Designation: E 697 – 96 (Reapproved 2001)

Standard Practice for Use of Electron-Capture Detectors in Gas Chromatography[1]

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1. Scope

1.1 This practice is intended to serve as a guide for the use of an electron-capture detector (ECD) as the detection component of a gas chromatographic system.

1.2 This practice is intended to describe the operation and performance of the ECD as a guide for its use in a complete chromatographic system.

1.3 For general gas chromatographic procedures, Practice E 260 or Practice E 1510 should be followed except where specific changes are recommended in this practice for use of an ECD. For a definition of gas chromatography and its various terms, see Practice E 355. These standards also describe the performance of the detector in terms which the analyst can use to predict overall system performance when the detector is coupled to the column and other chromatographic components.

1.4 *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.* For specific safety information, see Section 3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 260 Practice for Packed Column Gas Chromatography[2]

E 355 Practice for Gas Chromatography Terms and Relationships[2]

E 1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs[2]

2.2 *CGA Standards:*

CGA P-1 Safe Handling of Compressed Gases in Containers[3]

CGA G-5.4 Standard for Hydrogen Piping Systems at Consumer Locations[3]

CGA P-9 The Inert Gases: Argon, Nitrogen and Helium[3]

CGA V-7 Standard Method of Determining Cylinder Valve Outlet Connections for Industrial Gas Mixtures[3]

CGA P-12 Safe Handling of Cryogenic Liquids[3]

HB-3 Handbook of Compressed Gases[3]

2.3 *Federal Standard:*

Title 10, Code of Federal Regulations, Part 20[4]

3. Hazards

3.1 *Gas Handling Safety*—The safe handling of compressed gases and cryogenic liquids for use in chromatography is the responsibility of every laboratory. The Compressed Gas Association (CGA), a member group of specialty and bulk gas suppliers, publishes the following guidelines to assist the laboratory chemist to establish a safe work environment. Applicable CGA publications include: CGA P-1, CGA G-5.4, CGA P-9, CGA V-7, CGA P-12, and HB-3.

3.2 The electron capture detector contains a radioactive isotope that emits β-particles into the gas flowing through the detector. The gas effluent of the detector must be vented to a fume hood to prevent possible radioactive contamination in the laboratory. Venting must conform to Title 10, Code of Federal Regulations, Part 20 and Appendix B.

4. Principles of Electron Capture Detection

4.1 The ECD is an ionizating detector comprising a source of thermal electrons inside a reaction/detection chamber filled with an appropriate reagent gas. In packed column GC the carrier gas generally fullfills the requirements of the reagent gas. In capillary column GC the make-up gas acts as the reagent gas and also sweeps the detector volume in order to pass column eluate efficiently through the detector. While the carrier/reagent gas flows through the chamber the device detects those compounds entering the chamber that are capable of reacting with the thermal electrons to form negative ions. These electron capturing reactions cause a decrease in the concentration of free electrons in the chamber. The detector response is therefore a measure of the concentration and the change in concentration of electrons **(1-17)**.[5]

4.2 A radioactive source inside the detector provides a source of β-rays, which in turn ionize the carrier gas to produce a source of electrons **(18)**. A constant or intermittent negative potential, usually less than 100 V, is applied across the reaction chamber to collect these electrons at the anode. This flow of“

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[2] *Annual Book of ASTM Standards*, Vol 3.06.

[3] Available from Compressed Gas Association, Inc., 1725 Jefferson Davis Highway, Arlington, VA 22202-4100.

[4] Available from Superintendent of Documents, Government Printing Office, Washington, DC 20402.

[5] The boldface numbers in parentheses refer to a list of references at the end of this practice.

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