Designation: E 1009 – 95 (Reapproved 2006)

Standard Practice for Evaluating an Optical Emission Vacuum Spectrometer to Analyze Carbon and Low-Alloy Steel[1]

This standard is issued under the fixed designation E 1009; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers evaluation of an optical emission vacuum spectrometer to analyze carbon and low-alloy steels. It covers instruments used for the analysis of solid samples taken from molten metal for production control or from products to confirm the composition. Both pre-installation and post-installation precision and accuracy are included in the evaluation.

1.2 While Tables 1-3 are specific for plain carbon and low-alloy steel, they could be supplemented by similar tables for other materials.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves[3]

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis

E 528 Practices for Grounding Basic Optical Emission Spectrochemical Equipment[3]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E 135 and Practice E 876.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accuracy*—the closeness of a spectrochemical determination to an accepted reference; it is affected by imprecision and bias.

3.2.2 *standard error (SE)*—although primarily a calculation that measures how well a calibration has been defined, standard error (SE) is used in this practice as an indicator of accuracy. It is CRM-dependent and instrument-operator dependent. Some expected maximum SE values are listed, but comparisons between instrument calibrations can strictly be done only when identical suites of calibrants are used.

4. Summary of Practice

4.1 After the spectrometer is calibrated, use this practice to evaluate the instrument and its calibration. Certified reference materials are run as unknowns and precision is compared to Table 1. Before comparing standard errors to those in Table 2, ascertain that the calibration does not include unrealistic inflections. Values equal to or less than those in Tables 1 and 2 indicate that the instrument is acceptable.

5. Significance and Use

5.1 Periodically throughout the useful life of an optical emission spectrometer it becomes necessary to evaluate its performance. This is especially true at manufacture and during installation. The objective at this time is to establish whether the instrument meets design specifications and performs to customer specifications. A manufacturer's objective may be to compare production line instruments. With data on many instruments, such an evaluation procedure would be a valuable contribution to the manufacturer's quality control plan.

5.2 Use of this procedure at installation can tell the manufacturer or user whether there has been a significant change in performance due to faulty shipping or handling of the instrument. At this time, the procedure could be the beginning of a quality control plan for the user. Once established, the data from the procedure provide a base for comparison of future runs, enabling operators to detect changes in performance.

5.3 Data produced by this practice make possible a comparison of different instruments, for example, X-ray and optical emission or optical emission and atomic absorption. While the

[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

data in the Tables 1-3 are valid for optical emission spectrometers, other instruments may produce better or worse performance values. In this manner, the data could be used by management to determine the suitability of a given instrument to perform a given determination with an acceptable precision and accuracy.

TABLE 1 Recommended Precision Requirements For Steel Using An Optical Emission Vacuum Spectrometer[A]

Element	Approximate Concentration, %	Standard Deviation
C	0.06	±0.002
	0.15	0.004
	0.50	0.010
	0.90	0.015
Mn	0.35	0.007
	0.60	0.015
	1.00	0.02
	1.50	0.03
P	0.006	0.0007
	0.04	0.0015
S	0.005	0.0015
	0.04	0.002
	0.06	0.004
Si	0.02	0.004
	0.30	0.006
	0.50	0.010
Ni	0.03	0.002
	0.10	0.003
	0.70	0.007
	1.60	0.03
Cr	0.04	0.002
	0.30	0.007
	0.80	0.015
Sn	0.003	0.0006
	0.02	0.0015
	0.05	0.002
V	0.01	0.0015
	0.03	0.002
	0.25	0.007
Mo	0.03	0.003
	0.30	0.008
Cu	0.02	0.0007
	0.15	0.003
Ti	0.02	0.002
	0.20	0.008
Al	0.006	0.002
	0.02	0.003
	0.07	0.004
Nb	0.02	0.0015
	0.07	0.003
B	0.001	0.00015
	0.07	0.003
Zr	0.05	0.002
Pb	0.01	0.0015
Se	0.02	0.002

[A] These precisions were generated from actual data in one laboratory; as such, they represent what *has* been done with proven, homogeneous materials.

TABLE 1 (*a*) Revised Data

Element	Approximate Concentration, %	Standard Deviation
C	0.015	0.0009
C	1.03	0.012
MN	0.067	0.0006
Mn	2.00	0.023
P	0.0032	0.0007
S	0.0024	0.0001
Si	0.01	0.0002
Ni	0.021	0.0004
Ni	2.00	0.005
CR	1.48	0.009
Mo	0.005	0.0004
Mo	0.50	0.002
Cu	0.015	0.0003
Cu	0.30	0.0007
Ti	0.0055	0.0004
Al	0.004	0.0004
Al	0.04	0.0007
Nb	0.01	0.0005
Nb	0.1	0.002
B	0.001	0.0001
B	0.005	0.0001
Zr	0.013	0.0007
Pb	0.002	0.0004
As	0.01	0.0003
As	0.055	0.002

[A] These precisions were generated from data that were collected on newer instruments than the original data.

5.4 While this practice is directed towards optical emission vacuum spectrometers in the analysis of carbon and low-alloy steel, its use is not restricted to that instrument or that matrix.

6. Instrumentation

6.1 The vacuum spectrometer shall be equipped with an argon-flushed sample stand for point-to-plane excitation.

6.2 The excitation parameters and radiations measured shall be selected to meet the specified performance.

NOTE 1—Ordinarily this selection is made by the vendor, or instrument manufacturer, based on experience.

6.3 Provision shall be made to compensate for spectral interferences. More than one spectral line may be provided for an element, depending on element concentration or the excitation used, but switching of lines shall be done automatically.

7. Analysis Time

7.1 Analysis time, excluding sample preparation, shall not exceed 30 s for single burns, or 60 s for multiple burns.

NOTE 2—This requirement may be waived if speed of analysis is not