

Designation: D 6806 – 02

Standard Practice for Analysis of Halogenated Organic Solvents and Their Admixtures by Gas Chromatography¹

This standard is issued under the fixed designation D 6806; 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 the determination of impurities, stabilizers and assay of halogenated organic solvents and their admixtures by gas chromatography.

1.2 It is not the intent of this practice to provide a specific method of gas chromatography. The intent of this practice is to define what is required for a user to demonstrate that a method to be used is valid. The reason for this approach, as opposed to stating a method, is that gas chromatography is such a dynamic field that methods are often obsolete by the time they are validated. The use of this practice allows the user to use most effective technology and demonstrate that the method in use complies with a standard practice and is valid for the analysis of halogenated organic solvents and their admixtures.

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

ASTM D

2.1 *ASTM Standards:* E 180 Practice for Determining the Precision and Bias of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals²

3. Terminology

3.1 Purity and assay are used interchangeably in this standard.

3.1.1 Purity or assay are defined as 100 - sum(impurities + stabilizer) when impurities and stabilizers are expressed in %;

3.1.2 Or $100 - [sum(impurities + stabilizer)/10\ 000]$ when impurities are expressed in ppm.

3.2 Accuracy is defined per A2.2.1 of Practice E 180 or the agreement between an experimentally determined value and the accepted reference value.

3.3 Precision is defined per A2.1.7 of Practice E 180 or the degree of agreement of repeated measurements of the same property. It is generally expressed in standard deviations or percent relative standard deviation $(s/X) \cdot 100$, also known as coefficient of variation.

4. Summary of Practice

4.1 This practice will define the requirements for a gas chromatographic (GC) method to be valid for the determination of impurities, stabilizers and assay of halogenated organic solvents and their admixtures.

5. Method Requirements

5.1 The GC method must give adequate separation of the impurities and stabilizers common to the product in question so that the instrument response (area counts, milivolts, etc.) to the individual impurities or stabilizers can be measured with adequate precision and accuracy as defined in Section 6. Process knowledge from the supplier or manufacturer of the product is a resource of information as to what those impurities and stabilizers are. GC-Mass Spectrometry (GC-MS) is another resource to initially determine what components are present to be measured. See Table 1 for a list of possible impurities and stabilizers for each product.

5.2 Non polar capillary columns of about 0.32 mm by 30 m generally work well. Table 2 provides a list of columns that may prove suitable for the analysis of the halogenated organic products.

5.3 *Instrument Conditions*—The following GC conditions are often appropriate for the analysis of halogenated organic products and their admixtures though actual conditions should be optimized for the analysis being performed.

5.4 The separation is determined to be adequate by preparing standards of known amounts of the impurities and stabilizers in concentrations near enough to the expected concentrations in the sample for the instrument response to be linear over the concentration range of interest. Repeated injections (minimum of three) of this standard are made and the accuracy and precision of the method for the individual impurities and stabilizers are determined.

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¹ This practice is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.04 on Test Methods.

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² Annual Book of ASTM Standards, Vol 15.05.