



Designation: D 2743 – 68 (Reapproved 1998)

Standard Practices for Uniformity of Traffic Paint Vehicle Solids by Spectroscopy and Gas Chromatography¹

This standard is issued under the fixed designation D 2743; 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 These practices provide general information on the instrumental techniques available for detecting adulteration or nonuniformity of the chemical nature of the vehicle solids in purchased lots of traffic paints by means of the individual or combined use of infrared and ultraviolet spectroscopy and gas chromatography. The procedures given are applicable when traffic paint is selected and purchased on the basis of prequalification laboratory or road performance tests, or both, and a *reference sample* of the original paint so evaluated and selected is retained and compared with test samples representative of subsequent purchased and delivered lots of such paint and which are required to be the same as the original reference sample.

1.2 Although not specifically provided for in these practices, the methods given may also be applied, with appropriate modification, to evaluating the acceptability of traffic paints that have been purchased on the basis of composition specifications. In such cases, application is limited to the vehicle solids as before, as well as the availability of a suitable standard or range of standards representative of the vehicle solids that are acceptable and with which samples of subsequent delivered lots will be compared.

1.3 The techniques provided are wholly adequate for detecting gross adulteration of the vehicle solids where completely different drying oils, resins, or polymers, or combinations of these have been substituted for those originally contained in the reference sample. In cases of lesser adulteration or modification, these methods have been found adequate for detecting vehicle solids, adulterations, or modifications as low as 5 weight % of the vehicle solids.

1.4 These techniques have been developed on the basis of cooperative work with alkyd, chlorinated rubber-alkyd, and poly(vinyl toluene) type paints involving the detection of nonuniformity when such extraneous materials as rosin, fish

oil, hydrocarbon resin, and chlorinated paraffin have been added. The procedures given may be, but are not necessarily completely applicable to all other types of vehicle solids or extraneous additions, or both.

1.5 The methods provided appear in the following order:

	Section
Method A—Infrared Spectral Analysis of Total Vehicle Solids	10-12
Method B—Infrared Spectral Analysis of Unsaponifiable Matter from Vehicle Solids	13-15
Method C—Gas Chromatographic Analysis of Oils and Oil Acids Separated from Vehicle Solids	16-18
Method D—Ultraviolet Spectral Analysis of Total Vehicle Solids	19,20, and 21

1.6 *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:

- D 1259 Test Methods for Nonvolatile Content of Resin Solutions²
- D 1397 Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions³
- D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints²
- D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints²
- D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints²
- E 105 Practice for Probability Sampling of Materials⁴
- E 131 Terminology Relating to Molecular Spectroscopy⁵

3. Terminology

3.1 For definitions of terms and symbols, refer to Terminology E 131.

¹ These practices are under jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 03.06.

4. Summary of Methods

4.1 Each of the methods given requires both a reference and a test sample of traffic paint and a preliminary separation and removal of the pigment component in each.

4.2 *Method A* involves infrared spectral analysis of cast films of the total vehicle solids to detect spectral differences between the reference and test samples caused by gross or minor adulteration of the test sample.

4.3 *Method B* involves infrared spectral analysis of cast films of the unsaponifiable matter that has been separated from the vehicle solids in order to detect spectral differences between the reference and test samples caused by lesser adulterations of an unsaponifiable nature and which was not readily evident when using Method A.

4.4 *Method C* involves gas chromatographic analysis of prepared methyl esters of the separated fatty acids obtained from the vehicle solids in order to detect chromatographic differences between the reference and test samples caused by either gross or lesser adulteration of the drying oil fraction with extraneous drying oils which may not have been readily evident by the use of Methods A and B.

4.5 *Method D* involves quantitative ultraviolet spectral analysis of the total vehicle solids dissolved in a nonaromatic spectral grade solvent to give precise concentrations in order to detect ultraviolet spectral absorbance differences between the reference and test samples caused by minor or sophisticated adulterations of the vehicle solids and which may not be readily detected by Method A. Method D is to be used as an alternative to Methods B and C.

5. Selection of Test Methods and Significance and Use

5.1 All of the methods provided involve comparisons between the spectra or chromatograms of the reference and test samples to determine if they show significant differences. It is not possible at this time to establish quantitative limits as a guide to whether a spectral or chromatographic difference is truly significant. Certainly the presence or absence of a moderate or strong peak in the test sample which is not evident in the reference is significant. A persistent difference in the ratios of two peaks of one spectrum as compared to the reference sample is significant. On the whole, some judgment must be exercised in this respect and it is advisable to refer to published data on infrared or gas chromatography in order to establish, where feasible, the possible overall nature of the adulterant or its functional group which might be causing the comparison spectra to differ.

5.2 *Method A* is rapid and the most convenient of the procedures given. It should be utilized first in order to detect nonuniformity of the test sample. Significant spectral differences from that of the reference sample can be taken as an indication of adulteration and in such cases the use of the other methods is not necessary. As a general rule, Method A is sufficient to detect gross or major adulteration of the vehicle solids. However, where Method A shows no significant spectral differences, it cannot be assumed that the test sample is completely acceptable since changes in the type of drying oil, polyol, and certain dibasic acids in alkyd resins, addition of certain aliphatic or nonfunctional hydrocarbon resins, and

many minor adulterations may not always show characteristic infrared spectral differences. Therefore, in such cases it is best to proceed to additional tests as given in Methods B and C or else alternatively directly to Method D.

5.3 *Method B* is useful in detecting adulterations that are unsaponifiable or else have an unsaponifiable component that has escaped detection in Method A only because the adulterant may have been small in amount and therefore its strong spectral peaks may have been masked over by the rest of the vehicle solids. Some care should be taken in interpreting spectral differences in Method B to avoid an erroneous conclusion that the test sample is unacceptable because its spectrum is different. Apparent but unreal differences can occur as a result of incomplete saponification, failure to remove all saponifiable material, and varying degrees of contamination of the unsaponifiable fraction with sterols, etc., present in the vehicle solids. After it has thus been firmly established that a real spectral difference does exist, further tests are unnecessary, except that it is wise to resort again to the published literature on infrared to attempt to identify the possible nature of the adulterant. Where Methods A and B indicate acceptability of the test sample, it is still not always possible to rule out adulteration caused by changes or modifications in the saponifiable portion, that is, the type of fatty acid, dibasic acids, and polyol. In such cases, it is best to continue on to Method C for determination of the oil acids, and to other gas chromatographic methods for the polyol and dibasic acids when such equipment is available.

5.4 *Method C* is extremely sensitive in detecting adulterations and changes that have been made in the oil or fatty acid portion of the vehicle solids. It can, for example, detect whether linseed, coconut, oiticica, etc., has been substituted for soya oil and vice versa, or whether fish or tall oil has partially or wholly replaced some other drying oil, etc. Consequently, when the results of Methods A and B suggest that the test sample is acceptable and where a drying oil component is known to be present, Method C should be used additionally for more complete assurance of product uniformity. Where the results from Method C along with those from Methods A and B indicate product uniformity, it is a fairly safe assumption that the product has not been significantly altered.

5.5 *Method D* is intended as an alternative to Methods B and C and where the results from Method A indicate apparent product acceptability. Method D, by the use of quantitative ultraviolet spectral absorbance data, is an extremely sensitive procedure for the detection of complete or even partial adulteration of the test sample. However, considerable caution must be exercised in the preliminary pre-drying of the vehicle solids since it is at this stage that the components are extremely sensitive to oxidative changes. Even minor oxidative changes can seriously affect the absorbance data obtained in ultraviolet spectral analysis and may give an impression that the two samples being compared are different when in fact they are the same. When these considerations are provided for, and the comparison spectra are identical in Method D as well as in Method A, then it can be assumed that the sample is acceptable. Significant differences in the spectra from Method D would