Application Note

Long Term Stability Test for 19 Elemental Wavelengths

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Keywords: metallurgy, non-ferrous, halogens

1 Introduction

Long term stability is a very important figure of merit for any ICP spectrometer. Good stability enhances lab productivity and provides the user with increased confidence in the instrument and its results. High and low concentration standards were made for a 19-element method. Depending on the sensitivity of each element, the low standard was either 0.01, 0.1 or 0.2 ppm and the high standard 1 or 2 ppm. An analysis was performed over a period of 3 hours 40 min to provide stability data.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the “profile” function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample preparation

The standard was diluted in 5% HNO_3 in order to get the required concentration.

4 Instrument specification

The work was done on a JY ULTIMA 2 radial ICP spectrometer. The specifications of this instrument are listed in Tables 1 and 2.

Table 1: Specification of spectrometer

Parameter	Condition
Mounting	Czerny Turner
Focal length	1 m
Thermoregulation	Yes
Nitrogen purge	Yes
Grating number of grooves	2400 gr/mm
1st order resolution	0.010 nm
2nd order resolution	0.005 nm
Order	2nd order

Table 2: Specification of RF Generator

Parameter	Condition
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air

5 Operating conditions

The operating conditions are listed in Table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1000 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.1 L/min
Nebulizer gas flowrate	1 L/min
Nebulizer flowrate	3.4 bars (51 psi)
Sample uptake	1 mL/min
Type of nebulizer	Concentric
Type of spray chamber	Cyclonic
Argon humidifier	Yes
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The line with the highest sensitivity was used for each of the elements, as there were no interference problems. The conditions were the same for all elements.

Table 4: Analysis conditions

Element	Slits µm	Analysis Mode	Integration Time (sec)
All elements	20 x 15	Direct peaking	8

7 Discussion

7.1 Protocol

After a calibration with two points, a stability test was performed mixing the different samples. The sequence is shown in Table 5

Table 5: Sequence

	Time	Theoretical concentration for P in ppm	Theoretical concentration for Ca, Sr in ppm	Theoretical concentration for Al, Mo, Zn Pb, Co, Cd, Ni, Mn, Fe, Cr, Mg, V, Cu, Ti in ppm
1	0	20	1	2
2	10 min	2	0.1	0.2
3	23 min	0.4	0.02	0.04
4	1 h	20	1	2
5	1 h 10	2	0.1	0.2
6	2 h 45	20	1	2
7	3 h 10	2	0.1	0.2
8	3 h 30	0.4	0.02	0.04
9	3 h 40	20	1	2

7.2 Results

7.2.1 First solution

Sequence	Al Conc	167.020 nm RSD	P Conc	178.229 nm RSD	Mo Conc	202.030 nm RSD	Zn Conc	213.856 nm RSD
1	1.9894	0.85	19.7947	0.70	2.0306	1.49	2.0959	0.93
4	1.9675	2.17	19.4976	1.60	2.0334	1.90	2.0405	2.71
6	1.9684	3.28	19.8627	2.27	2.0239	0.68	2.0154	0.65
9	2.0371	1.77	19.7663	0.69	2.0540	0.64	2.0300	1.12
Conc	1.9906	2.02	19.7303	1.31	2.0354	1.17	2.0454	1.35
SD	0.0326		0.1604		0.0130		0.0352	
RSD	1.64		0.81		0.63		1.72	

Sequence	Pb Conc	220.353 nm RSD	Co Conc	228.616 nm RSD	Cd Conc	228.802 nm RSD	Ni Conc	231.604 nm RSD
1	2.0305	1.43	2.0714	1.27	2.0872	1.46	2.0472	1.33
4	2.0092	0.77	2.0558	2.17	2.0336	0.86	2.0317	1.26
6	2.0021	1.02	2.0300	1.23	1.9989	1.83	1.9646	3.06
9	1.9920	0.70	2.0522	1.12	2.0285	2.41	2.0473	0.68
Conc	2.0084	0.98	2.0523	1.45	2.0371	1.64	2.0227	1.58
SD	0.0163		0.0171		0.0367		0.0395	
RSD	0.81		0.83		1.80		1.95	

Sequence	Mn Conc	257.610 nm RSD	Fe Conc	259.940 nm RSD	Cr Conc	267.716 nm RSD	Mg Conc	2798.553 nm RSD
1	2.0288	2.58	2.0772	1.83	2.0074	3.12	2.0557	0.62
4	2.0385	0.88	2.0665	1.66	2.0149	2.48	2.0524	0.85
6	2.0065	2.08	2.0137	1.38	1.9558	1.97	2.0097	1.49
9	2.0179	0.53	2.0421	0.99	2.0272	0.57	2.0187	1.06
Conc	2.0229	1.52	2.0499	1.46	2.0013	2.04	2.0341	1.00
SD	0.0138		0.0282		0.0314		0.0233	
RSD	0.68		1.38		1.57		1.15	

Sequence	V Conc	292.402 nm RSD	Ca Conc	317.933 nm RSD	Cu Conc	324.754 nm RSD	Ti Conc	334.941 nm RSD
1	2.0418	0.69	1.0273	1.30	1.9721	1.10	1.9918	1.71
4	2.0411	1.34	1.0295	3.46	1.9510	1.42	2.0144	2.29
6	1.9950	1.79	1.0100	0.76	1.90008	0.55	1.9751	0.12
9	2.0344	0.91	1.0215	2.09	1.9343	1.23	1.9687	0.85
Conc	2.0281	1.18	1.0221	1.90	1.9396	1.07	1.9875	1.25
SD	0.0223		0.0088		0.0301		0.0204	
RSD	1.10		0.86		1.55		1.0275	

Sequence	Ca Conc	393.366 nm RSD	Al Conc	396.152 nm RSD	Sr Conc	407.771 nm RSD
1	0. 9826	1.23	1.9072	3.60	0.9758	1.20
4	0.9733	1.31	1.9625	1.28	0.9875	0.40
6	0.9749	1.30	1.9738	2.07	0.9918	1.06
9	0.9921	0.85	1.8871	2.44	0.9915	2.99
Conc	0.9807	1.17	1.9326	2.35	0.9867	1.41
SD	0.0086		0.0421		0.0075	
RSD	0.87		2.17		0.76	

7.2.2 Second solution

Sequence	Al Conc	167.020 nm RSD	P Conc	178.229 nm RSD	Mo Conc	202.030 nm RSD	Zn Conc	213.856 nm RSD
2	0.1972	0.69	1.9545	1.96	0.2040	1.47	0.2049	2.41
5	0.1933	1.80	1.9852	0.69	0.2010	0.64	0.2058	0.94
7	0.1867	0.18	1.9835	1.24	0.1992	0.26	0.2054	0.24
Conc	0.1924	0.89	1.9744	1.30	0.2014	0.79	0.2054	1.20
SD	0.0053		0.0713		0.0024		0.0004	
RSD	2.75		0.87		1.21		0.22	

Sequence	Pb Conc	220.353 nm RSD	Co Conc	228.616 nm RSD	Cd Conc	228.802 nm RSD	Ni Conc	231.604 nm RSD
2	0.2024	0.22	0.2024	0.78	0.2024	2.78	0.2016	2.31
5	0.2050	4.35	0.2054	0.29	0.2065	0.39	0.2064	0.49
7	0.2052	1.12	0.2017	0.10	0.1960	1.85	0.1965	2.47
Conc	0.2042	1.90	0.2031	0.39	0.2016	1.67	0.2015	1.76
SD	0.0015		0.0020		0.0053		0.0050	
RSD	0.76		0.97		2.63		2.46	

Sequence	Mn Conc	257.610 nm RSD	Fe Conc	259.940 nm RSD	Cr Conc	267.716 nm RSD	Mg Conc	279.553 nm RSD
2	0.2028	0.93	0.2001	0.90	0.1979	1.87	0.2077	0.32
5	0.2027	0.17	0.2070	2.19	0.2003	1.23	0.2094	2.39
7	0.2001	0.86	0.1998	1.35	0.1941	1.26	0.2026	2.31
Conc	0.2019	0.65	0.2023	1.48	0.1974	1.45	0.2066	1.67
SD	0.0015		0.0041		0.0031		0.0035	
RSD	0.75		2.01		1.58		1.70	

Sequence	V Conc	292.402 nm RSD	Ca Conc	317.933 nm RSD	Cu Conc	324.754 nm RSD	Ti Conc	334.941 nm RSD
2	0.1963	2.30	0.1000	2.08	0.1887	0.77	0.1975	0.51
5	0.1983	2.83	0.1006	1.98	0.1868	0.25	0.2021	1.11
7	0.1967	1.72	0.969	2.97	0.1831	1.80	0.1998	2.83
Conc	0.1971	2.29	0.0992	2.34	0.1862	0.94	0.1998	1.48
SD	0.0011		0.0020		0.0029		0.0023	
RSD	0.54		1.98		1.55		1.17	

Sequence	Ca Conc	393.366 nm RSD	Al Conc	396.152 nm RSD	Sr Conc	407.771 nm RSD
2	0.0937	0.63	0.2032	1.46	0.0960	1.39
5	0.0976	1.57	0.2026	0.29	0.0945	1.99
7	0.0942	1.40	0.1975	1.32	0.0938	1.23
Conc	0.0952	1.20	0.2011	1.02	0.0947	1.54
SD	0.0021		0.0032		0.0012	
RSD	2.24		1.57		1.21	

7.2.3 Third solution

Sequence	Al Conc	167.020 nm RSD	Co Conc	228.616 nm RSD	Cd Conc	228.802 nm RSD	Ni Conc	231.604 nm RSD
3	0.0357	2.47	0.3944	0.73	0.0391	1.93	0.0417	0.47
8	0.0346	8.23	0.3903	2.98	0.0391	1.66	0.0400	0.45
Conc	0.0352	5.35	0.3923	1.86	0.0391	1.80	0.0409	0.46
SD	0.008		0.0029		0.0000		0.0012	
RSD	2.31		0.74		0.12		3.02	

Sequence	Pb Conc	220.353 nm RSD	Co Conc	228.616 nm RSD	Cd Conc	228.802 nm RSD	Ni Conc	231.604 nm RSD
3	0.2037	8.08	0.0396	1.19	0.0410	1.29	0.0406	2.36
8	0.2027	12.5	0.0388	1.60	0.0386	1.82	0.0389	3.29
Conc	0.2032	10.3	0.0392	1.39	0.0398	1.56	0.0397	2.82
SD	0.0007		0.0005		0.0017		0.0013	
RSD	0.35		1.38		4.32		3.17	

Sequence	Mn Conc	257.610 nm RSD	Fe Conc	259.940 nm RSD	Cr Conc	267.716 nm RSD	Mg Conc	279.553 nm RSD
3	0.0404	2.00	0.0393	2.18	0.0399	1.56	0.0414	1.16
8	0.0401	0.39	0.0389	2.19	0.0377	0.92	0.0419	1.21
Conc	0.0402	1.20	0.0391	2.19	0.0388	1.24	0.0416	1.19
SD	0.002		0.0003		0.0016		0.0003	
RSD	0.52		0.72		4.00		0.79	

Sequence	V Conc	292.402 nm RSD	Ca Conc	317.933 nm RSD	Cu Conc	324.754 nm RSD	Ti Conc	334.941 nm RSD
3	0.0395	2.46	0.0185	2.58	0.0328	1.81	0.0397	2.07
8	0.0381	5.26	0.0190	1.48	0.0273	0.92	0.0391	0.87
Conc	0.0388	3.86	0.0188	2.03	0.0300	1.36	0.0394	1.47
SD	0.0010		0.0003		0.0039		0.0004	
RSD	2.56		1.74		1300		0.99	

Sequence	Ca Conc	393.366 nm RSD	Al Conc	396.152 nm RSD	Sr Conc	407.771 nm RSD
3	0.0193	1.52	0.0387	3.93	0.0191	1.30
8	0.0196	1.79	0.0404	1.08	0.0192	2.33
Conc	0.0195	1.65	0.0396	2.50	0.0192	1.82
SD	0.0002		0.0012		0.0001	
RSD	1.05		2.98		0.45	

8 Summary

The inherent stability of the JY ICP spectrometers is shown by the excellent results. The accuracy also shows that the lower concentrations of 0.2, 0.1 and 0.01 ppm can be easily determined on a routine basis. Results gave a precision of 0.8 to 2.1% RSD, depending on the element, for the period of 3 hours 40 min. Since less re-calibration is required, productivity is enhanced.

ISO 9001:2000
Certificate No A 25

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