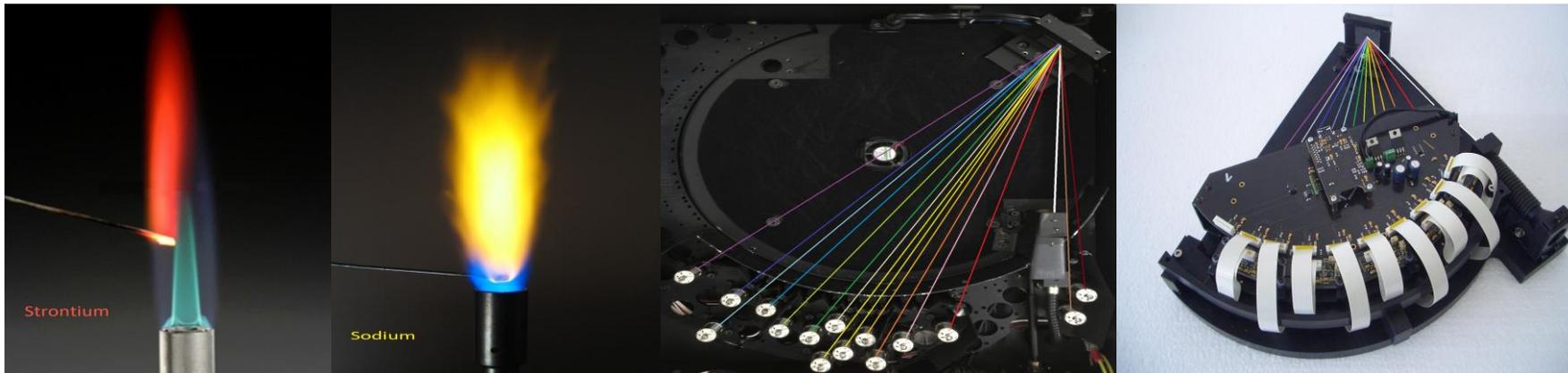


Chemical Analysis of metal samples using Optical Emission Spectroscopy (OES)

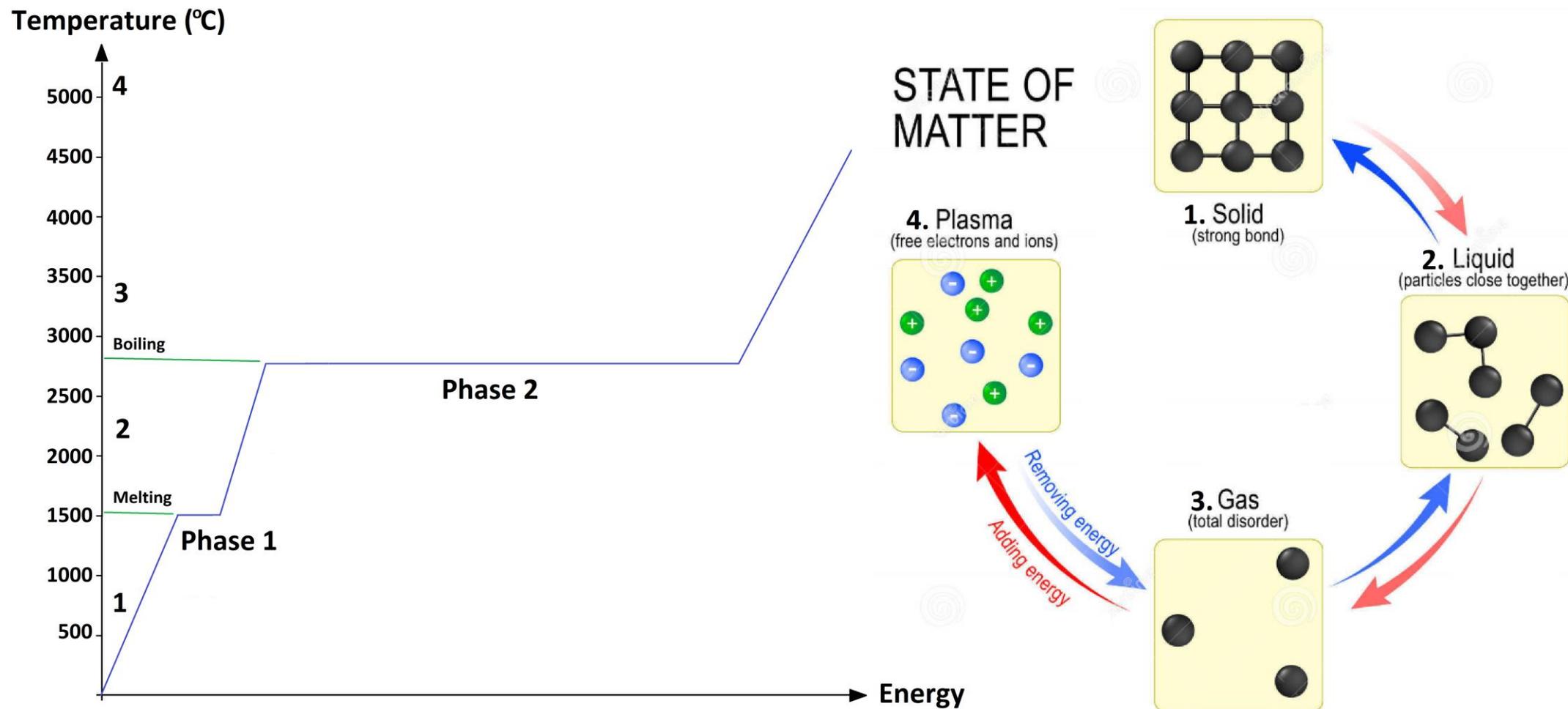
Glen Thiele
Spectrosource Pty Ltd



What is Optical emission spectroscopy (OES) also known as Atomic emission spectroscopy (AES)?



Aggregate state of matter



Atomic physics – How photons are emitted

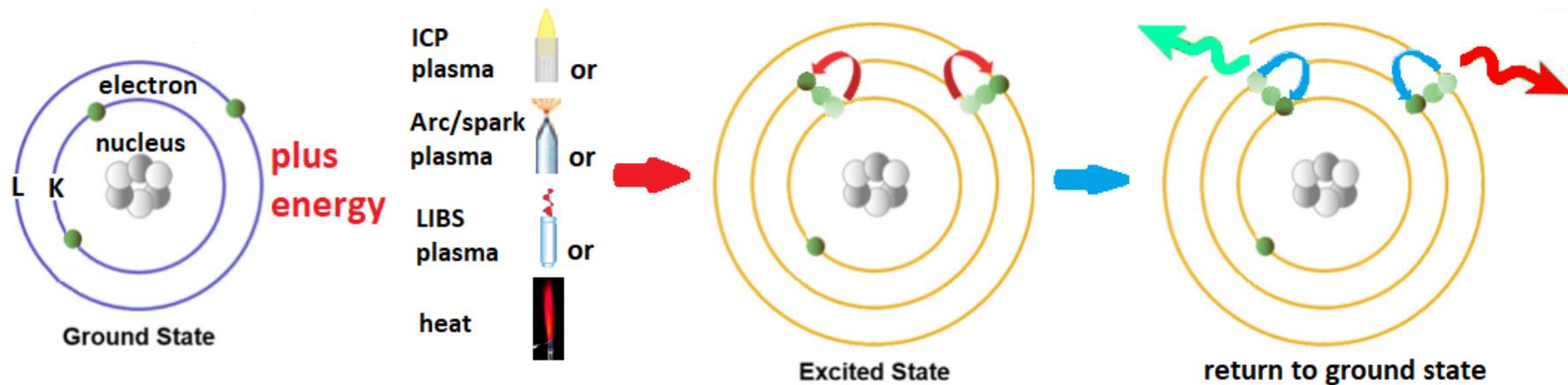


Figure 1: Bohr model (1913) Lithium --> excitation --> relaxation --> photon emission

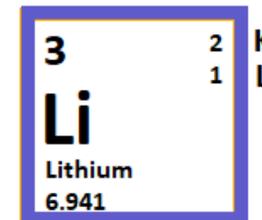


Figure 2: Lithium emission spectrum

Optical emission Spectrometers



ICP OES



LIBS OES

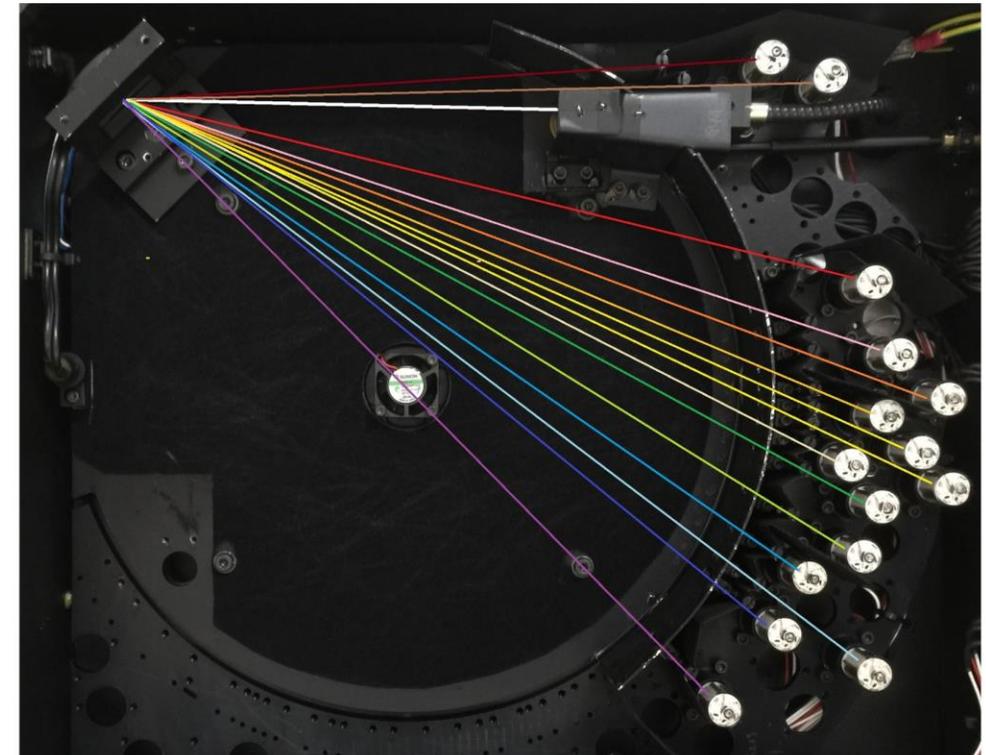
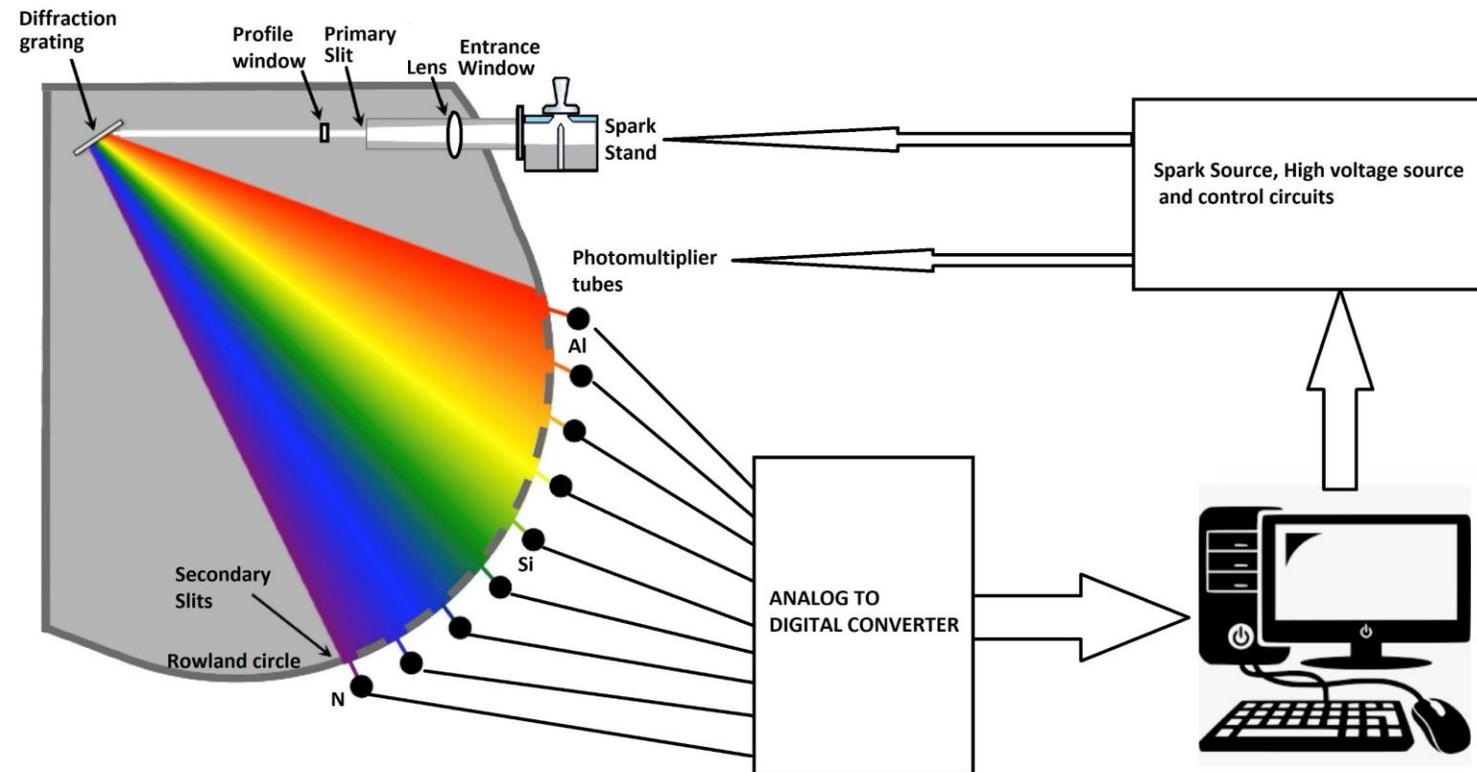


Arc/Spark OES

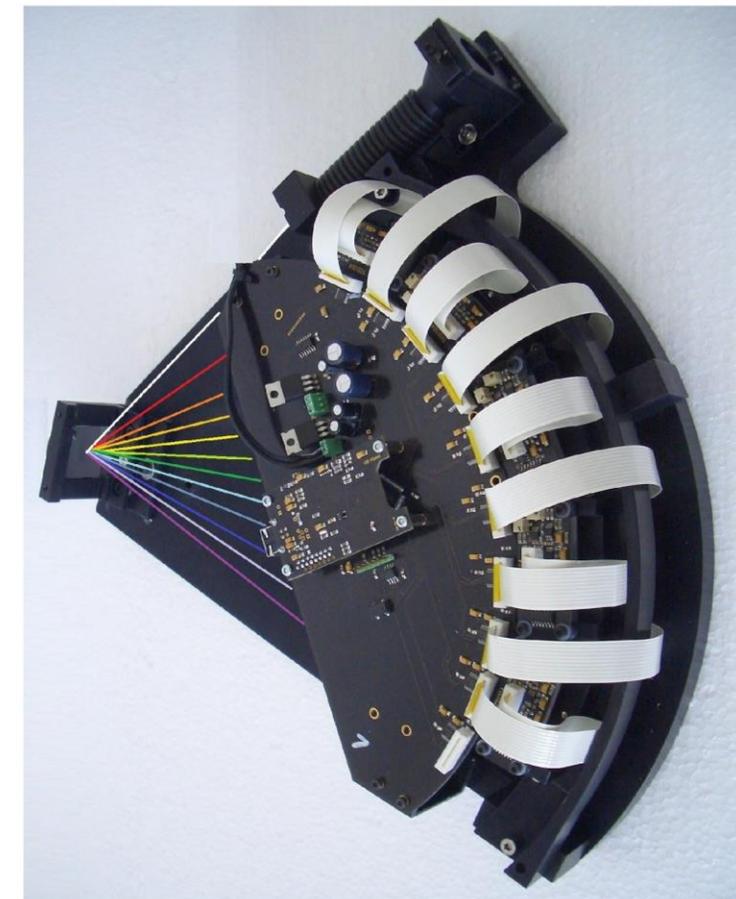
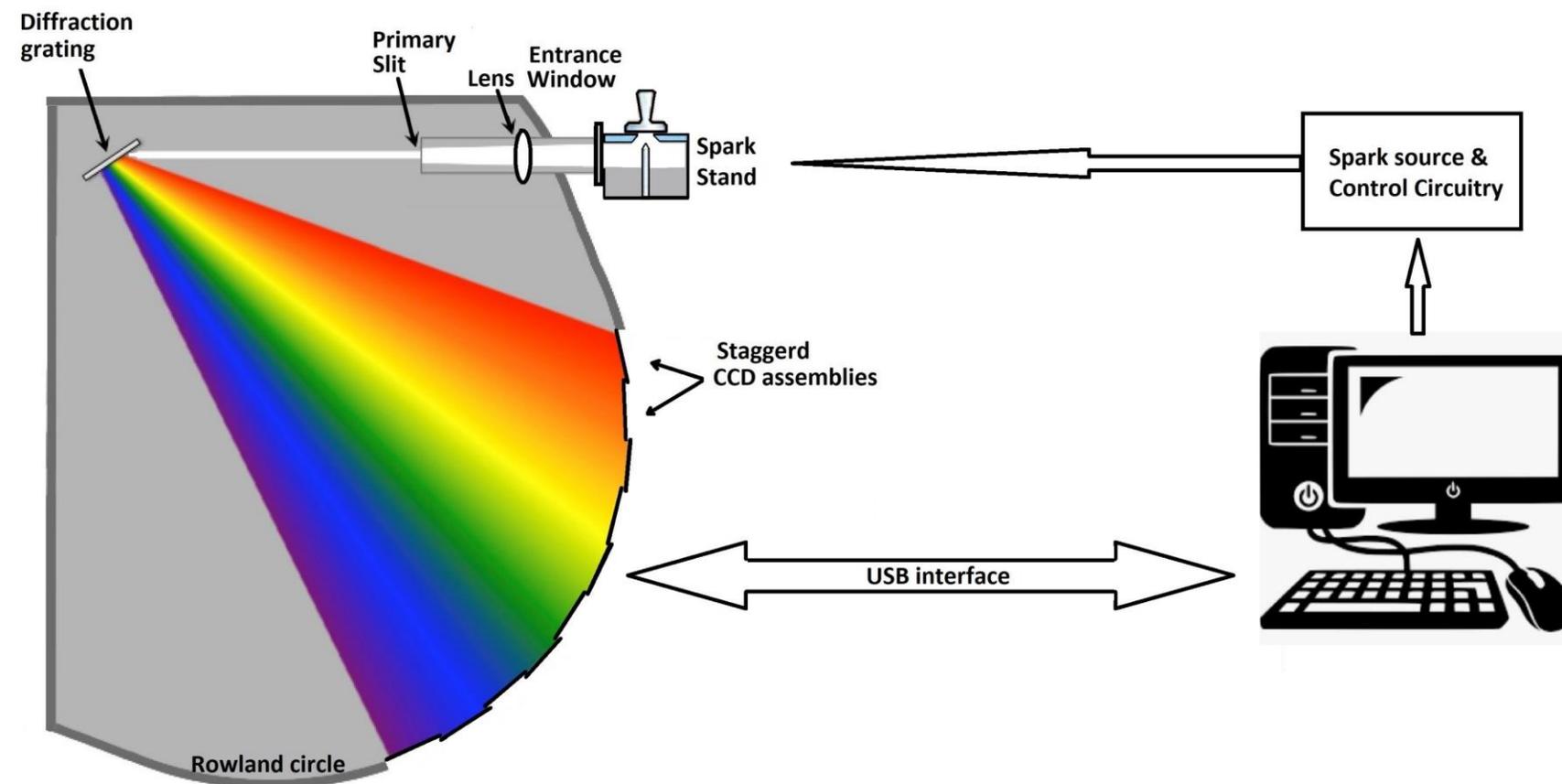
Why Use Spark OES Analysis?

- Most widely used, reliable and reproducible metal analysis technique.
- Wide choice of instruments.
- Excellent LOD
- CCD based spectrometers allow a high number of elements and bases.
- Detection of N is possible with high end portable and high end laboratory spectrometers.
- Detection of O is possible with high end laboratory spectrometers.

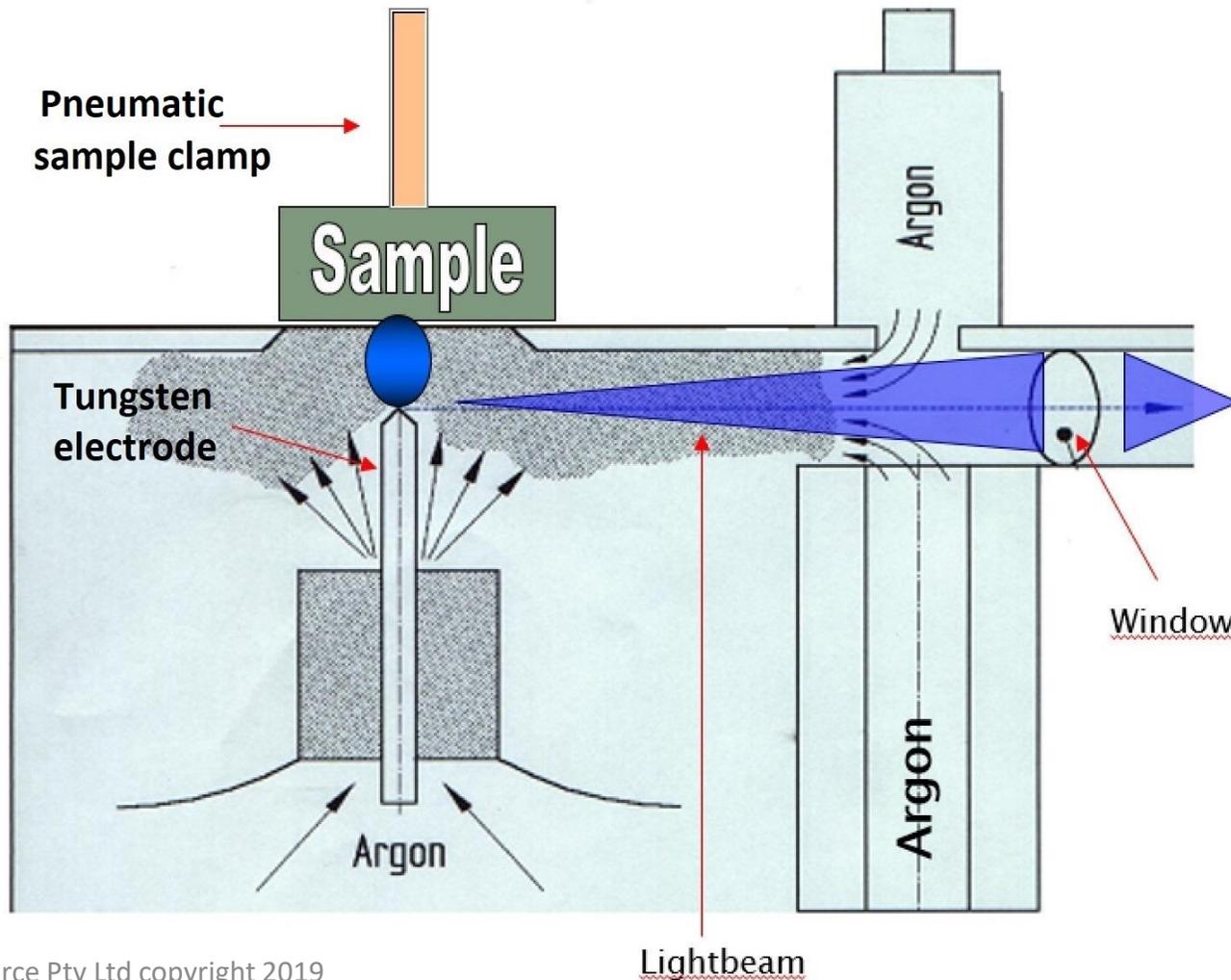
PMT based Spark OES architecture



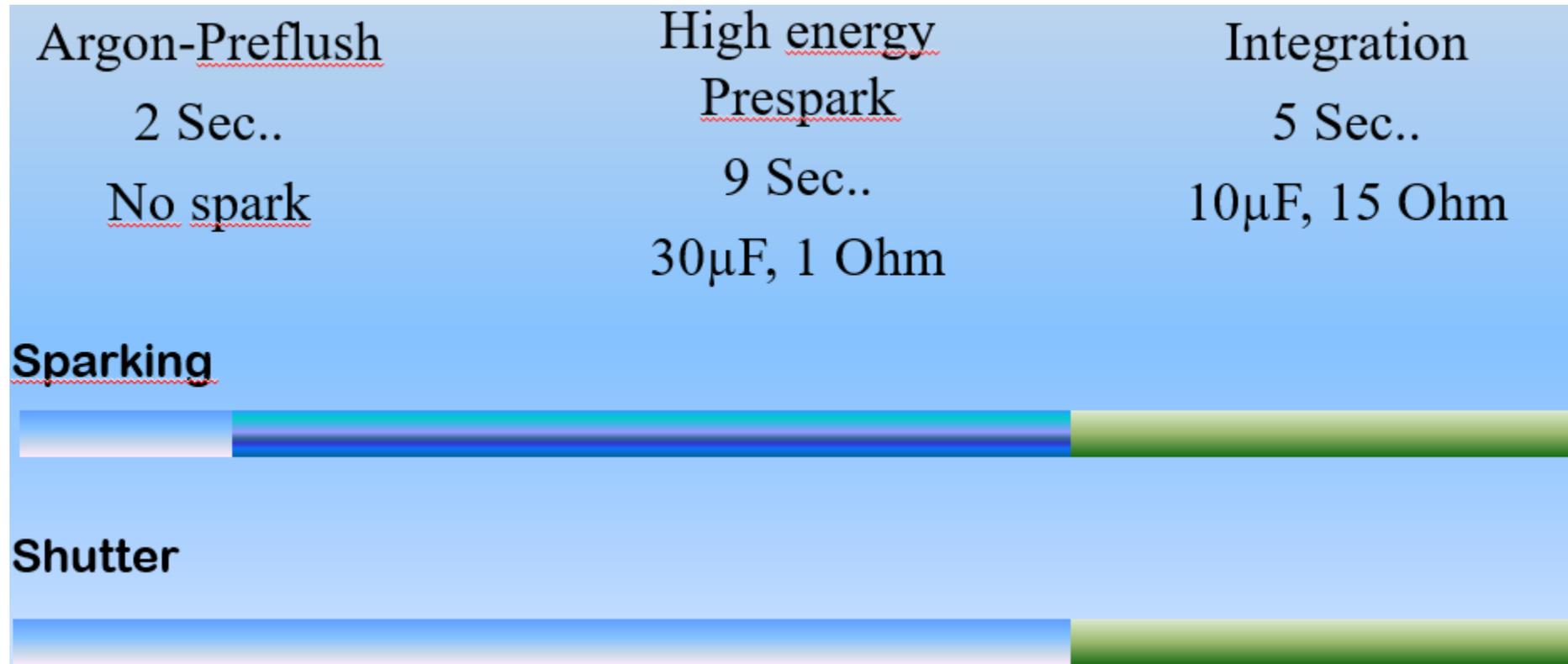
CCD based Spark OES architecture



Spark Stand architecture



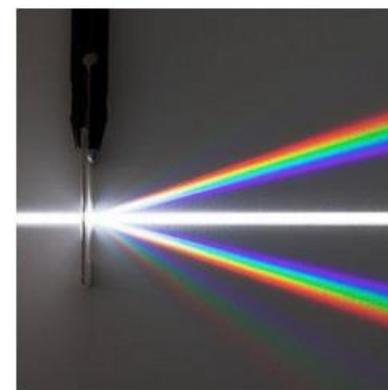
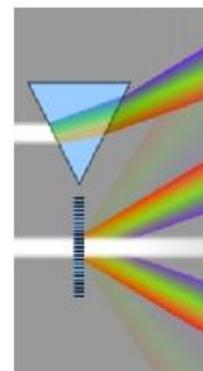
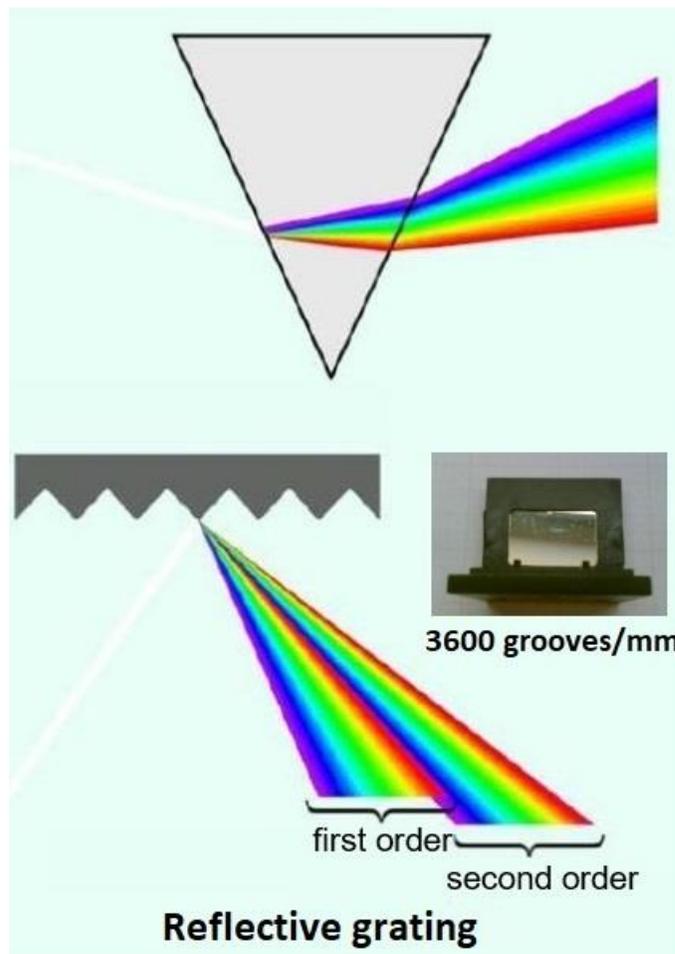
Typical spark sequence



Grating properties

Prism:

- non-linear refraction
- affected by temperature due to influence of medium density

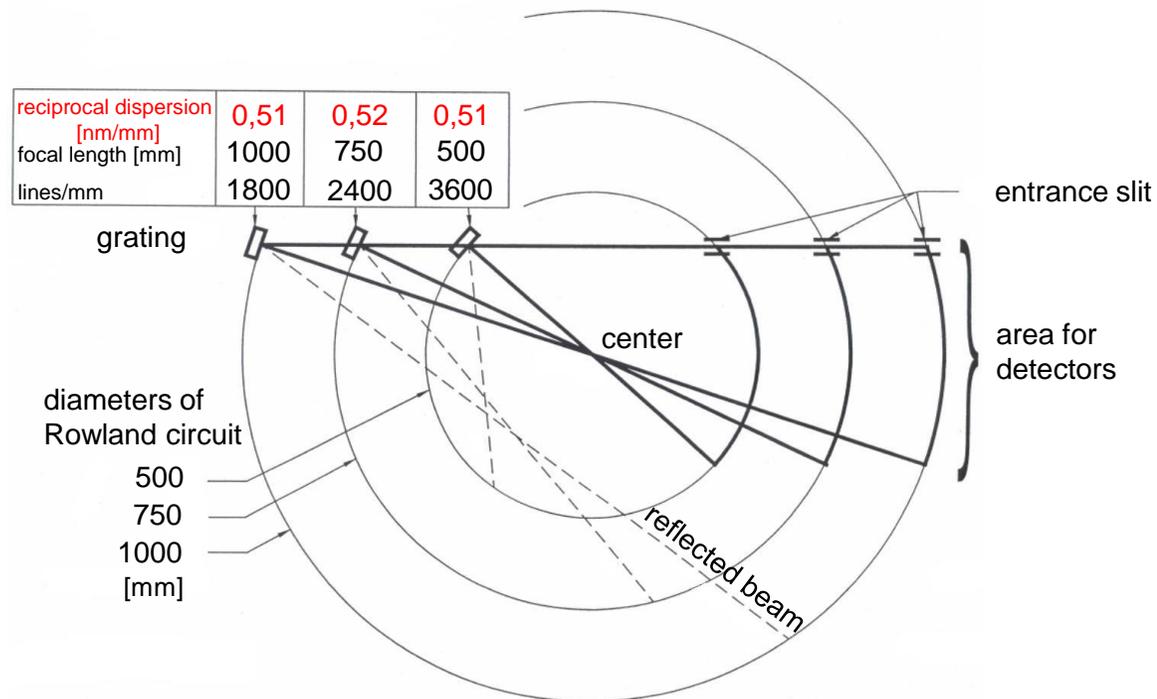


Transmission grating

Grating:

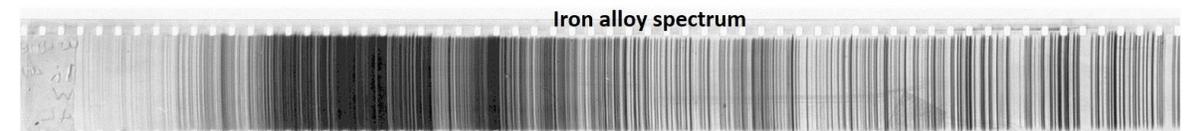
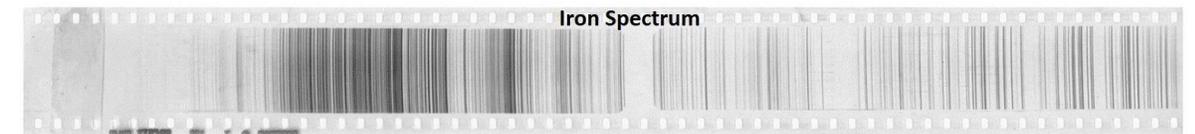
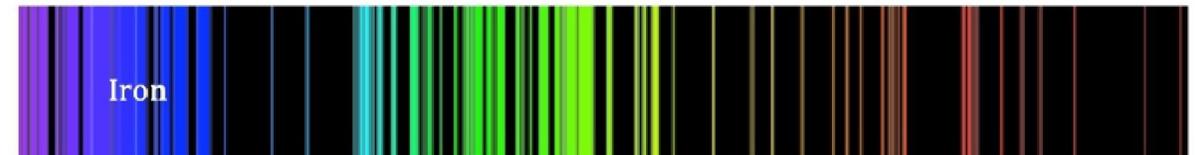
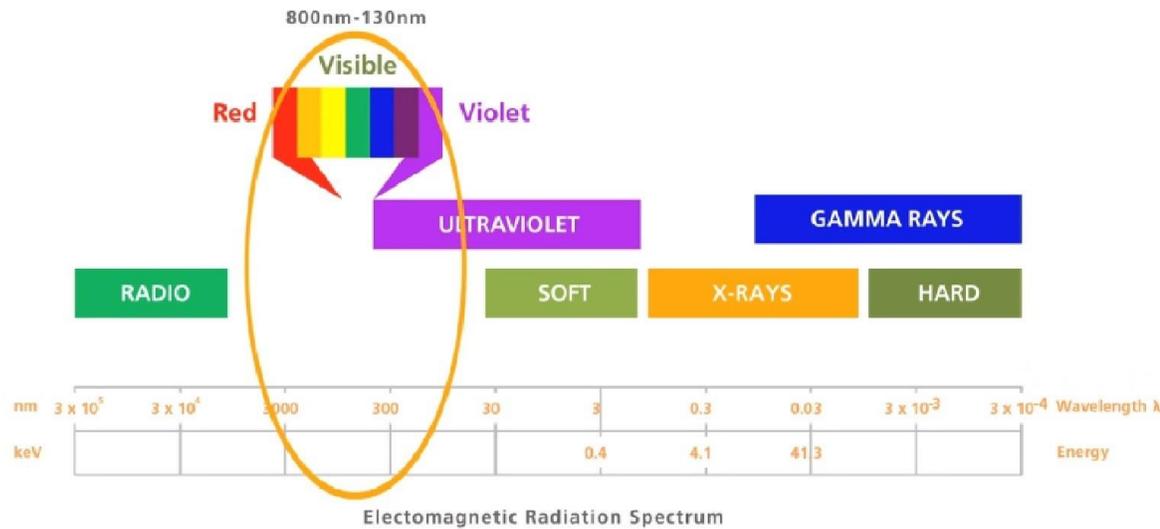
- linear diffraction
- different orders

Reciprocal dispersion as criteria of line separation



3600 grooves/mm grating

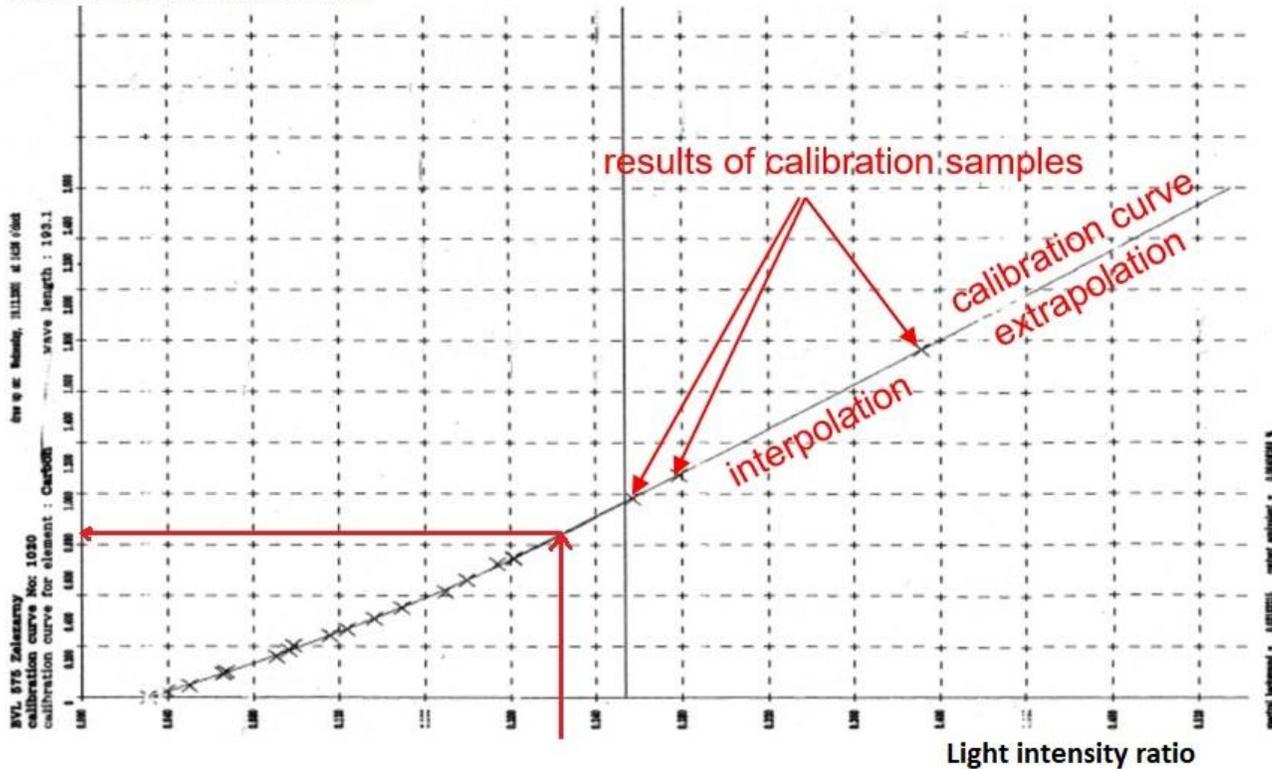
Spark OES Spectrum 130nm – 800nm



Calculation of results from Calibration Curve

Calibration curve for Carbon in low alloy steel

Element concentration ratio



result comparison to previously
measured set of calibration samples

Spectrosource Pty Ltd copyright 2019

standard analyse measuring program

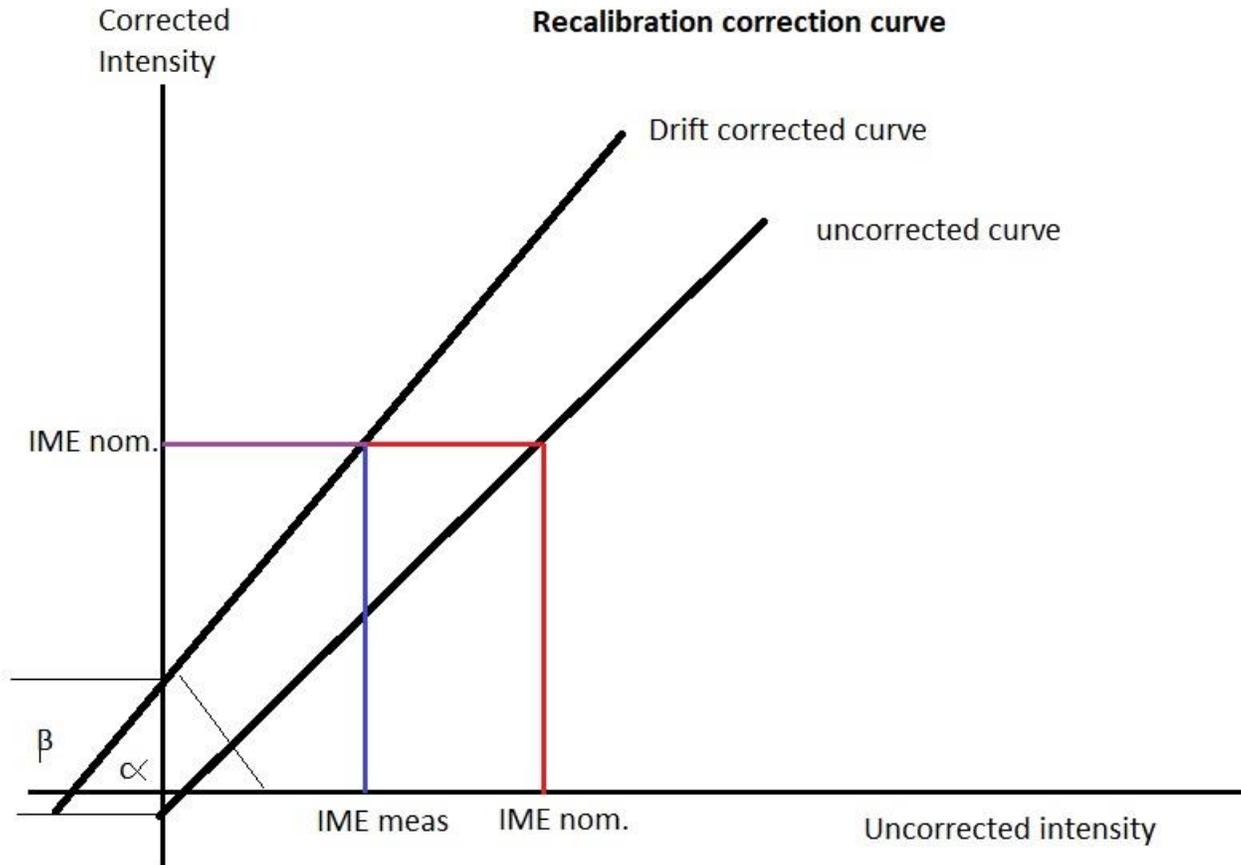
measuring program	program no	sample identification	measurement no.	grade no.	description
low alloyed steel	11	demo software	average	1.7131	16 MnCr 5

no.	C %	Si %	Mn %	P %	S %	Cu %	Al %	Cr %	Mo %
1	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.06	0.06
2	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.06	0.06
3	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.09	0.06
4	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.16	0.06
5	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.09	0.06
6	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.09	0.06
mitte	0.18	0.30	1.26	0.015	0.022	0.09	0.106	1.08	0.06

no.	Ni %	V %	Ti %	Nb %	Co %	W %	Pb %
1	0.04	0.02	0.029	0.039	0.05	0.06	0.003
2	0.04	0.02	0.029	0.039	0.05	0.06	0.003
3	0.04	0.02	0.029	0.039	0.05	0.06	0.003
4	0.04	0.02	0.029	0.039	0.05	0.06	0.003
5	0.04	0.02	0.029	0.039	0.05	0.06	0.003
6	0.04	0.02	0.029	0.039	0.05	0.06	0.003
mitte	0.04	0.02	0.029	0.039	0.05	0.06	0.003

Emission spectrometry is a
relative measurement method

Recalibration, Standardization or Drift correction



recalibration - program

measuring-program: Recalibration Steel, program-No: 10, measuring-No: average, all samples: 101 102 108 104, recalibration sample: 108

no.	Fe	C	Si	Cr	Cr
Soll	2500			1 660	
1	2459	124.0	944.0	5549	1.705
2	2431	123.0	927.0	5411	1.715
mittel	2445	123.5	935.5	5480	1.710

recalibration - values

Program No.: 10

element	alpha	beta
Fe	1.00000	0.0000
C	0.93204	0.0029
Si	1.00521	0.0024
Mn	0.91408	-0.0002
P	0.84835	0.0074
S	1.02584	-0.0032
Cu	0.99889	0.0027
Al	1.02454	-0.0012
Cr	1.02413	-0.0089
Cr	0.97830	0.0011
Mo	0.98026	-0.0004
Ni	0.91375	0.0013
Ni	0.94486	0.0077
U	0.92629	0.0005
Ti	0.99945	-0.0004
Nb	0.95061	0.0056
B	1.00386	-0.0029
Zr	0.93152	0.0022
Mn	1.03149	0.0013

Do you want to save these values?

yes no

no.	Mo	Ni	Ni	Zr
Soll			0.5810	
1	484.0	3100	0.6063	202.0
2	475.0	3035	0.6072	202.0
mittel	479.5	3068	0.6068	202.0

measuring values

Start end print report file report prog Samp avert bander calc graph direct print save

Belecmenue 2019090822 recal steel ... pre service 2019090822 recal cast 5... Desktop 09:25

Type Calibration

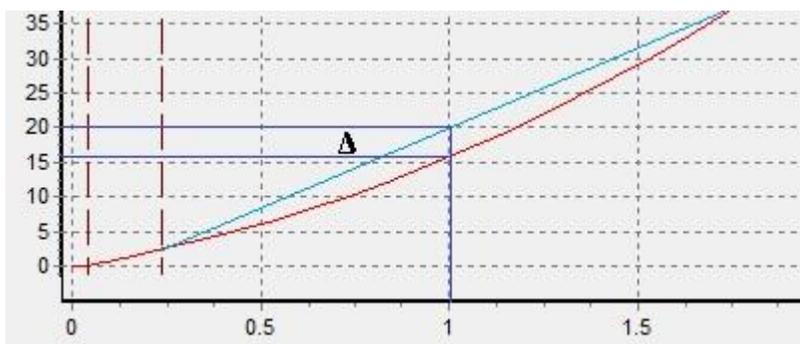


Illustration of exaggerated concentration error Δ in a calibration curve where Type calibration correction can be used to improve accuracy.

Type - Calibration

type no. 12 type description SS 2205 measuring-no average- measuring program High alloyed Cr>5% Ni>4% Program No. 13

No	C %	Si %	Mn %	P	% Mo %
nom	0.017	0.360	1.420	0.0	450 3.180
1	0.011	0.356	1.416	0.0	.17 3.163
2	0.008	0.350	1.417	0.0	.34 3.162
3	0.011	0.364	1.417	0.0	.28 3.164
4	0.008	0.347	1.409	0.0	.33 3.106
mittel	0.009	0.354	1.415	0.0	.28 3.149

No	Ni %	V %	Ti %	Nb	% Fe %
nom	5.930				66.417
1	5.98	0.076	0.010	0.0	66.37
2	5.98	0.076	0.011	0.0	66.18
3	5.95	0.077	0.011	0.0	66.29
4	5.99	0.076	0.010	0.004	66.30
mittel	5.98	0.076	0.010	0.005	66.28

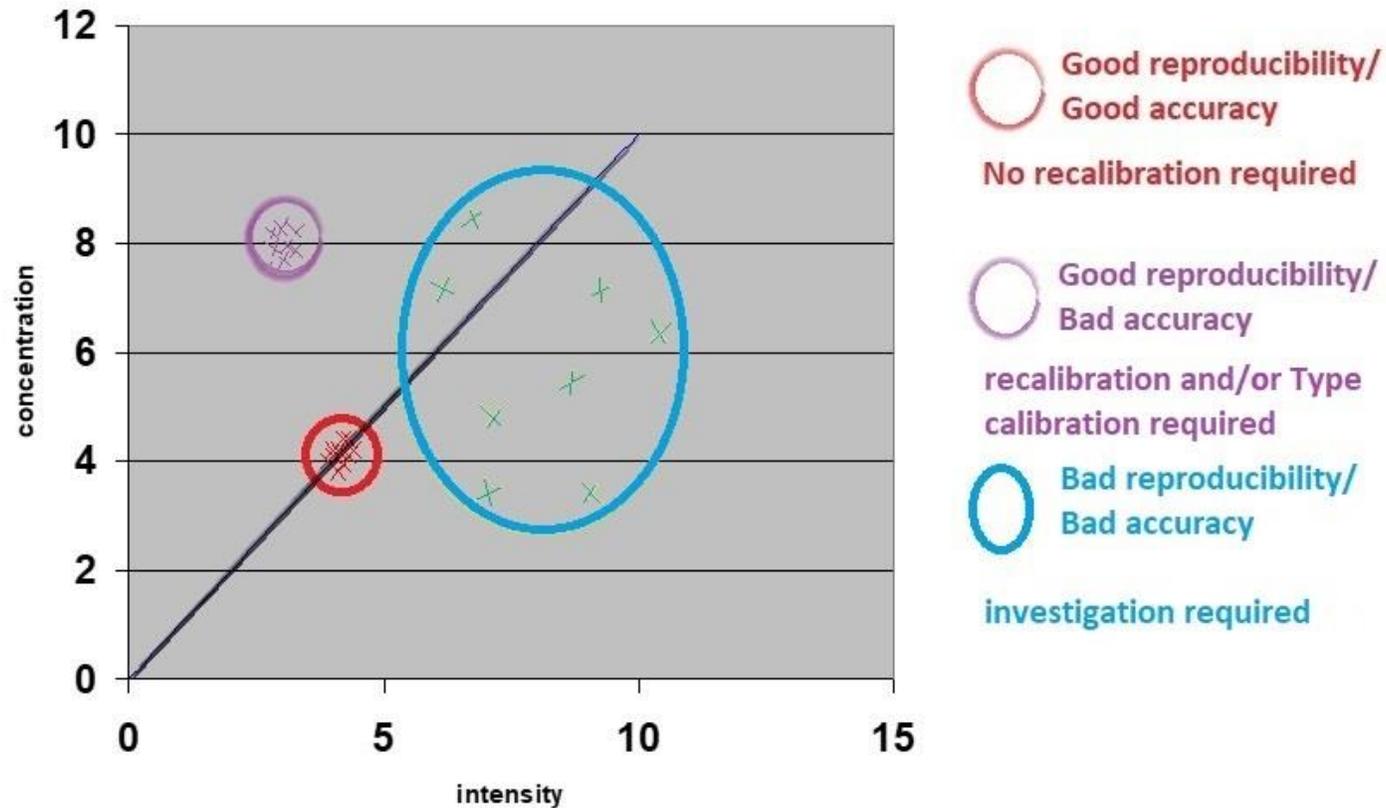
Type Correction Value
Type No: 122

Element	additive	multiplicativ
C	0.0076	
Si	0.0057	
Mn		1.003581
P	0.0034	
S	0.0010	
Cr		1.007635
Mo		1.009992
Ni		0.992337
N	-0.0011	
Fe	0.1324	

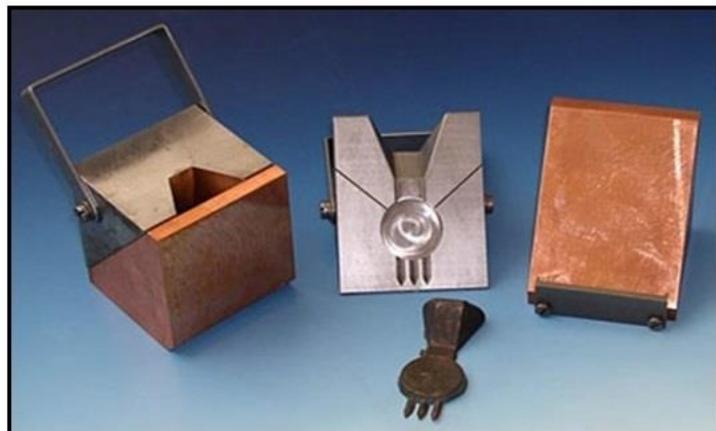
do you want to save this values ?

Start End printprotocol file protocol Type aver. Standart calc. rect pr direct print save

Measurement accuracy and reproducibility



The analytical Process



Sample taking



Sample preparation



Chemical analysis



Type Calibration and measure program



The screenshot displays two windows from the Spectrosource software: 'Type - Calibration' and 'Type Measuring'.

Type - Calibration Window:

- type no: 1, type description: BAS SUS 5/49, measuring no: average, measuring program: Cast Iron low alloy, Program No: 51
- Table 1:

No	C %	Si %	Mn %
Soll	3.800	1.900	0.610
1	3.81	1.86	0.628
2	3.75	1.82	0.635
mittel	3.78	1.84	0.632
- Table 2:

No	Ni %	V %	Ti %
Soll	1.000	0.510	0.002
1	0.944	0.525	0.002
2	0.949	0.536	0.003
mittel	0.947	0.531	0.002
- Table 3:

%	Cr %	Mo %
0	0.010	
3	0.013	0.017
6	0.013	0.018
4	0.013	0.018
- Table 4:

%	Fe %	Ceq %
0	92.104	
0	91.94	0.00
8	92.00	0.00
9	91.97	0.00
- Modal dialog: 'Type Correction Value' for Type No: 1. It lists elements and their additive/multiplicative values. A 'do you want to save this values?' dialog is also present.

Type Measuring Window:

- measuring program: Cast Iron low alloy, Program No: 51, type description: BAS SUS 5/49, type no: 1, on of Measured: average, Grade-No: no grade found, Grade-Description: no grade found
- Table 1:

No	C %	Si %	Mn %	P %	S %	Cu %	Al %	Cr %	Mo %
1	3.80	1.89	0.608	0.040	0.010	0.006	0.038	0.010	0.017
2	3.86	1.84	0.610	0.041	0.010	0.008	0.038	0.010	0.018
mittel	3.83	1.87	0.609	0.040	0.010	0.007	0.038	0.010	0.018
- Table 2:

No	Ni %	V %	Ti %	Nb %	Pb %	Mg %	Sn %	Fe %	Ceq %
1	1.000	0.509	0.002	0.000	0.043	0.080	0.082	91.86	4.44
2	1.003	0.501	0.003	0.000	0.047	0.081	0.059	91.87	4.48
mittel	1.002	0.505	0.002	0.000	0.045	0.080	0.070	91.87	4.46
- Table 3: 'type listing' for BAS SUS 5/49

element	nominal%	meas. value%
Fe	92.1040	91.9665
C	3.8000	3.7790
Si	1.9000	1.8394
Mn	0.6100	0.6316
S	0.0100	0.0129
Cu	0.0060	0.0102
Al	0.0400	0.0342
Cr	0.0100	0.0129
Ni	1.0000	0.9468
V	0.5100	0.5309
Ti	0.0020	0.0023
Sn	0.0700	0.0692
Mg	0.0800	0.0638

Material Data Base / Grade Table

grade table

delete save Load print close

226 Copy convert

index	grade no.	grade identification
12880	1.2880	1.2880
13202	1.3202	S 12-1-4-5
13247	1.3247	HS2-9-1-8
13316	1.3316	S 9-1-2
13318	1.3318	S 12-1-2
13333	1.3333	S 3-3-2
13342	1.3342	SC 6-5-2
13343	1.3343	S 6-5-2
13505	1.3505	100 Cr 6 (W3)
13520	1.3520	100 CrMn 6
13533	1.3533	17 NiCrMo 14
14001	1.4001	1.4001

double click

grade table entries

print close

index 13505 grade no. 1.3505

grade identification 100 Cr 6 (W3)

element no.	symbol
1	Fe.
2	C
3	Si
4	Mn
5	P
6	S

element no	minimal	maximal	extended mini	extended maximal	do not check
2	0,9	1,05	0,81	1,155	Air probe
3	0,15	0,35	0,135	0,385	never
4	0,25	0,45	0,225	0,495	never
7	0	0,3	0	0,33	never
9	1,35	1,65	1,3	1,805	never
12	0	0,3	0	0,33	never

- software compares analysis results with element concentration ranges and reports grade matches
- virtually unlimited material data entries

- international material standards as well as customised data base can be loaded
- Specific material data base can be linked to a measuring program

Thank you.

For more information or feedback contact Glen Thiele

mobile: 0420323577

email: info@spectrosource.com.au

- References
- (1) V. Thomsen, Modern Spectrochemical Analysis of Metals: An Introduction for Users of Arc/Spark Instrumentation (ASM International, Materials Park, Ohio, 1996).
- (2) Power Point Presentations – definitions and images: Belec Spectrometrie Opto-Electronik (2012 – 2016).
- (3) Various definitions and images: Wikipedia.
- (4) Melting Spectro Analysis Version 6: G Henderieckx – Gietech BV