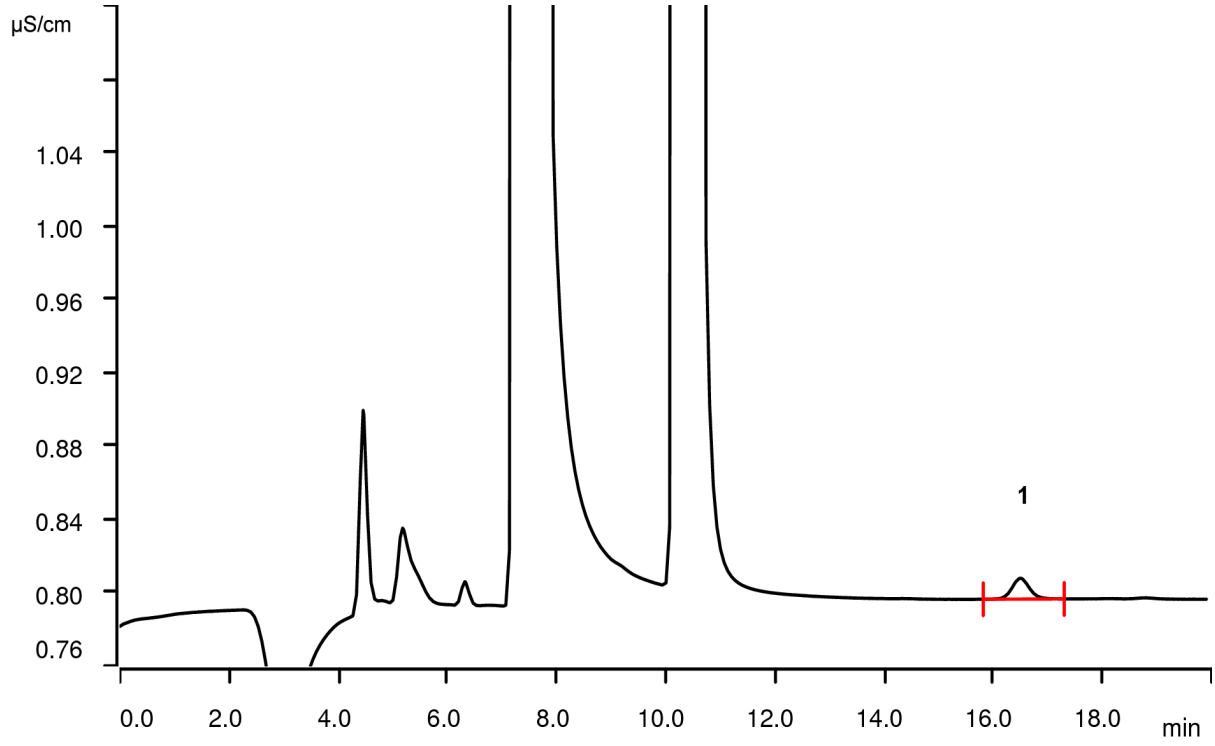
IC Application Note CIC–031

Sulfur determination in ammonia gas applying Combustion IC

Sulfur species in ammonia gas analyzed as sulfate after pyrohydrolysis by IC on a Metrosep A Supp 5 - 150/4.0 column.



Chromatogram of the absorber solution after pyrohydrolytic combustion of ammonia gas and the determination of the hydrolyzed combusted sulfur species as sulfate.

Sulfur species are critical contaminants in ammonia gas. They can cause high-temperature sulfidation of metals, form aggressive complexes with other elements, or react subsequently in processes where the ammonia gas is used. The concentration of such impurities tends to be very low, but they may not exceed critical levels of 0.5 mg/L. Although this level is very close to the system blank of the Combustion IC system, the setup can be used to prove that such critical limits are not exceeded.

Results

		Concentration (absorber sol.) [mg/L]	Concentration (gas) [mg/kg]
1	Sulfur	0.003	0.2

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Sample

Ammonia gas.

Sample preparation

Injection of 200 µL of ammonia gas directly into the combustion oven. Absorption of the combustion gases is followed by the subsequent injection to the ion chromatograph applying the intelligent Partial Loop Injection Technique with Inline Matrix Elimination.

Columns

Metrosep A Supp 5 - 150/4.0	6.1006.520
Metrosep A Supp 5 Guard/4.0	6.1006.500
Metrosep A PCC 2 HC/4.0	6.1006.340
Metrosep I Trap 1 - 100/4.0	6.1014.200
Metrosep A Trap 1 - 100/4.0	6.1014.000

Solutions CIC

Eluent	3.2 mmol/L sodium carbonate 1.0 mmol/L sodium hydrogen carbonate
Suppressor regenerant	500 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	100 mg/L hydrogen peroxide

Analysis

Conductivity after sequential suppression

Parameters

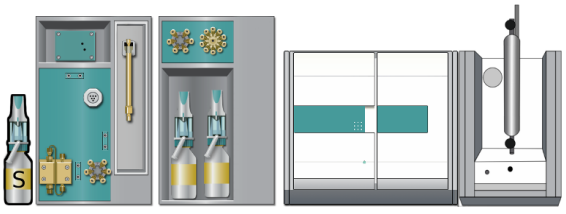
Flow rate	0.7 mL/min
Injection volume (IC)	200 µL (MiPT)
P_{max}	15 MPa
Recording time	20 min
Column temperature	30 °C

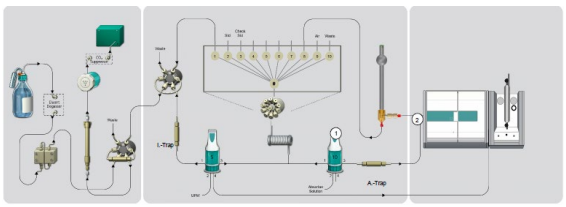
Combustion parameters

Argon	100 mL/min
Oxygen	300 mL/min
Oven temperature	1050 °C
Injection volume sample	200 µL
Post-combustion time	60 s
Post-cooling time	420 s
Initial volume of absorption solution	2.0 mL
Absorber solution feed	0.2 mL/min
Water inlet	0.2 mL/min
Post-combustion rinsing volume	1.0 mL

Instrumentation

930 Compact IC Flex Oven/SeS/PP/Deg	2.930.2460
IC Conductivity Detector	2.850.9010
MSM Rotor A	6.2832.000
Adapter sleeve for Suppressor Vario	6.2842.020
800 Dosino (regeneration)	2.800.0010
IC equipment: Dosino Regeneration	6.5330.190
920 Absorber Module	2.920.0010
Combustion Oven	2.136.0750
LPG Module	2.136.0740





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Version 1, published in April 2020