Glow Discharge Atomic Emission Spectrometry and its performance in Accordance with ASTM E1009

LECO Corporation; Saint Joseph, Michigan USA

Instrument: GDS850A

Standard Practice for Evaluating an Optical Emission Vacuum Spectrometer to Analyze Carbon and Low Alloy Steel

ASTM E1009 is used to determine both accuracy and precision of a spectrometer using Certified Reference Materials (CRM) with proven homogeneity and which have been established as satisfactory for calibration. NIST SRM 1761 – 1767 have been used in this evaluation.

Accuracy

Accuracy is determined by computing the standard error (SE) of each calibration curve and comparing the result to the values provided in E1009-Table 2. Results lower than the values given are considered satisfactory. The equation used to determine SE is shown below:

$$SE = \sqrt{\sum dc^2 / f}$$

dc = difference between certified and calculated values

f = degrees of freedom (number of reference materials)

The results of evaluation for accuracy of a GDS-850A spectrometer are shown in Table 1. An example of the standard error calculation is shown in Table 2.

Table 1: Elements, Concentration Ranges and Standard Error (SE) for Steel

Element	*Concentration Range*	*Max Allowable[1] Std Error, %*	*Std Error GDS850A*
C	0.02 – 1.0	0.009	0.0024
Mn	0.02 – 2.0	0.012	0.0058
P	0.002 – 0.04	0.002	0.0004
S	0.002 – 0.04	0.002	0.0004
Si	0.01 – 0.6	0.012	0.0040
Ni	0.01 – 2.0	0.014	0.0069
Cr	0.02 – 1.5	0.012	0.0023
V	0.004 – 0.3	0.005	0.0024
Mo	0.004 – 0.5	0.005	0.0015
Cu	0.001 – 0.5	0.007	0.0027
Ti	0.005 – 0.3	0.005	0.0014
Al	0.005 – 0.07	0.005	0.0010
B	0.0001 – 0.005	0.001	0.0001

Note 1: ASTM E1009 Table 2

Table 2: Example of Calculation of Standard Error (SE) for Mn

Given %	*Calculated %*	*Difference (d) %*	*d²*
0.022	0.021	0.0009	0.00000076
0.067	0.068	-0.0011	0.00000110
0.144	0.150	-0.0056	0.00003136
0.678	0.668	0.0104	0.00010816
1.210	1.205	0.0050	0.00002500
1.580	1.582	-0.0020	0.00000400
2.000	2.008	-0.0080	0.00006400

$$\sum d^2 = 0.000234$$

$$\sum d^2 / n = 0.0000335$$

$$SE = \sqrt{\sum d^2 / n} = 0.0058$$

Precision

The second part of the standard is to determine the precision of the instrument by evaluating 4 to 6 reference materials of proven homogeneity. Each of the reference materials is analyzed ten times, randomly, in two different 4-hour periods, for a total of 20 evaluations. The standard deviation of the results are computed and compared to the values provided in E1009-Table 1. The standard deviations calculated for fourteen elements are shown in Table 3. All of the collected data and calculated statistics for SRM 1762 are shown in Table 4.

Spectroscopy Performance Note

Delivering the Right Results

Table 3: Standard Deviations Calculated from Precision Data

Element	*Certified %*	*STDEV*	*Element*	*Certified %*	*STDEV*
C	0.015	0.0007	V	0.0040	0.0002
	1.03	0.011		0.30	0.0018
Mn	0.022	0.0002	Mo	0.0050	0.0003
	2.00	0.021		0.50	0.0029
P	0.0020	0.0001	Cu	0.0014	0.0001
	0.040	0.0005		0.51	0.0060
S	0.0024	0.0001	Ti	0.0055	0.0002
	0.035	0.0009		0.31	0.0060
Si	0.010	0.0002	Al	0.0040	0.0003
	0.63	0.0059		0.069	0.0008
Ni	0.021	0.0002	Nb	0.010	0.0005
	1.99	0.020		0.10	0.0020
Cr	0.024	0.0002	B	0.0010	0.00002
	1.48	0.0108		0.0054	0.00007

Table 4: Collected Precision Data – NIST 1762 (n=20)

Run	*C*	*Mn*	*P*	*S*	*Si*	*Ni*	*Cr*	*V*	*Mo*	*Cu*	*Ti*	*Al*	*B*
1	0.34	1.98	0.033	0.030	0.35	1.14	0.92	0.20	0.35	0.12	0.090	0.069	0.0050
2	0.35	1.97	0.034	0.031	0.34	1.18	0.91	0.19	0.34	0.12	0.092	0.070	0.0051
3	0.34	1.98	0.033	0.030	0.34	1.16	0.91	0.19	0.34	0.12	0.090	0.070	0.0050
4	0.34	1.99	0.033	0.031	0.34	1.16	0.92	0.20	0.35	0.12	0.089	0.070	0.0051
5	0.33	2.02	0.032	0.030	0.34	1.14	0.93	0.20	0.35	0.12	0.090	0.071	0.0049
6	0.33	1.99	0.032	0.030	0.35	1.15	0.91	0.20	0.35	0.12	0.090	0.071	0.0049
7	0.34	1.99	0.033	0.030	0.35	1.16	0.92	0.20	0.35	0.12	0.091	0.070	0.0051
8	0.34	2.00	0.032	0.029	0.35	1.16	0.92	0.20	0.35	0.12	0.094	0.071	0.0050
9	0.34	2.00	0.033	0.030	0.35	1.17	0.92	0.20	0.35	0.12	0.090	0.071	0.0050
10	0.34	2.01	0.033	0.030	0.35	1.16	0.93	0.20	0.35	0.12	0.092	0.071	0.0050
11	0.34	2.00	0.034	0.031	0.35	1.17	0.92	0.20	0.35	0.12	0.095	0.071	0.0051
12	0.34	2.02	0.033	0.032	0.35	1.16	0.94	0.20	0.36	0.12	0.094	0.071	0.0050
13	0.34	2.01	0.033	0.030	0.34	1.17	0.93	0.20	0.35	0.12	0.092	0.071	0.0049
14	0.34	2.03	0.033	0.031	0.35	1.16	0.94	0.20	0.36	0.12	0.097	0.072	0.0049
15	0.34	2.03	0.033	0.032	0.34	1.16	0.93	0.20	0.35	0.12	0.095	0.072	0.0049
16	0.33	2.04	0.033	0.031	0.35	1.16	0.93	0.20	0.36	0.12	0.095	0.072	0.0049
17	0.34	2.02	0.033	0.030	0.35	1.17	0.93	0.20	0.35	0.12	0.089	0.072	0.0049
18	0.34	2.02	0.033	0.030	0.35	1.16	0.93	0.20	0.35	0.12	0.092	0.072	0.0050
19	0.33	2.04	0.033	0.030	0.35	1.16	0.93	0.20	0.36	0.12	0.096	0.072	0.0048
20	0.34	2.01	0.033	0.031	0.35	1.17	0.93	0.20	0.35	0.12	0.095	0.072	0.0050
Avg	**0.34**	**2.01**	**0.033**	**0.031**	**0.35**	**1.16**	**0.93**	**0.20**	**0.35**	**0.12**	**0.092**	**0.071**	**0.0050**
SD	**0.004**	**0.021**	**0.0004**	**0.0008**	**0.003**	**0.008**	**0.008**	**0.002**	**0.004**	**0.001**	**0.0026**	**0.0008**	**0.0001**

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