



Designation: D5197 – 21

Standard Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)¹

This standard is issued under the fixed designation D5197; 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 presents a procedure for the determination of formaldehyde (HCHO) and other carbonyl compounds (aldehydes and ketones) in air. Other carbonyl compounds that have been successfully quantified by this method include acetaldehyde, acetone, propanal (propionaldehyde), 2-butanone (methyl ethyl ketone), butyraldehyde, benzaldehyde, isovaleraldehyde, valeraldehyde, o-tolualdehyde, m-tolualdehyde, p-tolualdehyde, hexanal, and 2,5-dimethylbenzaldehyde.

1.2 This test method involves drawing air through a cartridge containing silica gel coated with 2,4-dinitrophenylhydrazine (DNPH) reagent. Carbonyl compounds readily form stable derivatives with the acidified DNPH reagent. The DNPH derivatives are analyzed for parent aldehydes and ketones using high performance liquid chromatography (HPLC) or ultra-high performance liquid chromatography (UHPLC). UHPLC systems use higher pressures and smaller particle sizes in columns compared to HPLC systems. The sampling procedure is a modification of U.S. EPA Method TO-11A (see 2.2).

1.3 This test method is based on the reaction of carbonyl compounds with DNPH in the presence of an acid to form stable derivatives according to the reaction shown in Fig. 1, (where: both R and R^1 are alkyl or aromatic groups (ketones), or either, or both R or R^1 is a hydrogen atom (aldehydes)). The determination of formaldehyde and other carbonyl compounds, as DNPH derivatives, is similar to that of U.S. EPA Method TO-11A in that it uses HPLC or UHPLC for separation of carbonyl compounds followed by UV adsorption or photodiode array detection. This test method exceeds the stated applicability of TO-11A to include other carbonyl compounds that can be determined as stated in 10.2.4. This test method is suitable for determination of formaldehyde and other carbonyl compounds in the airborne concentration range from approximately 10 ppb_{v/v} (12 $\mu\text{g}/\text{m}^3$), requires sampling for 1 h at 1 L/min) to

1 ppm_{v/v} (1.2 mg/m^3). Lower concentrations in air may be determined using higher sampling volume and with control of contamination, appropriate selection of flow rate and sampling duration.

1.4 The sampling method gives a time-weighted average (TWA) sample. It can be used for long-term (1 to 24 h) or short-term (5 to 60 min) sampling of air for formaldehyde. Shorter sampling times or low flow rates will result in higher detection limits and may result in greater variation in co-located sampler results. Tests should be performed over a duration and a flow rate that allows the data quality objective of the project to be achieved. Sample times for other carbonyls, such as acetaldehyde, may be limited to short term (1).² The data provides total concentrations of carbonyl compounds from which time weighted average concentrations can be calculated.

1.5 This test method instructs the user on how to prepare sampling cartridges from commercially available chromatographic grade silica gel cartridges³ by the application of acidified DNPH to each cartridge.

1.6 The sampling flow rate, as described in this test method, has been validated for sampling rates up to 1.5 L/min for formaldehyde. This flow rate limitation is principally due to the high pressure drop (>8 kPa at 1.0 L/min) across user prepared silica gel cartridges which have a particle size of 55 to 105 μm . These cartridges are not generally compatible with battery-powered pumps used in personal sampling equipment (for example, those used by industrial hygienists).

1.7 Alternatively, pre-coated DNPH silica gel cartridges are commercially available and may be substituted provided they can be demonstrated to meet blank and analyte trapping acceptance criteria (2). Some of these use silica gel of a larger particle size that results in a lower pressure drop across the cartridge. These low pressure drop cartridges may be more suitable for sampling air using battery-powered personal sampling pumps.

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 The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ The cartridge used in the development and performance evaluation of this test method was the Sep-Pak Plus Silica cartridge. Other manufactures make similar products.