

Oxygen and nitrogen determination in steel granulates and drillings



Suitable analyzers

- ELEMENTRAC ONH-p2
- ELEMENTRAC ON-p 2

Used accessories

- Graphite crucibles (90180 & 90185)
- Weighing boat (88400-0477)
- Suitable calibration material (NIST or other)



ELEMENTRAC ONH-p2

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: Disable

III) Outgasing

Enable pulse: Disable
 Pre-heat: Disable

Time: 10 sec
 Power: 6000 W

IV) Stabilizing

Time: 60 sec
 Power: 4500 W

V) Analyzing

Minimum furnace temp: 42 °C
 Power duration: 180 sec
 Power: 4500 W

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

VI) Post waiting

Time: 20 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	7	25	90	0.05	8
Low & high nitrogen	Enable	14	25	90	0.05	8

Sample preparation

Granulates or drillings do not require a special treatment before they can be analyzed with the ELEMENTRAC ONH-p2. The general procedure of preparing chips is described in the ASTM E 1806 or DIN EN ISO 14284.

Procedure

- Prepare the ELTRA analyzer (exchange anhydrous, 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 an outer (90185) with inner crucible (90180) on the electrode tip, close furnace
 - (2) Weigh calibration material, place it in the sample drop mechanism and start analysis
 - (3) Used inner 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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Nitrogen

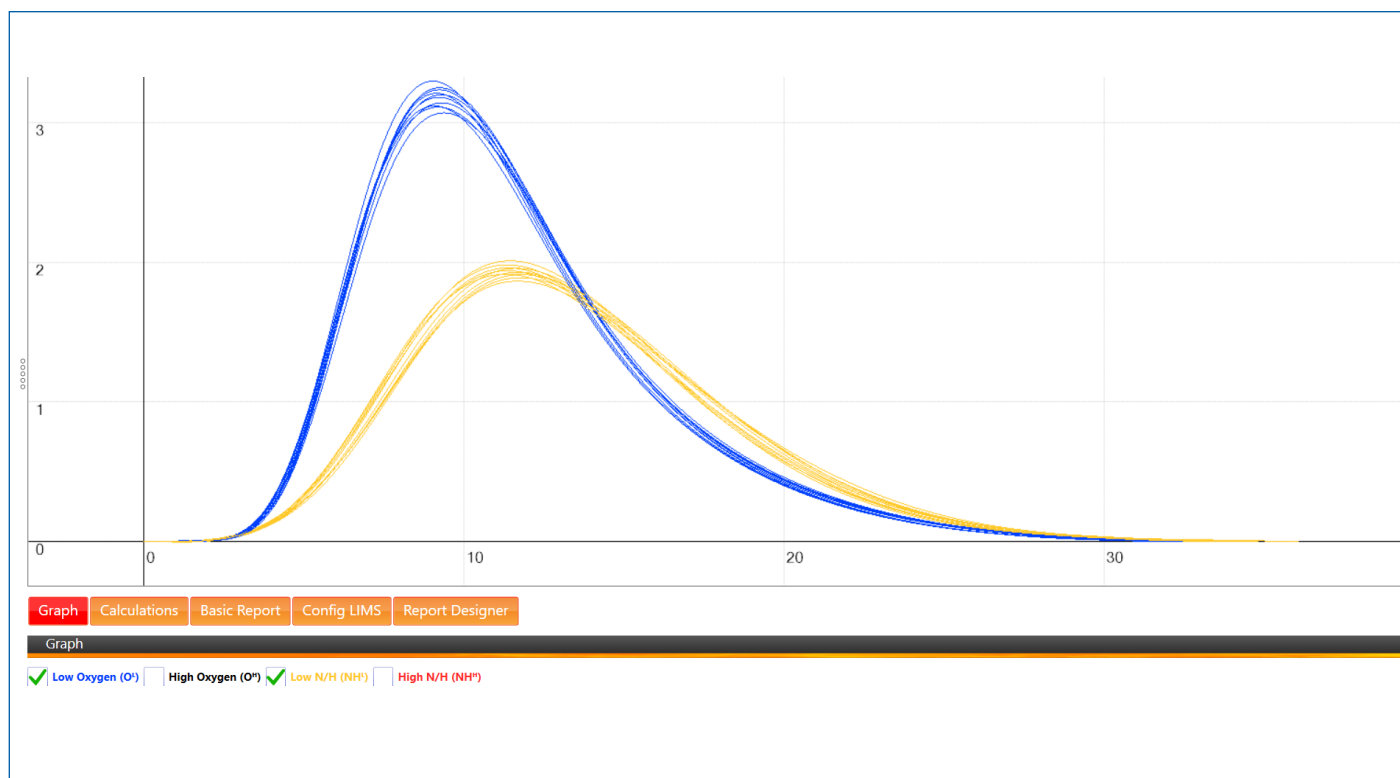


Oxygen

Typical results

Steel granulate ZRM 284-2*

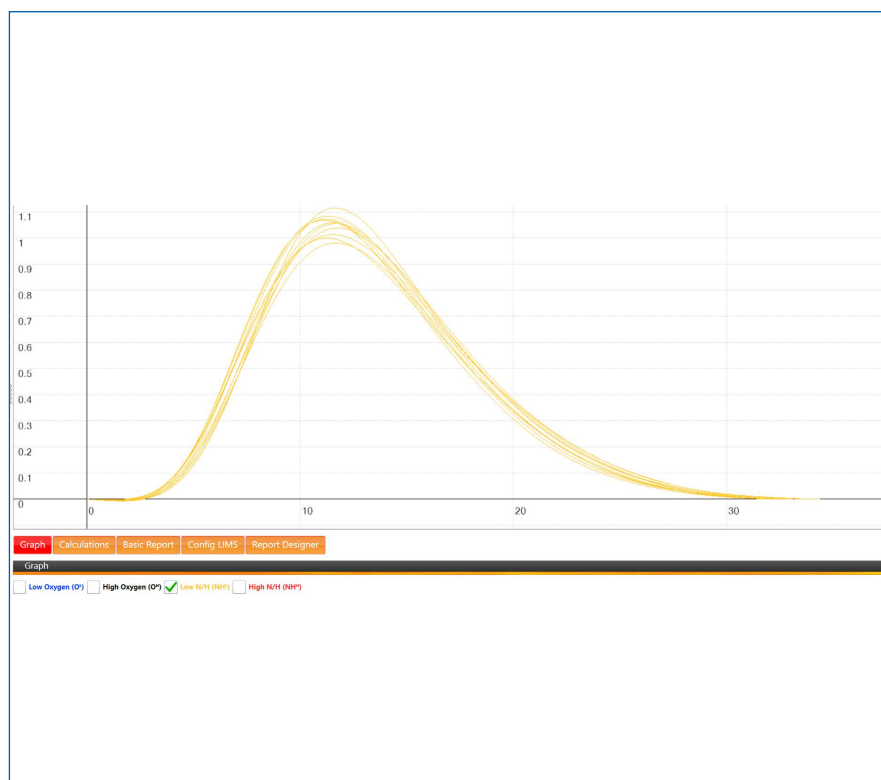
Weight (mg)	Oxygen (ppm)	Nitrogen (ppm)
1018	99.7	148.8
1057	96.5	152.6
1036	99.3	150.3
1027	99.1	150.6
1015	97.7	151.5
1021	102.0	150.1
1020	98.4	153.4
1035	99.2	150.0
1029	99.8	149.0
1011	98.1	153.4
Mean value		
	99.0	151.0
Deviation / Relative deviation (%)		
	1.5 (1.5)	1.7 (1.1)
* Certified value: O 99 +- 7 ppm; N 151 +- 2 ppm		



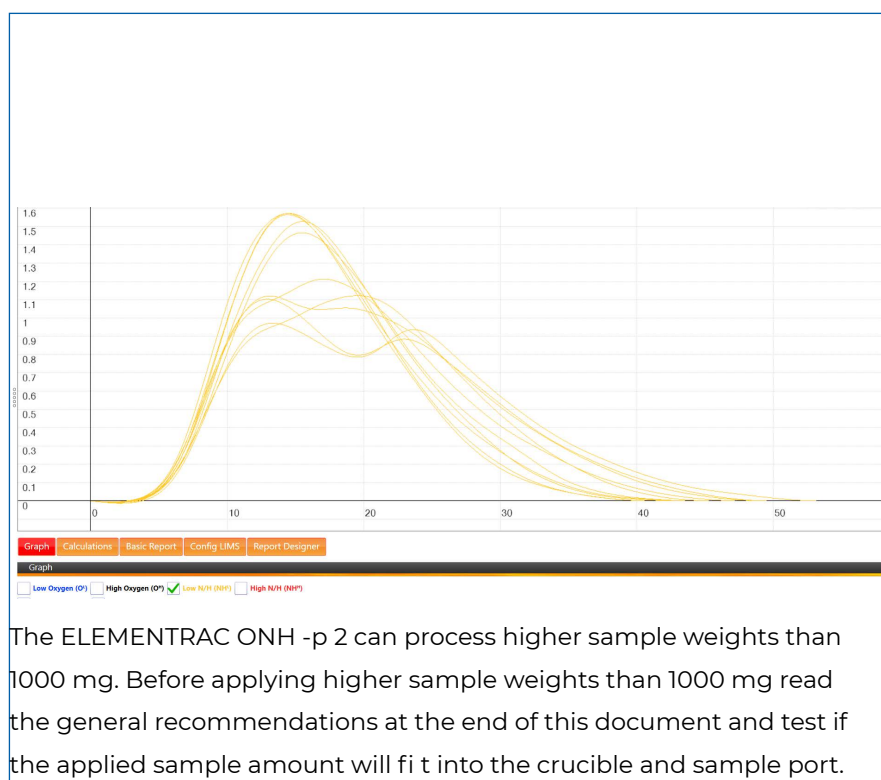
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Typical results	
Steel granulate AR 954 Lot 218B	
Weight (mg)	Nitrogen (ppm)
1015	81.67
1014	84.36
1024	81.36
1007	82.47
1008	82.03
1038	82.18
1039	83.74
1060	82.08
1061	82.90
1051	81.55
Mean value	
	82.43
Deviation / Relative deviation (%)	
	0.97 (1.2)
* Certified value: certified content N 83 ± 8 ppm	



Typical results	
Steel granulate AR 954 Lot 218B	
Weight (mg)	Nitrogen (ppm)
2048	85.99
2005	82.74
2068	80.94
2007	85.00
2078	81.72
2026	80.99
2025	84.01
2013	81.09
2028	83.23
2001	84.64
Mean value	
	83.04
Deviation / Relative deviation (%)	
	1.84 (2.2)
* Certified value: certified content N 83 ± 8 ppm	



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The ELEMENTRAC ONH-p2: recommendations

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 inner crucible for each measurement. Single crucibles (90190) can also be used for this application with the same settings.

Recommended sample weight

Higher samples weights in general do not guarantee an improved repeatability for oxygen and nitrogen determination. When higher sample weights are used the melting of samples and the release of nitrogen and forming of CO takes more time. This could influence the repeatability in a negative way. As long the sample is homogeneous sample weights between 200 and 1000 mg provide a good precision for oxygen and nitrogen measurement. The optimal sample weight is influenced additionally by the shape of the granulate (e.g. small leaves, needles). Eltra recommends to test the optimal sample weight without applying power (see next recommendation).



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Application of higher sample weights

Depending on the sample geometry an application of higher sample weights could be useful to improve the repeatability of the oxygen and nitrogen 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) A higher stabilizing and analysis power of 5000 W could improve the repeatability of measurements. With higher sample amounts and applied power an increased post waiting time (e.g. 30 or 40 seconds) improves the repeatability additionally.



Subject to technical modification and errors

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Irritating results (minor determination or high deviation)

Not consistent results could be traced to several reasons. Please check the chemicals (esp. the scrubber for the TC cell) when results are increasing or decreasing from measurement to measurement. A leakage check is recommended additionally.

Please clean the furnace, upper and lower electrode, sample port to remove traces of powder. When irritating results occur by using high sample weights (e.g 500-1000 mg or more) reduce the sample weight. Alternatively increase the outgasing time for a more efficient removal of atmospheric gases.

Unstable (or too high) oxygen results for granulates

The measured oxygen content is influenced heavily by the sample preparation. When a drill or a sectioning machine is used make sure that the applied speed is not too high and a sufficient cooling is applied. Otherwise the surface of the sample will be burned and contaminated with additional oxygen.

Minor determination of oxygen and nitrogen 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 approx. 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) and apply a longer outgasing time (20 seconds).