Application Note 1046





Oxygen and nitrogen determination in titanium samples (reduced power, less dust)



Suitable analyzers

- ELEMENTRAC ONH-p
- ELEMENTRAC ON-p

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 Standby flow: 0

Furnace cooling: 35 / 45 °C A flow of 10 l/h could improve precision Catalyst furnace: 650 °C when there is a long time span between

two measurements

II) Outgassing and stabilizing

Setting/Phase	Time [sec]	Power [W]	Flow [l/h]	Cool down [sec]
Outgassing (1. cycle)	10	0	27	NN
Outgassing (2. cycle)	35	5700	27	0

:	Setting/Phase	Minimum time [sec]	Maximum time [sec]	Power [W]	Flow [l/h]	Stability [V]
	Stabilizing	70	70	5500	27	0

III) Analysis

Power duration:80 secDrift compensation:onPower:5500 WOpen furnace:yes

Flow: 27 l/h

Channel	Minimum time [sec]	Maximum time [sec]	Integration delay [sec]	Comparator factor [%]
Low and high O	25	70	7	0.3
Low and high N	30	70	10	0.3

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IV) Post waiting

Post waiting time: 35 sec Granulate mode: off

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

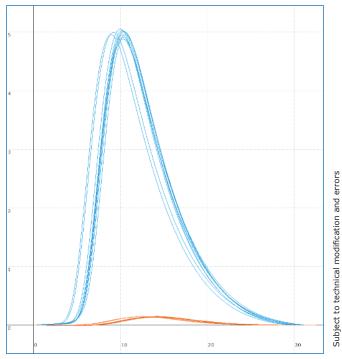
- Prepare ELTRA analyzer (e.g. exchange anhydrone, sodium hydroxide, copper oxide if necessary); clean the furnace, sample drop mechanism, electrode tip (if necessary)
- Run three blanks with empty crucibles
- Calibrate the analyzer with suitable calibration material (NIST or other)
 - (1) Fill one empty 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.

EMENTADO ELEMENTRAC ONH-p

→ Now start with the actual analysis.

Typical results				
ELTRA 91205-1003#1116B ¹⁾				
Weight (mg)	Oxygen (ppm)	Nitrogen (ppm)		
102.7	893.2	100.8		
103.4	917.2	101.8		
102.7	892.0	105.1		
101.9	878.9	98.4		
103.5	886.7	93.9		
103.2	904.5	97.8		
102.3	908.8	96.5		
103.8	882.9	103.3		
103.4	860.7	99.7		
103.4	877.8	94.0		
Mean value				
	890.3	99.1		
Deviation / Rel. deviation (%)				
16.7 / 1.9 % 3.7 / 3.8 %				



 $^{^{1)}}$ certified values: O: 890 ppm \pm 50 ppm; N: 99 ppm \pm 10 ppm

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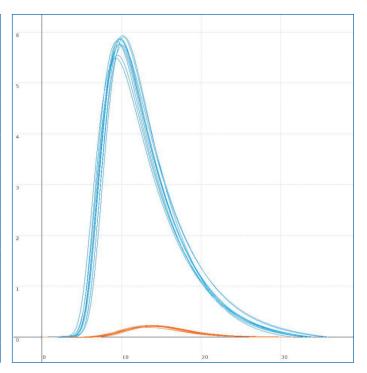




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Typical results				
LECO 502-891 ²⁾				
Weight (mg)	Oxygen (ppm)	Nitrogen (ppm)		
119.4	1150.6	95.8		
115.7	1114.3	86.5		
117.8	1159.5	104.7		
123.1	1149.7	98.9		
116.4	1205.1	97.7		
116.4	1206.7	105.1		
112.4	1183.0	101.5		
118.5	1180.6	106.0		
116.3	1120.3	93.8		
118.0	1171.1	107.4		
Mean value				
	1171.1	100.4		
Deviation / Rel. deviation (%)				
	37.9/3.2%	6.6/6.6%		



 $^{^{2)}}$ certified values: O: 1170 ppm \pm 90 ppm; N: 100 ppm \pm 10 ppm