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.

Designation: D5466 – 21

Standard Test Method for Determination of Volatile Organic Compounds in Atmospheres (Canister Sampling, Mass Spectrometry Analysis Methodology)[1]

This standard is issued under the fixed designation D5466; 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 describes a procedure for sampling and analysis of selected volatile organic compounds (VOCs) in ambient, indoor, and workplace atmospheres. The test method is based on the collection of whole air samples in stainless steel canisters with specially treated (passivated) interior surfaces.

1.2 For sample analysis, a portion of the sample is subsequently removed from the canister and the collected VOCs are selectively concentrated by adsorption or condensation onto a trap, subsequently released by thermal desorption, separated by gas chromatography, and measured by a low resolution mass spectrometric detector. This test method describes procedures for sampling into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).[2]

1.3 This test method is applicable to specific VOCs that have been determined to be stable when stored in canisters (see Table 1). Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (**1-4**).[3] Information on storage stability is also available for polar compounds (**5-7**). This test method has been documented for the compounds listed in Table 1 and performance results apply only to those compounds. A laboratory may determine other VOCs by this test method after completion of verification studies that include measurement of recovery as specified in 5.7 and that are as extensive as required to meet the performance needs of the customer and the given application.

1.4 The procedure for collecting the sample involves the use of inlet lines, air filters, flow rate regulators for obtaining time-integrated samples, and in the case of pressurized samples, an air pump. Typical long-term fixed location canister samplers have been designed to automatically start and stop the sample collection process using electronically actuated valves and timers (**8-10**). Temporary or short-term canister samplers may require the user to manually start and stop sample collection. A weatherproof shelter may be required if the sampler is used outdoors. For the purposes of this test method, refer to Practice D1357 for practices and planning ambient sampling events.

1.5 The organic compounds that have been successfully measured single-digit micrograms per cubic metre (μg/m^3 (or single digit parts-per-billion by volume (ppbv)) concentration with this test method are listed in order of approximate retention time in Table 1. The test method is applicable to VOC concentrations ranging from the detection limit to approximately 1000 μg/m^3 (300 ppbv). Above this concentration, smaller sample aliquots of sample gas may be analyzed or samples can be diluted with dry ultra-high-purity nitrogen or air or equivalent.

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

1.7 *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. Safety practices should be part of the user's SOP manual.*

1.8 *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.*

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] This test method is based on EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)" January 1999.

[3] The boldface numbers in parentheses refer to the list of references at the end of the standard.

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TABLE 1 Volatile Organic Compounds Determined by the Canister Method

NOTE 1—See 5.7 for requirements to add to this list.

Compound (Synonym)	Formula	Molecular Weight	Vaper Pressure kPa (25°C)	Listed in the U.S. EPA TO-14A/TO-15	CAS Number
Freon 12 (Dichlorodifluoromethane)	Cl_2CF_2	120.91	568	X/X	75-71-8
Methyl chloride (Chloromethane)	CH_3Cl	50.49	506	X/X	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	X/X	76-14-2
Vinyl chloride (Chloroethylene)	CH_2=CHCl	62.50	344	X/X	75-01-4
1,3-Butadiene	$(CH_2=CH)_2$	54.09	279	/X	106-99-0
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	X/X	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	64.52	12.3	X/X	75-00-3
Acetonitrile	C_2H_3N	41.05	9.9	O/X	75-05-8
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	X/X	75-69-4
Acrylonitrile	C_2H_3N	53.03	11.0		107-13-1
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	X/X	75-35-4
Dichloromethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	X/X	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	X/X	76-13-1
Trans-1,2-Dichloroethylene	$C_2H_2Cl_2$	96.95	44.13	O/X	156-60-5
1,1-Dichloroethane	CH_3CHCl_2	98.96	57.3	X/X	74-34-3
Methyl *tert*-Butyl Ether	$(CH_3)_3COCH_3$	88.15	32.7	O/X	1634-04-4
2-Butanone Methyl Ethyl Ketone	C_4H_eO	77.11	13.3	O/X	78-93-3
Chloroprene	C_4H_5Cl	88.54	25.06	O/X	126-99-8
cis-1,2-Dichloroethylene	CHCl=CHCl	96.94	60.3		156-59-2
Bromochloromethane	CH_2BrCl	129.38	15.6	O/X	74-97-5
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	X/X	67-66-3
Ethyl tert-Butyl Ether	$C_6H_{14}O$	102.18	32.7	X/X	637-92-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	X/X	107-06-2
Methyl chloroform (1,1,1,-Trichloroethane)	CH_3CCl_3	133.41	74.1	X/X	71-55-6
Benzene	C_6H_6	78.12	80.1	X/X	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	X/X	56-23-5
Tert-Amyl Methyl Ether	$C_6H_{14}O$	102.18	9	O/X	994-05-8
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	X/X	78-87-5
Ethyl Acrylate	$C_5H_8O_2$	100.12	2	O/X	140-88-5
Trichloroethylene (Trichloroethene)	$ClCH=CCl_2$	131.29	87	X/X	79-01-6
Methyl Methacrylate	$C_5H_8O_2$	100.12	3.9	X/X	80-62-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$CH_3CC=CHCl$	110.97	4.59	X/X	542-75-6
Methyl Isobutyl Ketone	$C_6H_{12}O$	100.16	2.13	O/X	108-10-1
trans-1,3-Dichloropropene (trans-1,3-Dichloropropylene)	$ClCH_2CH=CHCl$	110.97	3.07	X/X	542-75-6
1,1,2-Trichloroethane (Vinyl trichloride)	$CH_2ClCHCl_2$	133.41	2.53	X/X	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	2.8	X/X	108-88-3
Dibromochloromethane	$CHBr_2Cl$	208.28	7.32	O/X	124-48-1
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	1.56	X/X	106-93-4
n-Octane	C_8H_{18}	114.23	1.47	X/X	111-65-9
Tetrachloroethylene (Perchloroethylene)	$Cl_2C=CCl_2$	165.83	1.87	X/X	127-18-4
Chlorobenzene	C_6H_5Cl	112.56	1.20	X/X	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	1.33	X/X	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3\text{-}(CH_3)_2C_6H_4$	106.17	1.20	X/X	108-38-3
p-Xylene (1,4-Dimethylxylene)	$1,4\text{-}(CH_3)_2C_6H_4$	106.17	1.20	X/X	106-42-3
Bromoform	CH_2Br_3	252.73	0.747	X/X	75-25-2
Styrene (Vinyl benzene)	$C_6H_5CH=CH_2$	104.16	0.67	X/X	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	0.67	X/X	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2\text{-}(CH_3)_2C_6H_4$	106.17	0.93	X/X	95-47-6
4-Ethyltoluene	C_9H_{12}	120.19	1.38	X/O	622-96-8
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5\text{-}(CH_3)_3C_6H_6$	120.20	0.267	X/X	108-67-8
1,2,4-Trimethylbenzene	$1,2,4\text{-}(CH_3)_3C_6H_6$	120.20	0.665	X/X	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3\text{-}Cl_2C_6H_4$	147.01	0.286	X/X	541-73-1
Chloromethylbenzene	$C_6H_5CH_2Cl$	126.58	0.133	O/X	100-44-7
Benzyl chloride (α-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	0.123	X/O	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2\text{-}Cl_2C_6H_4$	147.01	0.181	X/X	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4\text{-}Cl_2C_6H_4$	147.01	0.232	X/X	106-46-7
1,1,2,3,4,4–Hexachloro–1,3-butadiene	Cl_6C_4	260.76	0.04	X/X	7-68-3
1,2,4 - Trichlorbenzene	$C_6H_3Cl_3$	181.44	0.133	O/X	120-82-1

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

E355 Practice for Gas Chromatography Terms and Relationships

[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.