



Designation: E 406 – 81 (Reapproved 1996)

Standard Practice for Using Controlled Atmospheres in Spectrochemical Analysis¹

This standard is issued under the fixed designation E 406; 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 provides general recommendations relative to the use of gas shielding during and immediately prior to specimen excitation in optical emission spectrochemical analysis. It describes the concept of excitation shielding, the means of introducing gases, and the variables involved with handling gases.

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.* Specific precautionary statements are given in Section 9.

2. Referenced Documents

2.1 ASTM Standards:

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

E 416 Practice for Planning and Safe Operation of a Spectrochemical Laboratory³

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology E 135.

4. Significance and Use

4.1 An increasing number of optical emission spectrometers are equipped with enclosed excitation stands and plasmas which call for atmospheres other than ambient air. This practice is intended for users of such equipment.

5. Reference to this Practice in ASTM Standards

5.1 The inclusion of the following paragraph, or suitable equivalent, in any ASTM spectrochemical method, preferably in the section on excitation, shall constitute due notification that this practice shall be followed:

X.1 *Gas Handling*—Store and introduce the gas in accordance with Practice E 406.

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6. Concepts of Excitation Shielding

6.1 Control of Excitation Reactions:

6.1.1 Nonequilibrium reactions involving variable oxidation rates and temperature gradients in the analytical gap produce spurious analytical results. The use of artificial gas mixtures can provide more positive control of excitation reactions than is possible in air, although air alone is advantageous in some instances.

6.1.2 Methods of introducing the gas require special consideration. Temperature gradients in both the specimen and the excitation column can be controlled by the cooling effect of the gas flow. Also, current density can be increased by constricting the excitation column with a flow of gas.

6.1.3 Control of oxidation reactions is possible by employing nonreactive or reducing atmospheres. For example, argon can be used to preclude oxidation reactions during excitation. A gas may be selected for a particular reaction, such as nitrogen to produce cyanogen bands as a measure of the carbon content of a specimen. Oxygen is used in some instances to ensure complete oxidation or specimen consumption. In point-to-plane spark analysis, a reducing atmosphere can be provided by the use of carbon or graphite counter electrodes in combination with an inert gas⁴ or by the use of special circuit parameters⁵ in ambient air.

6.2 Effects of Controlled Atmospheres:

6.2.1 Numerous analytical advantages can be realized with controlled atmospheres:

6.2.1.1 The elimination of oxidation during point-to-plane spark excitation can significantly reduce the so-called “matrix” effects and compositional differences. This can result in improved precision and accuracy.

6.2.1.2 The use of argon or nitrogen atmospheres in point-to-plane procedures can *increase* instrument response so that a wide range of concentrations can be covered with one set of excitation parameters, but because of the increased background, *small losses* in the detection limit can result from oscillatory high voltage spark excitation. Which effect occurs depends on wavelengths used.

⁴ Schreiber, T. P., and Majkowaki, R. F., “Effect of Oxygen on Spark Excitation and Spectral Character,” *Spectrochimica Acta*, Vol 15, 1959, p. 991.

⁵ Bartel, R., and Goldblatt, A., “The Direct Reading Spectrometric Analysis of Alloy Cast Iron,” *Spectrochimica Acta*, Vol 9, 1957, p. 227.

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.

³ *Annual Book of ASTM Standards*, Vol 03.06.