

C,S determination in CaF₂ Samples (with halogen trap)



Carbon Sulfur

Suitable analyzers

- ELEMENTRAC CS-i
- ELEMENTRAC CS-d
- Halogentrap (88600-0020)

Used accessories

- Ceramic crucibles (90149)
- Eltracel (88600-0010)
- Filling halogentrap (90234 | 90235)
- Glasswool (90311 | 90332)

**Application Settings****I) General**

Sample type:	Advanced	Lance purging:	off
Standby mode:	Custom	Purging while closing:	on
Standby flow:	180 l/h	Stabilize baseline:	on
Furnace purge through:	Exhaust	Stability:	0.01
Furnace purge time:	3 sec	Minimum time:	15 sec
Furnace purge flow:	180 l/h	Maximum time:	30 sec

II) Analysis

Flow:	180l/h	Chamber only:	1 sec
Ramping:	off	Lance and chamber:	1 sec
Voltage:	80 %	Auto comparator:	Off
Power duration:	25 sec	Drift compensation:	Off
		Post waiting:	1

Channel	Min time [sec]	Max time [sec]	Integration delay [sec]	Comparator level [mV]	Comparator peak [%]
Low & high C	20	120	12	10	1
High & high S	20	120	12	10	1

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Sample preparation

Make sure that the sample is disintegrated to a suitable sample size and is free from contaminations. Please dry the sample until mass constancy at 105°C. Pre heat the ceramic crucibles at least for 1h at 1000°C. Let the crucibles cool down in a desiccator.

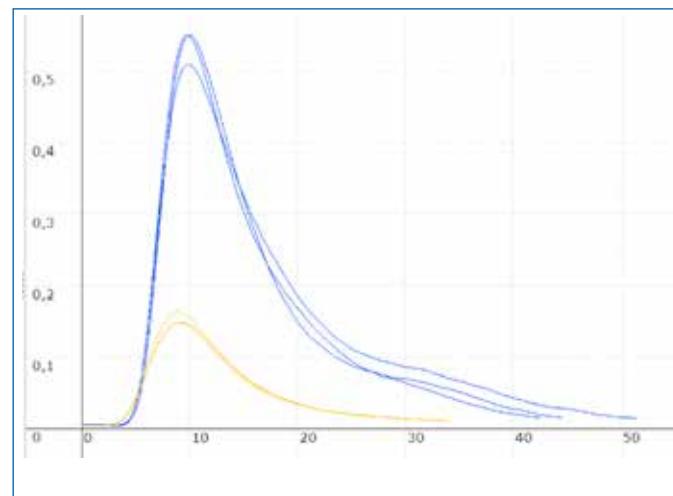
Procedure

- Prepare the ELTRA analyzer (e.g. exchange anhydride, sodium hydroxide, catalyst if necessary); clean combustion tube, heat shield and furnace area
- Fill the halogentrap consecutive with glasswool, potassiumiodide, glaswool, antimony, glaswool and antimony.
- Run three warm up samples (e.g. steel samples (92400-3050) with a minimum weight of 500 mg and 1.7 g tungsten as accelerator
- Calibrate the analyzer with suitable calibration material (NIST or other)
 - (1) Weigh in approx. 60 – 100 mg of the sample into the ceramic crucible
 - (2) Add 2 g of Eltracel (88600-0010)
 - (3) Place the crucible on the pedestal (use tongs!) and start analysis

Repeat steps (1) – (3) at least three times;
Mark the results and use the calibration function in the software.

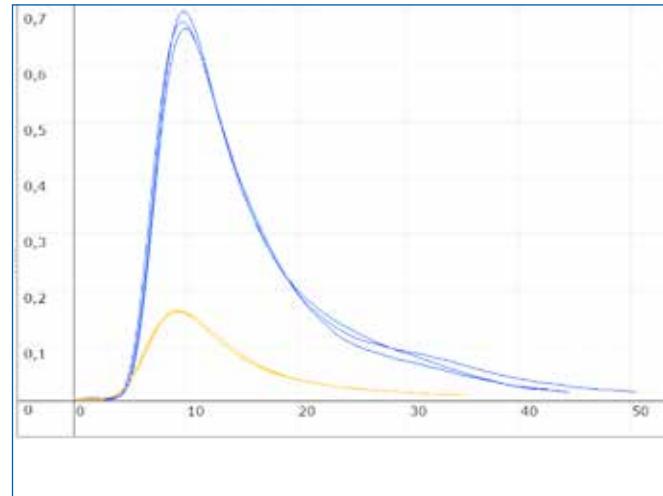
► Now start with the actual analysis.

Typical results		
CaF ₂ sample No. 1		
Weight (mg)	Carbon (%)	Sulfur (%)
53.9	0.0337	0.061
58.0	0.0397	0.058
52.9	0.0346	0.063
Average		
	0.0360	0.060
Deviation / Relative deviation		
	0.0032 (9.0%)	0.002 (4.0%)

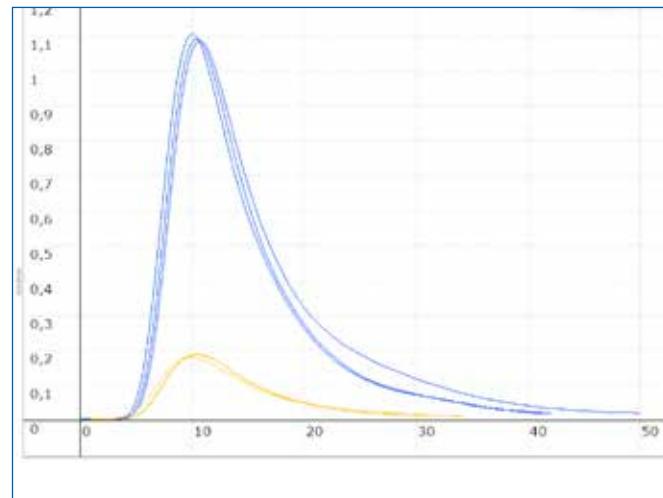


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Typical results		
CaF ₂ sample No. 2		
Weight (mg)	Carbon (%)	Sulfur (%)
52.5	0.0388	0.069
51.0	0.0398	0.068
51.0	0.0352	0.068
Average		
	0.0379	0.068
Deviation / Relative deviation		
	0.0024 (6.4%)	0.001 (1.2%)

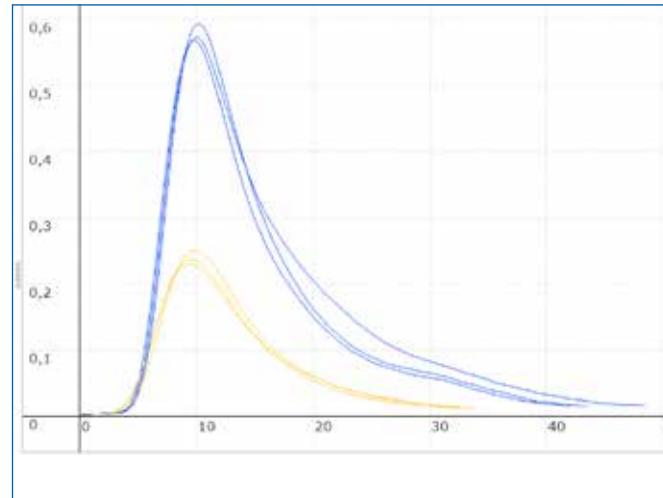


Typical results		
CaF ₂ sample No. 3		
Weight (mg)	Carbon (%)	Sulfur (%)
57.6	0.0641	0.072
52.3	0.0652	0.076
51.2	0.0629	0.079
Average		
	0.0641	0.075
Deviation / Relative deviation		
	0.0012 (1.9%)	0.004 (4.7%)



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Typical results		
CaF ₂ sample No. 4		
Weight (mg)	Carbon (%)	Sulfur (%)
50.8	0.0351	0.088
55.1	0.0319	0.089
54.3	0.0322	0.085
Average		
	0.0331	0.087
Deviation / Relative deviation		
	0.0018 (5.3%)	0.002 (2.1%)



Typical results		
CaF ₂ sample No. 5		
Weight (mg)	Carbon (%)	Sulfur (%)
57.5	0.0431	0.112
57.4	0.0406	0.110
55.9	0.0375	0.113
Average		
	0.0404	0.111
Deviation / Relative deviation		
	0.0028 (7.0%)	0.001 (1.0%)

