Designation: D8442 – 22

Standard Test Method for Determination of Cannabinoids in Cannabis Raw Materials and Resin Cannabis Products by Gas Chromatography and Flame Ionization Detection[1]

This standard is issued under the fixed designation D8442; 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 test method covers the analysis of cannabinoids in cannabis products by gas chromatography (GC) and flame ionization detection (FID).

1.2 This test method is applicable to cannabis raw materials and resin cannabis products as defined in Guide D8245, including those from hemp. Such material includes: biomass; plant material; flowers; resins; extracts; distillates; recovered solvents; and other intermediate processing material. The applicable concentration range of analysis will vary to some extent depending on the nature of the sample, for instance measurement of delta-9-tetrahydrocannabinol (Δ^9-THC) for regulatory purposes in hemp would require calibration to lower concentration levels compared to measurement of CBD in its isolate; however, in most cases, the test method is applicable to the determination of major and minor cannabinoids above about 0.1 mass% in concentration. Dilution of sample solutions is used to adjust concentrations to fall within appropriate calibration curves. Particular emphasis is placed on the determination of Δ^9-THC for regulatory compliance purposes and control. This test method can measure any cannabinoid that is eluted and detected from a GC column with sufficient resolution from any interfering compounds. Typical cannabinoids of interest that can be determined by this test method are shown in Table 1. Use of an HPLC technique is recommended if individual measurement of acids, such as THCA, is required.

1.3 The test method does not purport to identify all individual cannabinoids; however, individual users can adapt this test method for specific custom analyses to meet their needs.

1.4 *Units*—Values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4626 Practice for Calculation of Gas Chromatographic Response Factors

D8245 Guide for Disposal of Resin-Containing Cannabis Raw Materials and Downstream Products

D8270 Terminology Relating to Cannabis

D8282 Practice for Laboratory Test Method Validation and Method Development

D8334/D8334M Practice for Sampling of Cannabis/Hemp Post-Harvest Batches for Laboratory Analyses

D8375 Test Method for Determination of Cannabinoid Concentration in Dried Cannabis and Hemp Raw Materials using Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology D8270 for guidance on terminology relating to cannabis.

[1] This test method is under the jurisdiction of ASTM Committee D37 on Cannabis and is the direct responsibility of Subcommittee D37.03 on Laboratory.

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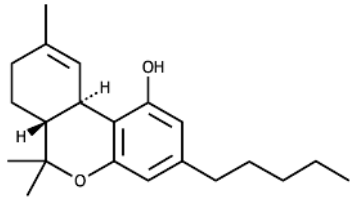
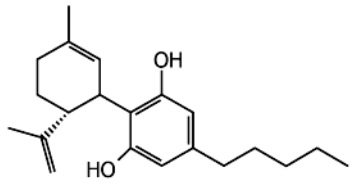
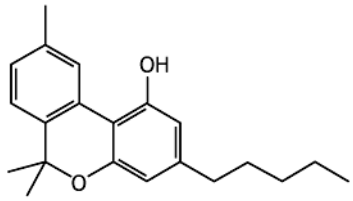
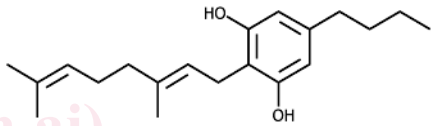
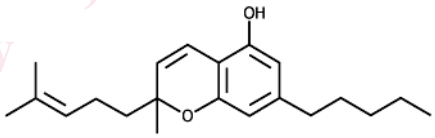
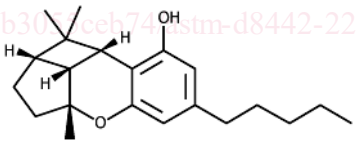
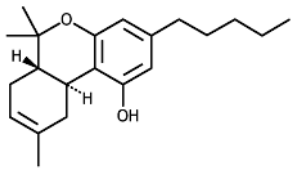
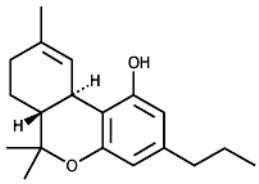
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

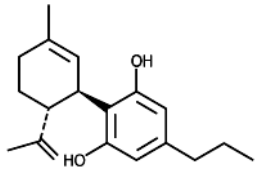
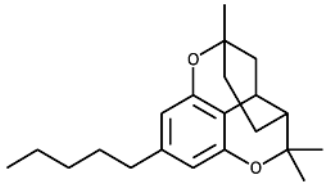
TABLE 1 Typical Cannabinoids Measured

Name	CAS #	Abbreviation	Chemical Structure
Δ9-Tetrahydrocannabinol	1972-08-3	THC or d9-THC	
Cannabidiol	13956-29-1	CBD	
Cannabinol	521-35-7	CBN	
Cannabigerol	25654-31-3	CBG	
Cannabichromene	20675-51-8	CBC	
Cannabicyclol	21366-63-2	CBL	
Δ8-Tetrahydrocannabinol	5957-75-5	d8-THC	
Tetrahydrocannabivarin	31262-37-0	THCV	

3.1.2 *decarboxylation, n*—removal of a carboxylate group from a cannabinolic acid usually through application of heat or by exposure to ultraviolet light.

3.1.3 *distillate, n*—intermediate or final product resulting from separation by distillation often used in combination with terms to denote specific fractions.

TABLE 1 *Continued*

Name	CAS #	Abbreviation	Chemical Structure
Cannabidivarin	24274-48-4	CBDV	
Cannabicitran	31508-71-1	CBT	

3.2 *Definitions:*

3.2.1 *CBC*—cannabichromene

3.2.2 *CBD*—cannabidiol

3.2.3 *CBDV*—cannabidivarin

3.2.4 *CBG*—cannabigerol

3.2.5 *CBL*—cannabicyclol

3.2.6 *CBN*—cannabinol

3.2.7 *CBT*—cannabicitran

3.2.8 *CRM*—certified reference material

3.2.9 *FID*—flame ionization detector

3.2.10 *GC*—gas chromatography

3.2.11 *HPLC*—high performance liquid chromatography

3.2.12 *ILS*—interlaboratory study

3.2.13 *LC*—liquid chromatography

3.2.14 *PPE*—personal protective equipment

3.2.15 *PTFE*—polytetrafluoroethylene

3.2.16 *PTV*—programmed temperature vaporization

3.2.17 *THC*—tetrahydrocannabinol

3.2.18 *THCV*—tetrahydrocannabivarin

3.2.19 Δ^9-*THC*—delta-9-tetrahydrocannabinol

3.2.20 Δ^8-*THC*—delta-8-tetrahydrocannabinol

3.2.21 *WCOT, n*—wall-coated open tubular

4. Summary of Test Method

4.1 The sample is analyzed by GC and FID. Standards are used to cover the desired concentration range of the analysis. Cannabinoid peaks are identified by means of their retention times. Generally, a known amount of sample is weighed and diluted with a solvent. Many samples of interest, for example, distillates, require little sample preparation. Biomass samples shall be dried and decarboxylated separately before their extraction, dilution, and analysis.

4.2 *FID*—Unknown peaks may be quantified using the average FID response factor for calibrated cannabinoids, however, it is desirable to identify and calibrate components with authentic standards. The FID produces a highly sensitive and linear response that is validated by the guidelines of Practice D8282.

5. Significance and Use

5.1 Gas chromatography and flame ionization detection provides a rapid means to identify and quantify cannabinoids in a variety of samples of interest. This test method allows producers of cannabis products to improve and optimize the quality of their products. For example, hemp extractors can use it to determine the efficiency of extraction processes and to verify that products meet regulatory requirements, ensuring safety and quality of products.

5.2 Cannabinoids, such as CBD and THC can be monitored throughout the production process. The determination of Δ^9-THC is often required for regulatory purposes and the determination of other THC isomers is often of interest. The United Nations Office on Drugs and Crime provides experimental details and guidance for use of GC to analyze cannabis related samples, including conditions suitable for decarboxylation of cannabinoid acids.[3]

5.3 Post-decarboxylated methodology is used. In decarboxylation, heat is used to liberate carbon dioxide from carboxylic acid cannabinoids, forming their corresponding neutral cannabinoids, for example, THC from THCA. It should be recognized that the hot temperature of the GC injection port itself is capable of effecting at least some decarboxylation (250 °C – Table 2), and many sample types, such as distillates, require no decarboxylation because it would have occurred during material processing. Therefore, some knowledge of sample properties and material processing is useful. Resulting determinations are for the total cannabinoid content of specific isomers, for example, total Δ^9-THC. For those samples requiring decarboxylation, the method is validated per Practice D8282 through the use of reference materials, spike and recovery of knowns, or through comparison with LC results.

[3] Recommended Methods for the Identification and Analysis of Cannabis and Cannabis Products, MANUAL FOR USE BY NATIONAL DRUG ANALYSIS LABORATORIES, United Nations, New York, 2009.

TABLE 2 Typical GC Conditions

Instrument	GC equipped with a split/splitless injection port
Column	Capillary column: 15 m, 0.25 mm inside diameter, 0.25 µm, Rxi-35Sil MS
Flow conditions	1 mL/min hydrogen carrier (constant flow)
Inlet	260 °C
Detector	FID
FID	Temperature: 350 °C 40 mL/min H2, 400 mL/min Air
Oven	80 °C (hold 1 min) to 320 °C at 20 °C/min (hold 3 min)
Sample injection	1 µL, split 10:1

For example, carrying out the decarboxylation procedure of a standard containing known amounts of CBDA and CBN should yield the correct amounts of CBD and CBN, where CBN is not significantly changed and the mass of CBD formed from CBDA should be 0.877 × CBDA. The same ratio applies to THC and THCA. For other cannabinoids of interest, the conversion factor is derived from the ratio of formula masses of the neutral to that of its acid. If the observed conversion deviates significantly from expected results, then corrective action is necessary.

5.4 As an aid, users are referred to other methods for determination of individual cannabinoids and their acids, for example, Test Method D8375.

NOTE 1—Other ASTM standards are in development.

6. Interferences

6.1 Coelution of cannabinoids with other species, if occurring, will result in inaccurate determinations. Cannabinoids in cannabis and minimally processed products are generally amenable to GC analysis, however, if validation indicates inaccurate recovery of any cannabinoids of interest, then corrective action must be taken, for example, switching to a higher resolution column. More sophisticated methods, such as Test Method D8375, shall be used if greater selectivity, specificity, and the need for determination of neutrals and cannabinoid acids individually is needed.

7. Apparatus

7.1 *Chromatograph*—Use a GC that has the following performance characteristics (see Practice E355):

7.1.1 *Column Temperature Programmer*—The gas chromatograph shall be capable of linear programmed temperature operation over a range sufficient to elute the last compounds of interest within a suitable analysis time. The programming rate shall be sufficiently reproducible to obtain a retention time repeatability of 0.01 min.

7.1.2 *Inlet*—A split/splitless inlet with liner optimized for cannabis analysis is recommended. PTV and programmable cool on-column injection systems have also been used successfully. As part of routine quality assurance, users shall verify adequate standard recovery and cannabinoid peak shapes, of which indicate the need for inlet maintenance, for example, from accumulation of particulates within its inlet liner.

7.1.3 *Detector*—The FID shall meet or exceed the specifications in accordance with Practice E594.

7.1.4 *Column*—The test method uses a WCOT (capillary) column. Columns and conditions used must provide adequate separation of the cannabinoids of interest. Fused silica, glass, and metal capillaries of 10 m to 60 m lengths with 0.25 mm, 0.32 mm, or 0.53 mm internal diameters and bonded stationary phases more polar than 100 % dimethyl-polysiloxane and with film thicknesses of 0.1 µm to 0.5 µm have been successfully used. An example chromatogram showing elution order and baseline separation of several typical cannabinoids of interest using the conditions of Table 1 is illustrated in Fig. 1. It is up to the user to validate the suitability of alternative columns and conditions.

7.1.5 *Microsyringe*—A microsyringe with a 23 gauge or smaller stainless-steel needle is used for sample introduction. Syringes of 0.1 µL to 10 µL capacity are commercially available. Automatic syringe injection is recommended. Other sample introduction devices are allowed provided that repeatable injection areas of less than 10% relative standard deviation are achieved.

7.2 *Data Acquisition System*—Use of an electronic integrating device or computer is mandatory. The device shall have the following capabilities: *(1)* graphic presentation of the chromatogram; *(2)* digital display of chromatographic peak areas; *(3)* measurement of area and retention times; and *(4)* calculation and use of response factors in accordance with Practice D4626, for example, external standardization.

7.3 Analytical balance capable of meeting the requirements of Practice D4307, for example, able to measure mass to a precision of 0.0001 g.

7.4 Grinder/homogenizer for biomass samples; cryogenic grinder optional.

7.5 Laboratory oven, able to minimally reach and maintain a temperature of 110° C.

7.6 Hot plate optional.

7.7 Vortex mixer optional.

7.8 Centrifuge optional.

7.9 Solvent dispenser optional.

7.10 Dispensing pipettes optional.

7.11 Cartridge or syringe filters, 0.45 µm or smaller, PTFE membrane.

7.12 Centrifuge tubes optional.

7.13 Glass vials.

7.14 Class A volumetric flasks.

7.15 Forceps optional.

8. Reagents and Materials

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such