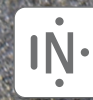


Oxygen and nitrogen determination in nickel powder



Nitrogen



Oxygen

Suitable analyzers

- ELEMENTRAC ONH-p2
- ELEMENTRAC ON-p 2

Used accessories

- Graphite crucibles (90180 & 90185)
- Suitable nickel capsules (e.g. 90257 or 88400-0066)
- 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

Oxygen and nitrogen determination in nickel powder



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

For safe analysis the powder has to be filled in a nickel capsule. The filling volume should not exceed 80 % of the nickel capsule. A sealing of the capsule is not required.

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)) or 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 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.



ELEMENTRAC ONH-p

Oxygen and nitrogen determination in nickel powder



Nitrogen

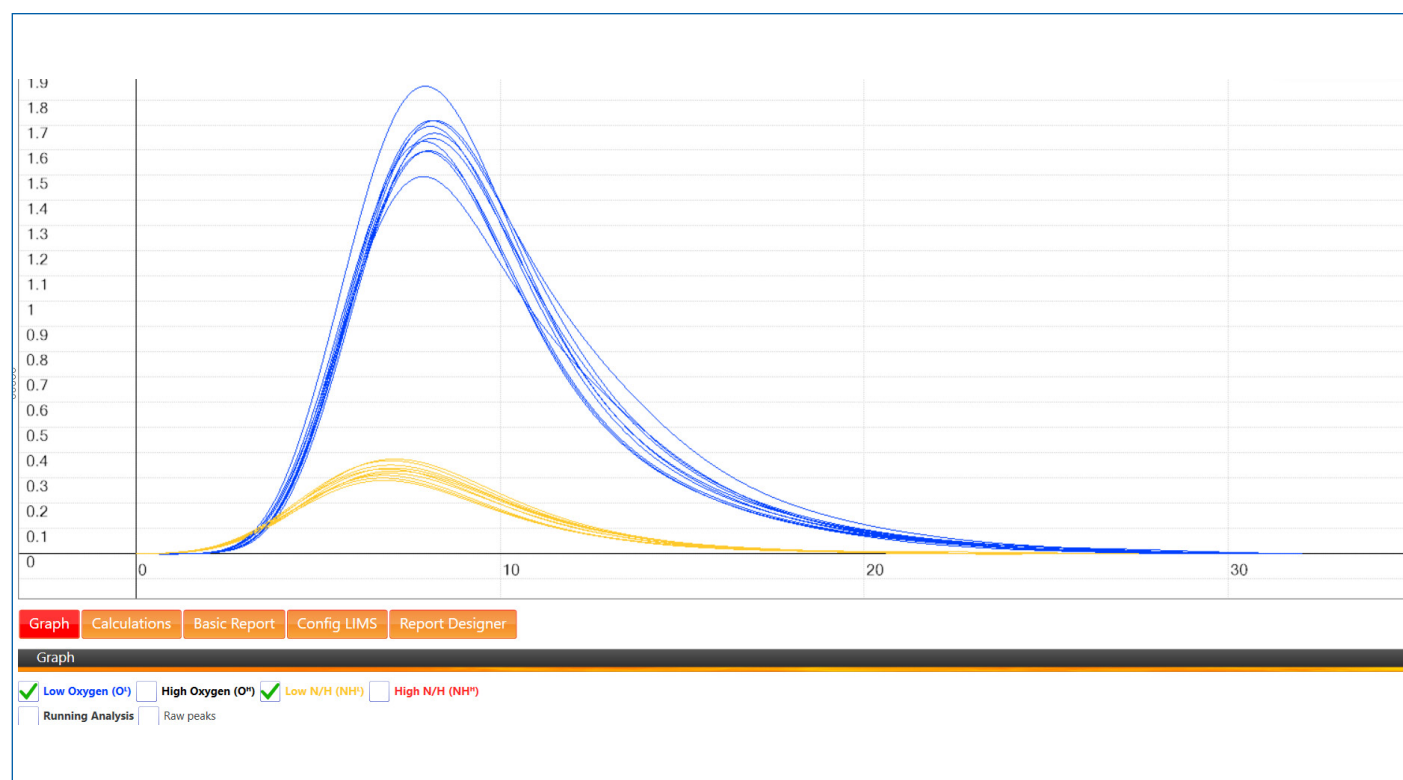


Oxygen

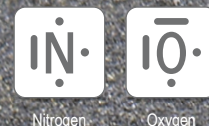
Typical results

Nickel powder ASTM AMPM 2010

Weight (mg)	Oxygen (ppm)	Nitrogen (ppm)
260	198	108
250	199	111
225	202	105
243	199	108
227	203	108
287	202	106
233	203	107
291	203	105
270	202	109
255	199	108
Mean value		
	201	107
Deviation / Relative deviation (%)		
	2.0 (1.0)	1.7 (1.6)
* Certified value: not available		



Oxygen and nitrogen determination in nickel powder



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. Single usage is recommended.

Recommended sample weight

For this application sample weights of approx. 250-300 mg have been used. It is possible to use higher (e.g. 500 - 1000 mg) or lower sample weights (e.g. 50 mg). With lower sample weights the homogeneity of the sample becomes very important. It may be possible that the deviation for oxygen and nitrogen measurements increases. Heavier sample weights may do not fit into the capsules. In this case large nickel crucibles (90256) may be required. ELTRA recommends to fill a capsule only to 80 % of its maximum volume to avoid sample loss during the transfer from the sample port to the hot crucible. A sealing of the capsules is not required.

Blank value of the capsule

For calibration steel pins (e.g. 91100-1004) have been used. After calibration three empty nickel capsules have been analyzed with a nominal weight of 250 mg. These values have been defined as "blank".

The influence of the blank value of the nickel capsules depends on the applied sample amount and the measured sample concentrations. ELTRA recommends to run 2-3 empty nickel capsules with a nominal weight which was used for sample analysis (here: 250 mg). The measured results indicate if the blank value is significant or not.

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 and sample port to remove traces of powder. When irritating results occur when using high sample weights (e.g 500-1000 mg or more) reduce the sample weight.

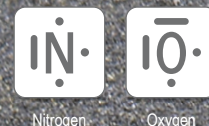
Alternatively increase the outgasing time for a more efficient removal of atmospheric gases.

Please check the blank value of the capsules to ensure a stable blank value.



ELEMENTRAC ONH-p

Oxygen and nitrogen determination in nickel powder



Unstable oxygen results

Depending on the fineness of the sample and sample storage additional oxidation of the nickel powder could take place. For safe and stable oxygen results a storage under argon atmosphere may be suitable.

Minor determination of oxygen and nitrogen after a long measurement break

The results in this application note have been obtained with an analyzer which was warmed up with 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-heating function (application setting) and apply a longer outgasing time (20 seconds).