



## ELEMENTRAC ONH-ps

# SIMULTANEOUS OXYGEN, NITROGEN AND HYDROGEN ANALYSIS IN STEEL



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# SIMULTANEOUS OXYGEN, NITROGEN AND HYDROGEN ANALYSIS IN STEEL



## SUITABLE ANALYZERS

- | ELEMENTRAC ONH-ps

## USED OR RECOMMENDED ACCESSORIES

- | Graphite crucibles (inner/outer) 90180 & 90185
- | Graphite crucible (single) 88400-0471
- | Suitable calibration material (NIST or other)

## INTRODUCTION

The ELEMENTRAC ONH-ps allows the simultaneous determination of oxygen, nitrogen and hydrogen in steel samples. This application note provides suitable analysis settings for the correct measurement of all three elements. Depending on the current concentrations and the crucibles used, slightly different settings must be applied to achieve optimal repeatability of results. At the end of the application note you will find some recommendations for troubleshooting when deviating results occur.

## ANALYZER PREPARATION

Before starting a large number of analyses, ensure that the analyzer is in good working condition. Make sure that upper and lower electrode are clean and not worn. Chemicals should be maintained according recommendations in the manual and the furnace should be clean. It could be useful in general to process 2-3 set up samples to warm up the analyzer. The measurement of control samples, CRM's or drift samples depends on the quality regulation in your lab.

## SAMPLE PREPARATION

Customer samples that need to be measured for oxygen, nitrogen, and hydrogen must be prepared to a suitable size that fits into the sample port, with an appropriate mass of approximately 500-1000 mg. If cut-off machines or drills are used for sample preparation and the process is not performed properly, the results may be affected. Immediately before analysis, cleaning customer samples and/or certified reference materials (CRMs) with acetone p.a. may improve repeatability.

## ADJUSTING OF SETTINGS

The fastest cycle time of approximately 3 minutes for O/N/H analysis is only achievable when the optional chiller is used for cooling. When using the heat exchanger, a longer post-analysis waiting time or different power settings may be required.



## SETTINGS

| Set the temperature of the chiller to 40°C

APPLICATION		ONH STEEL	ONH STEEL	ONH STEEL; H < 1 ppm
<b>SETTING NUMBER</b>		A	B	C
<b>CRUCIBLE</b>		90180 /90185	88400-0471	90180/90185
<b>TOPIC</b>	<b>SETTING</b>	<b>VALUE</b>		
<b>GENERAL</b>	Samples type	Advanced	Advanced	Advanced
	Use Argon	Off	Off	Off
	Catalyst [°C]	700	700	700
	Flow [l/h]	40	40	40
	Standby flow [L/h]	40	40	40
<b>PURGING</b>	Purging while closing	On	On	On
	Closing purge time [Sec]	2	2	5
<b>OUTGASING</b>	Enable pulse	On	On	On
	Pre-heat	On	On	On
	Pre-heat offset [°C]	2	2	2
	Pre outgas	Off	Off	Off
	Time [s]	10	10	10
	Power [W]	6000	6000	6000
<b>STABILIZING</b>	Stab. Baseline <sup>1</sup>	On	On	On
	Power [W]	4500	4200	4500
	Stability [V]	0.05	0.05	0.05
	Min Time [Sec]	65	65	75
	Stab. Set Time [Sec]	5	5	5
	Max time [Sec]	100	100	100
<b>ANALYZING</b>	Min Furnace Temp [°C]	40	40	40
	Power duration [Sec]	180	180	180
	Power [W]	4500	4200	4500
	Second Cycle	Off	Off	Off
	Open furnace	On	On	On
	Manual Load	Off	Off	Off
	Cooling delay [Sec]	5	5	5
	Peak finding	Drift comp.	Drift comp.	Drift comp.
<b>POST WAITING</b>	Time [Sec]	10	10	15
<b>CHANNEL HYDROGEN (H)</b>	Int. Delay [Sec]	3	3	3
	Min time [Sec]	25	25	18
	Max time [Sec]	45	45	25
	Comp. Factor [%]	0.1	0.1	0.1
	Peak max [V]	10	10	10
	Enable sample detection	Off	Off	Off
<b>CHANNEL LOW OXYGEN (OL)</b>	Int. Delay [Sec]	6	6	6
	Min time[Sec]	35	35	35
	Max time [Sec]	70	70	70
	Comp. Factor [%]	0.1	0.1	0.1
	Peak max [V]	10	10	10
	Enable sample detection	Off	Off	Off
<b>CHANNEL HIGH OXYGEN (OH)</b>	Int. Delay [Sec]	6	6	6
	Min time [Sec]	35	35	35
	Max time [Sec]	70	70	70
	Comp. Factor [%]	0.1	0.1	0.1
	Peak max [V]	10	10	10
	Enable sample detection	Off	Off	Off
<b>CHANNEL NITROGEN N</b>	Int. Delay [Sec]	17	17	17
	Min time [Sec]	35	35	35
	Max time [Sec]	70	70	70
	Comp. Factor [%]	0.1	0.1	0.1
	Peak max [V]	22	22	22
	Enable sample detection	Off	Off	Off

<sup>1</sup> Can optionally be set to off and a fixed stabilizing time of 65 seconds

## ANALYSIS OF OXYGEN, NITROGEN AND HYDROGEN IN STEEL SAMPLES USING INNER AND OUTER CRUCIBLES (SETTING A)

### ANALYZER PREPARATION

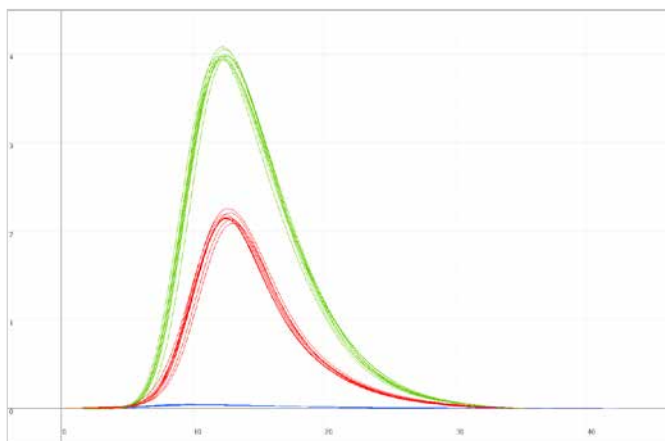
- Prepare analyzer according the description in the operation manual. Apply settings according the first column for the 90180 /90185 crucibles (Setting A). The combination of inner and outer crucibles in general provides the best repeatability and precision.
- The samples have been cleaned with acetone before analysis (details see end of the application note)

### TYPICAL RESULTS

#### STEEL PIN JK 54 <sup>1</sup>

WEIGHT [mg]	OXYGEN [ppm]	NITROGEN [ppm]	HYDROGEN [ppm]
1102.9	46.9	234.4	2.3
1099.8	45.5	235.8	2.1
1095.1	44.1	232.1	2.1
1096.8	45.6	227.1	2.2
1095.9	44.6	232.0	2.0
1110.6	45.5	227.8	2.5
1101.3	46.1	232.2	2.7
1101.6	44.9	226.7	2.1
1088.5	43.8	225.1	2.4
1093.7	50.6	234.1	2.3
<b>MEAN VALUE</b>	<b>45.8</b>	<b>230.8</b>	<b>2.3</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>1.9 / 4.2%</b>	<b>3.7 / 1.6%</b>	<b>0.2 / 9.0%</b>

<sup>1</sup> Certified value: Oxygen: 46 ± 4 ppm / Nitrogen: 229 ± 7 ppm / Hydrogen: 2.3 (informative)

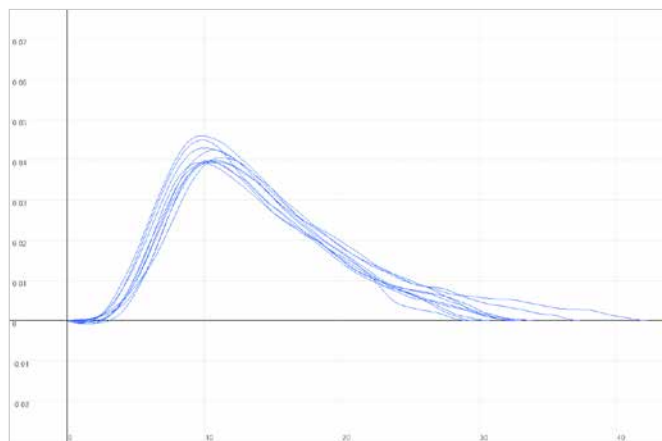


Measuring curves of CRM JK 54

Oxygen: red curves

Nitrogen: green curves

Hydrogen: blue curves



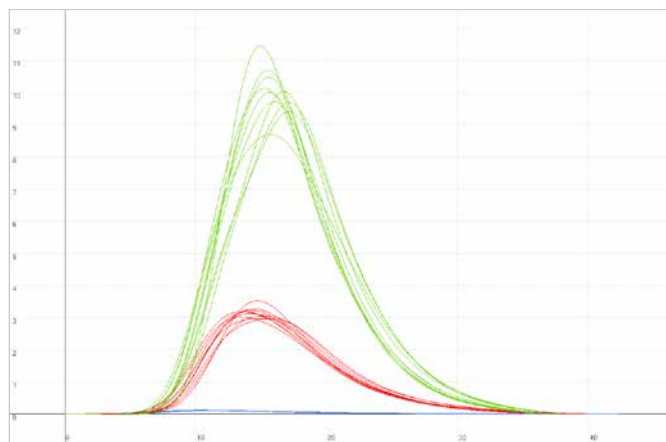
Measuring curve (Hydrogen) of CRM JK 54

**TYPICAL RESULTS**

**STEEL PIN 502-855( LOT 0718)<sup>1)</sup>**

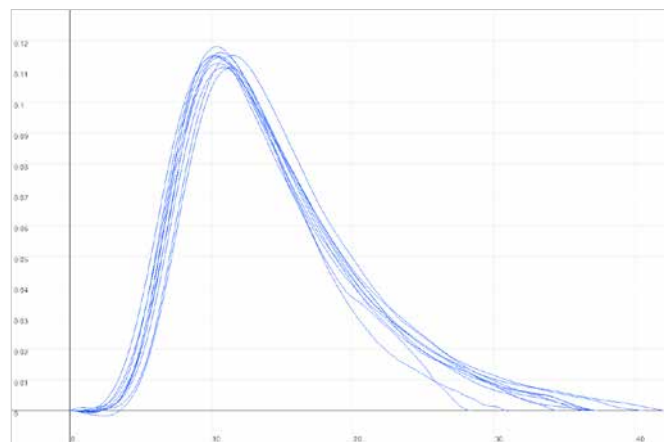
WEIGHT [mg]	OXYGEN [ppm]	NITROGEN [ppm]	HYDROGEN [ppm]
1002.8	124.2	672.2	6.0
1000.8	122.0	674.7	7.1
1003.7	120.4	668.8	6.5
1001.9	117.9	678.6	6.4
999.3	121.7	684.9	7.0
1001.0	121.6	663.9	6.1
1005.6	118.8	672.8	5.9
1005.0	118.0	659.7	6.4
1001.5	119.5	673.4	6.7
1004.0	120.1	671.9	6.8
<b>MEAN VALUE</b>	<b>120.4</b>	<b>673</b>	<b>6.5</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>2.0 / 1.7 %</b>	<b>6.8 / 1 %</b>	<b>0.4 / 6.2 %</b>

<sup>1)</sup> Certified value: Oxygen: 121 ± 3 ppm / Nitrogen: 673 ± 12 ppm / Hydrogen: 6.7± 0.5 ppm



**Measuring curves of 502-855**

Oxygen: red curves  
Nitrogen: green curves  
Hydrogen: blue curves



**Measuring curve (Hydrogen) of 502-855**

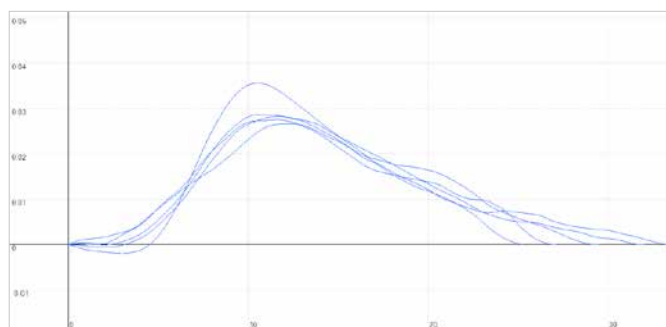
The given settings (A) can also be used for hydrogen-only analysis down to a concentration of approximately 1 ppm (0.0001%) hydrogen with a sample mass of 1000 mg.

## TYPICAL RESULTS

### BAM STEEL H2 <sup>1)</sup>

WEIGHT [mg]	HYDROGEN [ppm]
998.4	1.23
998.6	1.16
997.9	1.18
1000.1	1.20
988.2	1.18
<b>MEAN VALUE</b>	<b>1.19</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>0.03 / 2.2 %</b>

<sup>1)</sup> Certified value: Hydrogen 1.26 ± 0.07 ppm



Measuring curve (Hydrogen) of BAM CRM H2

## RECOMMENDATIONS FOR CORRECT MEASUREMENTS OF LOWER HYDROGEN CONCENTRATIONS

As mentioned above settings (A) can be used for reliable oxygen, nitrogen and hydrogen measurement with a lowest hydrogen concentration of approximately 1ppm (0.0001%). For correct analysis of lower concentrations an adjustment of the stabilizing time and measuring time may be required. See settings (C) and the following measuring data.

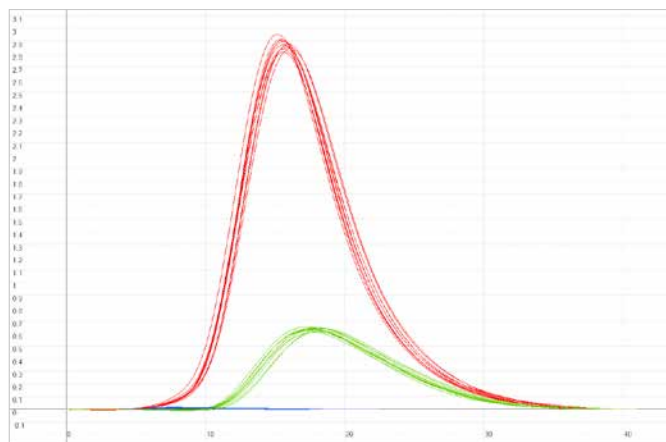
## ANALYSIS OF OXYGEN, NITROGEN AND LOW HYDROGEN IN STEEL SAMPLES USING INNER AND OUTER CRUCIBLES (SETTING C)

### TYPICAL RESULTS

#### STEEL (BALL) BS 2023 <sup>1</sup>

WEIGHT [mg]	OXYGEN [ppm]	NITROGEN [ppm]	HYDROGEN [ppm]
1049.9	74.3	50.8	0.3
1047.9	76.8	52.3	0.6
1044.7	72.5	52.3	0.4
1052.4	77.0	50.8	0.4
1052.0	76.3	52.0	0.3
1051.4	75.6	51.3	0.3
1052.8	72.2	51.0	0.3
1051.7	75.5	51.3	0.3
1051.6	73.8	52.1	0.3
1053.0	76.4	51.9	0.7
<b>MEAN VALUE</b>	<b>75.0</b>	<b>51.6</b>	<b>0.4</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>1.7 / 2.3 %</b>	<b>0.6 / 1.5 %</b>	<b>0.15 / 47 %</b>

<sup>1</sup> Certified value: Oxygen: 73 ± 9 ppm / Nitrogen: 50 ± 5 ppm / Hydrogen: 0.7± 0.3 ppm

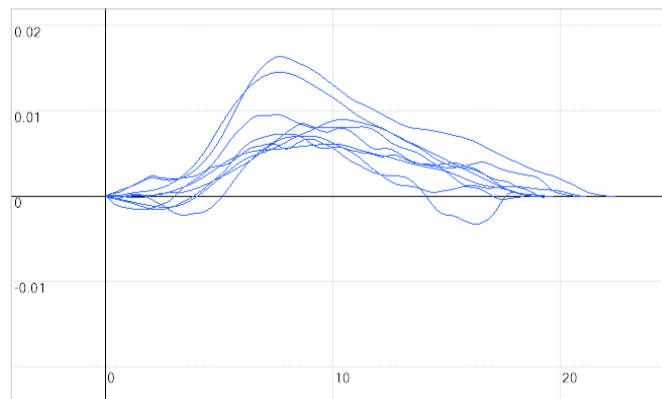


Measuring curves of BS 2023

Oxygen: red curves

Nitrogen: green curves

Hydrogen: blue curves



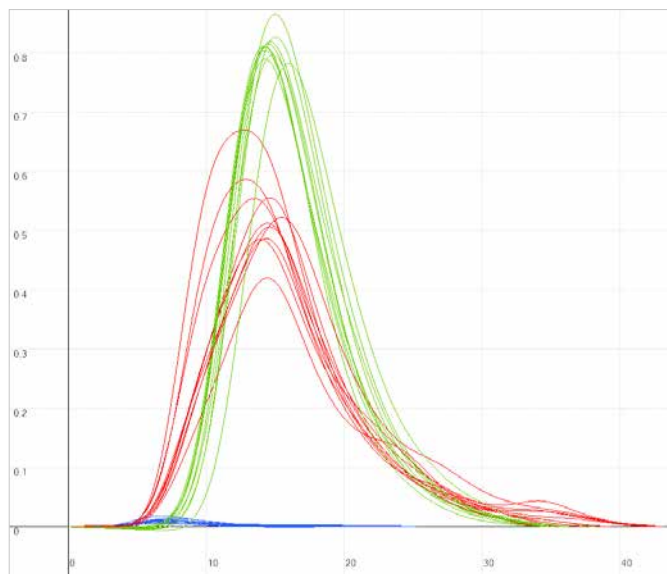
Measuring curve (Hydrogen) of BS 2023

**TYPICAL RESULTS**

**STEEL PIN 502-863 <sup>1</sup>**

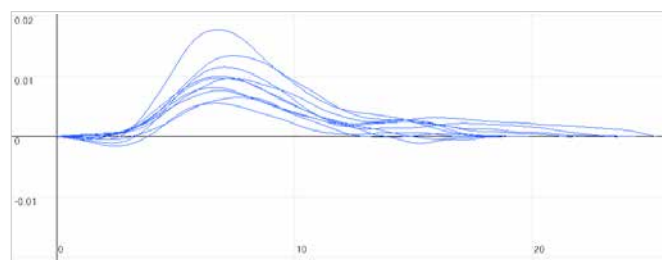
WEIGHT [mg]	OXYGEN [ppm]	NITROGEN [ppm]	HYDROGEN [ppm]
992.6	12.4	60.2	0.3
992.9	11.5	56.7	0.2
995.3	12.2	58.6	0.3
993.6	9.5	56.0	0.2
1001.3	11.1	57.0	0.2
997.1	13.4	55.7	0.1
997.0	14.1	56.2	0.3
996.8	15.5	53.9	0.5
991.2	13.3	56.4	0.5
995.1	10.8	57.1	0.2
<b>MEAN VALUE</b>	<b>12.4</b>	<b>56.8</b>	<b>0.3</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>1.8 (14 %)</b>	<b>1.8 (3.6 %)</b>	<b>0.1 / 41 %</b>

<sup>1</sup> Certified value: Oxygen: 14 ppm (informative)/ Nitrogen: 56 ± 3 ppm / Hydrogen: 0.4 ppm (informative)



**Measuring curves of 502-863**

Oxygen: red curves  
Nitrogen: green curves  
Hydrogen: blue curves



**Measuring curve (Hydrogen) of 502-863**

**NOTE**

The signal intensity for samples containing less than 1 ppm (0.0001%) hydrogen is typically low in the IR cell. Cleaning of the sample with acetone, adjusting the measuring time and applied sample mass could improve the repeatability of measurements.

## ANALYSIS OF OXYGEN, NITROGEN AND HYDROGEN IN STEEL SAMPLES USING INNER AND OUTER CRUCIBLES (SETTING B)

### INTRODUCTION

In addition to inner/outer crucibles, single crucibles can also be used for the determination of oxygen, nitrogen, and hydrogen. Generally, the deviation is slightly higher with single crucibles. Cleaning the sample or reference material with acetone p.a. can improve repeatability.

### ANALYZER PREPARATION

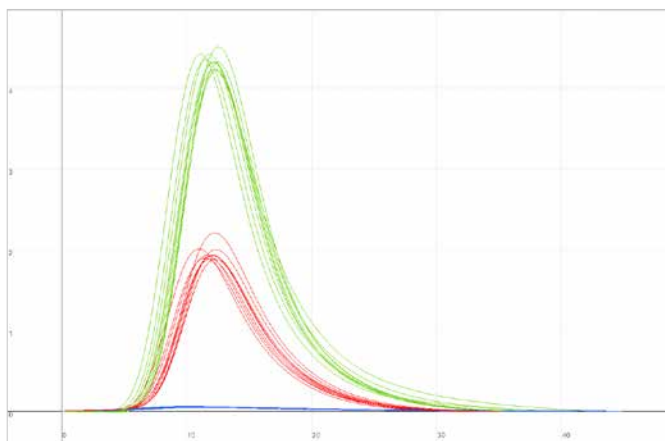
- Prepare the analyzer according to the instructions in the operation manual. Apply the settings listed in the second column for the 88400-0471 crucibles. Single crucibles generally require reduced analysis power (here, 4200 W) to achieve the same analysis temperature.
- If "bubbling" occurs, increase the post-waiting time to 60 seconds or try reducing the sample mass. In general, the resulting precision is slightly lower compared to measurements with inner and outer crucibles.

### TYPICAL RESULTS

#### STEEL PIN JK 54 <sup>1</sup>

WEIGHT [mg]	OXYGEN [ppm]	NITROGEN [ppm]	HYDROGEN [ppm]
1089.5	46.3	231.2	2.0
1099.5	44.2	244.4	2.3
1075.8	44.2	232.3	2.0
1093.5	47.5	211.8	1.7
1100.9	44.1	232.8	2.1
1099.0	52.6	221.6	1.9
1081.3	46.4	221.8	2.7
1091.1	44.6	228.5	1.9
1100.6	44.4	229.9	2.0
1095.7	44.1	232.6	1.9
<b>MEAN VALUE</b>	<b>45.9</b>	<b>228.7</b>	<b>2.1</b>
<b>DEVIATION / RELATIVE DEVIATION [%]</b>	<b>2.7 / 5.8 %</b>	<b>7.0 / 7.5 %</b>	<b>0.29 / 14 %</b>

<sup>1</sup> Certified value: Oxygen: 46 ± 4ppm / Nitrogen: 229 ± 7ppm / Hydrogen: 2.3 (informative)

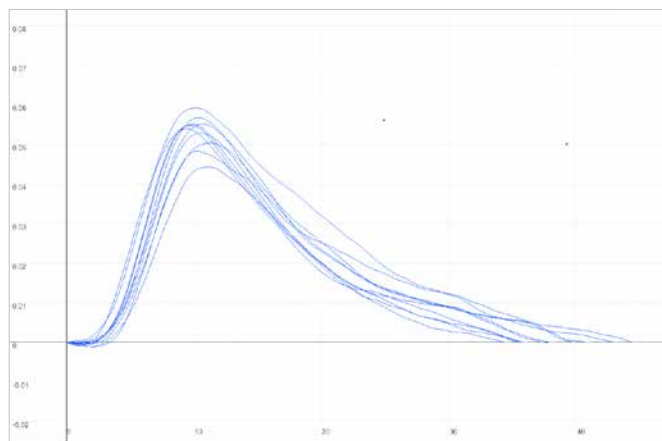


Measuring curves of CRM JK 54 (single crucibles)

Oxygen: red curves

Nitrogen: green curves

Hydrogen: blue curves



Measuring curve (Hydrogen) of CRM JK 54 (single crucibles)

## RECOMMENDATIONS FOR RELIABLE MEASUREMENTS

In general measurements with the ELEMENTRAC ONH-ps are easy, fast and reliable. If unexpected results occur, please consider the following recommendations.

ERROR	RECOMMENDATION
<b>O/N SHOW HIGH DEVIATION</b>	<ul style="list-style-type: none"> <li>Check if recommended crucibles are used (see application list).</li> <li>Check if crucibles show cracks which causes a higher deviation. (Increase Postwaiting time and reduce Outgasing power)</li> <li>Check if upper and lower electrode are in good condition.</li> <li>When high alloyed steel is analyzed increase applied stabilizing and analysis power up to 5000 W.</li> <li>When application show a lot of dust decrease the applied power for stabilizing and analyzing (-200/-300 W).</li> <li>When no chiller is utilized increase the postwaiting time (60 seconds) and reduce applied power for stabilizing and measuring (-200/-300 W).</li> <li>Clean samples or calibration materials with acetone p.a. (see example below)</li> </ul>
<b>ONLY O SHOWS DRIFT AND HIGHER DEVIATION</b>	<ul style="list-style-type: none"> <li>Check if upper electrode is worn. Maybe the nickel layer is removed and the electrode has to be replaced (very high oxygen results).</li> <li>Oxygen measurement is sensitive vs. dust and stability of furnace temperature: Clean the furnace mechanically, increase the stabilizing time up 80 seconds and increase postwaiting time (60 seconds).</li> </ul>
<b>ONLY N SHOWS DRIFT AND VERY HIGH N VALUES</b>	<ul style="list-style-type: none"> <li>Replace the NaOH/ Anhydrone filter before the TC cell (even when it looks fine): The chemical is saturated, CO<sub>2</sub> gets into the TC cell and causes a signal.</li> </ul>
<b>ONLY H SHOWS DEVIATION / DRIFT</b>	<ul style="list-style-type: none"> <li>For low hydrogen concentrations (&lt; 1 ppm) it may be useful to reduce the minimum analysis time to 20 seconds to get a good repeatability, for very high concentrations (&gt; 8 ppm) a minimum analysis time of 45 seconds may be required</li> <li>Samples with a high content of precious metals may require a higher analysis and stabilizing power (5000 W).</li> </ul>



BS 2023 with wool inside



Cleaning in acetone

### CLEANING WITH ACETONE

- Samples could have residues (e.g. oil) on the surface which falsifies the result. CRMS could have dust from other pins or from present wool/paper on the surface.
- Placing the sample or CRM in a small vessel containing pure analysis-quality acetone removes surface contaminants and can improve analytical precision. Before analysis, remove the sample or CRM from the acetone and allow it to air dry to eliminate any acetone residues. Start analysis immediately.

The effect of cleaning with acetone is illustrated in the following for the CRM JK 54. The CRM was measured 10 times (n=10) first without cleaning with acetone, afterwards with cleaning with acetone.

	WITHOUT CLEANING WITH ACETONE		WITH ACETONE CLEANING	
	Measured Concentration / relative Deviation	Lowest measured values/ highest measured value	Measured Concentration / relative Deviation	Lowest measured values/ highest measured value
<b>PPM OXYGEN</b>	48.0 ± 3.4	44.0 / 55.8	45.8 ± 1.9	43.8 / 50.6
<b>PPM NITROGEN</b>	226.4 ± 4.4	216.5 / 231.4	230.8 ± 3.7	225.1 / 235.8
<b>PPM HYDROGEN</b>	2.6 ± 0.5	2.0 / 3.7	2.3 ± 0.2	2.0 / 2.7

After cleaning with acetone the results usually show a smaller deviation. Oxygen and hydrogen values are more effected than nitrogen.