IC Application Note CIC-13

Analysis of certified LPG applying Metrohm Combustion IC with a nested method setup



This Application Note looks at the determination of fluorine and sulfur in certified liquefied petroleum gas (LPG) applying Metrohm Combustion IC. The determination sequence is set up in nested mode: while the absorption solution of the previously combusted sample is analyzed via IC, the combustion of the next sample takes already place.

Results

	Mean [mg/kg] (n = 9)	RSD [%] (n = 9)
Fluorine	411	2.6
Sulfur	456	4.0



Sample

Liquefied butane containing certified amounts of dimethylsulfide or fluorobenzene respectively

Sample preparation

The sample is analyzed by Combustion IC with flame sensor technology and intelligent Partial Loop Injection Technique with Inline Matrix Elimination.

Columns

Metrosep A Supp 16 - 150/4.0	6.1031.430
Metrosep A Supp 16 Guard/4.0	6.1031.500
Metrosep A PCC 1 HC/4.0	6.1006.310

Solutions

Eluent	7.5 mmol/L sodium carbonate 0.75 mmol/L sodium hydroxide
Suppressor regenerant	100 mmol/L sulfuric acid
Rinsing solution	STREAM
Absorber solution	100 mg/L hydrogen peroxide

Parameters

Flow rate	0.8 mL/min
Injection volume (IC)	20/40 µL (S- / F-LPG)
P _{max}	20 MPa
Recording time	18 min
Column temperature	45 °C

Combustion parameters

Combustion volume	5 μL (F-LPG) 10 μL (S-LPG)
Argon	100 mL/min
Oxygen	300 mL/min
Oven temperature	1050 °C
Post-combustion time	60 s
Initial volume of absorption solution	2.0 mL
Water inlet	0.1 mL/min

Analysis

Conductivity after sequential suppression

Instrumentation

930 Compact IC Flex Oven/SeS/PP/Deg	2.930.2560
IC Conductivity Detector	2.850.9010
920 Absorber Module	2.920.0010
Combustion Module (oven and gas module)	2.136.0730





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Nested combustion method

Nested combustion means that, while the absorption solution of sample 1 is being analyzed with IC, the combustion of sample 2 takes already place. This is made possible by MagIC Net, the software that accesses ample information of the next sample and takes logical decisions. MagIC Net distinguishes between the single calibration standard and samples that need to be combusted. While the latter are transported to the oven, the standard is excluded from the combustion step and is directly used for calibration tasks using MiPT.



Legend:

- 1 Fill loop with standard/check standard/ultrapure water and inject
- 2 Empty and rinse buffer tube from standard/check standard/ultrapure water injection
- 3 Empty absorber tube and prepare fresh absorption solution
- 4 Total Combustion time
- 5 Absorption solution is parked in the buffer tube for analysis in next sample line
- 6 Fill loop from the buffer tube
- 7 Empty and rinse buffer tube from sample followed by the rinsing of the absorption tube

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