

Low oxygen analysis in copper (carrier gas argon)



Suitable analyzers

- ELEMENTRAC ONH-*p*
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Used accessories

- Graphite crucibles (90190)

Description of the challenge

The measurement of oxygen in copper is a standard application with the carrier gas helium. When the carrier gas argon is used different settings are required to get a good repeatability of the oxygen measurements.



Application Settings

I) General

Sample type:	Advanced
Use argon:	on
Furnace Mode:	on
Catalyst:	650 °C
Cooling low:	40 °C;
Cooling high:	45 °C
Standby Flow:	10 l/h

II) Outgasing and stabilizing

Setting/Phase	Cooldown [sec]	Time [sec]	Power [W]	Flow [l/h]
Outgasing (Phase 1)	NN	30	4000	22
Outgasing (Phase 2)	0	20	3200	22

Setting/Phase	Min/Max time [sec]	Power [W]	Flow [l/h]	Stability [V]
Stabilizing (Phase 1)	85/85	3200	22	0.001

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III) Analyzing

Flow:	22 l/h
Power duration:	180 Sec
Power:	3100 W
Second cycle:	off
Open furnace:	on
Drift compensation:	on
Manual load:	off
Auto comparator:	off

Element	Minimum time [sec]	Maximum time [sec]	Integration delay [sec]	Comperator factor [%]
Low Oxygen	30	80	5	0.1
High Oxygen	30	80	5	0.1

IV) Postwaiting

Time:	15 sec
Granulate mode:	off

Sample preparation

For a calibration sample no sample preparation is required. In some cases the repeatability could be improved when the sample is washed in acetone p.a. immediately before the analysis, followed by a short drying at air.

Copper samples could show an improved deviation regarding their oxygen results when they are treated with acid, washed with water and acetone. Procedures, used acids can differ according to local standards. Please refer to your corresponding standard.

Procedure

- Prepare the ELTRA analyser and exchange anhydron, sodium hydroxide, copper oxide when it is necessary. Clean sample drop mechanism, furnace, electrode tip.
- Run three blanks with empty crucibles
- Calibrate the analyser

Please lock in a suitable calibration sample (e.g. ELTRA 91000-1002 (or something comparable)) with the sample weight in the ELEMENTS software and process the analysis.

When 3 calibration samples have been measured use the calibration function of the software.

Now the analyser is ready to run samples.

- Run the samples in the same way as the calibration samples.



ELEMENTRAC ONH-p

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Typical results	
ELTRA 91000-1004 (certified oxygen: 8 ppm $\pm$ 1 ppm)	
Weight (mg)	ppm oxygen
988.1	7.2
989.7	8.1
1004.3	9.0
973.7	7.2
986.0	7.7
985.2	8.5
999.3	7.3
983.5	7.5
992.6	9.1
991.7	7.0
Average values	
	7.9
Deviation / Relative deviation (%)	
	0.8 (9.8)

