PICKERING LABORATORIES

Analysis of Formaldehyde

Formaldehyde is widely used in manufacturing of building materials, cars, plywood, polymers, glues and adhesives. It is also commonly used in household items as a preservative and disinfectant. Due to high toxicity and suspected carcinogenicity products are tested to determine free formaldehyde content and ensure the safety of personal care products and other items. Free formaldehyde testing is also important to monitor production of resins and other polymers and to ensure the quality of final materials.

The HPLC method with post-column derivatization allows for quantifying free formaldehyde in a wide range of products and materials, from shampoos to emulsion polymers and phenolic resins. The method is simple, selective and very sensitive. Potential interferences are either separated from formaldehyde on the HPLC column or don't react with post-column reagent and so are not interfering with the detection.

Method

Analytical Conditions

Column: Waters Atlantis® HILIC Silica, 5 um, 4.6 x 250 mm

Column Temperature: 40 °C

Flow Rate: 0.5 mL/min

Mobile Phase: 15% 0.05 M Ammonium Acetate in water
85% Acetonitrile

Injection Volume: 10-50 uL

Sample Preparation

Leather samples (According to ISO 177226-1)
Weigh approximately 2 g of leather pieces to the nearest 0.01 g into a 100 ml glass Erlenmeyer flask. Add 50 ml of 0.1% sodium dodecylsulfonate or sodium dodecylsulfate solution (previously preheated at 40 °C) and fit the Erlenmeyer flask with a glass stopper. Shake the contents of the flask in the water bath for 60 min at 40 °C. Immediately filter the warm extract solution by vacuum through a glass fiber filter into a flask. Cool the filtrate and analyze.

Fabric Samples (According to ISO 14184-1)
Cut specimens into small pieces, and weigh approximately 1 g of the pieces to an accuracy of 10 mg. If the formaldehyde content is low, increase the test specimen weight to 2.5 g in order to achieve a sufficient accuracy. Put the weighed pieces into a 250 ml flask with a stopper and add 100 ml of water. Cap tightly and place in a water bath at 40 °C for 60 min. Shake the flask at least every 5 min. Filter the solution into another flask through a glass filter and analyze.

Resins and Other Samples
Extract samples with water by shaking for 60 min at 40 °C. Choose sample/extraction solution ratio based on expected Formaldehyde content. Use additional dilutions as necessary.

Post-Column Conditions

Post-Column System: Onyx PCX, Pinnacle PCX or Vector PCX

Reactor Volume: 0.5 mL

Reactor Temperature: 100 °C

Reagent: 0.81 M Ammonium Acetate
0.12 M Glacial Acetic Acid
0.05 M 2,4-Pentanedione

Reagent Flow Rate: 0.4 mL/min

Detection: FLD, λ_{EX} : 412 nm, λ_{EM} : 510 nm or UV/Vis 410 nm

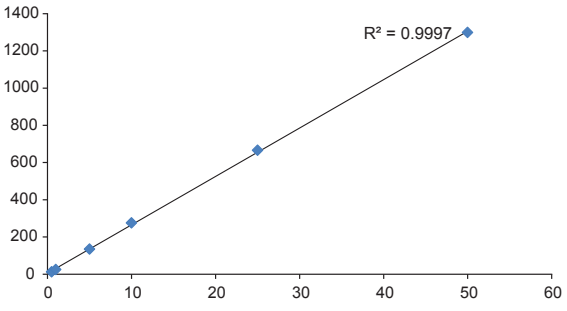

Fig 1. *Calibration curve for Formaldehyde (0.5 – 50 ppm)*

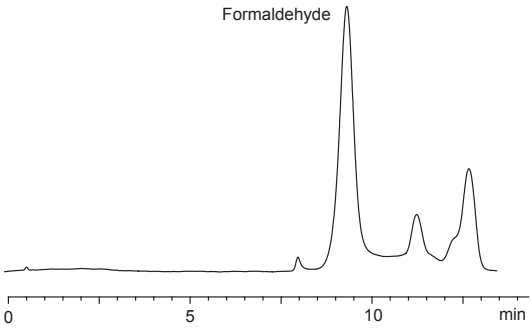

Fig 2. *Chromatogram of phenolic resin sample containing 0.02% of Formaldehyde*

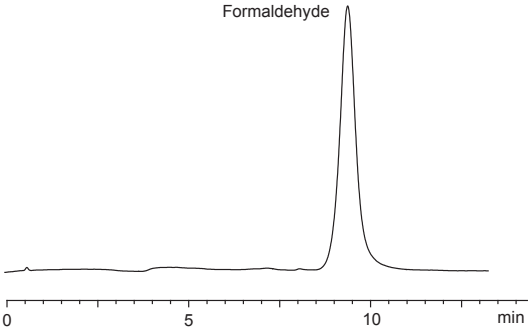

Fig 3. *Chromatogram of phenolic resin containing 0.1% of Formaldehyde*

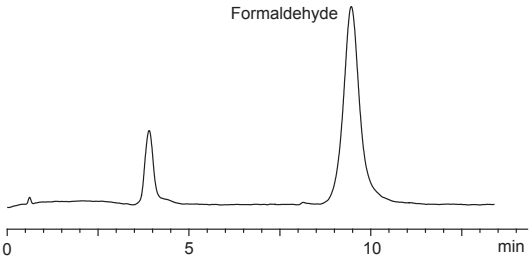

Fig 4. *Chromatogram of shampoo sample spiked with 0.01% of Formaldehyde*

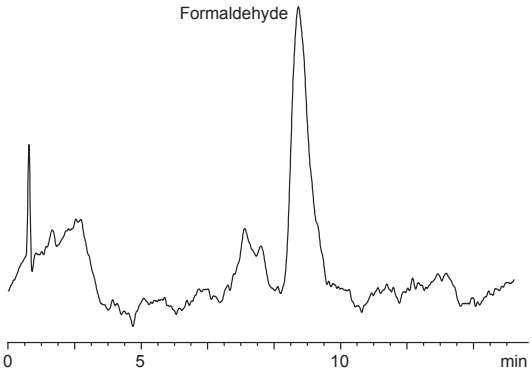

Fig 5. *Chromatogram of leather sample containing 5 ppm of Formaldehyde*

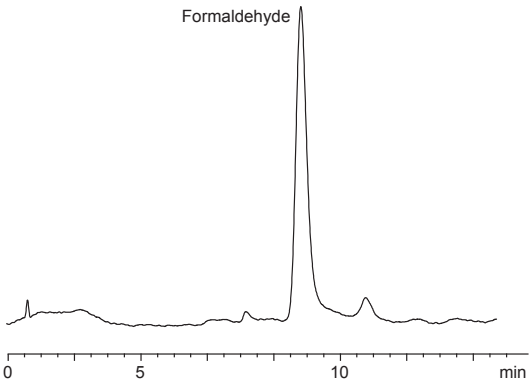

Fig 6. *Chromatogram of fabric sample containing 160 ppm of Formaldehyde*