

Nitrogen determination in silicon nitride



Suitable analyzers

- ELEMENTRAC ONH-*p*
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Used accessories

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



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 span between
 two measurements

II) Outgassing and stabilizing

| Setting / Phase | Time [sec] | Power [W] | Flow [l/h] |
|-----------------------|------------|-----------|------------|
| Outgassing (1. cycle) | 20 | 0 | 27 |
| Outgassing (2. cycle) | 30 | 5800 | 27 |
| Stabilizing | 75 | 5800 | 27 |

III) Analysis

Power duration: 80 sec
 Power: 5800 W
 Flow: 27 l/h

Drift compensation: on
 Open furnace: yes

| Channel | Minimum time [sec] | Maximum time [sec] | Integration delay [sec] | Comparator factor [%] |
|---------|--------------------|--------------------|-------------------------|-----------------------|
| High N | 35 | 80 | 10 | 0.3 |

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IV) Post waiting

Post waiting time: 20 sec
Granulate mode: off

Sample preparation

Make sure that the sample has a good homogeneity and is dry.
Drying one hour at 105°C maybe suitable.

Procedure

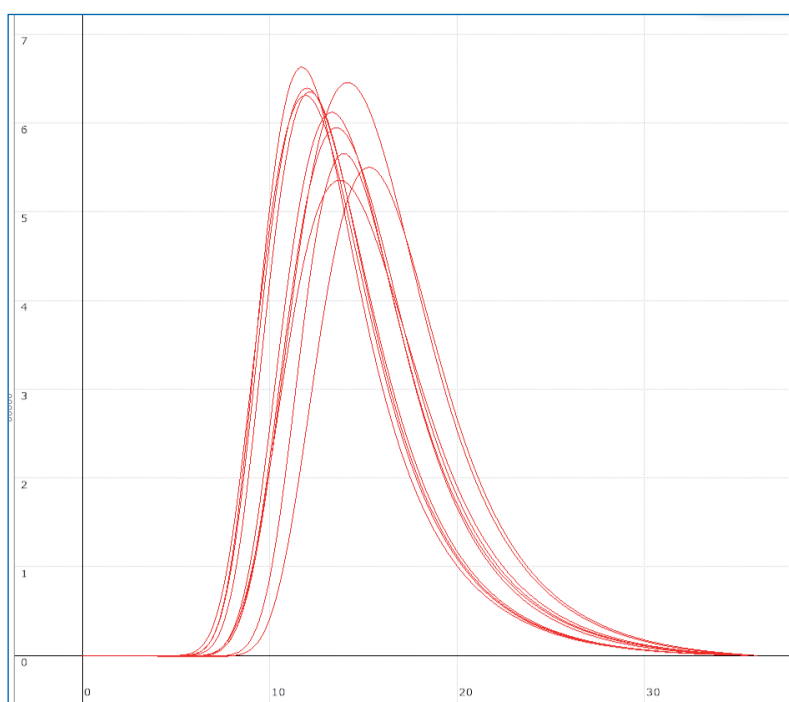
- Prepare ELTRA analyzer (e.g. exchange anhydron and/or sodium hydroxide if necessary); clean the furnace, sample drop mechanism, electrode tip (if necessary)
- Run three blanks with empty crucibles
- Calibrate the analyzer with suitable calibration material (NIST or other)
 - (1) Fill the inner crucible (90180) with a small amount of approx. 30 mg graphite (90800), put the inner crucible in the outer crucible (90185) and place them on the electrode tip. Close furnace.
 - (2) Weigh calibration material and place it in a nickel capsule (88400-0066) and seal it; place the nickel capsule 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 | | |
|----------------------------|-------------|--------------|
| ERM ED 101 ¹⁾ | | |
| Id | Weight (mg) | Nitrogen (%) |
| ERM ED 101 | 8.98 | 37.86 |
| ERM ED 101 | 10.16 | 38.20 |
| ERM ED 101 | 10.53 | 37.94 |
| ERM ED 101 | 10 | 38.25 |
| ERM ED 101 | 10.35 | 38.17 |
| ERM ED 101 | 10.36 | 38.21 |
| ERM ED 101 | 12.08 | 38.37 |
| ERM ED 101 | 9.96 | 38.20 |
| ERM ED 101 | 9.89 | 37.80 |
| ERM ED 101 | 10.5 | 38.33 |
| Mean value | | |
| | | 38.13 |
| Deviation / Rel. deviation | | |
| | | 0.19 / 0.51 |



¹⁾ certified value: N 38.1 ± 0.2