

Oxygen determination in copper samples



Suitable analyzers

- ELEMENTRAC ONH-p 2
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Used accessories

- Graphite crucibles (90190)
- Suitable calibration material (NIST or other)



ELEMENTRAC ONH-p 2

Application Settings

I) General

Sample type: Advanced
 Use argon: Off
 Catalyst: 650 °C
 Cooling low: 40 °C

Cooling high: 60 °C
 Flow: 40 l/h
 Standby Flow: 40 l/h

II) Purging

Purging while closing: Enable
 Closing purging time: 2 sec

III) Outgasing

Enable pulse: Enable
 Pre-heat: Disable

Time: 10 sec
 Power: 6000 W

IV) Stabilizing

Time: 60 sec
 Power: 3600 W

V) Analyzing

Minimum furnace temp: 41 °C
 Power duration: 180 sec
 Power: 3600 W

Open furnace: Enable
 Cooling delay: 5
 Peak finding: Drift compensation

VI) Post waiting

Time: 10 sec

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Channel Settings

Channel	Enable	Integration delay [sec]	Minimum time [sec]	Maximum time [sec]	Comparator factor [%]	Peak max [V]
Low & high oxygen	Enable	8	20	90	0.05	8
Low & high nitrogen	Disable	-	-	-	-	-

Sample preparation

Make sure that the surface of the copper is free from contaminations; otherwise clean the sample with acetone p.a. and let it air dry.

Procedure

- Prepare the ELTRA analyzer (exchange anhydron, copper oxide if necessary). Clean sample drop mechanism, furnace, electrode tip (if necessary).
- Run three blanks with empty crucibles
- Calibrate the analyzer with suitable calibration material (NIST or other)
 - (1) Place one empty crucible (90190) on the electrode tip, close furnace
 - (2) Weigh calibration material, place it in the sample drop mechanism and start analysis
 - (3) Used graphite crucible has to be given into waste

Repeat steps (1) - (3) at least three times;

Mark the results and use the calibration function in the software.

➔ Now start the actual analysis.

Notice:

General recommendations for this application can be found at the end of this document.

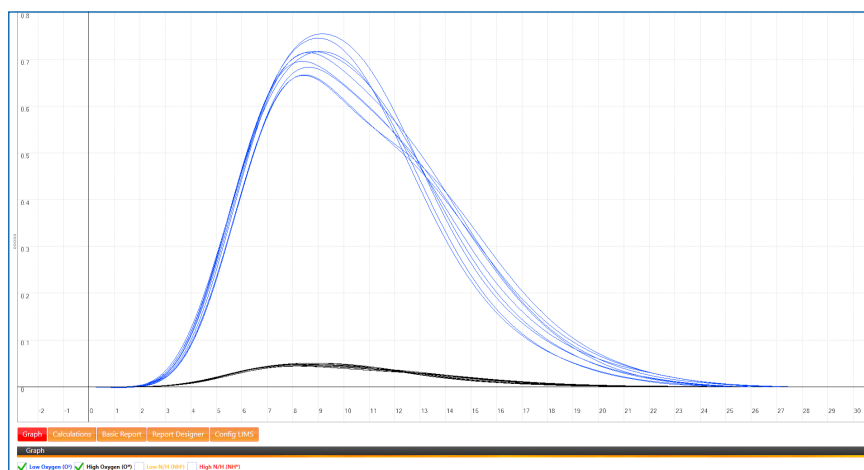


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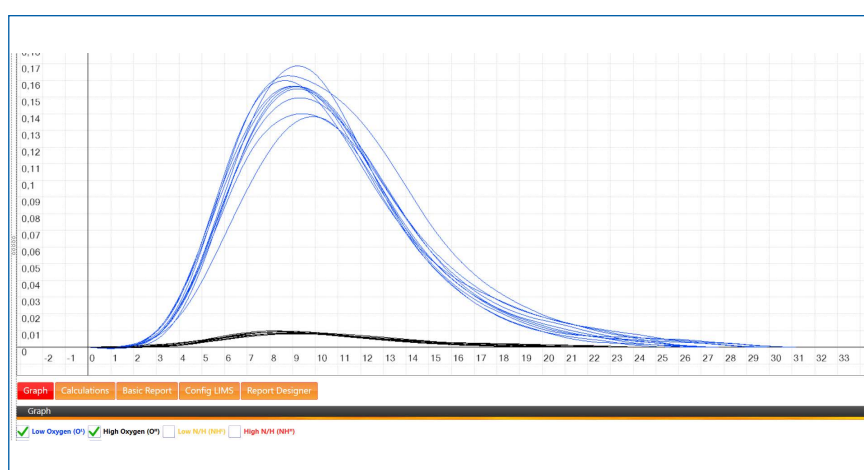
Oxygen determination in copper samples



Typical results	
ELTRA 91000-1004 (LOT 918B)*	
Weight (mg)	Oxygen (ppm)
1013	7.80
1006	5.83
1014	6.54
1010	7.02
1010	7.24
1014	7.41
1012	7.19
1013	7.71
1011	8.09
1012	6.32
Mean value	
	7.12
Deviation / Relative deviation (%)	
	0.71 (9.9)
* Certified content: Oxygen 7 +/- 2 ppm	



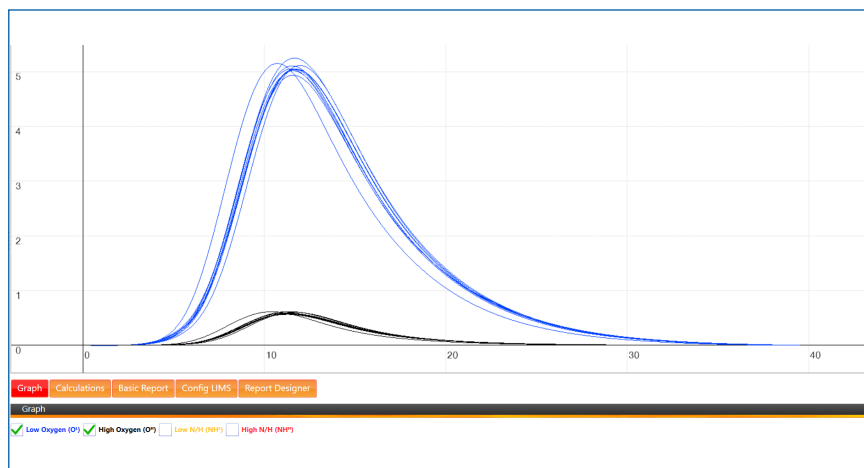
Typical results	
LECO 501-953* (LOT 918B)	
Weight (mg)	Oxygen (ppm)
1001	3.18
1010	3.08
1003	3.02
1003	3.09
1003	2.99
1000	2.92
1003	2.95
995	3.56
1002	2.67
1001	2.99
Mean value	
	3.0
Deviation / Relative deviation (%)	
	0.2 (7.4)
* Certified content: Oxygen 3.2 +/- 0.42 ppm	



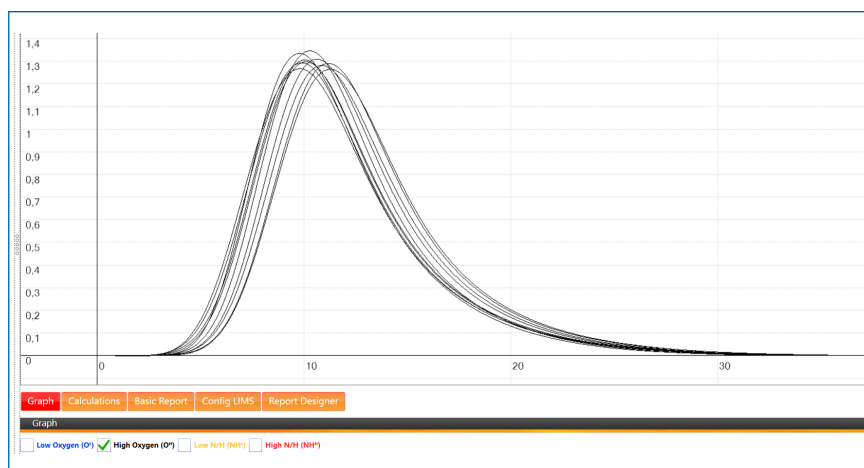
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Typical results	
ELTRA 91000-057 (LOT 20181113)*	
Weight (mg)	Oxygen (ppm)
1001	186.74
1009	187.32
1010	185.51
967.7	188.58
994.1	187.09
998.1	187.09
1015	188.92
1011	188.49
1084	188.66
1004	188.32
Mean value	
	187.70
Deviation / Relative deviation (%)	
	1.03 (0.5)
* Certified content: Oxygen 187 +/- 6 ppm	



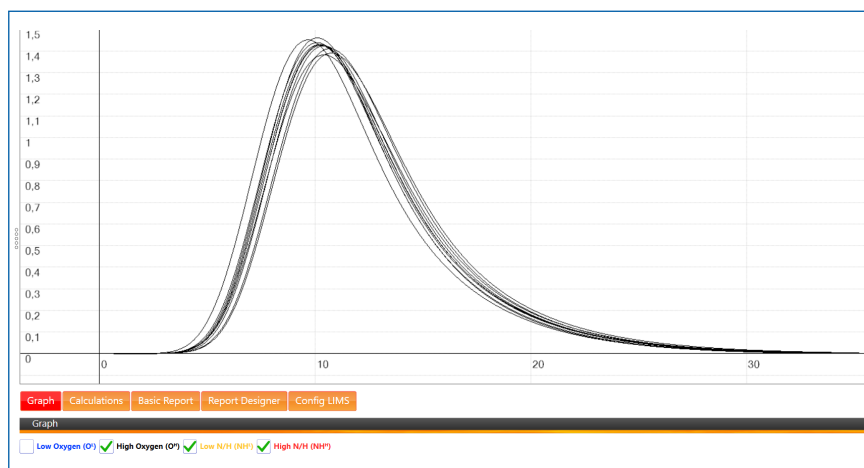
Typical results	
AR148* (LOT 518B)	
Weight (mg)	Oxygen (ppm)
1004	491.93
1002	493.19
1010	492.36
1004	490.23
1003	493.41
1010	491.83
1004	495.35
1007	496.19
1012	494.40
1014	491.16
Mean value	
	493.01
Deviation / Relative deviation (%)	
	1.88 (0.4)
* Certified content: Oxygen 493 +/- 9 ppm	



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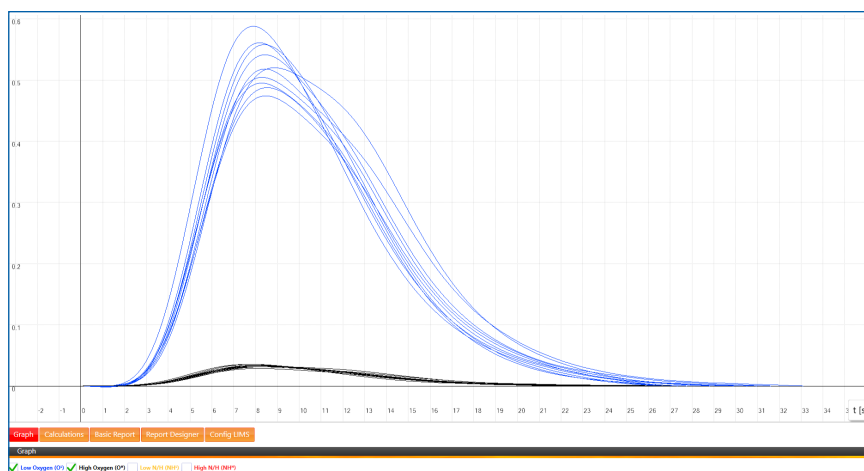


Typical results	
LECO 501-149 (LOT 0576)*	
Weight (mg)	Oxygen (ppm)
1002	544.95
1002	543.70
1003	542.63
1004	543.92
1001	542.08
1003	541.74
1002	545.00
1000	542.33
1004	542.44
1002	543.26
Mean value	
	543.21
Deviation / Relative deviation (%)	
	1.16 (0.2)
* Certified content: Oxygen 543 +- 8 ppm	



The ELEMENTRAC ONH -p 2 can process higher sample weights than 1000 mg. Before applying higher sample weights than 1000 mg read the general recommendations at the end of this document and test if the applied sample amount will fit into the crucible and sample port.

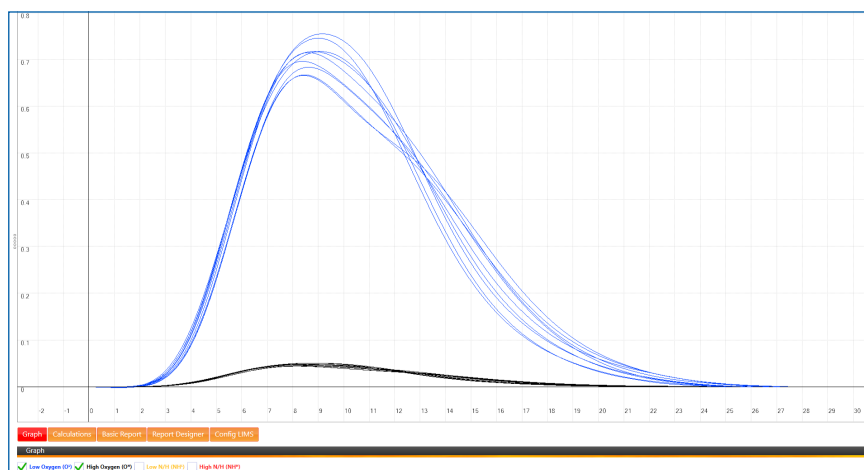
Typical results	
ELTRA 91000-1004 (LOT918B) with 2000 mg sample weight (2 pins)	
Weight (mg)	Oxygen (ppm)
2018	6.57
2017	7.05
2020	6.97
2020	6.32
2023	7.32
2017	6.51
2014	7.67
2016	7.25
2020	7.45
2019	7.16
Mean value	
	7.03
Deviation / Relative deviation (%)	
	0.44 (6.2)
* Certified content: Oxygen 7 +- 2 ppm	



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Typical results	
ELTRA 91000-1004 (LOT918B)* with 3000 mg sample weight (3 Pins)	
Weight (mg)	Oxygen (ppm)
3027	6.99
3032	6.83
3032	7.29
3029	6.91
3027	7.06
3029	7.12
3031	7.21
3028	6.81
3032	6.82
Mean value	
	7.00
Deviation / Relative deviation (%)	
	0.18 (2.5)
* Certified content: Oxygen 7 +- 2 ppm	



Typical results	
ELTRA 91000-1002 (LOT534985510)* with 2000 mg sample weight (2 Pins)	
Weight (mg)	Oxygen (ppm)
2010	321.28
2009	319.19
2011	324.01
2027	327.50
2004	322.59
2010	322.63
2002	320.85
2022	322.18
2025	324.31
2021	325.63
Mean value	
	323.02
Deviation / Relative deviation (%)	
	2.43 (0.8)
* Certified content: Oxygen 323 +- 8 ppm	



Oxygen determination in copper samples



The ELEMENTRAC ONH-p2

Cleaning of the furnace & upper electrode

Furnace and upper electrode have been cleaned after every 10-15 samples.

Usage of crucibles

Data for this application note has been obtained by using a new single crucible for each measurement. Sometimes it may be possible to remove the melted copper sample and use the crucible for a second or third time. Multiple usage of one crucible will cause a higher contamination with copper dust and could influence the repeatability of measurements.

ELTRA recommends the usage of single crucibles (90190) for best results. It may be possible to use inner (90180) and outer crucibles (90185) for this application when the applied power during stabilizing and analyzing is increased to a value of 3900 W.



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Colour of copper residues

The colour the applied copper (e.g. shiny copper or grey) does not influence the correctness of measurements. With lowering the applied power the residues in the crucibles look more shiny copper. A reduced power increases the risk of lower determination of oxygen and can cause a higher RSD.

Analyzing copper samples with very low oxygen concentrations (e.g. < 10 ppm)

Esp. for the analysis of low oxygen concentrations a sample preparation according to ASTM 2575-1019 may be required. The sample preparation does only affect the preparation of customer samples, but not the calibration samples like e.g. ELTRA 91000-1004. The ASTM 2575 (2019) standard recommends to etch a sample for three minutes in concentrated HCl, followed by treatment with a copper pickle solution (equal parts of the concentrated acids HNO₃, H₃PO₄, CH₃COOH) at 70 °C for one minute; followed by three rinsing cycles in distilled water and methanol. Afterwards the sample should be dried in hot air. Further information is available in the standard ASTM 2575.

Oxygen determination in copper samples



Minor determination of oxygen in copper after a long measuring pause

The results in this application note have been obtained with an analyzer which was warmed up by 3 blanks and 3 calibration samples. All consecutive measurements have been processed with a medium cycle time of 3 minutes for one sample. When the measurement process is interrupted for more than 30 minutes it is recommended to process a blank analysis to warm the analyzer up again. When the analyzer in general is used in a discontinuous way ELTRA recommends to activate the pre heat function (application setting), apply a longer outgasing time (20 seconds) and higher stabilizing and analysing power settings (4000 W).

General minor determination of oxygen in copper

When the oxygen concentration in selected copper samples is measured too low a higher stabilizing and analysis power (Max 4500 W) should be applied. In some copper sample the oxygen is not only bounded to copper, but also to other elements like manganese or iron. For a complete release of oxygen in these samples a higher analysis and stabilizing power is required.

Application of higher sample weights (e.g. 2-3 gram)

Depending on the sample geometry an application of higher sample weights could be useful to improve the repeatability of the oxygen measurements. When higher sample weights have to be processed with the ONH-p2 the following details have to be taken into account:

- (1) Check if the sample will fit into the furnace and the crucible. This could be tested by applying the sample to the furnace and click the "clean furnace" button in the analyzer status window. When the sample falls without blocking into the crucible and the height of the sample is lower than the crucible an analysis may be possible with this sample.
- (2) When two pieces are applied (e.g. pins) make sure that these pins are applied in a vertical way to the sample port: This procedure reduces blocking.
- (3) A higher stabilizing and analysis power of 4000 W has to be applied for significant higher sample weights than 1000 mg.

