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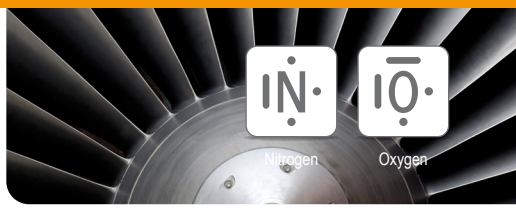
ELEMENTAL ANALYZERS

Oxygen and nitrogen determination in titanium samples



Suitable analyzers
- ELEMENTRAC ONH-*p 2*
- ELEMENTRAC ON-*p 2*

Used accessories
- Graphite crucibles (90180 & 90185)
- Nickel baskets (88600-0012)
- Suitable calibration material (NIST or other)

ELEMENTRAC ONH-*p 2*

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:	45 °C		

II) Purging

Purging while closing:	Enable
Closing purging time:	2 sec

III) Outgasing

Enable pulse:	Enable	Time:	10 sec
Pre-heat:	Disable	Power:	6500 W

IV) Stabilizing

Time:	60 sec
Power:	5600 W

V) Analyzing

Minimum furnace temp:	45°C	Open furnace:	Enable
Power duration:	180 sec	Cooling delay:	5
Power:	5600 W	Peak finding:	Drift compensation

VI) Post waiting

Time:	25 sec

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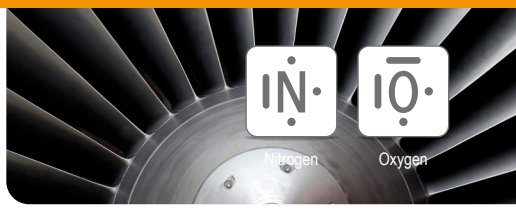

Channel Settings

Channel	Enable	Integration delay [sec]	Minimum time [sec]	Maximum time [sec]	Comparator factor [%]	Peak max [V]
Low & high oxygen	Enable	9	25	75	0.05	8
Low nitrogen	Enable	17	20	60	0.05	8
High nitrogen	Disable	-	-	-	-	-

Sample preparation
Make sure that the surface of the titanium is free from contaminations - otherwise clean the sample with acetone p.a. and let it air dry. Customer sample may require additional sample treatment (see recommendations at the end of this document)

ELEMENTRAC ONH-*p*

Procedure

- Prepare the ELTRA analyzer (exchange anhydrone, NaoH; 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 the crucible (90180 + 90185) on the electrode tip, close furnace
(2) Weigh calibration material, place it in a nickel basket and apply the nickel basket into the sample drop mechanism and start analysis
(3) Used graphite crucible (90180) 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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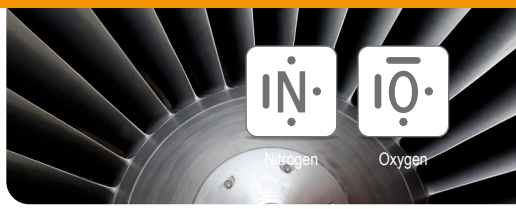
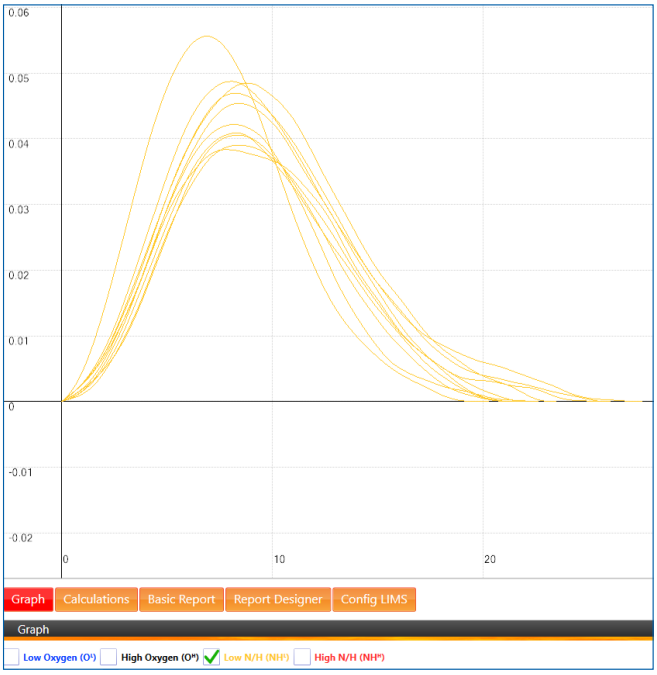
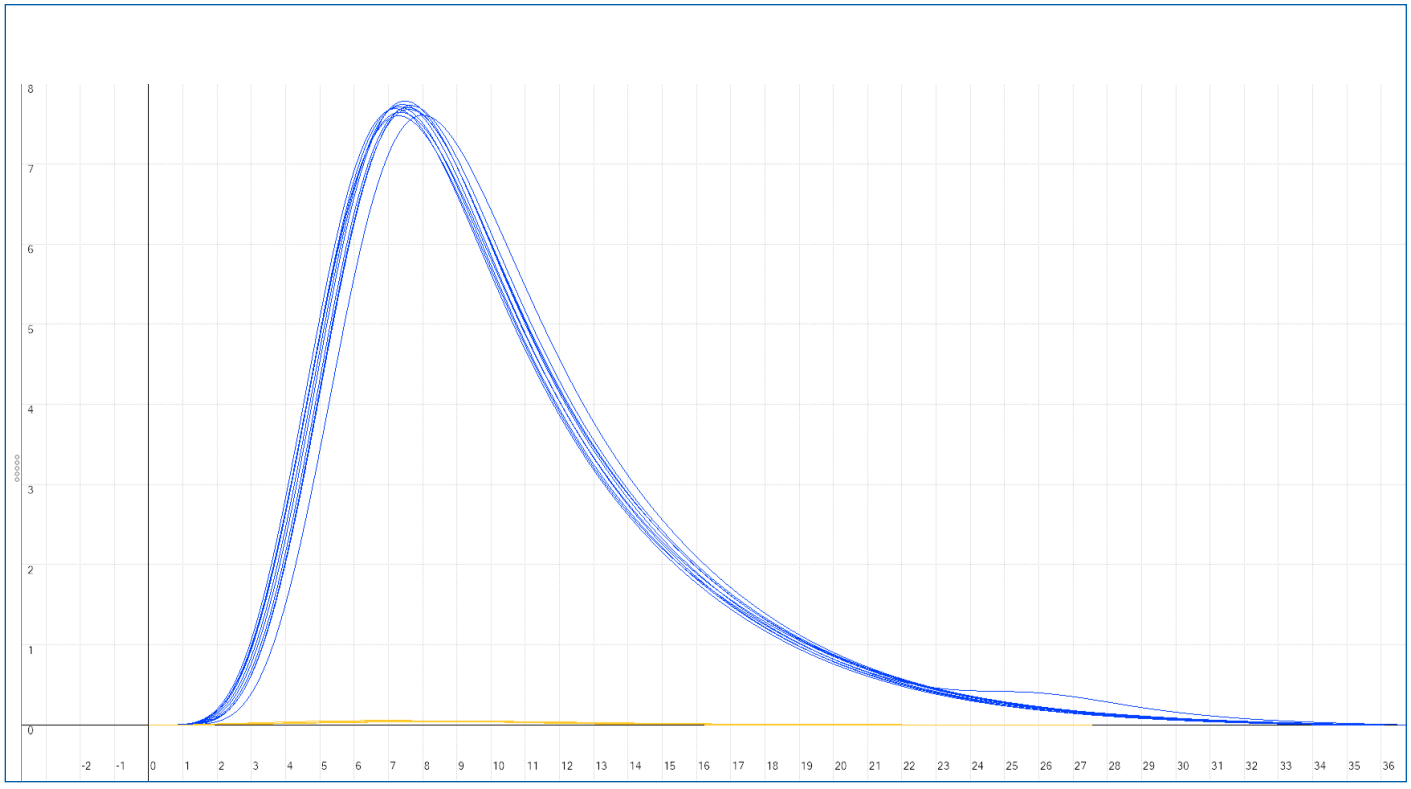


Typical results		
AR 631 (Lot 1019A) *1		
Weight (mg)	**Oxygen (%)**	**Nitrogen (%)**
102	0.359	0.0060
102	0.306	0.0067
102	0.356	0.0062
102	0.362	0.0067
101	0.360	0.0051
101	0.364	0.0053
101	0.367	0.0068
102	0.356	0.0051
102	0.358	0.0052
102	0.365	0.0056
Mean value		
	0.361	0.0059
Deviation / Relative deviation (%)		
	0.0036 (1%)	0.0007 (12%)
*1 Certified value: O 0.361 ± 0.0150 % N 0.0058 ± 0.0024 %		





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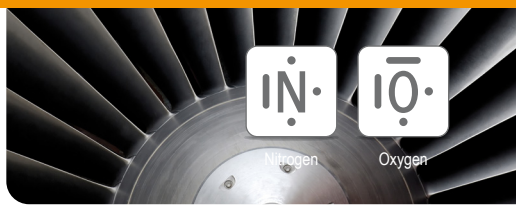
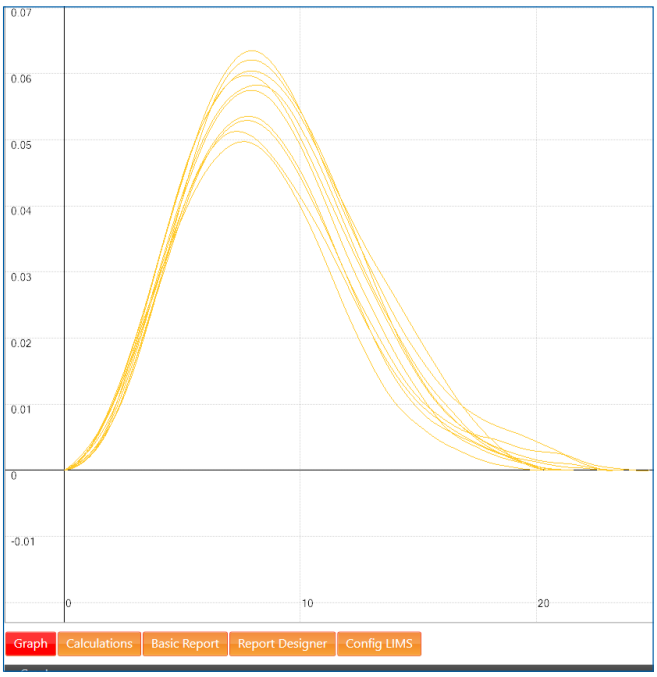
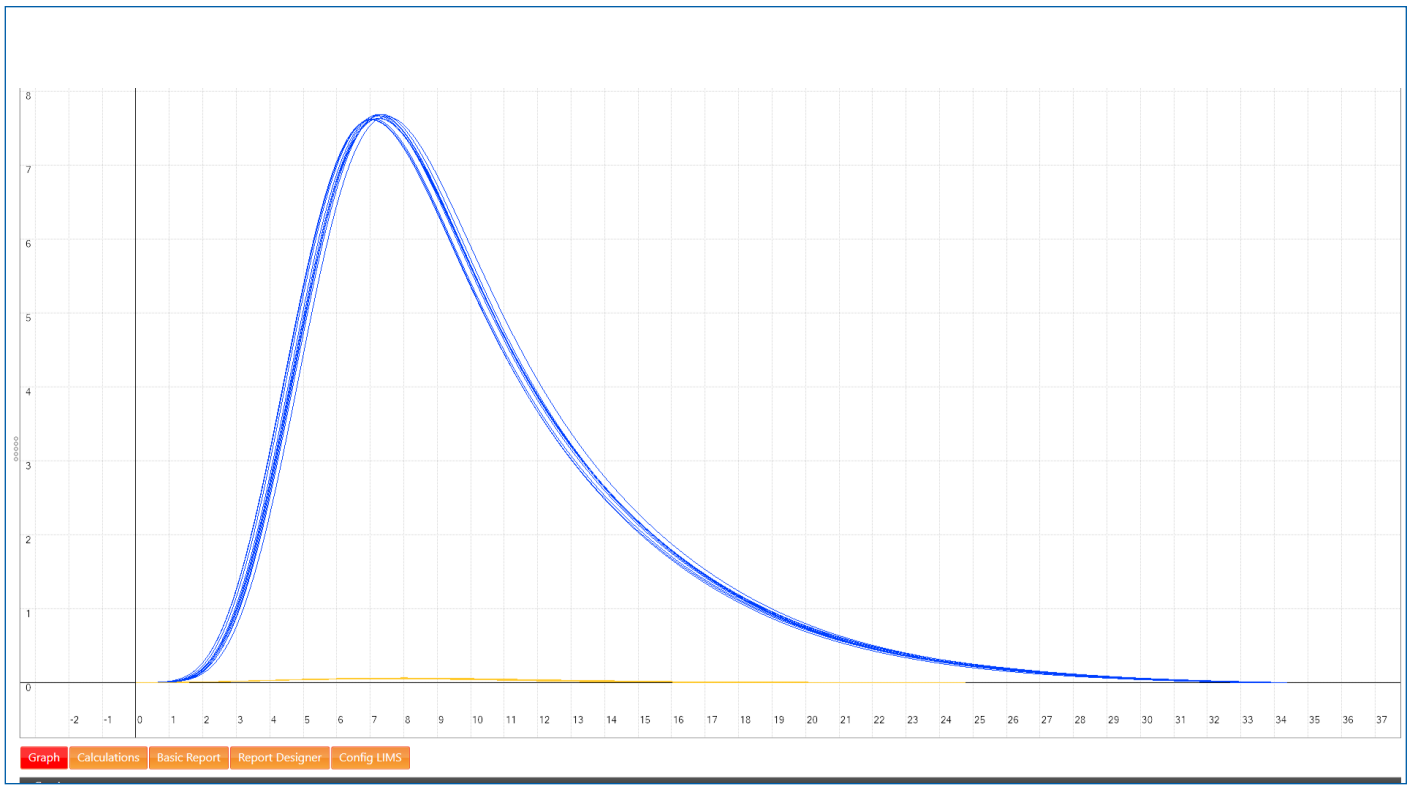


Typical results		
LECO 502-876 (Lot 0301) *1		
Weight (mg)	**Oxygen (%)**	**Nitrogen (%)**
116	0.310	0.0057
115	0.307	0.0065
116	0.309	0.0055
116	0.313	0.0071
115	0.312	0.0060
115	0.309	0.0051
116	0.308	0.0069
115	0.308	0.0056
115	0.309	0.0065
115	0.311	0.0066
Mean value		
	0.3100	0.0061
Deviation / Relative deviation (%)		
	0.0018 (0.6%)	0.0007 (10.9%)
*1 = Certified value: O 0.311 ± 0.008; N 0.006 ± 0.001		





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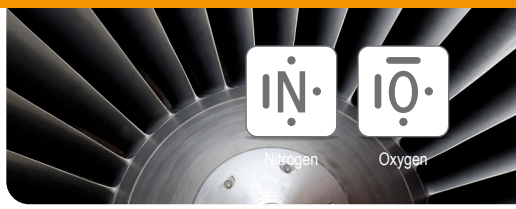
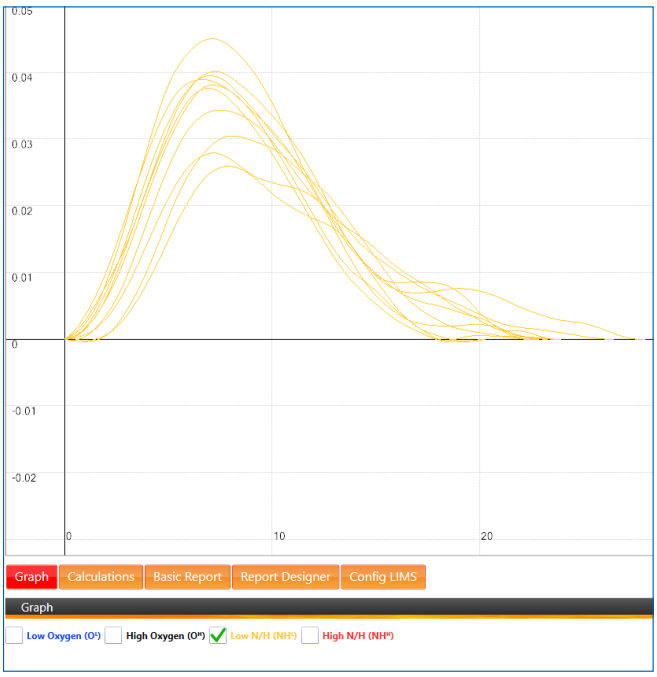
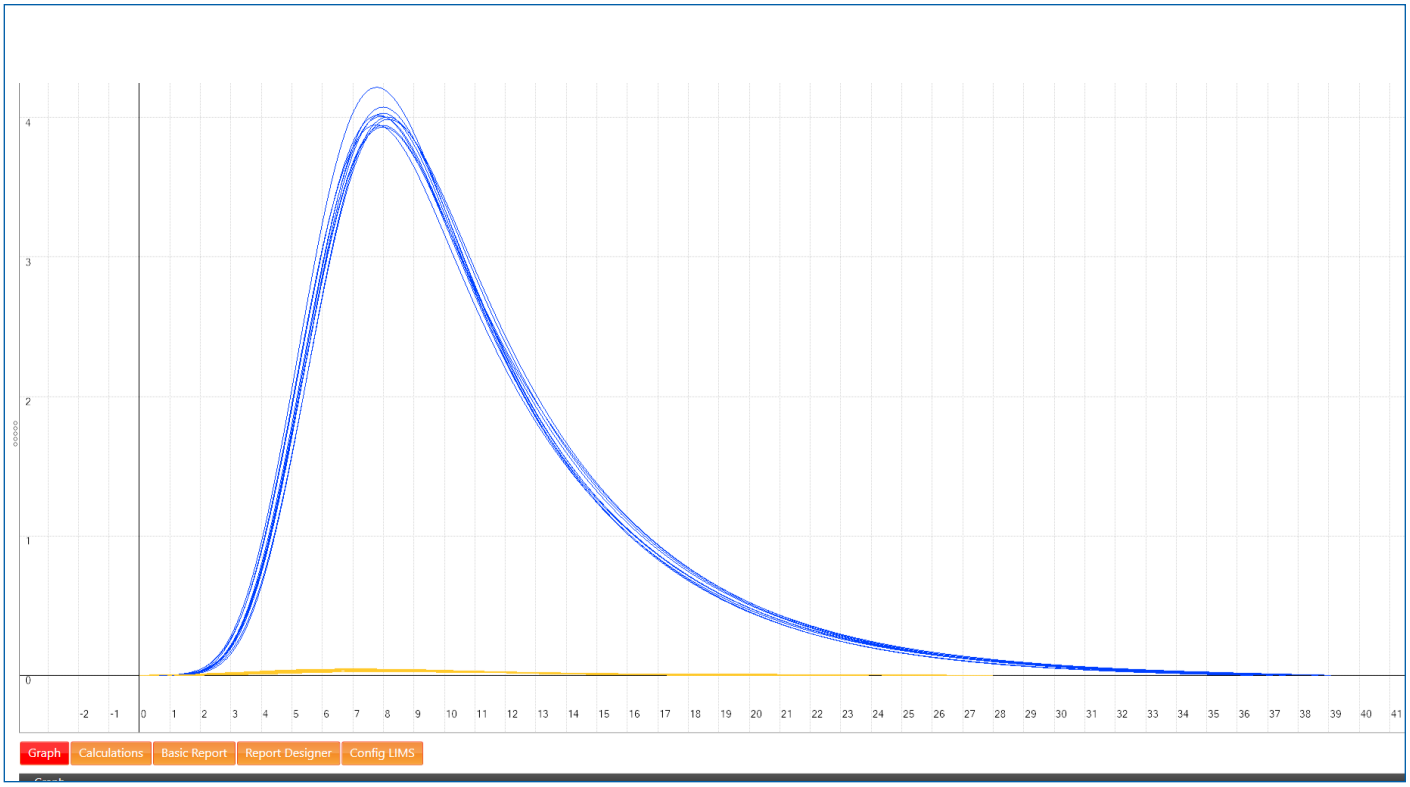


Typical results		
AR 649 (Lot 319G) *1		
Weight (mg)	**Oxygen (%)**	**Nitrogen (%)**
103	0.113	0.0086
103	0.113	0.0066
104	0.115	0.0064
104	0.111	0.0075
104	0.112	0.0070
104	0.116	0.0046
103	0.115	0.0060
103	0.115	0.0064
103	0.117	0.0081
106	0.118	0.0078
Mean value		
	0.1150	0.0069
Deviation / Relative deviation (%)		
	0.0022 (1.9)	0.0012 (16)
*1 Certified value: O 0.115 ± 0.009%; N 0.0067 ± 0.0015%		





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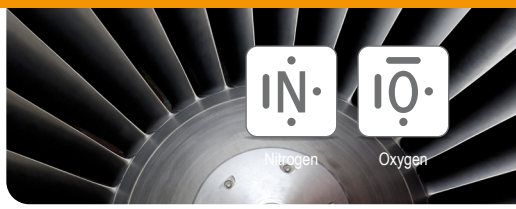
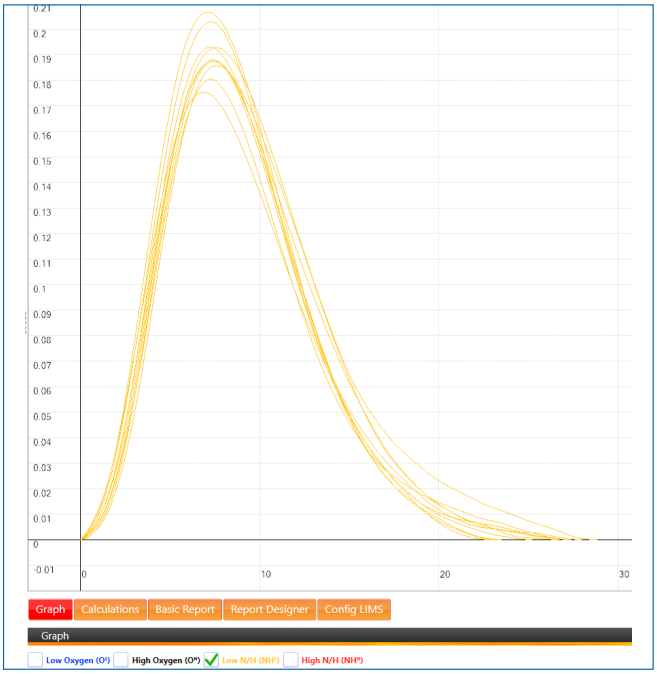
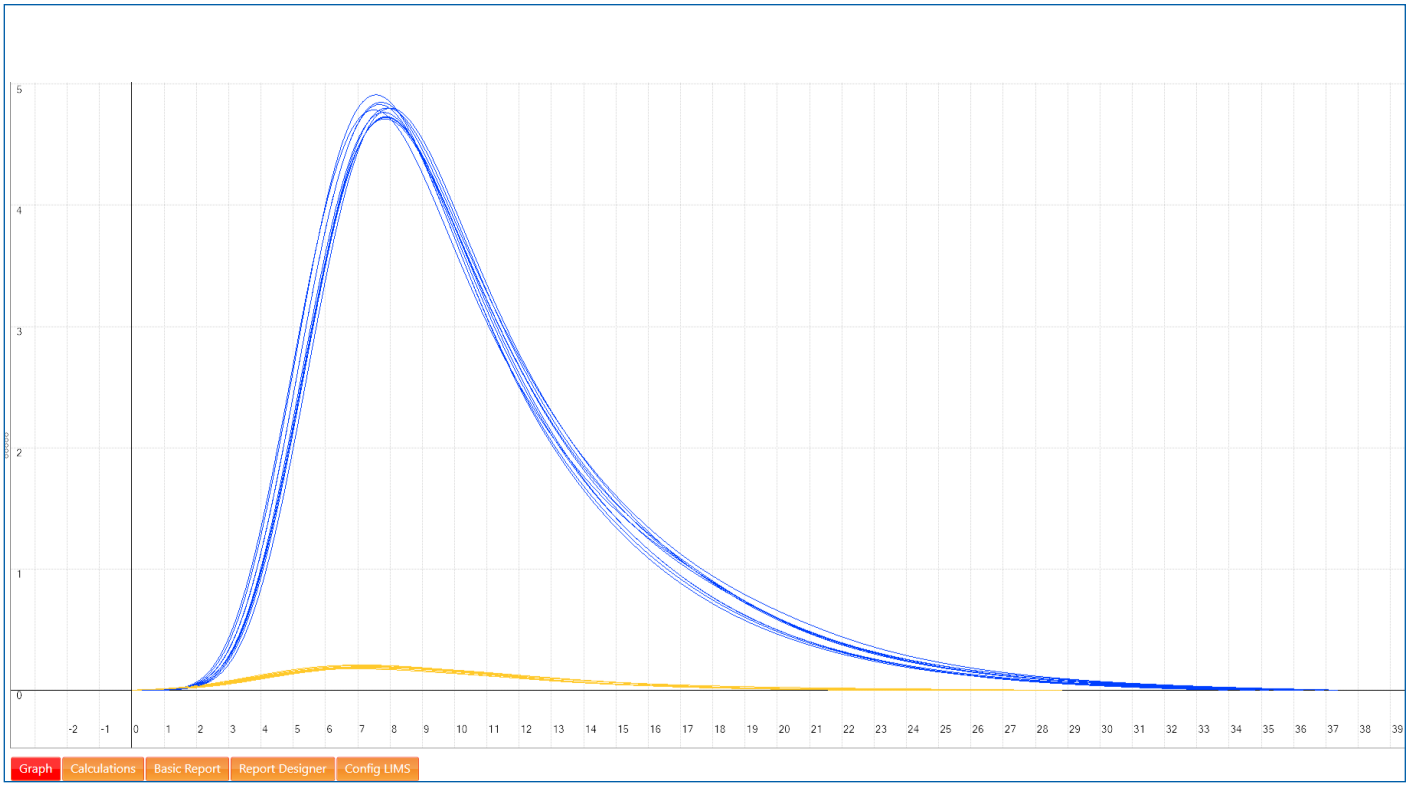


Typical results		
AR 642 Lot 319B		
Weight (mg)	**Oxygen (%)**	**Nitrogen (%)**
102	0.148	0.019
102	0.146	0.018
104	0.150	0.019
105	0.150	0.020
103	0.148	0.018
102	0.147	0.020
104	0.149	0.019
106	0.149	0.018
105	0.146	0.017
107	0.153	0.019
Mean value		
	0.149	0.019
Deviation / Relative deviation (%)		
	0.002 (1.4)	0.001 (5.8)
*1 Certified value: O 0.149 ± 0.006; N 0.0190 ± 0.004		





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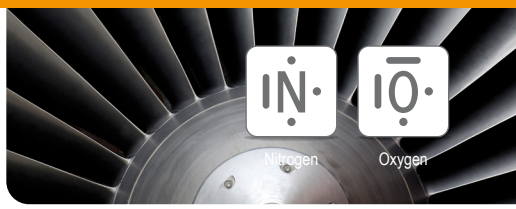


Formation of bubbles

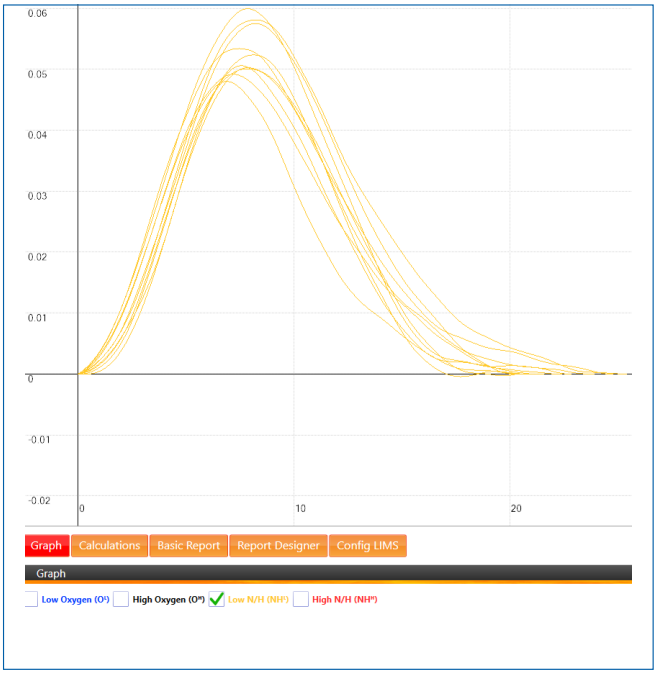
Some samples show during multiple measurements bubbles in the graphite crucible. Bubbles could be a sign for cleaning the upper and lower electrode and are an indicator for too hot furnace temperatures. Some sample profit from an alternative application setting like the following:

Setting	Alternative value
Outgasing	6000 W / 10 sec
Stabilizing	5800 W / 50 sec
Analysis power	5800 W

Please exchange the outer crucible every three measurements.

The following results have been obtained with this alternative settings without bubbles:

Typical results		
LECO 502-876 (Lot 0301) *1		
Weight (mg)	**Oxygen (%)**	**Nitrogen (%)**
114	0.306	0.0058
115	0.309	0.0060
115	0.311	0.0074
116	0.315	0.0068
115	0.310	0.0050
115	0.310	0.0057
115	0.312	0.0068
115	0.306	0.0053
115	0.315	0.0065
115	0.313	0.0056
Mean value		
	0.311	0.0061
Deviation / Relative deviation (%)		
	0.0034 (1.1)	0.0008 (12)
*1 Certified value: O 0.311 ± 0.008; N 0.006 ± 0.001		



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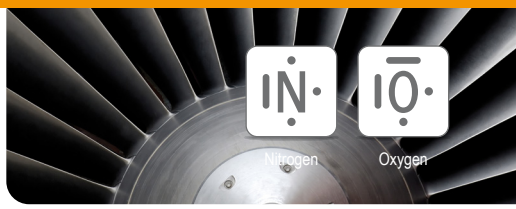

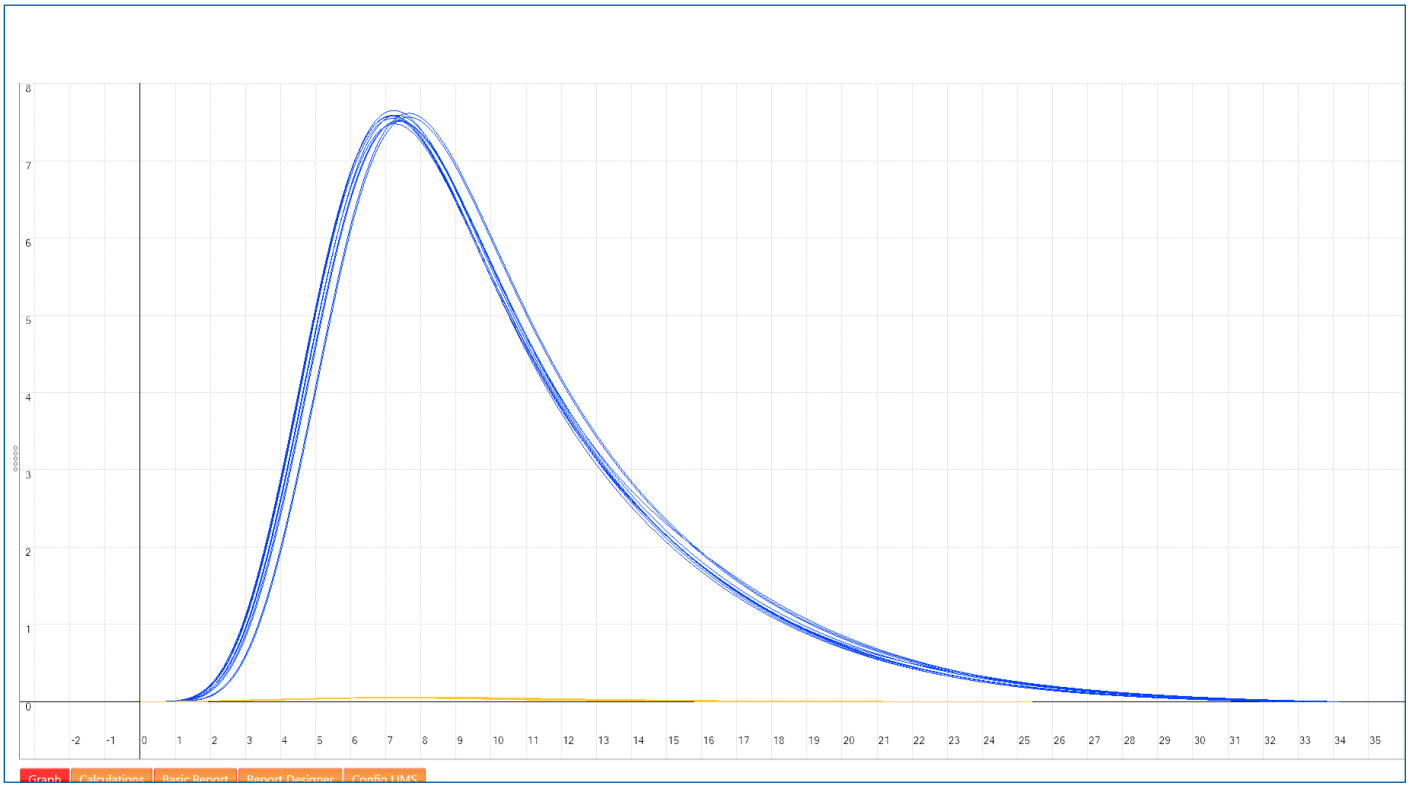

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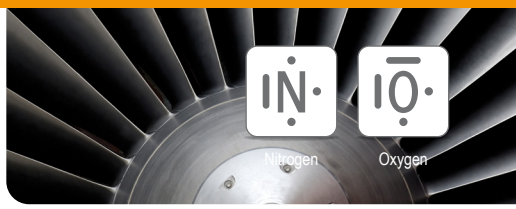

The ELEMENTRAC ONH-*p* 2: recommendations

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 every measurement. The outer crucible has been replaced every 10-15 measurements. When irritating results in combination with sputtering or bubbling samples have been obtained an earlier replacement of the outer crucibles may be required (see chapter irritating results).

ELEMENTRAC ONH-*p*

Preparation of measurements
Preparation of measurements encompass different procedures to achieve the best possible repeatability and reliability of oxygen and nitrogen measurements in titanium. These procedures affect the analyzer and its chemicals and sometimes preparation of the calibration material (CRM´s), nickel baskets (flux) and samples. .

For precise measurements of oxygen and nitrogen in titanium the analyzer should be well maintained. The upper electrode and furnace should be clean and the lower electrode should not look worn. Glass wool, anhydrone, sodium hydroxide and copper oxide should be replaced according the recommendations in the operation manual.

Calibration material (CRM-s) and nickel flux (baskets)
Titanium based calibration material with certified oxygen and nitrogen content is available from different suppliers (e.g. ELTRA). This calibration usually does not have to be prepared separately. Typically the calibration material is available in the shape of small pins with a typical weight of 90 -130 mg. When this calibration material is not well prepared, stored or transported dust could cover the calibration material. In this case treatment with acetone could be useful to remove dusty residues:
Calibration material and customer samples have to be placed into a nickel basket (flux). These baskets are available in superior quality at ELTRA (88600-0012) and do not require an additional preparation. When baskets from other suppliers are used they may require an etching process according the ASTM E 1409-13. The baskets have to be immersed for 50 -60 seconds into a fresh cleaning solution of 75 ml acetic acid, 25 ml HNO_3 and 2 ml HCl. The edged baskets should be rinsed in running water for 2-3 minutes, followed by removing the excess water with a paper towel. Afterwards the baskets has be washed with acetone and stored under acetone.
Pre edged baskets like ELTRA 88600-0012 could require a cleaning with acetone when contamination with dust is possible.

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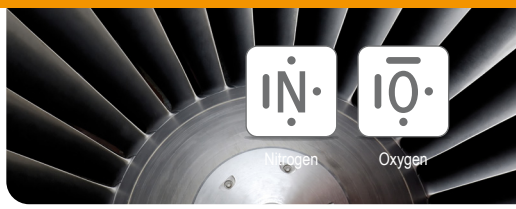

Sample preparation (for solid samples according ASTM E 1409-13)

The surface of a sample could contains more oxygen than the bulk sample. For a correct oxygen measurement mechanical surface treatment with a file or a lathe maybe required. Alternatively chemical treatment with pickle solution (HF acid and H2O2) could be useful. Further information are available in the ASTM E 1409 standard.

Application and storage of titanium based powders

Powders (e.g. 50-100 mg) have to be filled in a suitable nickel capsule (e.g. ELTRA 90257). The capsule with powder has to be applied into the nickel basket. Sample preparation like treatment with acids, solvents or any mechanical treatment is not applicable for powders. Never the less the oxygen content in titanium based powders can increase over time when the powder sample is not stored under inert gas atmosphere (nitrogen or argon):
When powders are analyzed disable the setting "Purging while closing".

Typical sample weight

According the ASTM E 1409 the sample weight should be between 0.1 and 0.15 g for solid samples. For powder analysis ELTRA recommends sample weights between 0.05 and 0.15 g.

Irritating results

Too low O/N values

In most cases too low results for oxygen and esp. nitrogen are caused by not sufficient temperature in the furnace or crucible. It may be suitable to run 2-3 Blanks after a longer measuring pause (e.g. after 60 Minutes) to warm up the analyzer. Some titanium samples like TiAl6V4 require a higher power in comparison to standard titanium. In this case the applied stabilizing and analysis power should be increased to 6000 W. When titanium based powders are measured with a too low O/N content the reduction of sample weight is recommended.

Too high O/N values

Too high nitrogen or oxygen contents could be caused by wrong sample preparation. E.g. the sample was burned during mechanical treatment which causes a higher oxygen content. Abrasive papers which get in contact with the sample should be free from oxygen, because excess oxygen could be introduced into the sample. When powders are measured with a too high oxygen content it maybe was not stored under an inert gas and oxidation of the sample occurred. Absurd high nitrogen concentrations (e.g. 1 % N instead of 0,005 %) are mainly caused by the saturated filter in front of the TC cell. Replace anhydrone and NaOH according the instructions in the operation manual.

Increased deviation for O/N measurements

A higher deviation could have several reasons:

a) Maintenance status ELEMENTRAC ONH-p 2 (dirty upper electrode, worn lower electrode, worn chemicals)
b) Not well prepared samples (e.g. different residues of acids, contamination with oxygen, overheating)
c) Not sufficient temperature (no warm up samples, too less power for special titanium samples)
d) Too many samples in short time. (Eltra recommends to increase the post waiting to 75 seconds)
e) Bubbling: Please try the alternative setting mentioned above.

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