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Standard Practice for Calculation of Gas Chromatographic Response Factors[1]

This standard is issued under the fixed designation D 4626; 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 describes a procedure for calculating gas chromatographic response factors. It is applicable to chromatographic data obtained from a gaseous mixture or from any mixture of compounds that is normally liquid at room temperature and pressure or solids, or both, that will form a solution with liquids. It is not intended to be applied to those compounds that react in the chromatograph or are not quantitatively eluted. Normal C_6 through C_{11} paraffins have been chosen as model compounds for demonstration purposes.

1.2 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are for information only.

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

2.1 *ASTM Standards:*

D 2268 Test Method for Analysis of High-Purity *n*-Heptane and *Iso*octane by Capillary Gas Chromatography[2]

D 2427 Test Method for Determination of C_2 Through C_5 Hydrocarbons in Gasolines by Gas Chromatography[2]

D 2804 Test Method for Purity of Methyl Ethyl Ketone by Gas Chromatography[3]

D 2998 Test Method for Polyhydric Alcohols in Alkyd Resins[4]

D 3329 Test Method for Purity of Methyl Isobutyl Ketone by Gas Chromatography[3]

D 3362 Test Method for Purity of Acrylate Esters by Gas Chromatography[3]

D 3465 Practice for Purity of Monomeric Plasticizers by Gas Chromatography[5]

D 3545 Test Method for Alcohol Content and Purity of Acetate Esters by Gas Chromatography[3]

D 3695 Test Method for Volatile Alcohols in Water by Direct Aqueous-Injection Gas Chromatography[6]

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[7]

E 260 Practice for Packed Column Gas Chromatography[8]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *response factor (R)*—a constant of proportionality used to convert the observed chromatographic response of specific compounds to either mass or volume percent composition. The observed response may be measured as peak areas or peak heights. Depending on the calculation formula, the response factor (*R*) is applied by either multiplying or dividing the observed response by the determined factor.

3.1.2 In this practice, the response factors determined are multiplying factors.

4. Summary of Practice [9]

4.1 Individual C_6 to C_{11} *n*-paraffins are precisely weighed and combined in an inert, tight-sealing glass vial. Different concentration levels of the blend components to cover concentration ranges of interest may be obtained by dilution with a suitable solvent. As diluent, a *n*-paraffin, such as *n*-dodecane, that is, higher boiling than the blend components is suitable. The quantitative blends are analyzed, in duplicate, by gas chromatography using either thermal conductivity, flame-ionization or other forms of detection. From the mass or volume composition of the blend and the raw area or peak height measurements, mass or volume response or relative response factors for each blend component are calculated.

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 06.04.

[4] *Annual Book of ASTM Standards*, Vol 06.03.

[5] *Annual Book of ASTM Standards*, Vol 08.02.

[6] *Annual Book of ASTM Standards*, Vol 11.02.

[7] *Annual Book of ASTM Standards*, Vol 05.02.

[8] *Annual Book of ASTM Standards*, Vol 14.02.

[9] Supporting data are available from ASTM Headquarters. Request RR: D02-1200.

5. Significance and Use

5.1 ASTM standard gas chromatographic methods for the analysis of petroleum products require calibration of the gas chromatographic system by preparation and analysis of specified reference mixtures. Frequently, minimal information is given in these methods on the practice of calculating calibration or response factors. Test Methods D 2268, D 2427, D 2804, D 2998, D 3329, D 3362, D 3465, D 3545, and D 3695 are examples. The present practice helps to fill this void by providing a detailed reference procedure for calculating response factors, as exemplified by analysis of a standard blend of C_6 to C_{11} *n*-paraffins using *n*-C_{12} as the diluent.

5.2 In practice, response factors are used to correct peak areas to a common base prior to final calculation of the sample composition. The response factors calculated in this practice are "multipliers" and prior to final calculation of the results the area obtained for each compound in the sample should be multiplied by the response factor determined for that compound.

5.3 It has been determined that values for response factors will vary with individual installations. This may be caused by variations in instrument design, columns, and experimental techniques. It is necessary that chromatographs be individually calibrated to obtain the most accurate data.

6. Apparatus

6.1 *Chromatograph*—Any gas chromatograph equipped with either a flame ionization, thermal conductivity or other detector may be used that meets the performance requirements of the method for which calibration is being performed.

6.2 *Recorder*—A recording potentiometer with a full-scale response time of 1 s or less may be used.

6.3 *Integrator or Computer*—Means must be provided for determining the detector response. Peak heights or areas can be measured by computer, electronic integration or manual techniques.

NOTE 1—Rapidly eluting peaks such as those produced by a capillary column are difficult to accurately measure manually. Therefore, peaks of this type must be measured by computer or electronic integration.

6.4 *Column*—Any column may be used that will satisfactorily separate the compounds of interest, including the solvent, if used.

6.5 *Sample Introduction*—Sample introduction may be by means of a constant volume liquid sample valve or by injection with a microsyringe through a septum.

6.6 *Blend Preparation Apparatus*—The specific equipment required to prepare liquid blends is described in Test Method D 4307.

7. Reagents and Materials

7.1 *Carrier Gas*, helium, hydrogen, or other suitable gases may be used depending on the detector and the requirements of the method being calibrated.

7.2 *Combustion Gases*—Air and hydrogen are required for flame ionization detectors.

7.3 *n-Paraffin Hydrocarbons*, C_6, C_7, C_8, C_9, C_{10}, C_{11} and C_{12}-99 % pure.

7.4 *Solvent*, used as a diluent to vary concentrations of blend components. A suitable solvent is one that is relatively nonvolatile, miscible with all sample components and, preferably, well resolved chromatographically from all mixture components. In this model, *n*-C_{12} is used.

8. Procedure

8.1 *Instrument Preparation*—Install the chromatographic columns and establish the flow rates and operating temperatures as specified in the method for which calibration is being performed. Refer to Practice E 260 for specific instructions. Condition the columns at their required operating temperature until a stable baseline is established at the required sensitivity.

8.2 *Calibration Blends*—Prepare appropriate calibration blends as described in Practice D 4307. The blends should resemble as closely as possible the components and concentrations expected in the test sample to be analyzed, because response factors may not be linear over large concentration ranges.

NOTE 2—For volume response factors, volumetric concentrations are calculated from gravimetric concentrations using component densities in accordance with Practice D 4307.

8.3 *Blend Analysis*—Analyze each prepared blend in duplicate using chromatographic conditions and injection technique that are identical to those used for test samples.

8.4 *Peak Measurements*—Determine the peak height or area of each *n*-paraffin in the blend, excluding *n*-C_{12} diluent, using the same measurement technique that is to be used for test samples. Where electronic integration or a computer is used, the various integration parameters must be the same for analysis of the blends and for the test samples.

9. Calculation

9.1 Calculate the response factor for each *n*-paraffin on a mass (weight) basis as follows:

$$R_M = M/A \tag{1}$$

where:

R_M = mass (weight) response factor for a specific *n*-paraffin, g/unit
M = mass (weight) of a specific *n*-paraffin in the blend, g, and
A = area or peak height of the specific *n*-paraffin peak, units.

9.1.1 Calculate the mass relative response factors as follows:

NOTE 3—For purposes of this model calculation *n*-heptane has been chosen as the standard reference compound.

$$RR_M(C_N) = R_M(C_N)/R_M(C_7) \tag{2}$$

where:

$RR_M(C_N)$ = mass (weight) relative response factor for a *n*-paraffin of carbon number N
$R_M(C_N)$ = mass (weight) response factor for a specific *n*-paraffin of carbon number N determined in 9.1, g/unit
$R_M(C_7)$ = mass (weight) response factor for a *n*-heptane determined in 9.1, g/unit

9.2 Calculate the response factor for each *n*-paraffin on a volume basis as follows:

$$R_V = V/A \tag{3}$$

where:

R_V = volume response factor for a specific *n*-paraffin, mL/unit,

V = volume of the specific *n*-paraffin in the blend, mL, and

A = area or peak height of the specific *n*-paraffin peak, units.

9.2.1 Calculate the volume response factors as follows:

$$RR_V(C_N) = R_V(C_N)/R_V(C_7) \quad (4)$$

where:

$RR_V(C_N)$ = volume relative response factor for a specific *n*-paraffin of carbon number N,

$R_V(C_N)$ = volume response factor for a specific *n*-paraffin of carbon number N determined in 9.2, mL/unit, and

$R_V(C_7)$ = volume response factor for *n*-heptane determined in 9.2, mL/unit.

10. Keywords

10.1 gas chromatography; response factor