ELTRA®
ELEMENTAL ANALYZERS

Hydrogen determination in steel samples

Suitable analyzers
- ELEMENTRAC ONH-*p 2*
- ELEMENTRAC OH-*p 2*

Used accessories
- Graphite crucibles (90180 & 90185)
- Suitable calibration material (NIST or other)

ELEMENTRAC ONH-*p 2*

Application Settings

I) General

Sample type:	Advanced	Cooling high:	60 °C
Use argon:	Off	Flow	40 l/h
Cooling low:	45°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:	45°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	40	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.

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Procedure

- Prepare the ELTRA analyzer (exchange anhydrone, 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) Place the crucible (90180 + 90185) on the electrode tip, close furnace
(2) Weigh calibration material, place it in the sample drop mechanism and start analysis
(3) 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.

Repeat steps (1) - (3) at least thre 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.

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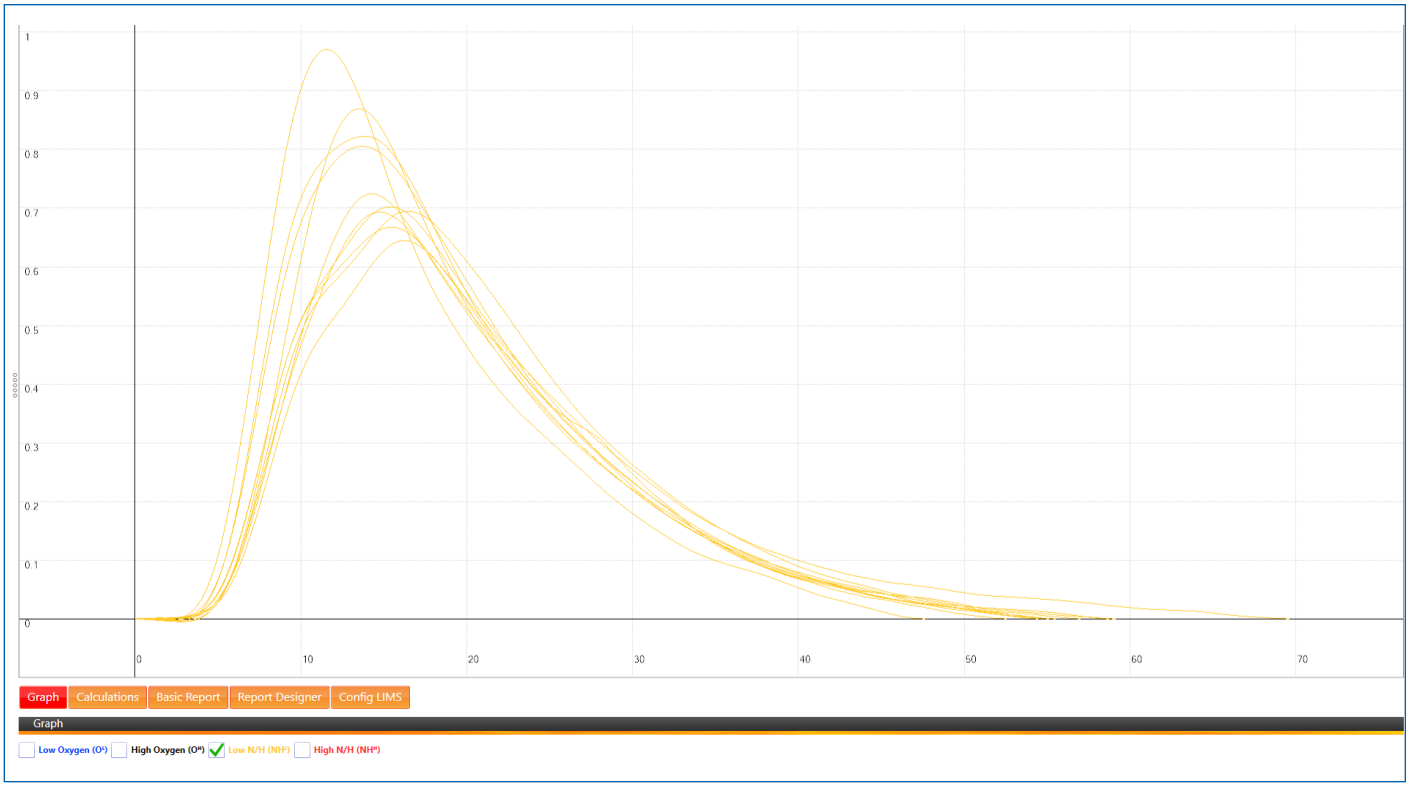
Typical results	
91400-1003 (Lot 419F) *1	
Weight (mg)	**Hydrogen (ppm)**
1023	6.84
1016	6.50
1014	6.77
1016	6.33
1009	7.22
1015	7.02
1012	6.35
1017	7.19
1009	6.54
1008	6.39
Mean value	
	6.72
Deviation / Relative deviation (%)	
	0.34 (5.1)
*1 Certified value: H 6.7 ± 1.2 ppm	



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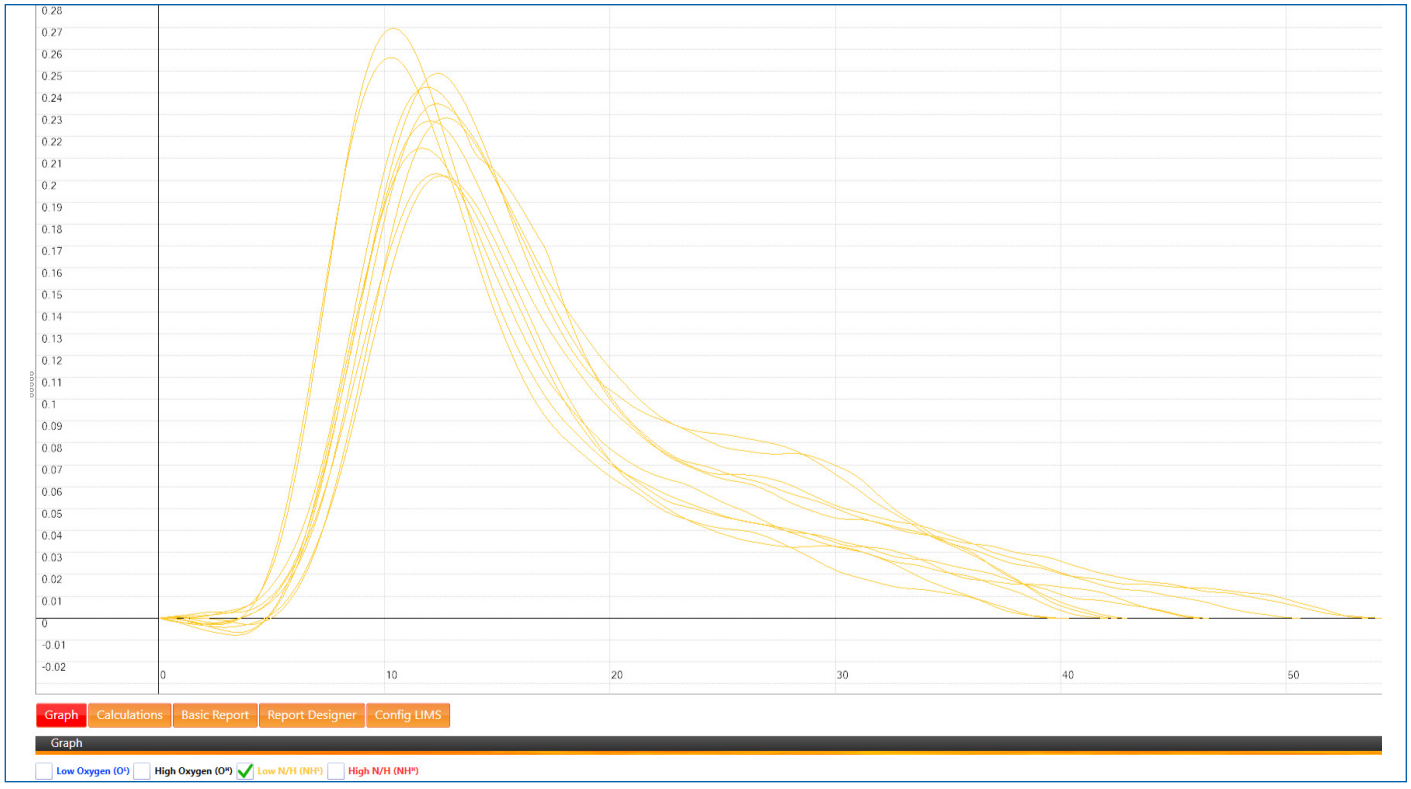
Typical results	
AR 555 (Lot 219C) *1	
Weight (mg)	**Hydrogen (ppm)**
1012	1.93
1008	2.44
1010	2.17
1013	2.56
1011	2.52
1014	2.36
1011	2.10
1011	2.60
1011	2.47
1010	2.07
Mean value	
	2.32
Deviation / Relative deviation (%)	
	0.24 (10%)
*1 = Certified value: H 2.3 ± 1.1 ppm	



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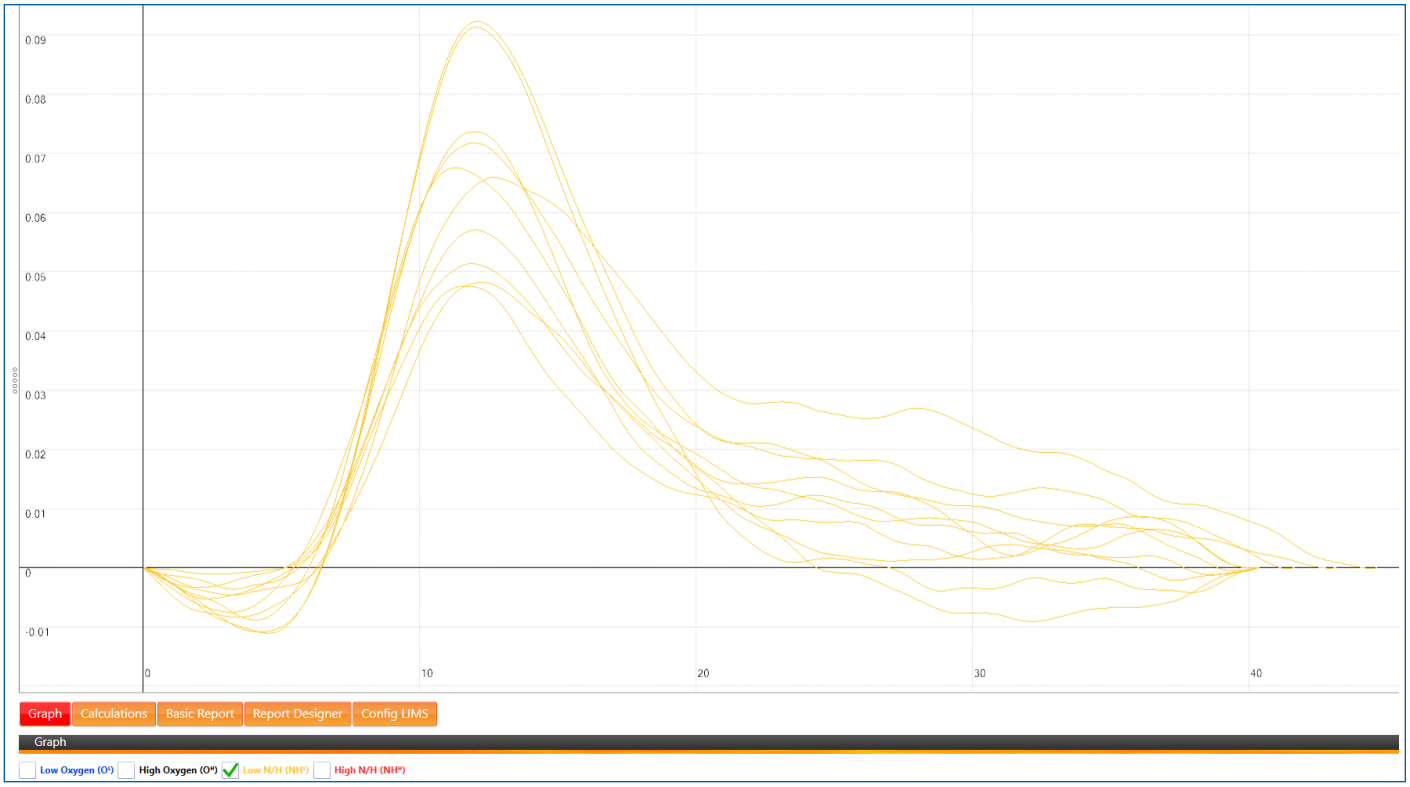
Hydrogen determination in steel samples

Typical results	
502-948 (Lot 0608) *1	
Weight (mg)	**Hydrogen (ppm)**
1008	1.09
1008	0.90
1005	0.97
1005	0.81
1006	0.81
1004	0.86
1005	0.88
1005	1.09
1003	0.78
1005	0.94
Mean value	
	0.91
Deviation / Relative deviation (%)	
	0.11 (12%)
*1 Certified value: H 0.9 ppm	



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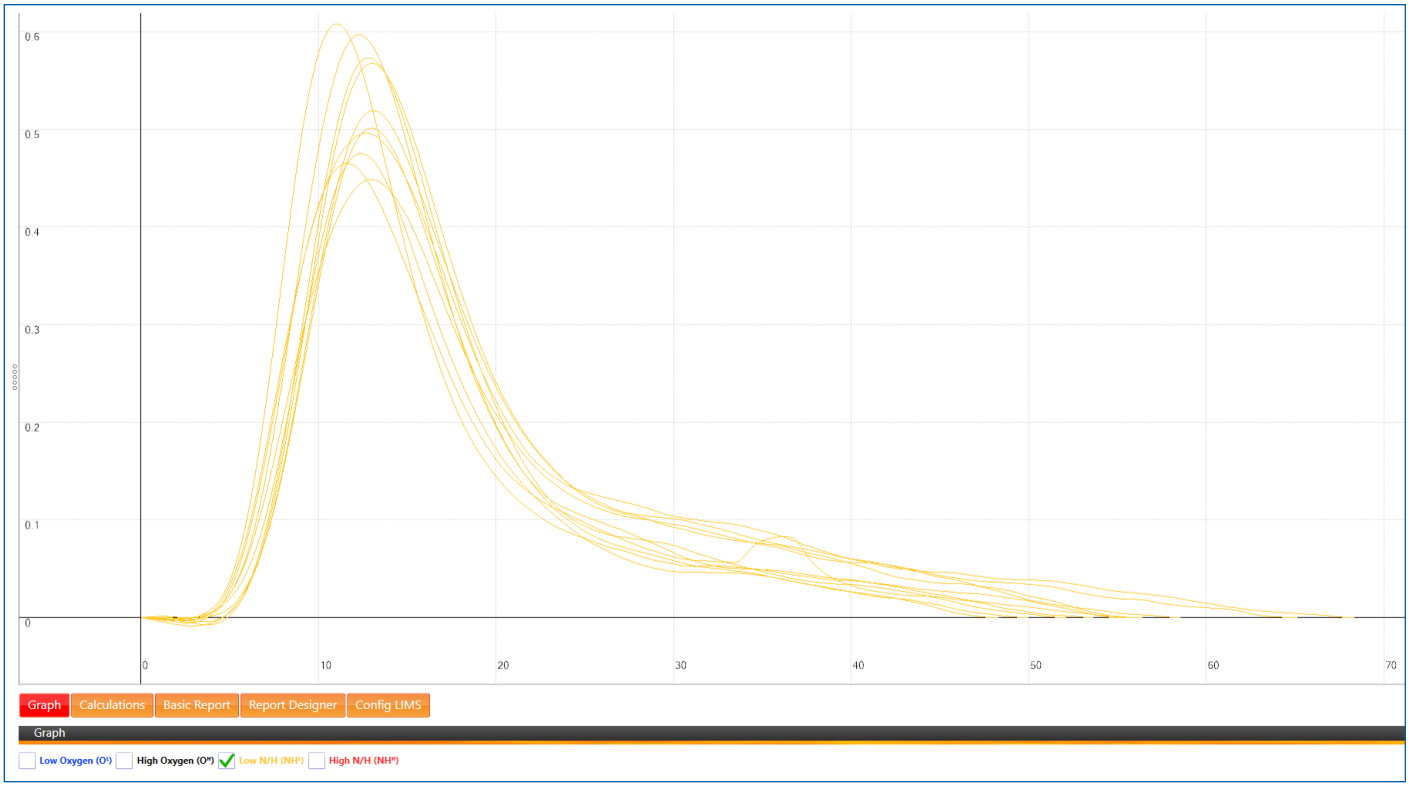
Hydrogen determination in steel samples

Typical results	
AR 555 (Lot 219C) *1	
Weight (mg)	**Hydrogen (ppm)**
2019	2.52
2013	2.30
2024	2.11
2019	2.32
2018	2.38
2023	2.53
2015	2.30
2020	2.16
2024	1.96
2021	2.56
Mean value	
	2.32
Deviation / Relative deviation (%)	
	0.2 (8.5)
*1 Certified value: H 2.3 ± 1.1 ppm	

Note:
The ELEMENTRAC ONH -p2 can process higher sample weights than 1000 mg.
Before applying these read the general recommendations at the end of this document and test if the applied sample amount will fi t into the crucible and sample port.



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Hydrogen determination in steel samples

The ELEMENTRAC ONH-*p* 2: recommendations

Cleaning of the furnace & upper electrode

Furnace and upper electrode have been cleaned after every 10-15 samples.

ELEMENTRAC ONH-*p*

Usage of crucibles

Data for this application note has been obtained by using a new inner crucible for each measurement. Single crucibles (90190) can also be used for this application with the same settings.

Application of higher sample weights (e.g. 2 gram)

Depending on the sample geometry an application of higher sample weights could be useful to improve the repeatability of the hydrogen measurements.

When higher sample weights have to be processed with the ONH-p2 the following details have to be taken into account:

(1) Check if the sample will fi t into the furnace and the 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.

(2) When two pieces are applied (e.g. pins) make sure that these pins are applied in a vertical way to the sample port: This procedure reduces blocking.

(3) A higher stabilizing and analysis power of 3900 W could be useful for reliable hydrogen measurement with higher sample weights (e.g. 2000 mg)

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Irritating results (minor determination or high deviation)

Not consistent results could be traced to several reasons. Please check the chemicals (esp. the srubber for the TC Cell and Schuetze reagent) when results are increasing or decreasing from measurement to measurement. A leakage check and cleaning of electrodes is recommended additionally.

Hydrogen measurements in general are sensitive versus the following fluctuations:

Gas quality

The carrier gas quality (nitrogen) must be 99,995 % or higher. Beside quality of the carrier gas the length of the gas connection could influence the quality of measurements. A gas purification furnace could improve the repeatability.

Carrier gas pressure

The repeatability of measurements is getting worse with falling carrier gas pressure. Esp. when only a few bar in the gas supply are left the base line can show a higher drift.

Fluctuations in the furnace temperature

The furnace temperature can increase unnoticed when the flow of cooling water is getting down or the temperature of the cooling water increases during the day. A large number of consecutive measurements could increase the furnace temperature additionally. An increased post waiting time (60 seconds) could provide a more stable furnace temperature and more repeatable results. In rare cases an external chiller may be required.

Applied power

The given application settings are suitable for common steel samples. With increasing content of chromium, titanium or other precious metals in the steel samples an increased analysis and stabilizing power (e.g. 4000 W) may required to obtain stable results.