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Determination of Phosphoric Acid in the Air of Workplace by Ion Chromatography

XIA NING* and JI-HUA ZHANG

Zhangqiu CDC of Shandong Province 250200, P.R. China

*Corresponding author: E-mail: sdzqnx@163.com

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In this work, a method is established to determine phosphoric acid in the air of workplace by ion chromatography. The phosphoric acid in the air of workplace was collected with membrane filter and then eluted with water. The eluent was directly injected to the after filtration with 0.22 μm membrane. The signals were detected by ion chromatography with conductivity detector. The separation and determination of the process was completed within 12 min at the flow rate of 1.00 mL/min using the eluent of KOH (30 mmol/L). The retention time of phosphoric acid was 10.507 min. In addition, in the range of 0.50-10.0 μg/mL a good linear relationship was obtained and the correlation coefficient $r > 0.999$, the detection limit was 0.004 μg/mL and the minimum detectable concentration was 0.003 mg/m^3 (to collect 75 L air sample count).

Key Words: Phosphoric acid, Ion chromatography.

INTRODUCTION

Phosphoric acid is a colourless oily liquid. The phosphoric acid vapour or mist has irritation to eyes, nose and throat and also its liquid can cause skin or eye burns. Its existence form in the air is aerosol. The permissible concentration-time weighted average (PC-TWA) of phosphoric acid exposure limits in the workplace air is 1 mg/m^3 and the permissible concentration-short term exposure limits (PC-STEL)[1,2] is 3 mg/m^3. At present, the national standard determination method for phosphoric acid in the air of workplace was ammonium molybdate spectrophotometric method[3]. However, the standard series had no changes of gradient and using a lot of harmful chemical reagents during detecting phosphoric acid with this method. In present study, we used microporous membrane filter to collect samples of phosphoric acid in the workplace air, then eluted with water and detected with ion chromatography, without using any toxic chemical reagents. The proposed method was used for the determination of phosphoric acid in the workplace air with a fast analysis, low toxicity reagents, high sensitivity and selectivity, less interference, simple operation and well repeatability.

EXPERIMENTAL

The phosphoric acid mist in the air was collected with microporous membrane, eluted with water, separated by the column chromatography, detected by conductivity detector, qualitative by the retention time and quantitative by the peak area.

Microporous membrane filter, aperture 0.8 μm; sampling clip, filter diameter 40 mm; small plastic sampling clip, filter diameter 25 mm; air sampler, flow rate 0-3 L/min and 0-10 L/min; Plug tube, 10 mL and 50 mL; volumetric flask, 50 mL; beaker, 50 mL; thermostatic waterbath; Φ13 mm sample filter, nylon 0.22 μm microporous membrane filter; ion chromatography: ICS-2000 ion chromatography (American dianne company), Chromelen chromatogram workstation.

Anionic analysis column: Dionex IonPac AS19 (4 mm × 250 mm), anionic protection column: Dionex IonPac AG19 (4 mm × 50 mm); suppressor: ASRS 4 mm; detector: conductivity detector; Eluent: 30 mmol/L KOH isocratic elution (using KOH eluent tank); flow rate of eluent: 1.00 mL/min; suppressor current: 75 mA; injection volume: 25 μL; column temperature: 30 ºC. The conductivity of the experimental water was lower than 1.0 μs/cm.

Standard solution: Accurately weighed 0.1389 g dried potassium dihydrogen phosphate (KH_2PO_4) and dissolved it into water, then transferred the solution into the 100 mL volumetric flask quantitatively and diluted to the mark. It was the standard stock solution of 1.00 mg/mL. The stock solution was diluted into100.0 μg/mL phosphate standard solution with water before using. We could also prepare it with the national recognized standard solution.

Short time sampling: Air samples were collected using a sampling clip with 5 L/min flow rate for 15 min at sampling points.

Long time sampling: Air samples were collected using a small plastic sampling clip with 1 L/min flow rate for 4-8 h at sampling points.

Personal sampling: A small plastic sampling clamp equipped with a microporous membrane, was worn on upper chest of the sample object in order to make the air intake as close as possible to the breathing zone. The flow rate of collecting air samples was 1 L/min for 2-8 h.

After collecting samples, the surface of the membrane exposed to dust was folded two times and then the folded membrane was put into a test tube with a plug for further transport or storage. The samples could be kept for 7 days at least at room temperature.

Control experiment: As a control, all the approach was the same with the above sample except not connected with the air sampler collecting air samples.

Samples preparation: After removing the sampled membrane from the test tube with a plug to a beaker, the tube was washed with 10 mL water and the washing liquid was also transferred into the beaker. When the membrane in the beaker was flattened and soaked in water, the beaker was covered with a vessel and heated in a boiling water bath for 10 min. The eluate was transferred into a 50 mL colorimetric tube quantitatively after cooling. Then washed the membrane and the beaker with *ca.* 20 mL waters for several times and put the washing liquid into the colorimetric tube. After being settled to permit and mixed adequately, the sample solution was filtrated with a sample filter for further determination. The eluate could be diluted if the concentration of phosphoric acid was over the range of measuring. And the dilution factor could be multiplied when calculated.

Standard curve: 0.25, 1, 2.5, 5 mL phosphoric acid standard solutions were added into five 50 mL volumetric flasks separately and then were all settled to permit with water to get 0.5, 2, 5, 10 μg/mL phosphoric acid standard series. The ion chromatograph was adjusted to the optimum determination conditions in accordance with the instrument operating conditions. The flow rate of eluent was 1.00 mL/min and the sample volume was 25 μL. Each point of the standard series was repeated determined 3 times and a standard curve could be drawn according to the peak area and the corresponding concentration of phosphoric acid (μg/mL).

Determination of samples: The sample solution and the control solution were determined using the operating conditions for the determination of the standard series. And then the peak areas of the sample solution and the control solution could be determined. The phosphate concentration could be obtained from the standard curve and the differences of the peak area value between the sample and the control.

Calculation: The sampling volume was converted into standard sample volume according to the following eqn. 1 if the temperature of the sampling point was below 5 ºC or above 35 ºC and the atmospheric pressure was below 98.8 KPa or above 103.4 Kpa.

$$V_0 = V \times \frac{293}{273 \times t} \times \frac{P}{101.3} \qquad (1)$$

In the eqn. 1: V_0: Standard sample volume, L; V: Sampling volume, L; t: Temperature of the sampling point, ºC; P: Atmospheric pressure of the sampling point, KPa.

The concentration of phosphoric acid in the air of workplace was calculated according to eqn. 2:

$$C = \frac{50\ c}{V_0} \qquad (2)$$

In the eqn. 2: The concentration of phosphoric acid (C) in the air of workplace, mg/m^3; Elution volume, 50 mL; The measured value of phosphoric acid in the sample solution, g/mL (c); Standard sample volume, L (V_0).

RESULTS AND DISCUSSION

Linear range and detection limit: The linear relationship was well if the concentration of the phosphoric acid ranged from 0.50-10.0 μg/mL and the correlation coefficient of the standard curve was above 0.999. The detection limit was 0.004 μg/mL. The minimum detection concentration was 0.003 mg/m^3 when the sample volume was 75 L.

Precision of the method: The standard curve was determined 6 times continuously and the relative standard deviation was 1.30-3.25 %.

TABLE-1
RESULTS OF THE PRECISION EXPERIMENT

Frequency	Value of peak areas of the standard curve (μS × min)				r
	0.50 (μg/mL)	2.00 (μg/mL)	5.00 (μg/mL)	10.0 (μg/mL)	
1	0.022	0.120	0.281	0.604	0.9993
2	0.022	0.114	0.276	0.600	0.9992
3	0.023	0.123	0.286	0.608	0.9994
4	0.022	0.124	0.290	0.623	0.9993
5	0.023	0.120	0.282	0.610	0.9992
6	0.022	0.116	0.280	0.606	0.9993
Mean	0.022	0.120	0.282	0.608	0.9993
S	0.0005	0.0039	0.0049	0.0079	–
cv (%)	2.27	3.25	1.74	1.30	–

S: Standard deviation. cv: Coefficient of variation.

Accuracy of the method: A certain amount of standard solution was added to the blank microporous membrane and the solution was detected as the samples for high, medium and low concentrations of spiked recovery test. Each level was detected 6 times separately. The standard recovery rates were in the range of 97.0-105.0 %.

Stability experiment of the storage sample: The experimental results showed that, the concentration of the phosphoric acid in the samples which stored at room temperature decreased less than 10 % when being measured at the 1st, 3rd, 5th, 7th day, respectively. It indicated that the samples could be stored at least 7 days at room temperature.

Interference studies: Other anions such as F^-, Cl^-, NO_2^-, SO_4^- and NO_3^-, had no interference in the determination of phosphate under this condition. Results of the interference experiment showed in Fig. 1.

This study showed that the precision and accuracy of the results conformed to the requirements of the experiment by collecting samples with microporous membrane filter, eluting with water and being detected with ion chromatography. It is provided with many advantages such as simple operation, high stability, high sensitivity and good selectivity, less interference, low cost, without chemical reagent and only 12 min to complete

TABLE-2
RESULTS OF THE STANDARD RECOVERY RATES

Spiked value	Determined value						Mean	Recovery rate (%)
3.00	2.82	3.01	3.00	2.85	2.91	2.86	2.91	97.0
5.00	5.17	5.05	5.26	5.30	5.18	5.10	5.18	103.6
7.00	7.52	7.49	7.14	7.16	7.39	7.42	7.35	105.0

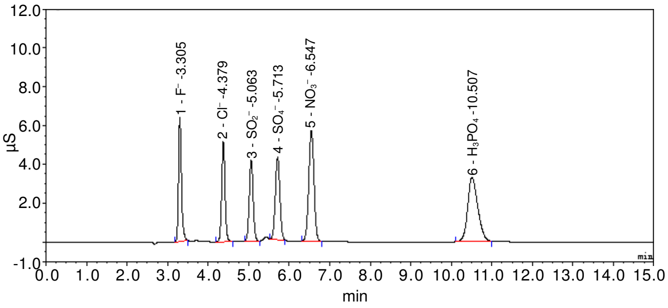

Fig. 1. Chromatogram of interference experiment

a sample analysis *etc*. In short, it was an ideal analytical method for determination of phosphoric acid in the air of workplace.

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