



Designation: E 172 – 90 (Reapproved 1999)

Standard Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis¹

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1. Scope

1.1 This practice provides for the description of the various parameters of sources used to excite the spectra in optical emission spectrochemical analysis. Recent research on sampling and excitation of metals has shown that the critical parameter to control is the current through the specimen and circuit although the actual mechanism of sampling is unknown. A specific current can be achieved by several combinations of capacitance, voltage, resistance, and inductance, depending on the values for all four. A practice consisting of component values only is insufficient to properly describe an excitation source.

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

3. Terminology

3.1 Definitions:

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *capacitor discharge*—an electrical discharge of current from a capacitor or combination of capacitors, the energy of which is obtained from either an a-c or d-c electrical supply.

3.2.2 *glow discharge*—a low pressure, usually 0.01 to 3 kPa, electrical discharge in an inert gas resulting from the passage of current through the gas.

3.2.3 *laser*—a device that generates coherent electromagnetic radiation in the ultraviolet, visible, or infrared regions of the spectrum.

3.2.4 *plasma*—a flowing stream of gaseous charged particles containing either atomic or molecular ions and free electrons. It is a good conductor of electricity and is affected by magnetic fields. It is differentiated from “gas containing ions” in that the motions of charged particles in a plasma must be described collectively, not individually.

3.2.5 *self-initiating capacitor discharge*—a discharge of current from a capacitor that occurs at such time as the voltage on the capacitor is sufficient to break down the analytical or control gap. Discharge may also take place by triggering a control device such as a thyratron tube. (This type of discharge has been traditionally known as a *spark source*).

4. Significance and Use

4.1 The purpose of this practice is to provide fundamental information on equipment used to excite the spectrum of the specimen in optical emission spectrochemical analysis.

4.2 The selection of equipment to analyze the specimen depends on the nature of the specimen, the elements to be determined, and the concentration levels expected. In certain cases the time required to complete the analysis may be an important factor.

5. Description of Excitation Source Power Supplies

5.1 Where an analytical gap is an element of the excitation system, it is recommended that the circuitry be described in an overall way as producing one of the four types of current forms described in 5.1.1–5.1.4. Where an analytical gap is not an element of the excitation system, it is recommended that the source be described as producing one of the three types of excitation described in 5.1.5–5.1.7.

5.1.1 *Continuous d-c Arc*—A self-maintaining d-c electrical discharge.

5.1.2 *Noncapacitive a-c Arc*—A series of electrical discharges in which the individual discharges are either self-initiating or initiated by separate means. These discharges are extinguished when the potential across the analytical gap falls to a value that no longer is sufficient to maintain the discharge. Each current pulse is in the reverse direction from the previous one. The magnitude of peak current in each half-cycle is approximately constant.

5.1.3 *Noncapacitive, Intermittent d-c Arc*—A series of electrical discharges in which d-c pulses are initiated by separate means, either mechanically or electrically. Each current pulse

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.

has the same polarity as the previous one and lasts for less than 0.1 s.

5.1.4 Capacitor Discharge (Note 1):

5.1.4.1 Triggered Capacitor Discharge—A series of electrical discharges from capacitors, the energy of which is obtained from either an a-c or d-c electrical supply. Each discharge may have either an oscillatory, bipolar, or unipolar character. It is initiated by separate means and is extinguished when the potential across the analytical gap or the current through the gap falls to a value that no longer is sufficient to maintain the discharge.

5.1.4.2 Self-Initiating Capacitor Discharge—A series of electrical discharges, each of which may be oscillatory or directional and has a comparatively high maximum instantaneous current resulting from the breakdown of the analytical gap or the auxiliary gap, or both, by electrical energy stored at high voltage in capacitors. Each discharge is self-initiated and is extinguished when the potential across the gap, or gaps, or the current through these gaps is no longer sufficient to maintain the discharge. A following discharge will occur when the voltage on the capacitor has built up to a sufficient level and a control gap has been activated. This control gap may be an air blast gap, a mechanical rotary gap, thyratron, or gated solid state device.

NOTE 1—The self-initiating capacitor discharge (SICD) differs from the triggered capacitor discharge mainly in the range of electrical parameters available to each and that the triggered capacitor discharge requires separate means for initiating each discharge. In general, an SICD has potentials of the order of 5000 to 40 000 V using capacitances between 0.001 and 0.02 μF . A triggered capacitor discharge has potential of 200 to 2000 V and uses capacitances of 1 to 250 μF .

5.1.5 Glow Discharge:

5.1.5.1 Hollow Cathode—An electrical discharge in an inert gas at a pressure normally between 10^{-2} and 10^{-3} Pa that vaporizes and excites the sample. The sample-holding device is frequently a metal or graphite rod with a hole along its axis. The discharge occurs within the cavity of the cathode.

5.1.5.2 Plane Cathode—The specimen, which must have a plane surface, is made the cathode of the glow discharge. The discharge operates in an inert atmosphere at a pressure of between 10^{-2} and 10^{-3} Pa.

5.1.5.3 Jet-Assisted Glow Discharge—One or more high velocity gas jets strike the surface of the sample cathode, increasing the sampling rate and the emission intensities. When the observation is made in a direction parallel to the sample surface, the jets are positioned so as to sweep the sputtered sample atoms away from the surface into the observation zone. One configuration spaces six jets around the circumference of a circular sputtering region, and angles the jets towards the center causing a plume of entrained sample atoms and ions to rise in a direction normal to the flat surface of the sample.

5.1.5.4 Pulse Glow Discharge—A glow discharge that is pulsed intermittently to a high current to improve its spectral characteristics. Observations are usually synchronized with the pulses.

5.1.6 Plasma:

5.1.6.1 d-c (Direct Current)—A plasma in an inert gas, argon or helium, produced by a direct current between electrodes in the gas stream.

5.1.6.2 IC (Inductively Coupled)—A plasma in a nonoxidizing atmosphere, argon, nitrogen, or helium produced by a radio frequency field between 1 MHz and 1 GHz. Only frequencies allowed by the appropriate international agreements for use in industrial equipment should be employed.

5.1.6.3 MI (Microwave Induced)—A plasma in an inert gas, usually helium, produced by a field of frequency greater than 1 GHz, most commonly 2.45 GHz.

5.1.7 Laser:

5.1.7.1 A laser consists of an active medium in which population inversion of energy levels and therefore stimulated emission of radiation may be obtained by optical pumping or electrical excitation. Laser action may either be continuous or discontinuous in the form of a laser pulse or a sequence of laser pulses. The medium may be a solid, liquid, or gas. A laser analytical system may or may not use auxiliary capacitor discharge excitation for enhanced light output from the vapor plume.

6. Required Electrical Parameters

6.1 The following parameters, including specimen polarity, should be specified in analytical practices to permit users to match the source characteristics employed by this practice. For each type source, specify where applicable the current, voltage, frequency, capacitance, inductance, resistance, pulse duration, gas, flow rate, pressure, and other significant parameters (Note 2).

NOTE 2—The actual detailed electrical circuits and parts are beyond the scope of this practice. The reader is referred to specific manufacturer's literature or to a comprehensive review of sources,³ which contains an extensive bibliography.

6.2 Continuous d-c Arc Source—List the current as measured during the first 5 s of arcing. For example:

Current, A, d-c 14.0

6.3 Noncapacitive a-c Arc Source—List the rms current, output potential of the transformer, and frequency of the power supply during operation. State whether the discharge is self-initiating or separately initiated. If separately initiated, list the parameters of the initiating circuit. For example:

Power Circuit	
Current, A, rms (root mean square)	2.3
Potential, V, rms	2400
Frequency, Hz	60
Ignitor	
Capacitance, μF	0.0025
Inductance, μF	residual
Resistance, Ω	2.5
Voltage, V	20 000

6.4 Noncapacitive Intermittent d-c Arc Source—Describe the primary power supply and list the rms current. State whether the initiating means is mechanical or superimposed high frequency. If the latter, list the parameters of the circuit. For example: The primary power supply is a half-wave rectified, 110-V, 60-Hz power line. It is initiated by a low-power spark synchronized to initiate each half cycle when the primary power supply reaches 80 V.

Current, A, d-c 6.5

³ *Emission Spectroscopy*, edited by Ramon M. Barnes; Dowden, Hutchinson, Ross, Stroudsburg, PA, 1976.