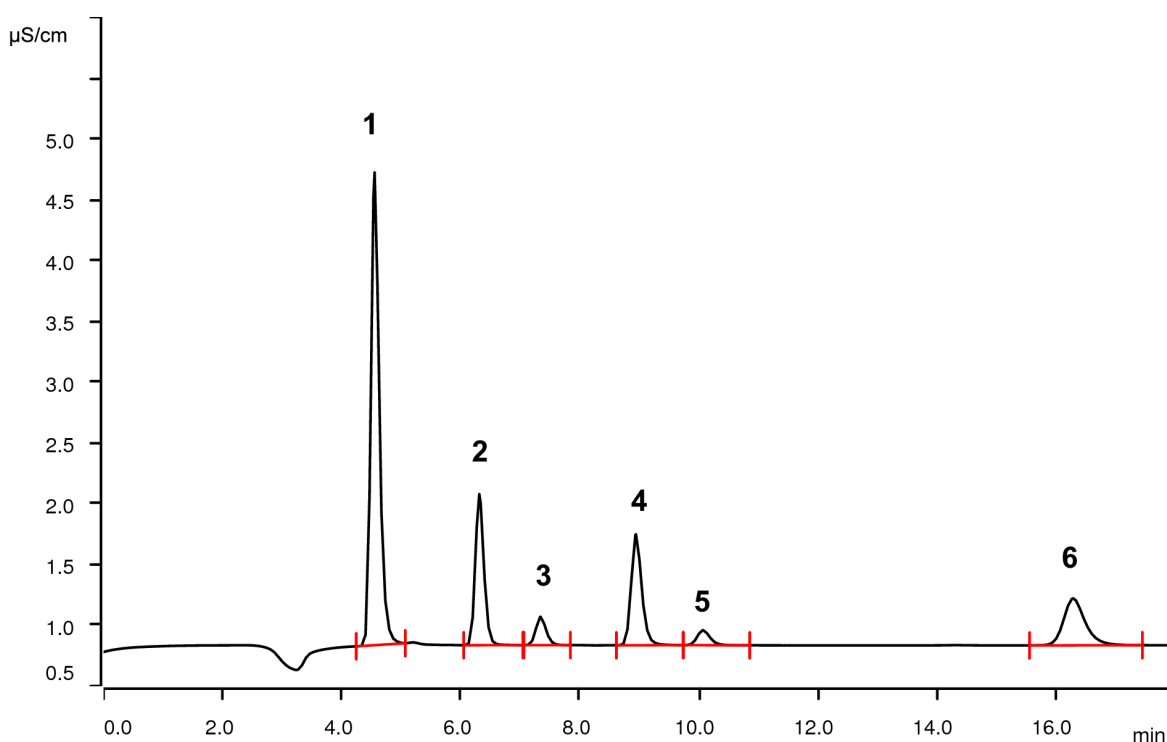


# Halogens and sulfur in LPG according to ASTM D7994



ASTM D7994 - 17 describes the determination of fluorine, chlorine, and sulfur in liquefied petroleum gas (LPG) by oxidative pyrohydrolytic combustion followed by ion chromatography. A synthetic butane sample is analyzed. 50  $\mu\text{L}$  of the sample is injected into the combustion system using the LPG Module. The combustion products are analyzed by IC applying intelligent Partial Loop Injection Technique after Inline Matrix Elimination.

## Results

	Mean [mg/kg]	RSD [%] n = 3
1 Fluorine	26.33	1.5
2 Chlorine	17.23	1.1
4 Bromine	37.83	1.2
6 Sulfur	13.08	0.5

Peak 3 and 5 are nitrite and nitrate, respectively. Both are not quantified.

## Sample

Liquefied butane gas

## Sample preparation

The sample is analyzed by Combustion IC and 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

## Solutions

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	150 mg/L hydrogen peroxide

## Parameters

Flow rate	0.7 mL/min
Injection volume (IC)	200 µL (MiPT)
P <sub>max</sub>	15 MPa
Recording time	20 min
Column temperature	30 °C

## Combustion parameters

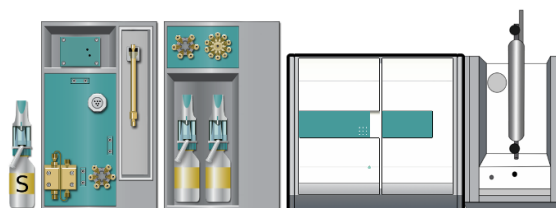
Argon	100 mL/min
Oxygen	300 mL/min
Oven temperature	1050 °C
Post-combustion time	120 s
Initial volume of absorption solution	2.0 mL
Water inlet	0.1 mL/min
Injection Volume (LPG)	50 µL

## Analysis

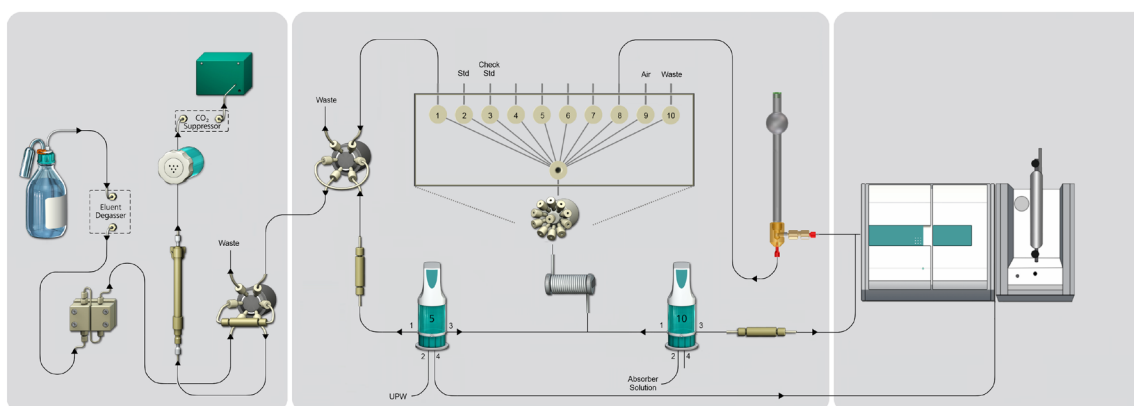
Conductivity after sequential suppression
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## Instrumentation

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



## System graphic



Liquid handling of absorber solution is performed by the 10 mL-Dosino, i.e., the initial volume is set into the absorber vessel before combustion. During combustion, absorber solution is dosed (0.2 mL/min) to the T-piece located at the end of the combustion tube to achieve immediate absorption of combusted products (Post-combustion rinse). The connection between absorption vessel, and after combustion has finished, the combustion tube is rinsed with 1 mL (final rinse). Water inlet into the combustion tube for pyrohydrolytic environment is carried out by the 5 mL-Dosino with a rate of 0.1 mL/min. As MagIC Net exactly records all volumes added to the absorber solution during and after combustion, no internal standard is required.

The transfer of samples and standards into the loop of the 920 Absorber Module is performed by the 5 mL-Dosino using partial loop injection (MiPT) giving full flexibility to injection volumes in a range from 4 to 200  $\mu\text{L}$ . A fixed volume of 200  $\mu\text{L}$  was used for all MiPT injection volumes in this work.

In the case of LPG analysis, the flame sensor is not utilized for combustion control. Therefore, the combustion time is dictated only by 1) the time required for injection of the sample by the LPG module and 2) the post-combustion time specified in the instrument method. The LPG module contains a 1  $\mu\text{L}$  sample loop that may be filled and injected up to 50 times. Ultimately, this configuration is capable of injecting a variable LPG sample volume ranging from 1 to 50  $\mu\text{L}$ . The time required for injection of the sample (1  $\mu\text{L} \times 50$  loop-fills) is approximately 5 minutes.