

Oxygen and hydrogen determination in titanium hydride



Suitable analyzers

- ELEMENTRAC OH-p
- ELEMENTRAC ONH-p
(ELEMENTS Software 1.3 or higher)

Used accessories

- Graphite crucibles (90180 and 90185)
- Nickel capsules, pressed (88400-0066)
- Graphite powder (90800)
- Suitable calibration material (pure chemical or NIST)



Application Settings

I) General

Furnace mode: on
 Furnace cooling: 35 / 45 °C

Standby flow: 0
 A flow of 10 l/h could improve precision when there is a long time distance between two measurements

II) Outgassing and stabilizing

Setting/Phase	Time [sec]	Power [W]	Flow [l/h]
Outgassing (1. cycle)	30	5000	27
Outgassing (2. cycle)	15	1100	18
Stabilizing	120	1100	18

III) Analysis

Flow: 18 l/h
 Drift compensation: on
 Open furnace: yes

Duration / Power cycle 1	Duration / Power cycle 2	Duration / Power cycle 3
110 sec / 1100 W	5 sec / 3000 W	150 sec / 4800 W

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IV) Post waiting

Post waiting time: 10 sec
 Granulate mode: off

Sample preparation

Usually no sample preparation is required for analysis of titanium hydride. Due to long storage of this powder the hydrogen content could decrease, whereas the oxygen content could increase.

Procedure

- Prepare ELTRA analyzer. If necessary exchange anhydrous, sodium hydroxide, schuetze reagent and clean sample drop mechanism, furnace, electrode tip.
- Run three blanks with empty crucibles
- Calibrate the analyzer
 - With copper or other suitable calibration material for oxygen
No sample preparation or additional flux is necessary
 - With pure TiH_2 or other suitable CRM from NIST or other supplier
Fill in 25-30 mg sample into a pressed nickel capsule; seal it and apply it to the sample load. Add approx. 30 mg of graphite powder in the inner crucible, place inner and outer graphite crucible on the graphite tip and start analysis
- Repeat the calibration process in minimum 3 times and use the calibration function in the ELEMENTS software



→ Now start with current analysis

Typical results		
ALFA AESAR TiH ₂ 1-3 Micron powder LOT: R18E035		
Weight (mg)	% Hydrogen	% Oxygen
31.34	4.027	2.42
31.04	4.023	2.50
31.10	4.021	2.44
25.15	4.013	2.39
26.62	4.026	2.41
29.14	4.015	2.44
25.61	4.031	2.41
26.47	4.010	2.43
28.64	4.031	2.39
27.66	4.028	2.49
Mean value		
	4.023	2.43
Deviation / Rel. deviation (%)		
	± 0.0076 (0.2%)	± 0.03 (1.5%)

