark and note the weight to the nearest 0.1mg (sample weight).

The following weights or dilutions are recommended for low concentrated PERACLEAN® products:

PERACLEAN® 0,1	dilute 500 mg to 100 ml in a volumetric flask (dilution factor 200)
PERACLEAN® 0,25	dilute 200 mg to 100 ml in a volumetric flask (dilution factor 500)
PERACLEAN® 0,4	dilute 100 mg to 100 ml in a volumetric flask (dilution factor 1000)
PERACLEAN® 1 Foam	dilute 100 mg to 200 ml in a volumetric flask (dilution factor 2000)

Photometric measurement

Place 1 ml (1000 µl) of the diluted peracetic acid sample in a clean 10 ml volumetric flask. Add 2 ml of the acetic acid solution, 0.5 ml of the potassium iodide solution and 1 ml of the ABTS solution in succession. Then made up the volumetric flask to 10 ml with high purity water and mix well.

After a reaction time of 15 minutes, transfer the now green colored sample solution into a 1cm cuvette and place it in the measuring cell of the photometer. Measure the extinction of the sample at 405nm and note the value *E* (sample).

To determine the required zero value, a blank must also be measured. The blank sample is prepared in exactly the same way, but without addition of peracetic acid solution (chemical blank).

The photometric measurement of the blank must be carried out before the measurement of the sample (note the instructions of the device manufacturer!).

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CALCULATION

$$\textbf{Peracetic acid}\ [\textbf{mg/kg}] = \frac{\boldsymbol{E}\ (\textbf{sample}) * \boldsymbol{F}\ (\textbf{PAA}) * \textbf{V Volumetric flask}\ [\textbf{ml}] * \textbf{1000}}{\textbf{sample weight}\ [\textbf{mg}]}$$

$$\textbf{Peracetic acid}\ [\textbf{wt\%}] = \frac{\boldsymbol{E}\ (\textbf{sample}) * \boldsymbol{F}\ (\textbf{PAA}) * \textbf{V Volumetric flask}\ [\textbf{ml}]}{\textbf{sample weight}\ [\textbf{mg}] * \textbf{10}}$$

ENVIRONMENT/DISPOSAL OF CHEMICALS

The disposal of laboratory quantities of hydrogen peroxide must be in accordance with local regulations.

LITERATURE

- Product information "Peracetic Acids", e.g. PERACLEAN® 15
- Determination of hydrogen peroxide and peracetic acid content by titration
- (Analytical Method for Peracetic acids)
- Manufacturers equipment descriptions
- The Analyst, June 1997, Vol. 122 (567-571), Ulrich Pinkernell, Hans-Joachim Lüke, Uwe Karst

  Westfälische Wilhelms-Universität, Münster, Germany

  "Selective Photometric Determination of Peroxycarbolic Acids in the Presence of Hydrogen Peroxide"

REMARKS

The method is based on the internal analytical method PA-198 [609/PM13].

Evonik Operations GmbH
Active Oxygens
Rodenbacher Chaussee 4
63457 Hanau, Germany
PHONE +49 6181 59-3295
FAX +49 6181 59-73295
www.active-oxygens.com

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The peroxide experts at Evonik

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