Application Work AW IC CH6-1124-0912

Determination of sulfite in ambient air by absorption and Ion Chromatography

Branch

Water, waste water, air, environmental protection

Keywords

IC; 850; conductivity detection; 920; Absorber Module; absorption; Metrosep A Supp 5 - 150/4.0; Metrosep A Supp 4/5 Guard/4.0; sulfate; air; matrix elimination; branch 2

Summary

This application work describes the determination of gaseous sulfite in air. Air is sampled through a vacuum pump into the absorption vessel of the 920 Absorber Module where gaseous sulfite is oxidized to sulfate by the means of a hydrogen peroxide solution. An aliquot of this absorbing solution is transferred into the IC system for analysis.

Samples

Air samples were collected for thirty minutes in hydrogen peroxide solution at a gas flow rate of 231 mL/min.

Instruments

850 Professional IC Anion - MCS - Prep 2	2.850.2150
IC Conductivity Detector	2.850.9010
Metrosep A Supp 5 - 150/4.0	6.1006.520
Metrosep A Supp 4/5 Guard/4.0	6.1006.500
920 Absorber Module	8.920.8002
Metrosep A PCC 1 HC/4.0	6.1006.310
MagIC Net 2.4™ Professional	6.6059.242
KNF Laboport N 840 FT.18 Vacuum pump	-
Zephyr 500 HR digital gas flow meter by Phenomenex	-
red-y gas flow meter by Voegtlin	-
Software get red-y 5 by Voegtlin	-



Reagents

Standards for IC, TraceCERT®, from Sigma-Aldrich Sulfate, $\beta(SO_4^{2^-}) = 1$ g/L in H₂O, No.90071

Sodium carbonate (Na₂CO₃), puriss. p.a., CAS 497-19-8 Sodium bicarbonate (NaHCO₃), puriss. p.a., CAS 144-55-8

Hydrogen peroxide solution (30%), TraceSELECT $\ensuremath{\mathbb{R}}$ Ultra from Sigma Aldrich, No 16911

Ultrapure water, resistivity >18 M Ω ·cm (25 °C), type I grade (ASTM D1193)

Sulfuric acid, H_2SO_4 , ultrapure 96%, CAS 7664-93-9, Sigma Aldrich 84720

Solutions

A stock solution with $c(H_2SO_4) = 2 \text{ mol/L}$ sulfuric acid was prepared out of the concentrated sulfuric acid.

Eluent	c (Na ₂ CO ₃) = 3.2 mmol/L	
	c (NaHCO ₃) = 1.0 mmol/L	
Supressor solution	100 mM H ₂ SO ₄	
Absorbing solution	100 ppm H ₂ O ₂	

Standard solutions

β [µg/L]	Std. 1	Std.2	Std.3
SO4 ²⁻	1	20	80

Standards were prepared out of a stock solution with a concentration of 1000 ppb sulfate.

Sample preparation

The PTFE-tubing coming from the absorption vessel was connected with a PVC tubing by four Oetiker hose clamps ("7 OET"). The PVC tubing was lead out of the window for sampling purposes. A connector was set on top of the absorption vessel and the two gas flow meters were put inline between the absorption vessel and the vacuum pump. By running the vacuum pump air was drawn through the sample tubing and the solution in the absorption vessel. Air flow was adjusted by the red-y gas flow meter of company Voegtlin and the corresponding software. The value for air flow was read off Phenomenex's gas flow meter.

2 mL of absorbing solution were set into the absorption vessel in advance of each analysis by the 10 mL-Dosino of the 920 Absorber Module. Sampling time was thirty minutes.

Analysis

Sample transfer to the IC system was performed by the 5 mL-Dosino of the 920 Absorber Module after sampling time was finished. Loop volume of 6-port-valve at the 920 Absorber Module was 250 μ L. The loop was completely filled with sample-containing absorbing solution (full loop).

Matrix elimination was achieved by flushing the sample onto the pre-concentration column at the IC side prior to separation.

Parameters

Flow rate eluent	0.7 mL/min
Temperature column	30°C
Recording time	20 min
Injection volume	250 µL
Data source	Conductivity detector
Channel	Conductivity
Temp. coefficient	2.3 %/°C
Polarity	+

Evaluation

Evaluation was performed using MagIC Net[™] 2.4 software based on automatic integration of the peak area.

The total mass of SO₂ in absorbing solution is calculated by MagIC NetTM 2.4 as a user-defined result in µg. The volume of air is achieved by multiplying the gas flow [mL / min] with the sampling time. The division of total mass of SO₂ by gas volume multiplied with a factor 1'000'000 leads to the amount of SO₂ in µg per m³.

Results

In total, 135 determinations were performed over a time period of five days. Weather was sunny with practically no wind. Values for SO_2 are smaller at night times and increase during day with a maximum in the afternoon. At the beginning of 19th September it started to rain resulting in lower values for SO_2 during daytime.

Comments

With the use of a vacuum pump and the 920 Absorber Module it is possible to sample air. In combination with a Metrohm IC system quantification of sulfite can be performed without further sample preparation.

Date

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Appendix

Result table of chosen determinations

Determination start	m SO ₂ [ug]	SO ₂ [ug/m3]
14.09.2012 16:22	0.054	7.8
15.09.2012 01:33	0.012	1.7
15.09.2012 16:14	0.040	5.8
16.09.2012 00:30	0.004	0.6
16.09.2012 17:02	0.041	5.9
17.09.2012 04:03	0.008	1.2
17.09.2012 16:55	0.072	10.4
18.09.2012 03:01	0.010	1.4
18.09.2012 16:48	0.074	10.7
19.09.2012 08:25	0.009	1.3

Graphical evaluation



time course of SO₂

Example sample chromatogram

(2 mL absorbing solution; 231 mL / min air flow)



Calibration

