

Hydrogen determination in titanium samples



Suitable analyzers

- ELEMENTRAC ONH-p2
- ELEMENTRAC OH-p 2

Used accessories

- Graphite crucibles (90180 & 90185)
- Tin pellets (90251)
- Suitable calibration material (NIST or other)



ELEMENTRAC ONH-p2

Application Settings

I) General

Sample type:	Advanced	Cooling high:	60 °C
Use argon:	Off	Flow	40 l/h
Cooling low:	40 °C	Standby Flow:	40 l/h

II) Purging

Purging while closing:	Enable
Closing purging time:	2 sec

III) Outgasing

Enable pulse:	Enable	Time:	15 sec
Pre-heat:	Enable	Power:	6000 W
Pre-heat offset:	2 C		

IV) Stabilizing

Time:	65 sec
Power:	3500 W

V) Analyzing

Minimum furnace temp:	41°C	Open furnace:	Enable
Power duration:	180 sec	Cooling delay:	5
Power:	3500 W	Peak finding:	Drift compensation

VI) Post waiting

Time:	20 sec
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Channel Settings

Channel	Enable	Integration delay [sec]	Minimum time [sec]	Maximum time [sec]	Comparator factor [%]	Peak max [V]
Low & high oxygen	Disable	-	-	-	-	-
Low & high hydrogen	Enable	12	30	200	0.05	8

Sample preparation

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

Procedure

- Prepare the ELTRA analyzer (exchange anhydron, Schuetze reagent 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) Add two tin pellets (90251) to an empty inner crucible (90180) and introduce the inner crucible into an outer graphite crucible (90185)
 - (2) Place the crucible (90180 + 90185+90251) on the electrode tip, close furnace
 - (3) Weigh calibration material, place it in the sample drop mechanism and start analysis
 - (4) Used graphite crucible (90180) 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

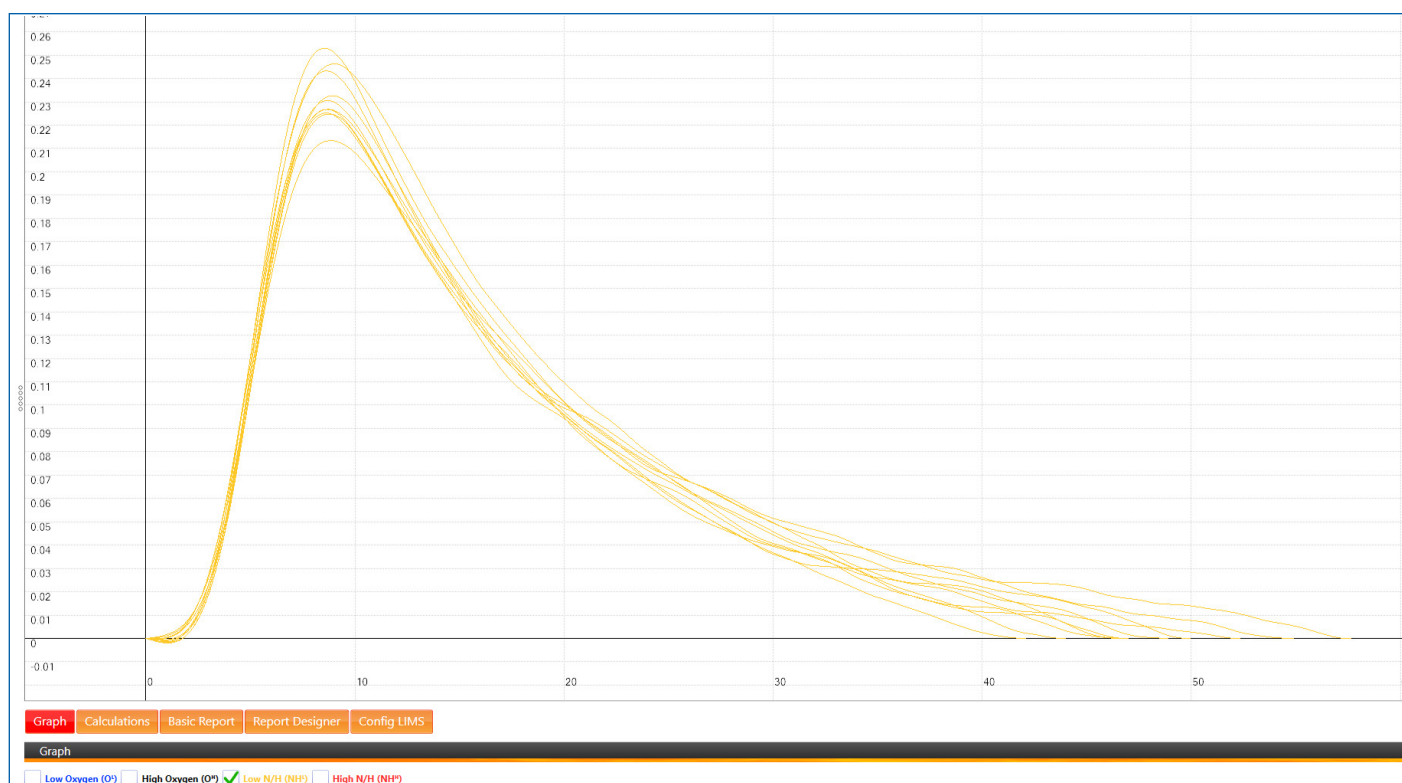
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Typical results

AR 631 (Lot 1019A) *1

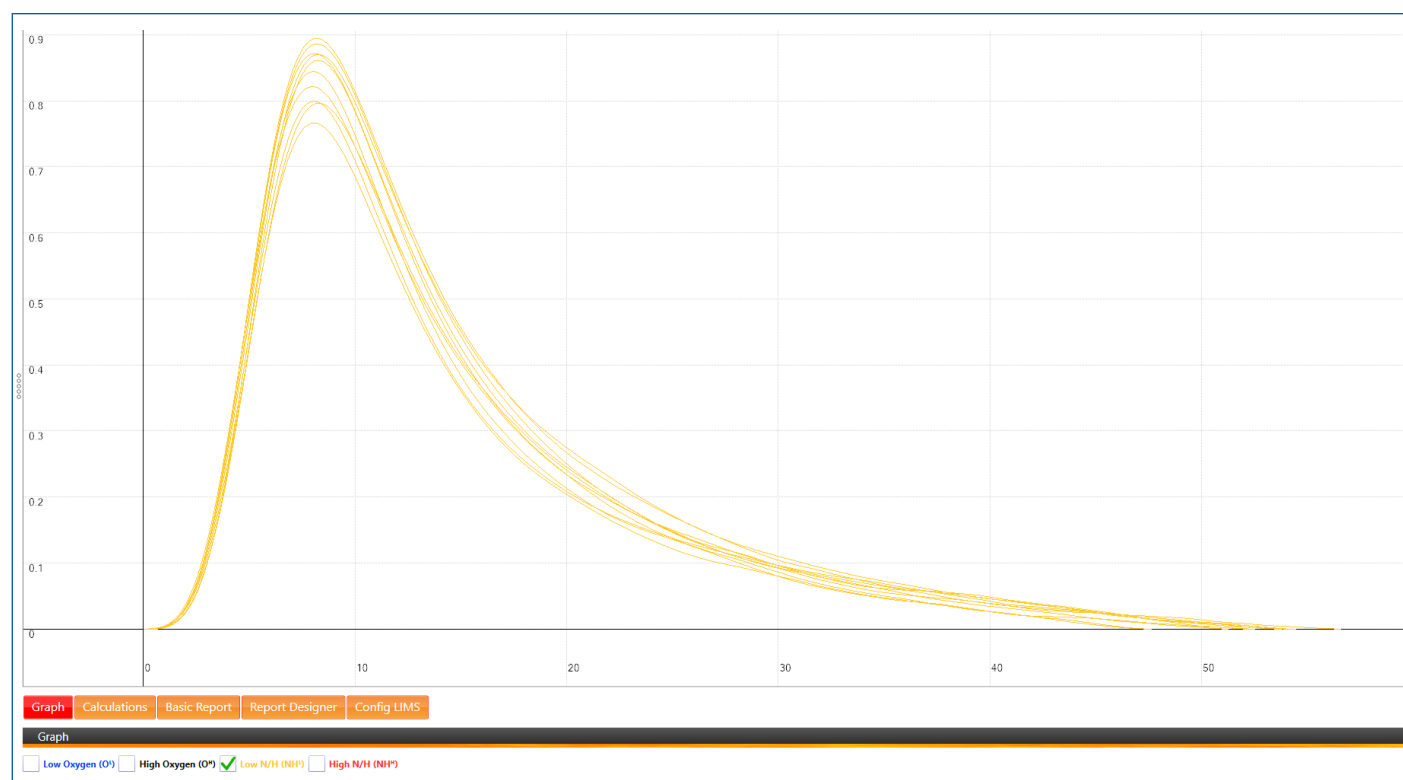
Weight (mg)	Hydrogen (ppm)
102	17.00
101	16.44
103	15.17
102	15.27
101	16.82
102	15.53
102	16.05
102	15.57
102	16.04
103	16.36
Mean value	
	16.02
Deviation / Relative deviation (%)	
	0.63 (4.0%)
*1 Certified value: H 16 ± 3 ppm	



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Typical results	
AR 642 (Lot 319B) *1	
Weight (mg)	Hydrogen (ppm)
106	48.84
105	54.03
106	44.40
107	51.69
105	54.77
105	49.43
106	47.01
104	50.71
105	48.30
103	52.73
Mean value	
	50.19
Deviation / Relative deviation (%)	
	3.24 (6.4%)
*1 = Certified value: H 50 ± 9 ppm	



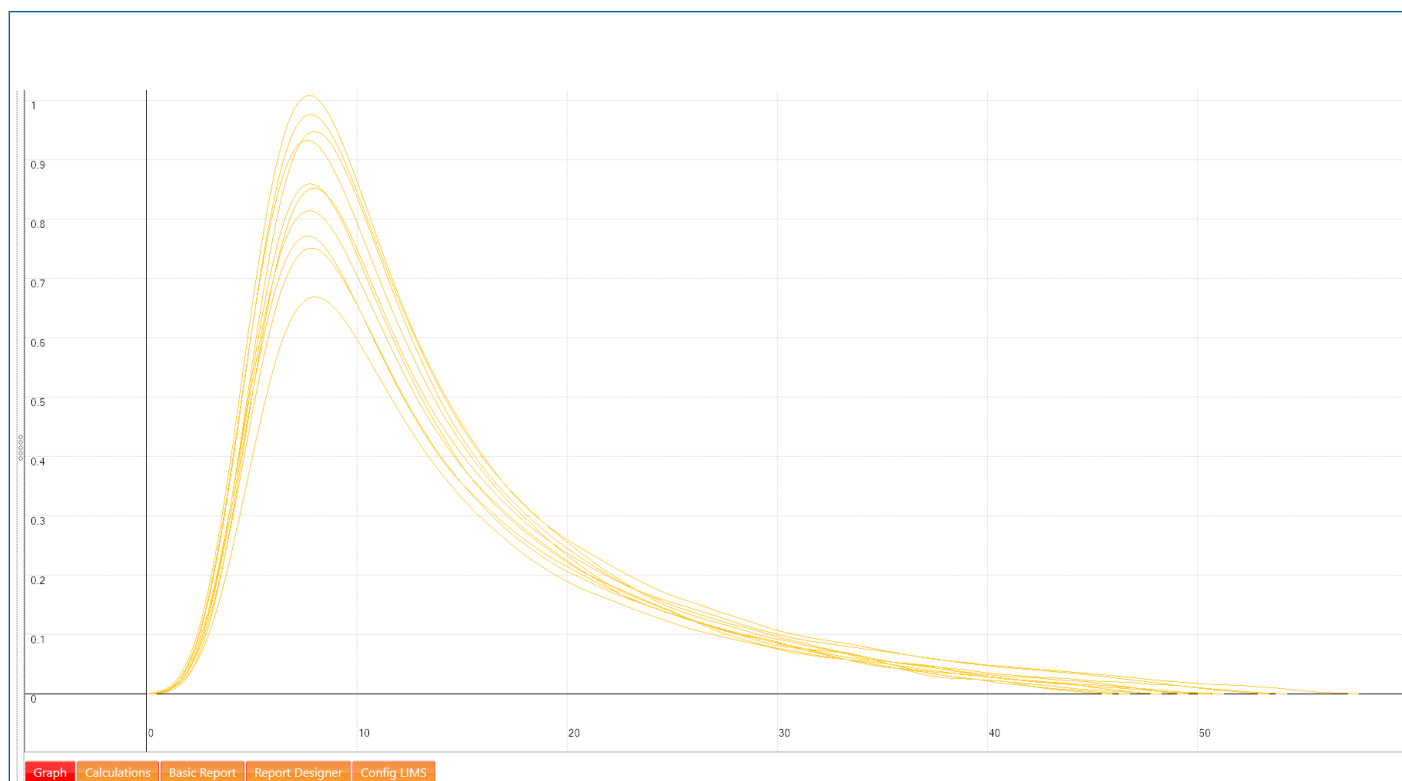
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Typical results	
AR 649 (Lot 319G) *1	
Weight (mg)	Hydrogen (ppm)
104	51.98
103	42.54
103	54.58
104	58.86
105	54.58
103	50.76
104	46.20
105	46.02
100	54.76
104	48.09
Mean value	
	50.84
Deviation / Relative deviation (%)	
	5.06 (10%)
*1 Certified value: H 50 ± 9 ppm	

Note:

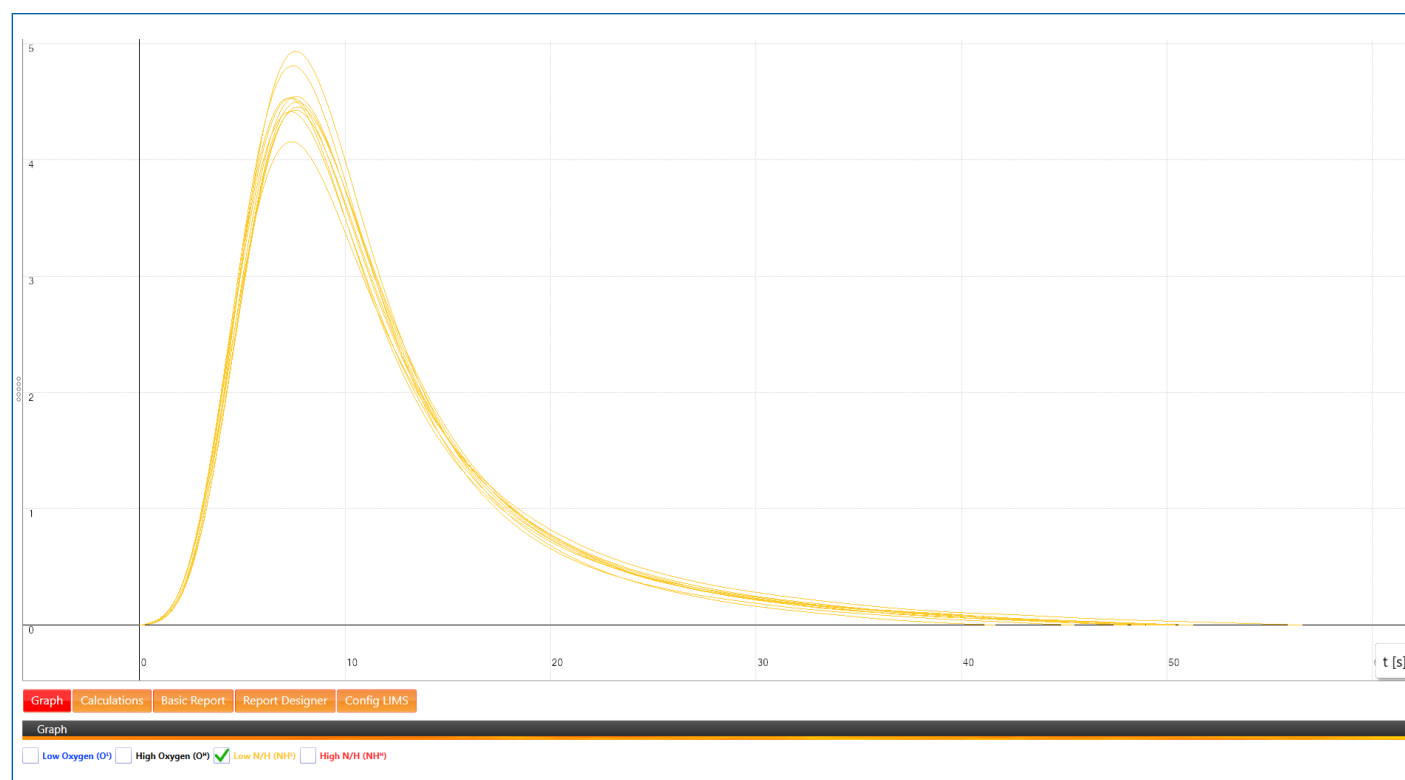
Cleaning with Acetone could be useful for customer samples to remove organic contaminations or dust residues which may contain traces of TiH₂. CRM's in general do not require a pre cleaning with acetone. A precleaning could improve the repeatability, but it is not a must. For comparison results of the CRM AR 642(Lot 319 B) are listed which have been pre cleaned with acetone. In comparison to the measurement without pre cleaning the repeatability is in the same range, but slightly worse.



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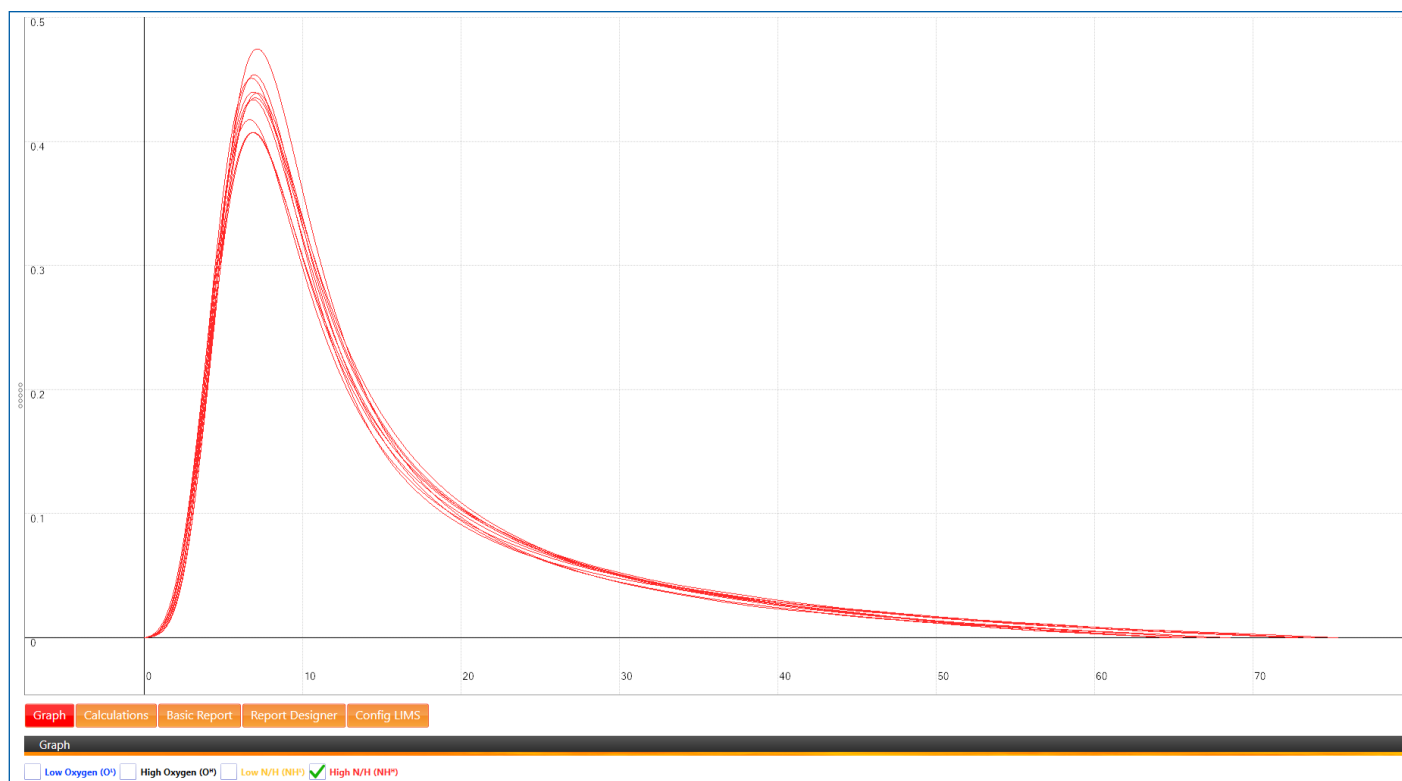
Typical results	
AR 642 (Lot 319B) *1	
Weight (mg)	Hydrogen (ppm)
104	190
103	191
104	190
105	189
103	190
104	188
102	189
103	191
104	193
104	193
Mean value	
	191
Deviation / Relative deviation (%)	
	1.53 (0.8)
*1 Certified value: H 192 ± 7 ppm	



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Typical results	
AR 589 (Lot 814C) *1	
Weight (mg)	Hydrogen (ppm)
254	191
251	194
251	189
254	192
252	192
252	192
251	188
254	198
252	191
252	187
Mean value	
	192
Deviation / Relative deviation (%)	
	3.2 (1.7)
*1 Certified value: H 192 ± 10 ppm	



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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 every measurement.

The outer crucible has been replaced every 10-15 measurements. When irritating results in combination with sputtering samples have been obtained an earlier replacement of the outer crucibles may be required (see chapter irritating results).

Irritating results

The accuracy and repeatability of results is influenced by many parameters. Regarding the ELEMENTRAC ONH-p2 the following maintenance steps should be considered:

- * Clean the upper electrode (and furnace) to remove tin dust
- * Replace the outer crucible. This is esp. recommended when the sample has contact with the upper electrode.

The following pictures (example 1 & 2) illustrate some invalid measurements:

- * Replace the chemicals (Schuetze reagent as well as NaOH and Magnesium perchlorate)



ELEMENTRAC ONH-p



Example 1:
Tin layer on the crucible



Example 2:
Sample which migrates
to the upper electrode

Irritating results as well as minor or over determination of hydrogen could also be due to wrong sample preparation (see next chapter)

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Sample preparation

The sample preparation is described in the ASTM E 1447-09(16) in the chapter 11. As mentioned above CRMs could be treated with Aceton followed by air drying, but it is not recommended strictly. Samples with unknown concentrations could be abraded with a file or maybe turned down with a lathe. In both cases treatment with Acetone is recommend before the sample is applied to the ELEMENTRAC ONH-p 2. The sample should only be treated with clean tweezers or forceps to avoid contaminations. When the sample is anyhow mechanically treated (with a file or lathe) the user should make sure that not too much heat is introduced into the sample. This overheating could cause minor determination of hydrogen.

In case of powder analysis no sample treatment is recommended, but for correct hydrogen analysis the sample should be filled in a nickel capsule (e.g. 90257, 88400-0066).

Sample weight

According the ASTM E 1447 the sample weight should not exceed 300 mg. Typical amounts for titanium samples are about 60- 300 mg for solid samples and 50 – 150 mg for powders. When lower sample weights have to be analyzed (e.g. in stents) the user should pay strong attention to the sample preparation to avoid any form of contamination. When the sample (e.g. stent) shows a high surface (volume) it may be useful to proceed a test if the sample will fit into the sample port and crucible.

Samples with high volume (surface)

Depending on the sample geometry a preliminary investigation may be useful to test if the sample will fit into the sample port and crucible. This could be tested by applying the sample to the furnace and click the “clean furnace” button in the analyzer status window. When the sample falls without blocking into the crucible and the height of the sample is lower than the crucible an analysis may be possible with this sample.