

## Method of Test for Persulfates in Denture Cleansers

### 1. Scope

This method is applicable to the determination of persulfates in denture cleansers.

### 2. Method

After extraction, persulfates are determined by high performance ion chromatography (HPIC).

#### 2.1. Equipment

##### 2.1.1. High performance ion chromatograph.

2.1.1.1. Detector: conductivity detector.

2.1.1.2. Column: IonPac<sup>®</sup> AS16, 5 µm, 4 mm i.d. × 25 cm, or an equivalent product.

2.1.1.3. Guard column: IonPac<sup>®</sup> AS16, 5 µm, 4 mm i.d. × 5 cm, or an equivalent product.

2.1.1.4. Anion self-regenerating suppressor: ASRS 300, or an equivalent product.

##### 2.1.2. Ultrasonicator.

#### 2.2. Chemicals

50% (w/w) sodium hydroxide solution, GR grade;

Deionized water, resistivity ≥ 18 MΩ · cm (at 25°C);

Potassium persulfate, reference standards.

#### 2.3. Apparatus

2.3.1. Volumetric flask: 20 mL and 100 mL.

2.3.2. Membrane filter: 0.22 µm, Nylon.

#### 2.4. Eluent

Dilute 6.86 mL 50% (w/w) sodium hydroxide solution with deionized water to 2000 mL, and filter with a membrane filter. Take the filtrate as the eluent.

#### 2.5. Standard solution preparation

Transfer about 28.1 mg of potassium persulfate reference standards into a 20 mL volumetric flask, dissolve and dilute with deionized water to the volume as the standard stock solution (equivalent to S<sub>2</sub>O<sub>8</sub><sup>2-</sup> 1000 µg/mL). When to use, dilute with deionized water to 2-25 µg/mL as the standard solutions.

#### 2.6. Sample solution preparation

Transfer about 0.5 g of the homogenized and well-mixed sample accurately weighed into a 100 mL volumetric flask. Add 80 mL of deionized water, and sonicate for 30 min. Add deionized water to the volume. Filter with a

membrane filter, and take the filtrate as the sample solution.

## 2.7. Identification and quantification

Accurately inject 25 µL of the sample solution and the standard solutions into HPIC separately, and operate according to the following conditions. Identify persulfates based on the retention time. Calculate the amount of persulfate in the sample by the following formula:

The amount of persulfate in the sample (%) =  $\frac{C \times V}{M} \times 10^{-4}$

where,

C: the concentration of persulfate in the sample solution calculated by the standard curve (µg/mL)

V: the final make-up volume of sample (mL)

M: the weight of sample (g)

HPIC operating conditions<sup>(note)</sup> :

Detector: Conductivity detector.

Column: IonPac® AS16, 5 µm, 4 mm i.d. × 25 cm.

Guard column: IonPac® AS16, 5 µm, 4 mm i.d. × 5 cm.

Anion self-regenerating suppressor: ASRS300, 4 mm.

Eluent: As described in section 2.4.

Flow rate: 1.0 mL/min.

Note: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

## Remark

1. Limits of quantification (LOQ) is 0.04%.
2. Further validation should be performed when interference compounds appear in samples.

## Reference

Huang, Z., Ni, C., Wang, F., Subhani, Q., Wang, M. and Zhu, Y. 2014. Simultaneous determination of peroxydisulfate and conventional inorganic anions by ion chromatography with the column-switching technique. J. Sep. Sci. 37: 198-203.

## Reference chromatogram

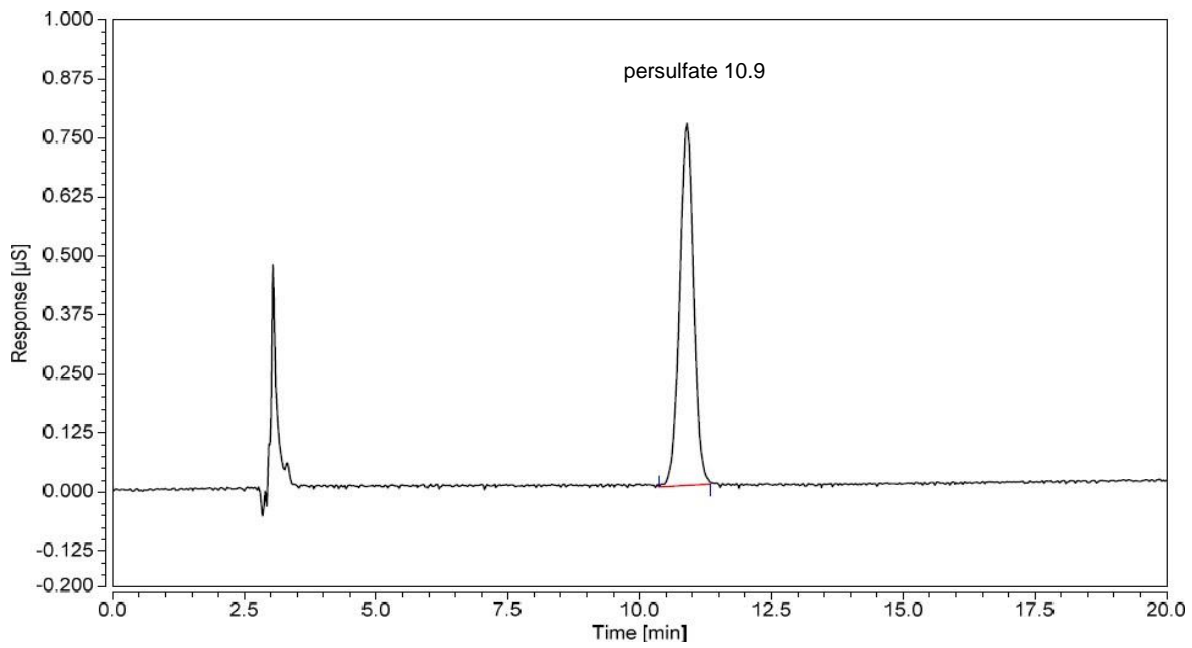


Figure. HPIC chromatogram of persulfate.