ASTM D5910-05 (2012) Standard Test Method for Determination of Free Formaldehyde in Emulsion Polymers by Liquid Chromatography

Significance and Use

4.1 With the need to calculate free formaldehyde levels in emulsion polymers, it is necessary to make the determination without upsetting any equilibria that might generate or deplete formaldehyde. This test method provides a means for determining ppm levels of free formaldehyde in emulsion polymers without upsetting existing equilibria.

1. Scope

1.1 This test method is used for the determination of free formaldehyde (HCHO) in emulsion polymers without upsetting existing formaldehyde equilibria. The procedure has been evaluated using acrylic, acrylonitrile-butadiene, carboxylated styrene-butadiene and polyvinyl acetate emulsion polymers. This test method may also be applicable for emulsion polymers of other compositions. The established working range of this test method is from 0.05 to 15 ppm formaldehyde. Emulsion polymers must be diluted to meet the working range.

1.2 This test method minimizes changes in free formaldehyde concentration that can result from changes in the physical or chemical properties of an emulsion polymer.

1.3 There are no known limitations to this test method when used in the manner described. The emulsion polymer test specimen must be prepared with a diluent that has a pH similar to that of the emulsion. Use of an inappropriate pH may upset formaldehyde equilibria and result in incorrect formaldehyde levels.

1.4 The values stated in SI units are to be regarded as 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Abstract

Sample Preparation: Formaldehyde (HCHO) is reacted with 2,4-dinitrophenylhydrazine (DNPH) to form a Schiff base (HCHO-DNPH derivatization product) which has an absorbing maximum (λmax) at 360 nm.

HPLC System: (Ctrl + Click to follow link)
Column: Licrosorb RP8 (250 × 4 mm, 10 µm, Merck)
For shorter retention times use: **Waters Symmetry Columns C8 5µm 4.6*150mm HPLC Column** or **Agilent HPLC Column ZORBAX Eclipse XDB-C8 Analytical 4.6*150mm 5-Micron** or **Ansys HPLC Packed Column Inertsil 5u C8 (Ctrl + Click to follow link)**

Injection Volume: 10-µL
Column temperature: 30°C
UV Wavelength: 345nm
Mobile Phase: Water–Acetonitrile (55:45, v/v)
Flow Rate: 1 mL/min
Expected retention time of the HCHO–DNPH Product: Approx. 11min. with Licrosorb RP8 (250 × 4 mm, 10 µm column, or between 6 to 10 min. with a 150 x 4.6mm. 5 µm C8 column.

References: http://www.astm.org/Standards/D5910.htm

G. Lunn, L.C. Hellwig. Handbook of Derivatization Reactions for HPLC. Wiley-Interscience Publication, 1998

Benassi, C. A., Semenzato, A., Zaccaria, F. and Bettero, A. 1990. High performance liquid chromatographic determination of free formaldehyde in cosmetics preserved with Dowicil 200. Journal of Chromatography 502.

Wu, Pai-Wen; Chang, Chieu-Chen; Chou, Shin-Shou. Determination of formaldehyde in cosmetics by HPLC method and acetylacetone method. Journal of Food and Drug Analysis 11(1): 8-15 2003