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Technical Bulletin

April, 2022

ANALYSIS OF 3M™ FORMALDEHYDE MONITOR 3721+/3720+ by HPLC

STANDARD PREPARATION

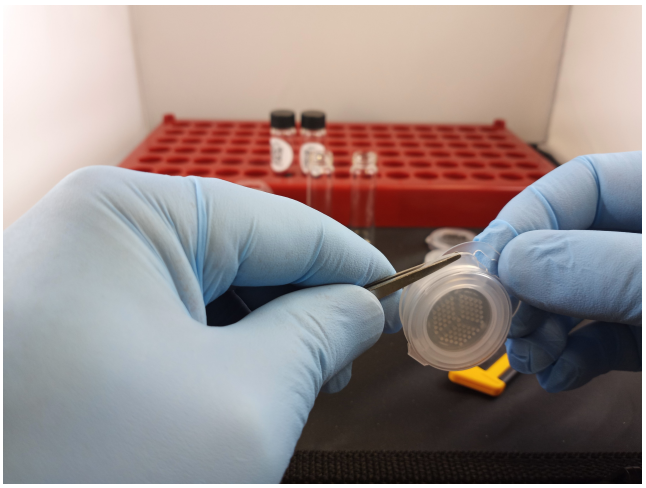
Accurately weigh the 2,4-dinitrophenylhydrazone (2,4-DNP) derivative of the aldehyde to be analyzed into a measured volume of HPLC grade acetonitrile to make a Stock Standard Solution equiv. to ca. 1.0 mg of derivative per ml in acetonitrile. Store the Stock Standard under refrigeration and make fresh monthly. Dilute Stock Standard Solution in acetonitrile weekly to make 3-5 Working Standards in the range of interest. Store Working Standards in a closed container under refrigeration when not in use. Prior to injection, dilute solution 1:1 with mobile phase (0.05 M KOAc buffer, pH5), and filter using a 0.45 ?m filter. We recommend using a Syringeless Filter Vial with 0.45 µm PVDF filter.

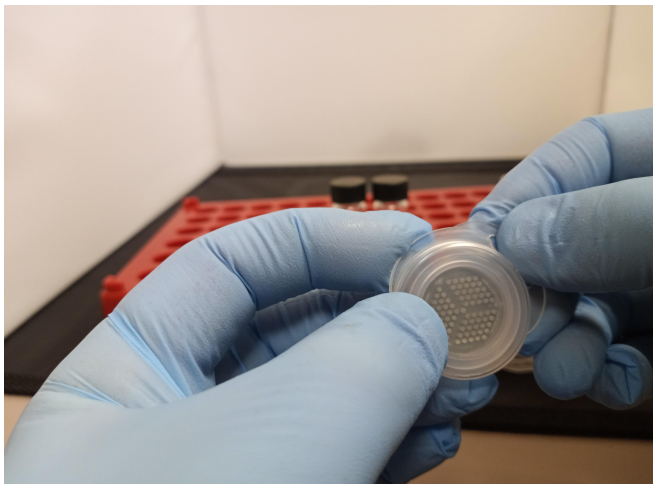
DESORPTION SOLVENTS AND DESORPTION EFFICIENCY

The IH Sampling Guide includes a list of monitors, analytes, suggested desorption solvent and reported desorption efficiency (DE). Acetonitrile is often the suggested desorption solvent for aldehydes. Other extraction solvents can be found in the NIOSH Manual of Analytical Methods or at the OSHA website. Labs who desire to use a different extraction solvent would need to determine their own DE values for each analyte on the appropriate monitor. A method for determining the DE is available (R.A. Dommer & R.G Melcher, (1978) AIHA Journal, 39:3, 240-246). To obtain an accurate value, it is good practice for individual labs to determine the DE at several (3-5) concentrations (µg of analyte per gram of media) that bracket the range of interest, and to determine the value at each concentration in triplicate. Some laboratories choose to re-determine DE values received from a 3rd party or to re-determine their own values from time-to-time. It has been shown that DE determinations may be made using multiple (at least 5) analytes in a single solution provided the analysis method used is capable of analyzing each analyte separately.

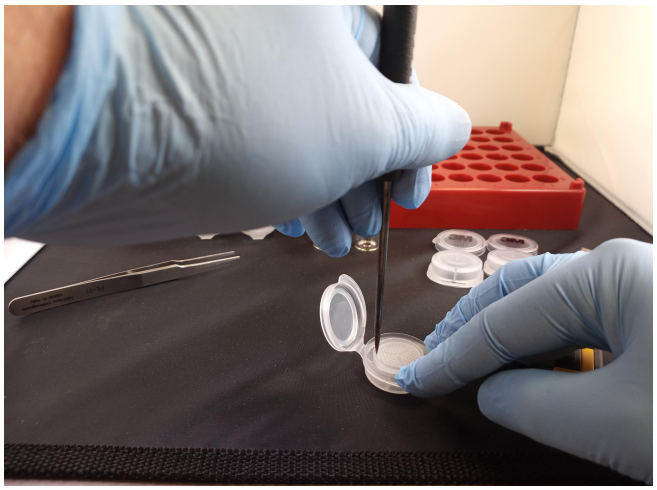
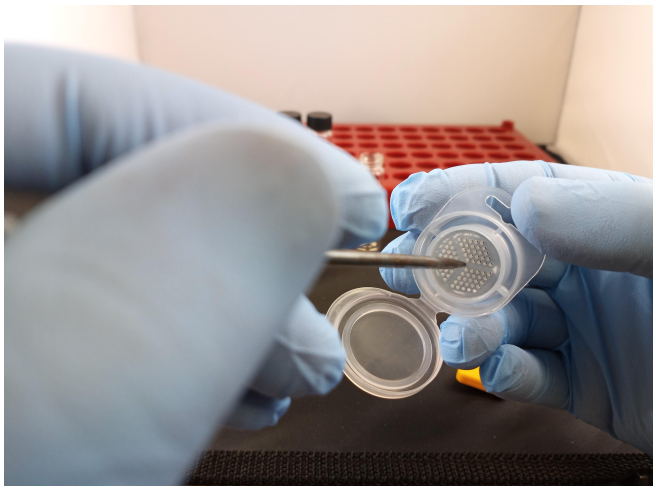
SAMPLE PREPARATION

Remove each Monitor to be tested from the Return Pouch. To open the face of the Monitor either use something flat as a wedge to open it, or just open with your fingers.





Place Monitor on a hard solid surface. Do Not hold the badge in the palm of your hand.

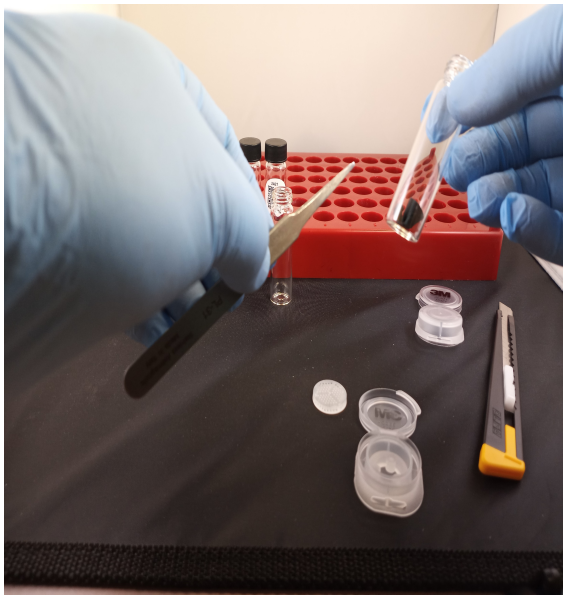
	
YES	**NO**

Using a lab pick (e.g., McMaster-Carr 3842A42) as a tool, remove the plastic Sampling Grid from the clear plastic Sampler Body exposing the yellow glass fiber wafer beneath. Place the tip of the pick either at the side or the middle. Push down hard and pry out the sampling grid.

If you do not have a pick, use a small sharp blade to make 2 incisions within 25 degrees for each other. Then pry the sampling grid out.

Using clean forceps, place the glass fiber wafer in an inert filtration vial (e.g., Syringeless Filter Vial, 0.45?m PVDF, Whatman Item No UN513VAQU).



Accurately pipet 1.0 ml of acetonitrile into the filtration vial. Agitate the vial carefully for one minute. Then add 1.0 ml of 0.05 M. KOAc Buffer pH5, cap the filtration vial and agitate again. Push the plunger to filter the solution. Reserve solution for analysis.

BLANKS

Most laboratory tests involve the analysis of an associated background blank, consisting of reagents or media that have not been exposed to the analyte tested. When typical blank values are detectable (attributed to analyzable substances acquired extrinsically), a blank test result is subtracted from the value obtained from each test to obtain a “blank corrected value”. It is good practice to initially analyze several monitors to determine whether blank values are detectable or significant relative to the laboratory’s reporting limit. If blank values are detectable and significant, it is good practice to measure and retain an average blank value for each manufactured lot of monitors analyzed and subtract that blank value from each measured sample. In some cases, an average blank value may be obtained from the media manufacturer.

At least once per analytical batch, remove a similar Monitor which has not been exposed. Remove the yellow filter disc from an unexposed Monitor and process this "BLANK" in exactly the same fashion as the Sample Preparation (above).

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC) ANALYSIS

Inject an aliquot of the Sample Preparation from each Monitor to be analyzed into a HPLC System using the following conditions.

HPLC Column	Restek Pinnacle RP II C18 150 x 3.2 mm, 5 µm, 110Å
Mobile Phase (a)	50% acetonitrile / 50% (0.05 M KOAc Buffer pH5)
Column Temp	40°C
Flow Rate	0.45 ml per min (nominal)
Injection Volume	10 microliter (nominal)
Detector Wavelength	355 nm

(a) Optimal mobile phase for formaldehyde. For higher aldehydes, increase % acetonitrile or use gradient.

Concomitantly, inject measured aliquots of a BLANK Preparation and three Standard Preparations in the range of interest (i.e. which bracket the concentrations of the Sample Preparations)

CALCULATION

Acquire the analytical data into a computer system in which chromatography data handling software has been installed. Using the software, compare the peak area ratio for analyte *vs* internal standard normalized for concentration from each Sample Preparation to the best-fit Calibration Curve obtained from BLANK and Standard Preparations and compute the Analyte Concentration in the Sample Preparation.

Calculate Exposure Level from Analyte Concentration as follows:

$$\text{EXPOSURE LEVEL } (ppm) = \frac{1000(C)(V)(F)(R)}{(M)(SR)(T)}$$

Where

C = Analyte Concentration Found (*µg/ml* Formaldehyde DNP Derivative)

V = Volume of Extraction Fluid (2.0 ml)

F = Mass Ratio: (HCHO/HCHO-DNP = 0.143)

R = Molar Volume @ 25°C (24.45 *l/mole*)

M = Molecular Wt of Aldehyde Derivative (b)

SR = Monitor Sampling *Rate* (*ml/min*)

T = Sampling Time (*min*)

(b) E.g., Mol Wt for HCHO-DNP is 210.1 g/mole

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