ELTRA®
ELEMENTAL ANALYZERS

Oxygen and nitrogen determination in steel pins

Suitable analyzers

- ELEMENTRAC ONH-*p 2*
- ELEMENTRAC ON-*p 2*

Used accessories

- Graphite crucibles (90180 & 90185)
- Suitable calibration material (NIST or other)

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Application Settings

I) General

Sample type:	Advanced	Cooling high:	60 °C
Use argon:	Off	Flow	40 l/h
Catalyst:	650 °C	Standby Flow:	40 l/h
Cooling low:	40 °C		

II) Purging

Purging while closing:	Enable
Closing purging time:	2 sec

III) Outgasing

Enable pulse:	Enable	Time:	10 sec
Pre-heat:	Disable	Power:	6000 W

IV) Stabilizing

Time:	60 sec
Power:	4400 W

V) Analyzing

Minimum furnace temp:	42°C	Open furnace:	Enable
Power duration:	180 sec	Cooling delay:	5
Power:	4400 W	Peak finding:	Drift compensation

VI) Post waiting

Time:	15 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	25	90	0.05	8
Low & high nitrogen	Enable	15	25	90	0.05	8

Sample preparation

Pins do not require a special treatment before they can be analyzed with the ELEMENTRAC ONH-p2. Other solid pieces may require a cleaning of the surface with acetone followed by drying at air. The general procedure of sample taking is described in the ASTM E 1806 or DIN EN ISO 14284.

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Procedure

- Prepare the ELTRA analyzer (exchange anhydrone, 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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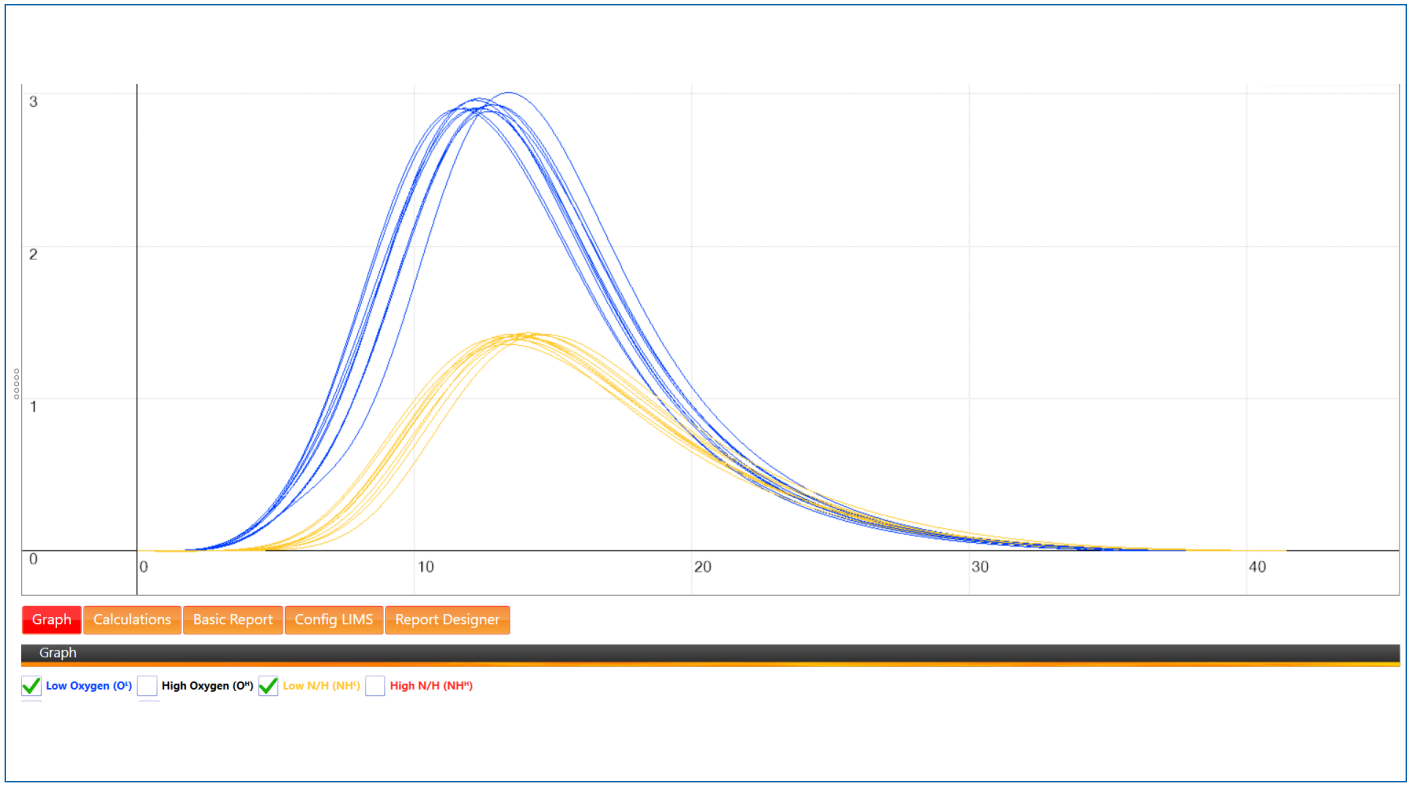
Typical results		
AR 1650 Lot 519C		
Weight (mg)	**Oxygen (ppm)**	**Nitrogen (ppm)**
1010	99.2	101.9
1004	99.7	102.0
1012	100.9	101.8
1011	98.64	101.5
1005	101.4	102.5
1009	100.7	102.0
1007	100.0	101.5
1006	98.2	102.2
1010	100.4	102.3
1002	100.4	101.84
Mean value		
	100.0	102.0
Deviation / Relative deviation (%)		
	1.0 (1.0)	0.32 (0.3)
* Certified value: O 100 ± 8 ppm; N 102 +- 9 ppm		



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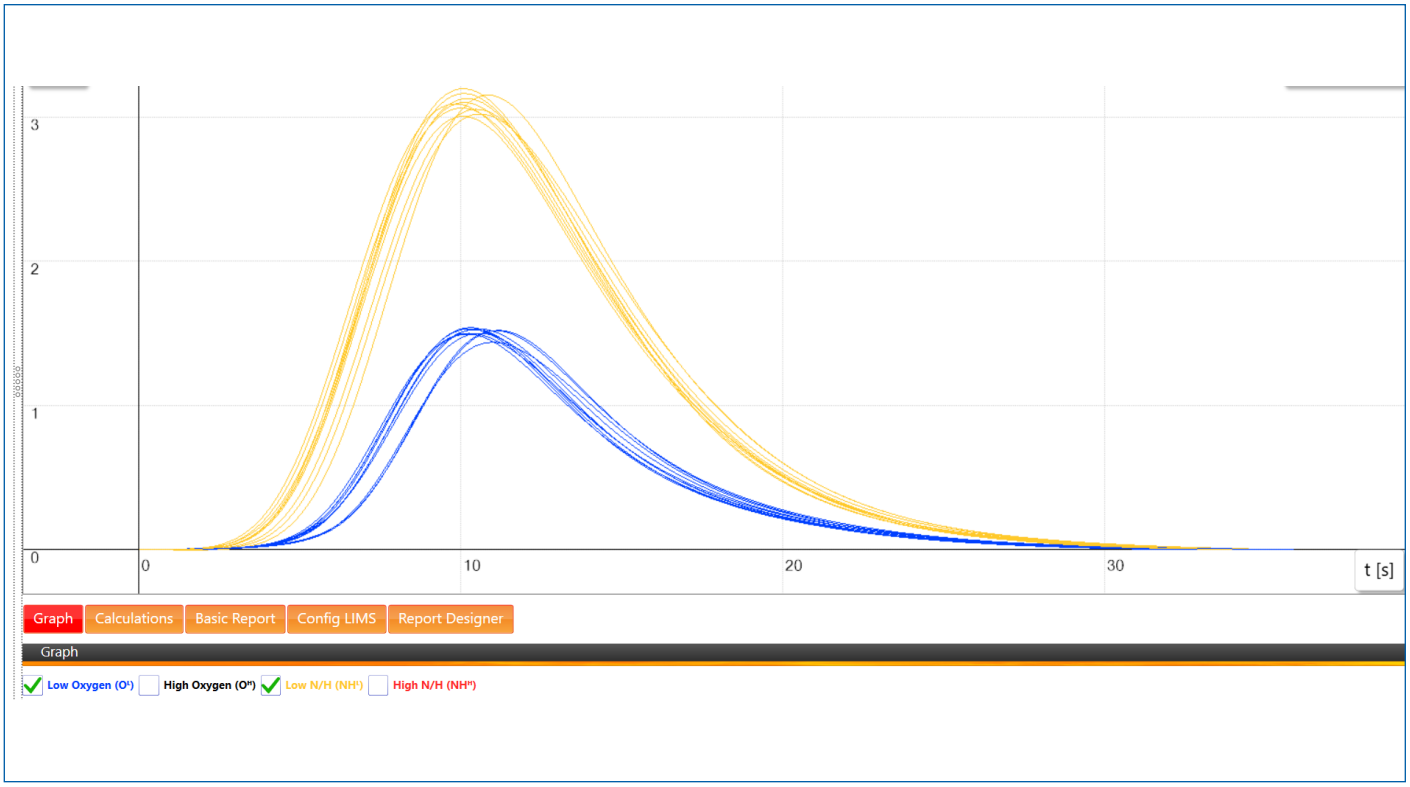
Typical results		
AR 1651 Lot 419G		
Weight (mg)	**Oxygen (ppm)**	**Nitrogen (ppm)**
1002	31.1	200.0
995	32.2	195.7
995	30.4	205.7
998	30.9	200.6
994	29.9	200.5
997	30.7	200.7
995	31.8	204.1
1002	29.9	204.4
998	32.41	204.0
997	30.6	204.2
Mean value		
	31.0	202.0
Deviation / Relative deviation (%)		
	0.8 (2.8)	3.0 (1.5)
* Certified value: O 31 ± 5 ppm; N 202 ± 7 ppm		



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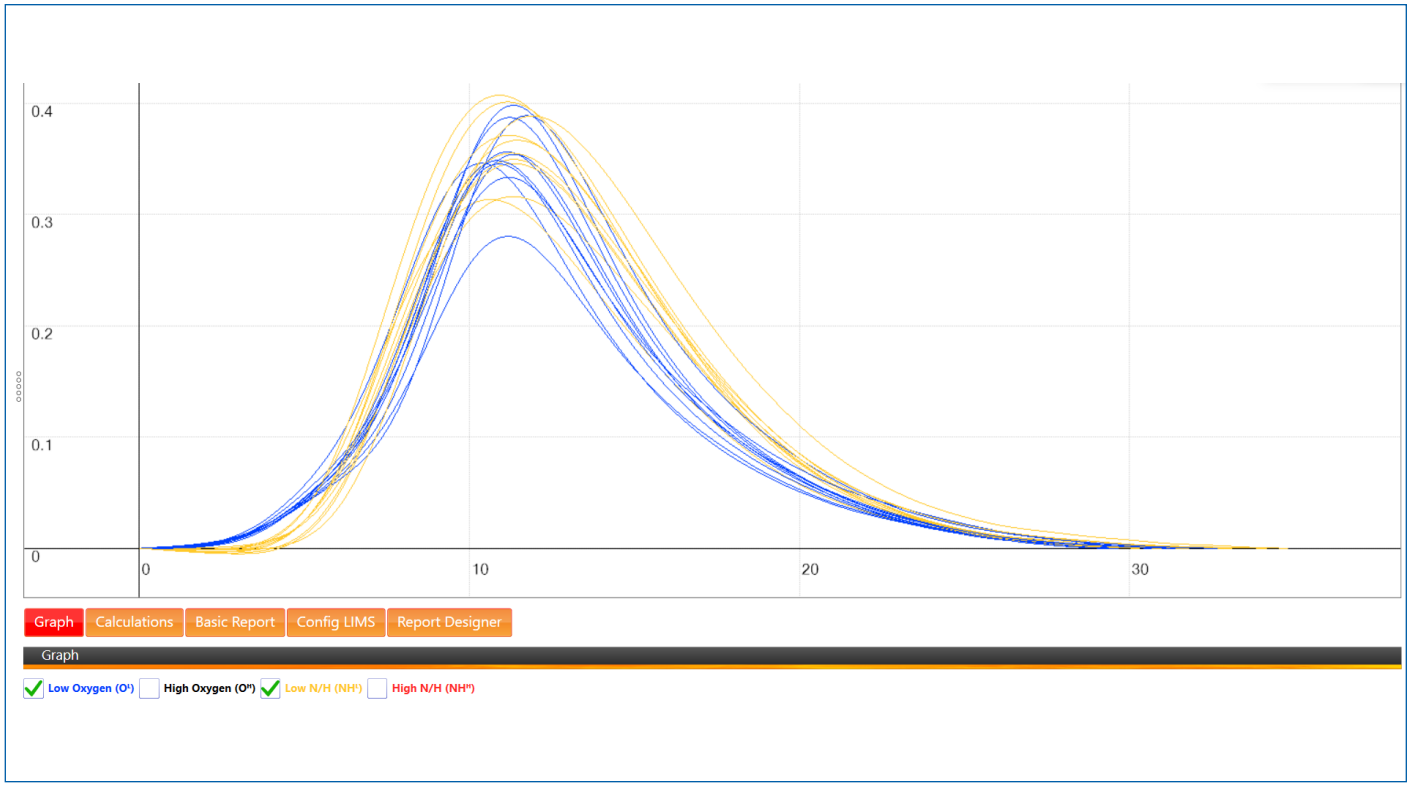
Typical results		
AR 1653 Lot 819D		
Weight (mg)	**Oxygen (ppm)**	**Nitrogen (ppm)**
973	6.82	26.04
980	6.83	27.28
984	5.64	33.07
983	7.47	29.46
983	7.52	28.54
971	7.04	29.01
978	7.64	33.37
978	6.69	31.38
980	7.21	33.52
985	6.73	30.38
Mean value		
	6.96	30.21
Deviation / Relative deviation (%)		
	0.58 (8.3)	2.61 (8.6)
* Certified value: O 7 ± 4 ppm; N 30± 10 ppm		



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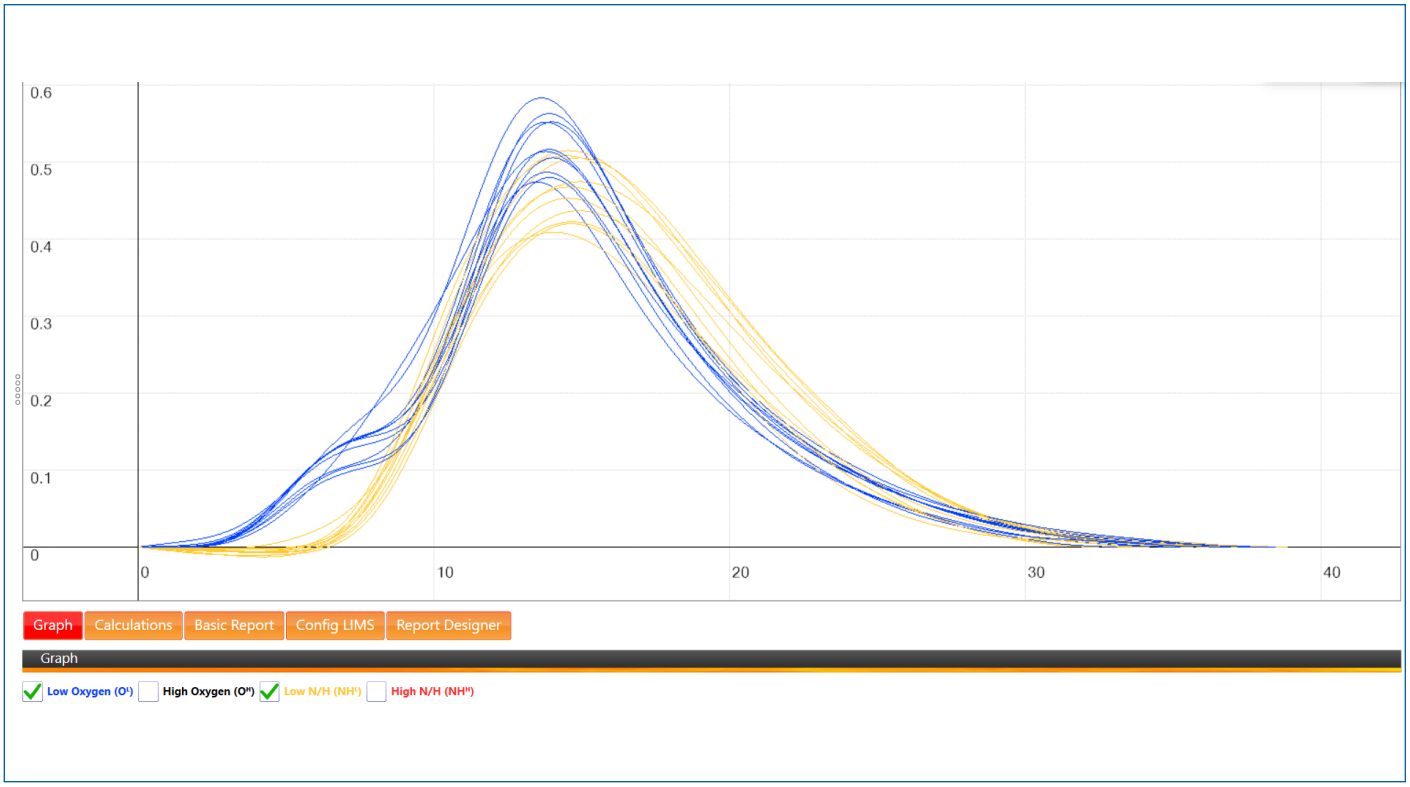
Oxygen and nitrogen determination in steel pins

Typical results		
AR 1653 Lot 819D		
Weight (mg)	**Oxygen (ppm)**	**Nitrogen (ppm)**
1955	7.39	33.61
1956	7.42	33.42
1962	7.78	33.61
1950	6.18	26.38
1953	6.84	27.36
1962	6.80	32.46
1959	7.48	29.38
1955	6.67	29.47
1970	6.92	25.73
1958	6.11	24.98
Mean value		
	6.96	29.64
Deviation / Relative deviation (%)		
	0.58 (8.3)	3.44 (11.6)
* Certified value: O 7 ± 4 ppm; N 30± 10 ppm		

The ELEMENTRAC ONH -p2 can process higher sample weights than 1000 mg.
Before applying these read the general recommendations at the end of this document and test if the applied sample amount will fi t into the crucible and sample port.



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The ELEMENTRAC ONH-*p* 2: 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.

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Application of higher sample weights (e.g. 2 gram)
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 fi t 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 4600 W has to be applied for significant higher sample weights than 1000 mg.

Irritating results (minor determination or high deviation)
Not consistent results could be traced to several reasons. Please check the chemicals (esp. the srubber 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,

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sample port to remove traces of powder.

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).