

Oxygen and nitrogen determination in titanium samples



Suitable analyzers

- ELEMENTRAC ONH-p
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Used accessories

- Graphite crucibles (90180 and 90185)
- Nickel basket high purity (88600-0012)
- Suitable calibration material (NIST or other)



Application Settings

I) General

Furnace mode: ON

Furnace cooling: 35/45 °C

Catalyst furnace: 650°C

Standby Flow: 0

A flow of 10 l/h could improve precision when there is a long time distance between 2 measurements.

II) Outgasing and stabilizing

Setting / Phase	Time [sec]	Power [W]	Flow [l/h]
Outgasing	60	6000	27
Stabilizing	75	6000	27

A second outgasing cycle or an increased outgasing time could improve the precision for very low oxygen and nitrogen contents. To reduce dust it could be useful to split the outgasing cycle in 30 sec (0 W Power) and 30 sec (6000 W Power).

III) Analysis

Power duration: 70 sec

Drift compensation: on

Power: 6000 W

Open furnace: yes

Flow: 27 l/h

Channel	Minimum time [sec]	Maximum time [sec]	Integration delay [sec]	Comparator factor [%]
Low and High O	35	70	7	0.3
Low and High N	25	70	10	0.3

IV) Postwaiting

Postwaiting time: 35 sec

Furnace clean up: No

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Sample preparation

Make sure that the surface of the titanium is free from contaminations; otherwise clean the sample with acetone p.a. and let dry at atmosphere.

Procedure

- Prepare ELTRA analyzer (exchange anhydron, sodium hydroxide, Schuetze reagent if necessary), clean furnace, sample drop mechanism, electrode tip (if necessary)
- Run three blanks with empty crucibles
- Calibrate the analyzer with suitable calibration material (NIST or other)
 - (1) Fill one empty inner crucible (90180) in one outer crucible (90185) and place them on the electrode tip, close furnace
 - (2) Weigh calibration material and place it in a nickel basket (88600-0012) and seal it; place the nickel basket with the sample in the sample drop mechanism and start analysis
 - (3) After analysis give the inner crucible into waste and fill in a new one. The outer crucibles can be used approximately 10 times

Repeat steps (1) – (3) at least three times;
 Mark the results and use the calibration function in the software.

-> Now start with the actual analysis.



Typical results		
91205-1004 Titanium Standard (LOT 613 B)		
Weight (mg)	% O	ppm N
106.2	0.2107	49.9
104.5	0.2141	63
105.3	0.202	48.6
107.7	0.2151	63.2
101.5	0.2236	65.8
103.3	0.219	72.4
102	0.2256	69.7
105.2	0.2096	77.2
106.1	0.2177	68.9
105.8	0.2135	69.2
Average values		
	0.215	64.79
Deviation / Relative deviation (%)		
	0.007 / 3.21 %	9.21 / 14.21 %