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Standard Practices for Describing and Specifying the Spectrograph¹

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1. Scope

1.1 These practices list and cover those features of the spectrograph that are of importance to spectrochemical analysis. They are intended thus to aid in evaluating and comparing spectrographs. It is not the purpose of these practices to establish binding specifications or tolerances, but rather to call attention to important parameters, suggest methods to measure those parameters, and, in some cases, assign values which are indicative of acceptably good performance. Because of the great variety of demands imposed by spectrochemical techniques, rigid performance criteria are not feasible. These practices shall be used to guide authors of analytical procedures in adequately defining their methods.¹

1.2 *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:

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis²

E 116 Practice for Photographic Photometry in Spectrochemical Analysis²

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

3. Terminology

3.1 For definitions of terms used in these practices refer to Terminology E 135.

4. Summary of Practice

4.1 Certain features and components of the spectrograph that must be considered in spectrochemical analysis are discussed (Section 5). Those features which must be specified fully to determine the performance of a spectrograph are discussed and methods of specification are given (Section 6).

5. Description of Pertinent Spectrograph Components and Associated Equipment

5.1 The following are pertinent to spectrochemical analyses:

5.1.1 *Type of Dispersing Element*—A prism, grating, echelle, or a combination of these shall be used. For a prism, consider material, type of prism, prism angle, width of base, and height (Note 1). For a grating or echelle include the number of grooves per millimetre (or inch), size of ruled area, blaze, method of fabrication (original or replica), blank material and configuration, and coating (Note 2).

NOTE 1—*Example*—Crystal quartz, Cornu, 60° prism, 100-mm base, 45 mm high.

NOTE 2—*Example*—A 1200 grooves/mm replica grating with ruled area 42 mm high by 92 mm wide, blazed for 3000 Å first order, and ruled on an aluminized borosilicate blank ground to a radius of 3 m.

5.1.2 *Type of Mounting*, whether stigmatic or astigmatic, the focal length of the collimator, and of the camera, if different.

NOTE 3—*Example*—Wadsworth, 3.4 m stigmatic.

5.1.3 *Dispersion*—Linear reciprocal dispersion is given in angstroms per millimetre. If it is a function of wavelength as in the case of a prism instrument, specify the dispersion at 500-Å intervals over the usable wavelength region or at a wavelength critical to the application.

NOTE 4—*Example*—Linear reciprocal dispersion in first order is 4 Å/mm.

5.1.4 *Resolving Power*—As distinct from the working resolution of a spectrograph discussed in 6.1.4, the resolving power, R , is a theoretical limit which can only be approached in practice. It is defined as follows for a grating instrument:

$$R = \lambda / \Delta\lambda = nM \quad (1)$$

where:

λ = wavelength,

$\Delta\lambda$ = wavelength differences between two lines just resolved,

n = grating order, and

M = number of lines ruled on grating.

For the first order of a spectrograph with a grating of 1200 grooves/mm and 100 mm wide ruled area at 3000 Å, $R = 120\,000$ and $\Delta\lambda = 0.025$ Å.

5.1.5 *Slit*, fixed or adjustable, uni- or bilateral, and the width or widths available to be specified; also the maximum and

¹ This practice is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices.

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² *Annual Book of ASTM Standards*, Vol 03.05.

minimum slit heights to be specified.

NOTE 5—*Example*—Bilateral slit width continuously adjustable from 0 to 0.1 mm; height continuously adjustable from 0 to 15 mm.

5.1.6 Wavelength Coverage—The total wavelength range for which the instrument can be used and the wavelength range which can be photographed at a single setting.

NOTE 6—*Example*—The entire region from 2000 to 10 000 Å can be photographed. Up to 2400 Å may be photographed at a single setting of the instrument (first order).

5.1.7 Camera—The size of plates or film, or both, that can be accommodated should be specified.

NOTE 7—*Example*—Camera will accommodate two 100 by 250-mm (4 by 10-in.) or two 50 by 250-mm (2 by 10-in.) plates.

5.1.8 Illuminating System (External Optics)—Consider each component of the illuminating system and note its function.

NOTE 8—*Example*—A quartz relay lens ($F_D = 60$ mm) is used to image the source at unit magnification on an aperture 8 mm wide and adjustable in height from 1 to 6 mm. This aperture is imaged on the collimator at 11× magnification by a quartz lens ($F_D = 180$ mm) located at the slit.

NOTE 9—It is quite essential to describe fully all external optics. Wide variations in performance can be brought about by changing the method of illumination.

5.1.9 Accessory Equipment—Shutter, plate racking mechanism, and any other devices which contribute to the performance of the instrument.

5.1.10 Size—Over-all size, weight, and distribution of weight on each of the supports.

6. Specification of Performance

6.1 Adequate performance for a specific problem cannot be ensured by a set of general specifications. It is possible, however, to describe some of the design parameters and performance criteria which primarily determine the quality of performance to be expected from a spectrograph. In addition, for versatility the instrument shall meet or exceed certain minimum specifications which will be given. For more detailed information the literature should be consulted.^{3,4,5}

6.1.1 Dispersing Element—To specify a prism for spectrochemical analysis, include only the information listed in 5.1.1. Any serious defects will be reflected in the over-all performance of the spectrograph and will be detected by failure to meet one or more of the subsequent specifications in this section. The same remarks apply to gratings and echelles with one exception, the specifications of the maximum permissible intensities of ghosts and satellites. The intensities of Rowland ghosts in the first order of a grating having 600 grooves/mm (15 000 grooves/in.) shall not exceed 0.1 % level. There shall be no more than three ghosts at either side of a parent line. If

any additional Rowland ghosts are present, their intensity shall not exceed 0.01 % of the parent line. Ghost lines may be readily recognized and their relative intensity estimated by photographing a simple, strong line spectrum such as obtainable with a mercury vapor lamp. In order to obtain uniform illumination of the spectrographic slit,⁶ mount the lamp in front of the slit in such a way that irregularities of the filament and markings on the envelope do not interfere. Use a step sector or step filter, and a 10-μm slit width but no other external optics. A spectral range setting to include the Hg 4358 Å line is quite suitable, but other strong mercury lines could be used as well. Make preliminary tests to check stability of the lamp and uniformity of illumination. Adjust the exposure time to record a strong mercury line such as Hg 4358 Å to a transmittance value between 1 and 10 %. Take two exposures for the actual test, one at the exposure time determined above and the other 1000 times longer, such as 3 s and 3000 s. Note ghost intensity by comparing the two exposures. Steps at the 1000× exposure for the ghost lines should be weaker than their counterparts of the parent line in the 1× exposure if the ghost intensity is to be less than 0.1 %. Because of the reciprocity effect this test cannot be more than a coarse guide. For more precise measurements, photomultipliers commonly are used.

6.1.2 Speed—Express the theoretical optical speed of a spectrograph as the effective F number, which is given as follows:

$$\text{Effective } F \text{ number} = f/2\sqrt{wh/\pi \cos \theta} \quad (2)$$

where:

f = focal length of the camera lens or mirror,

w = width of dispersing element,

h = height of dispersing element, and

θ = angle between the normal to the dispersing element and the direction of the radiation leaving that element.

The actual optical speed attainable will depend also upon the number of reflecting and absorbing elements in the optical train, the illumination employed, the plate tilt angle, and in the case of a grating, its blaze. For the latter, the relative speed of the spectrograph should be specified at 500-Å intervals throughout the available wavelength in range in each usable order. A practical test for the comparative speed of a spectrograph under conditions in use may be performed by recording the iron spectrum using a controlled set of conditions, and estimating the time required for selected lines to produce lines of 50 % transmittance. An intensity distribution curve is thus obtained which is particularly useful in comparing the speed of an individual spectrograph using, say, two different gratings or two different illuminating systems. The test is less useful in comparing two or more spectrographs because of uncontrollable variations in illuminating systems, source parameters, peculiarities of the optical elements, etc. A typical test is outlined as follows:

6.1.2.1 Source—A d-c arc between an upper negative graphite rod and a lower iron bead in a graphite electrode (iron globule arc) as described in Practice E 116.

³ Sawyer, R. A., *Experimental Spectroscopy*, Prentice-Hall, New York, NY, Second Edition, 1951.

⁴ Harrison, G. R., Lord, R. C., and Loofbourow, J. R., *Practical Spectroscopy*, McGraw-Hill, New York, NY, 1948.

⁵ Grove, E. L., *Analytical Emission Spectroscopy*, Marcel Dekker, Inc., New York, NY, 1971.