QuantoLux

QLX1 Application Note

Analysis of Steels

Introduction

QLX1 is a portable optical emission spectrometer with laser excitation. It allows the user to perform positive material identification (PMI) and at the same time rapidly analyse the material to quantitatively determine its elemental composition in weight percent per element.

Due to its innovative design, **QLX1** is capable to perform a full analysis for a wide range of metals. Please also check for other application notes available for other metal bases. Multiple metal apps can be combined in one instrument – even added later.

The analytical performance of this instrument is comparable to mobile spark spectrometers, while **QLX1** also extends this to smaller samples, wires, etc. without the need of adapters - and at much reduced operational expenses and with more benefits on time-savings and maintenance. It is superior to handheld devices due to spectral range and resolution, bigger battery and larger argon reservoir.

This report presents the calibrated ranges and performance data for the analysis of steels.

QuantoLux QLX1

The **QLX1** has a flexible configuration that allows for easy upgrades in the future. The optical systems cover the UV & VIS range and make the instrument suitable for ferrous and non-ferrous apps. Every unit can be equipped or upgraded to other published **QLX1** apps!

Special services make updates available online. **QLXUp!** provides the instrument with the latest software version, while **AppUp!** delivers latest improvements and upgrades in calibrations.*

Calibration of Steel Alloys

The analytical app for steels is divided into five different sub-methods:

- Fe – Orientation
- Low Alloy Steel
- Stainless Steel
- Tool Steel
- Manganese Steel

Elements	Fe - Orientation		Low Alloy Steel		Cr-Cr/Ni Steel		Tool Steel		Mn Steel	
	min %	max %	min %	max %	min %	max %	min %	max %	min %	max %
C	0.005	4.50	0.005	1.50	0.01	2.50	0.10	2.50	0.01	1.70
Si	0.010	6.50	0.01	3.00	0.01	4.00	0.01	2.00	0.01	1.60
Mn	0.005	20.00	0.003	2.50	0.002	19.00	0.01	1.50	3	20.00
P*			0.003	0.10	0.003	0.10	0.003	0.06	0.003	0.11
S*			0.003	0.15	0.003	0.10	0.003	0.10	0.003	0.25
Cr	0.01	30.00	0.002	8.00	0.02	30.00	0.005	12.00	0.005	4.00
Mo	0.01	10.00	0.005	2.50	0.005	3.00	0.005	11.00	0.005	1.50
Ni	0.005	45.00	0.005	6.00	0.005	45.00	0.005	0.40	0.005	2.00
Cu	0.005	8.00	0.005	1.00	0.005	8	0.005	0.20	0.005	0.30
Al	0.005	2.00	0.005	1.20	0.005	2.00			0.005	0.25
As*			0.003	0.10						
B*			0.001	0.01	0.001	1.00				
Co	0.005	12.00	0.005	2.20	0.005	10.00	0.003	10.00		
Nb	0.005	3.00	0.003	0.35	0.005	3.00				
Sb	0.005	0.50	0.005	0.50						
Pb	0.01	0.25	0.01	0.20	0.01	0.30				
Sn*			0.002	0.13						
Ti	0.003	2.50	0.003	0.35	0.003	0.45				
V	0.010	10.00	0.005	1.00	0.01	1.00	0.01	11.00	0.01	0.25
W	0.10	19.00	0.05	3.30			0.05	19.00		
N*					0.03	0.80	0.05	19.00		
Fe	Balance		Balance		Balance		Balance		Balance	

*Figure 1: Calibration Ranges for Steels; elements marked with * require UV-Add-on*

The factory calibration of the ferrous app always includes all alloy groups and elements for a given configuration that are available at the time of production. Later, **AppUp!** can ensure that you are always up-to-date and benefit from element additions, range extensions, or performance improvements.

Sample Preparation

To achieve optimum results, the sample surface being analysed must be free from dirt, contaminations, grease, rust or anything else that you don't want to analyse.

Generally – but more importantly for low alloy steel – samples should be freshly prepared to avoid oxidation effects on the surface.

Please follow the motto: remove everything from the surface that you do not want to analyse.

A handy battery angle-grinder is suitable for most steel alloys.

Samples for this report were prepared with a disk grinder and 60 grid paper.

Measurement parameters

A typical measurement takes 3 seconds, this includes a pre-spark and several channel-specific integration windows. The analysis is an average of 2 or 3 subsequent readings on different spots on the sample. For PMI applications, however, most users just perform a single shot. If in doubt, a second reading is done.

The sample turnaround time is typically less than 15 seconds with multiple measurements and average calculation. In PMI with single-shots much faster.

Pulse energies, frequencies, and pulse duration are optimised per analytical method to achieve best results. All parameters are fixed in factory certified calibrations that are readily available for use.

Timings are stable for any quality or element. No extended pre-spark or longer integration times are required.

Use of Argon

In **QLX1** argon is primarily used for two purposes:

1. Increase plasma light in the sample stage, and
2. Create an atmosphere in the optics that is transparent for UV light

And then, argon in laser-OES is not needed to

- bridge the electrode gap for ignition
- remove condensate from the spark stand

The flows required are extremely low due to the geometry and small plasma size.

For **QLX1** applications Argon 4.8 quality (99.998%, "Argon for spectrometry") is recommended.

Argon flows of **QLX1** are minimised: only 2 l/h as constant flow and 10 l/h during the 3 second measurement. Per measurement 0.008 liter (8 ml) are consumed, the 24h consumption is around 50 liters.

Argon cost is about 80% reduced compared to spark spectrometry. Calculate your annual savings!

Standardisation

A classical 2-point standardisation is not necessary with **QLX1**. A drift monitor sample is checked in intervals and possible drifts are corrected. This process takes about 15 seconds to complete. A profile check is automatically conducted with each measurement. For users who need to document the status of the instrument, the software also still supports the classical workflows for a 2-point as well as a type standardisation.

Savings on time, more instrument uptime, no cost for SUS sample replacements.

Typical Precision

The precision obtained with **QLX1** is very much comparable to those achieved with classical spark spectrometers.

QLX1 applications are not optimized for low detection limits, but rather accuracy.

Precision is also referred to as "repeatability". A homogeneous, freshly prepared sample is analyzed a few times and average, and absolute (SD) & relative standard deviations (RSD) evaluated.

The following list shows expected minimum performances in two concentration groups:

Concentrations	RSD
0.001 – 0.5	<10%
0.5 – 45.0%	<4%

Precision is influenced by a few factors:

- sample preparation (i.e. surface quality)
- metallurgy (inclusions, segregation effects of certain elements, etc.)
- gas flows in probe head and optics (transparency)
- probe-on-sample stability

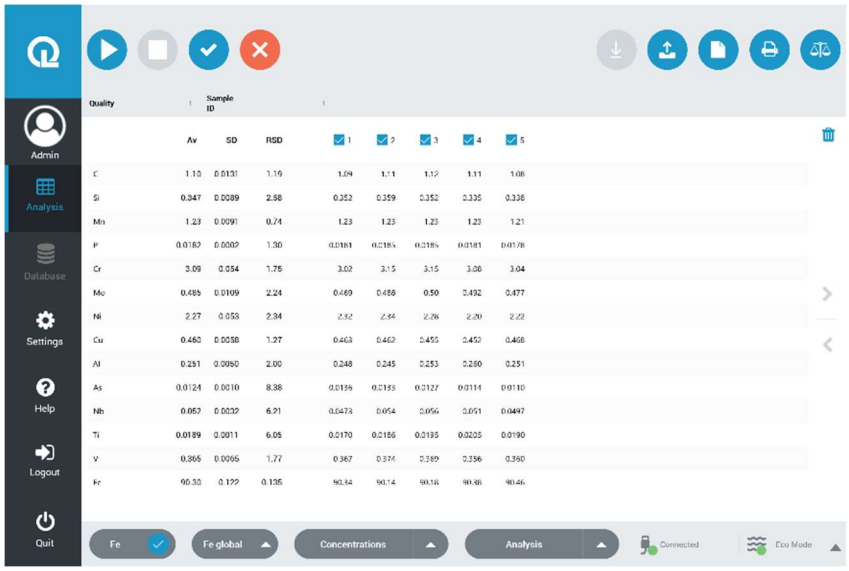

Figure 2: typical precision on a steel sample with stable positioning

No memory effects

QLX1 does not show any memory effect. Immediately after an alloyed sample, a low alloy or pure sample may be analysed without any memory effect. This is simply due to the technology used: there is no electrode which may carry residuals of the previous sample and there is no condensate in the chamber to be re-excited with the next measurement.

The same benefits apply to matrix changes. Without any effect you can switch between matrices.

And, by the way, you don't need your old brushes any more!

Huge time savings on electrode brushing, cleaning, matrix change, standardisation!

Accuracy

Certified Reference Materials (CRMs) are used for the creation of calibrations. The results of these samples are processed to obtain polynomial coefficients from a multivariate regression. Together with line switches, inter-element & matrix corrections they lead to the concentration of an element.

In OES the following factors influence the accuracy of results:

- Quality and accuracy of certified standards
- Precision of readings
- Calculation & correction of line interferences
- pre-spark energy and timing
- strategic read-out timing

The following calibration curves are examples for Carbon and Chromium in steels. Both are showing excellent correlations with the certified standards.

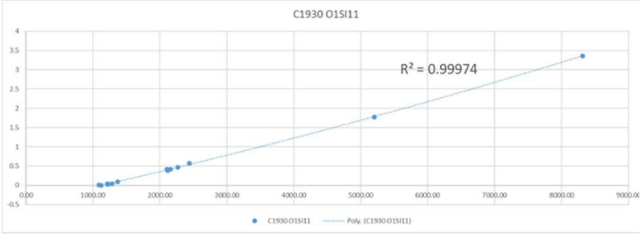

Figure 4: Carbon regression with high correlation to CRMs

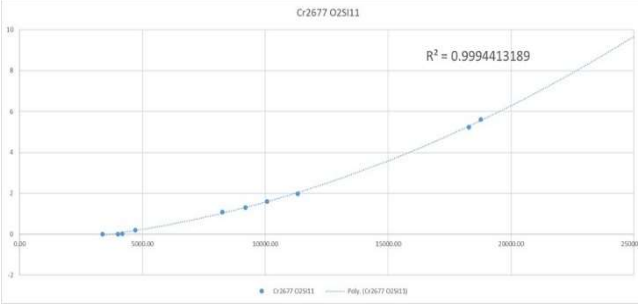

Figure 3: Chromium line 267.7 nm

Conclusion

The QuantoLux **QLX1** is a portable laser-OES with advanced technologies that offer substantial benefits in the field of identifying and analysing steel alloys. Metal processing industries, inspection companies and users in metal trading & recycling applications take special advantage of its features.

As a summary, following are some of the key productivity boosters and payback accelerators that the **QLX1** offers:

- full-blown analysis with Carbon and optionally further UV-elements like P, S, N, As, B, Sn, …
- truly portable solution with re-chargeable battery
- attached Argon mini flask with up to 40 liters
- Lower cost for argon due to drastically lower flow rates (~80%)
- Faster results lead to cost reductions in production processes
- Zero maintenance; no electrode or spark stand cleaning saves time and money
- No consumables costs (like setup samples, brushes, etc.)
- Reduced investment as **QLX1** is prepared to cover your future needs
- Simple and comfortable use
- Overall, much reduced operational expenses

Learn more about laser-OES

QuantoLux is offering various forms of trainings, including e-learning programs, to learn more about optical emission spectroscopy with lasers and specific applications.

For more details please consult the website at www.quantolux.com or contact our local partner.

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