Designation: D3271 – 87 (Reapproved 2012)

Standard Practice for Direct Injection of Solvent-Reducible Paints Into a Gas Chromatograph for Solvent Analysis[1]

This standard is issued under the fixed designation D3271; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice describes the techniques used to inject whole paint samples directly into a gas chromatograph to obtain a chromatogram from which the solvent composition may be established.[2,3]

1.2 This practice is not designed to be quantitative.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.* A specific hazard statement is given in 6.1.

2. Referenced Documents

2.1 *ASTM Standards:*[4]

E260 Practice for Packed Column Gas Chromatography

3. Summary of Practice

3.1 A suitable aliquot of whole paint is injected, by means of a syringe, into a gas chromatographic column in order to separate the solvents.

4. Significance and Use

4.1 Gas chromatographic separation of solvents present in whole paints is the preferred first step for identifying and quantitating solvent compositions, using auxiliary procedures and techniques.

5. Apparatus

5.1 *Gas Chromatograph*—Any instrument with temperature programming capability may be used. It should be equipped with either a thermal conductivity or flame ionization detector (see Practice E260).

5.2 *Recorder*—A 1 to 10-mV recorder with a full-scale response time of 2 s or less and a maximum noise of ±0.03 % of full scale.

5.3 *Column*—The resolution of the column must be such that under the operating conditions selected the distance from the base line to the depression between two adjacent peaks must be not more than 50 % of the smaller peak. Columns may be either packed or capillary.

6. Reagents and Materials

6.1 *Carrier Gas*—Helium or hydrogen for use with thermal conductivity detector units; and nitrogen, helium or argon for use with flame ionization detector units.

6.1.1 **Warning:**When hydrogen is used, special precautions should be taken to prevent gas leakage from causing a possible explosion.[5]

6.2 *Column Packing Material,* meeting requirements in 5.3. The following materials have been used satisfactorily:

6.2.1 Polyethylene glycol, molecular weight 20 000, and diisodecyl phthalate as liquid phases on a solid support of 60 to 80 mesh (250 to 175 µm) diatomaceous earth.

6.2.2 Porous beads of ethylvinylbenzene and divinylbenzene copolymer, 60 to 80-mesh (250 to 177-µm) have also been successfully used as column material.

6.3 *Liquid Charging Devices,* such as micro syringes. Disposable type is preferred.

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[1] This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[2] New York Society for Paint Technology, Technical Committee 76, "The Application of Gas Chromatography to the Analysis of Coating Solvents," *Journal of Paint Technology*, Vol 40, No. 516, January 1968, pp. 33–48.

[3] Esposito, G. G., and Swann, M. H., "Direct Analysis of Solvents in Lacquer by Programmed Temperature Gas Chromatography," *Official Digest Federation Society Paint Technology*, Vol 33, No. 440, September 1961, pp. 1122 –1131.

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[5] Consult Pamphlet G-5, available from the Compressed Gas Association, Inc., 500 5th Ave., New York, NY 10036.

6.4 *Septum Sample Vials,* PTFE-fluorocarbon-faced.

7. Procedure

7.1 Protect the injection port from the nonvolatile portion of the paint by using a borosilicate glass injection port sleeve (Note 1), glass wool plug, or by any other suitable means. If the whole paint sample is of sufficiently low viscosity, draw an aliquot directly into the syringe. When the whole paint sample is too viscous, dilute with a suitable solvent (Note 2) that will not interfere with the analysis. In order to minimize solvent losses due to evaporation, the whole paint sample should be placed in a PTFE-fluorocarbon-faced septum sample bottle prior to being drawn into the syringe. Proceed with normal techniques to obtain a chromatogram of the solvent or solvents present in the sample.

NOTE 1—Consult manufacturer of particular instrument used.

NOTE 2—Ethyl ether and methylene chloride have been found satisfactory for most sample types.

8. Keywords

8.1 chromotography, direct injection techniques; chromotography, gas; chromotography, solvent composition

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