ELTRA®
ELEMENTAL ANALYZERS

Nitrogen and (oxygen) analysis in steel granulates & powders with manual load

Suitable analyzers

- ELEMENTRAC ONH-*p*
- ELEMENTRAC ON-*p*
- (ELEMENTS Software 1.3 or higher)

Used accessories

- Graphite crucibles (90180 and 90185)
- Graphite powder (90800)
- Weighing boat (88400-0477)
- Suitable calibration material

ELEMENTRAC ONH-*p*

Description of the challenge

Sometimes steel samples have a very narrow certification for their oxygen, nitrogen or hydrogen content. When the samples are available as granulate or powder usually a capsule has to be used as sample carrier. Due to the blank value a nickel capsules could have a negative effect on the deviation of the measured element concentration. An application of the granulates or powders to the analyzer without capsule is also not recommended because the sample could cause blockage in the sample load. An alternative would be the analysis of these sample via the manual load procedure. In a manual load analysis no sample has to be applied in the sample load. The furnace opens after the crucible has been outgased, so that the sample can be directly given to the crucible.

Procedure and typical results of the manual load analysis are described in this application note. As reference material the following samples with different shapes have been chosen:

ZRM 077-3
carbon steel

CRM 090-1
unalloyed steel

ZRM 284-3
highly alloyed steel

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Application Settings

I) General

Sample type:	Advanced
Furnace Mode:	on
Catalyst:	500 °C
Cooling low:	40 °C
Cooling high:	45 °C
Standby Flow:	10 l/h

II) Outgasing and stabilizing

Setting/Phase	Cooldown [sec]	Time [sec]	Power [W]	Flow [l/h]
Outgasing (Phase 1)	NN	40	5200	27
Outgasing (Phase 2)	0	20	4200	27
Outgasing (Phase 3)	0	10	0	27

Setting/Phase	Min/Max time [sec]	Power [W]	Flow [l/h]	Stability [V]
Stabilizing (Phase 1)	40/40	0	40	0.001
Stabilizing (Phase 2)	70/70	0	27	0.001

III) Analyzing

Flow:	27 l/h
Power duration:	180 Sec
Power:	4200 W (for sample CRM 090-1 / 077-3) 5000 W (for sample 284-3)
Second cycle:	off
Open furnace:	on
Drift compensation:	on
Manual load:	on
Auto comparator:	off

Element	Minimum time [sec]	Maximum time [sec]	Integration delay [sec]	Comperator factor [%]
Low Oxygen	35	35	8	0.1
High Oxygen	35	40	8	0.1
Low N	30	40	18	0.1
High N	30	40	18	0.1

IV) Postwaiting

Time:	10 sec
Granulate mode:	off

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Sample preparation
For these kinds of samples no sample preparation is required. ELTRA recommends to use the weighing boat (part number 88400-0477) for weighing and applying of the sample.

Procedure
- Prepare the ELTRA analyzer and exchange anhydrone, sodium hydroxide, copper oxide when it is necessary. Clean Sample drop mechanism, furnace, electrode tip.
- Run three blanks with empty crucibles

At the end of the outgasing process the user is asked to confirm furnace opening. Please confirm opening and confirm 5-10 seconds later that the furnace should be close again.

- Calibrate the analyzer

Please lock in a calibration samples (e.g. ELTRA 91100-1007 (or something comparable)) with the sample weight in the ELEMENTS software. Press "Analysis Start".
"DO NOT APPLY THE CALIBRATION MATERIAL IN THE SAMPLE PORT"
Please apply the calibration sample after the furnace has opened after the outgasing process and confirm again when the calibration sample is applied to the empty crucible. The applying of the sample should not last a long time. The sample should be applied within 5-10 seconds.
Repeat the calibration process in minimum 3 times.
CAUTION: The calibration factor for a manual load sample is different than the calibration factor for a sample which was applied in the sample port.
When 3 calibration samples have been measured use the calibration function of the software.

- Run the samples in the same way as the calibration samples.

ELTRA recommends to shake the graphite crucible when a sample with a low density was applied to the crucible. Please have a look at the following pictures:


Applying sample


Before Shaking


After Shaking

Note: For the ZRM 284-3 sample 25 mg graphite was added to the inner graphite before the outgasing process. The graphite improves the repeatability of the nitrogen measurement and reduces bubbling.

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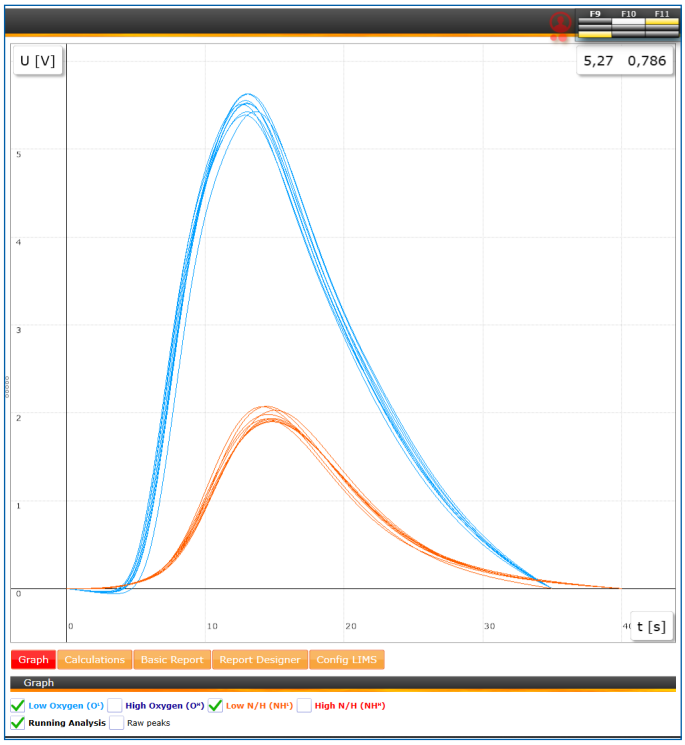
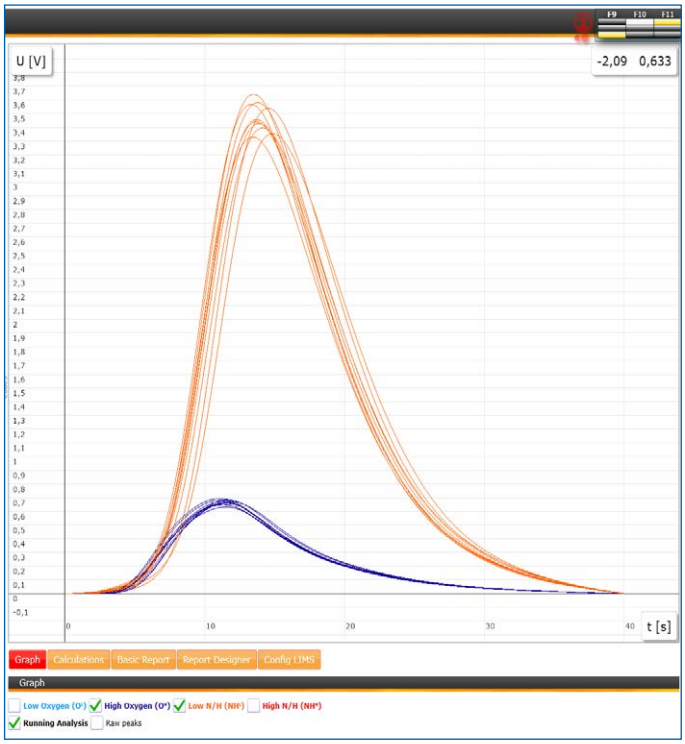
Typical results		
EURONORM: ZRM 077-3 (only N certified: N: 86 ppm ±1,8 ppm)		
Weight (mg)	**ppm oxygen** (informative; not certified)	**ppm nitrogen**
1010.6	196.4	84.6
1003.6	201.7	86.2
1021.9	204.4	87.6
1011.2	198.3	84.9
1001.6	198.9	83.9
1010.3	198.8	86.5
1017.1	205.8	87.6
1002.4	206.1	86.9
1049.0	205.7	85.3
1002.9	205.6	86.2
Average values		
	202.23	86.02
Deviation / Relative deviation (%)		
	3.77 (1.9%)	1.26 (1.5%)



Typical results		
EURONORM: ZRM 090-1 (only N certified: N: 146 ppm ±2 ppm)		
Weight (mg)	**ppm oxygen** (informative; not certified)	**ppm nitrogen**
1015.7	316.7	146.0
1073.0	308.9	145.7
1050.6	303.6	146.4
1012.1	327.6	147.6
1003.3	336.5	144.6
1030.3	311.1	143.3
1030.9	320.3	146.1
1030.0	336.3	147.2
1006.9	316.7	146.8
1017.5	328.0	146.0
Average values		
	320.6	146.0
Deviation / Relative deviation (%)		
	11.3 (3.5%)	1.30 (0.9%)



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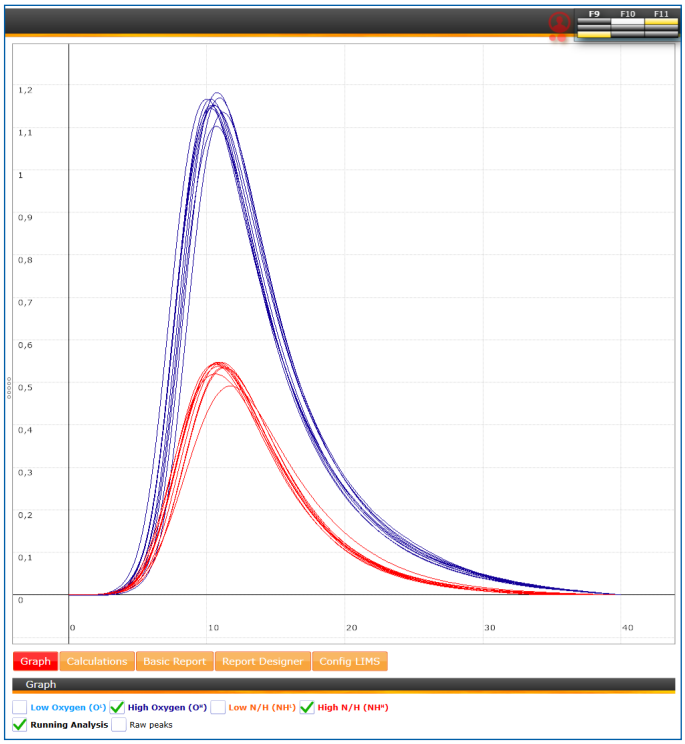
Typical results		
EURONORM: ZRM 284-3 (only N certified: N: 418 ppm ±8 ppm)		
Weight (mg)	**ppm oxygen** (informative; not certified)	**ppm nitrogen**
1003.7	459.3	418.8
1028.1	489.6	417.1
1033.3	470.6	420.4
1002.4	476.1	418.6
1027.9	478.7	421.2
1028.3	485.6	415.1
1010.3	491.8	421.0
1019.7	502.2	411.2
1020.8	511.5	416.3
1021.3	509.1	420.0
Average values		
	487.5	418.02
Deviation / Relative deviation (%)		
	16.9 (3.5%)	3.13 (0.7%)



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