



Formaldehyde Sampler Analysis Using the Chromotropic Acid Assay Method

This method is a variation of the original NIOSH Method 3500 for analyzing passive samplers for formaldehyde (SKC Catalog Nos. 526-100, 526-200, and 526-201). *Samples should be analyzed within 30 days of exposure.*

MATERIALS NEEDED:

Spectrophotometer	(Capable of holding 13 x 100 mm glass tubes and reading absorbance at 580 nm.)
Centrifuge	(Capable of 1500 G to spin 13 x 100 mm tubes)
Hot Water Bath	(Capable of 95 degrees C)
Oven	(Capable of drying glassware at 110 degrees C)
Vortex Mixer	
Pipets	(Various 0.5 - 10.0 ml)
Volumetric Flasks	(Various)
Tube Racks	
Test Tubes (centrifuge tubes)	(13 x 100 mm glass - Fisher)
Polyethylene Film	
PE Caps for Tubes	(KimKaps)

CHEMICALS NEEDED:

Sulfuric Acid, Concentrated	(Mallinkrodt AR)
Sodium Bisulfite, Granular	(Baker Analyzed)
Chromotropic Acid, Disodium Salt, Dihydrate	(Baker Analyzed)
Formaldehyde Standard, 4000 mg/L	(Hach Voluette)
Formaldehyde-Free Deionized Water	
Hot Tap Water (55 degrees C minimum)	

PROCEDURE:

It is imperative to avoid sources of formaldehyde, general aldehyde, alcohol and organic contaminations and that the method follows all good analytical lab practices. Although the chromotropic acid procedure is generally free from many interferences, strict attention to detail is mandatory if good results are to be obtained. *Analyze samples within 30 days of exposure.*

ANALYTICAL CURVE:

Preclean all glassware to be used in the analysis by rinsing 2x with hot tap water followed by 2x with DI water. Dry the glassware in the oven for 1 hour and keep covered with plastic until ready to use. The glassware is good for one day.

Make 100.0 ml of a 2.5% (w/v) stock sodium bisulfite solution with DI water. Make fresh daily.

Make a 400.0 µg/ml stock solution of formaldehyde solution using the Hach standard with DI water. This solution is good for one month at 4 degrees C.

Make 10.0 ml of an 8.8% (w/v) solution of chromotropic acid solution with DI water and store protected from the light. Make fresh daily.

Make a series of working formaldehyde standards as follows. Take 10 ml of the 400 µg/ml stock solution and dilute to 100 ml in DI water. Use this standard to make calibration standards according to the following table:

Flask size*	2.5% Bisulfite stock soln.	40 µg/ml formaldehyde std	Formaldehyde (µg/ml)
50 ml	5 ml	0	Blank
200 ml	20 ml	0.5 ml	0.1 µg
100 ml	10 ml	0.5 ml	0.2 µg
100 ml	10 ml	1.0 ml	0.4 µg
50 ml	5 ml	1.0 ml	0.8 µg
50 ml	5 ml	2.5 ml	2.0 µg
50 ml	5 ml	4.0 ml	3.2 µg
50 ml	5 ml	5.0 ml	4.0 µg

* make up to the mark with DI water

In duplicate, pipet 2.00 ml of each blank/standard into a 13 x 100 mm test tube (centrifuge tube).

Add 0.10 ml of chromotropic acid to each tube.

Add 3.00 ml of sulfuric acid slowly and with care, using appropriate protective measures, to each tube, cap and vortex for 5 seconds.

Heat these solutions in the water bath for 15 minutes. Make sure that the liquid level of the bath exceeds the level in the test tubes.

Cool the standards for 30 minutes in air or more quickly in a cold water bath, and vortex for 5 seconds. Read the values at 580 nm in the spectrophotometer. Graph concentration in µg/ml against absorbance. The slope should be between 0.24 and 0.28, the intercept 0.000 ± 0.010 , and the regression coefficient should be better than 0.995.

SAMPLES:

Samples from Formaldehyde Sampler Catalog No. 526-100 are color-developed inside the 526-100 vial, but the 526-200 and 526-201 samples require the adsorption disc to be removed from the sampler and placed inside a 13 x 100 mm test tube for color development.

Formaldehyde Sampler, Catalog No. 526-100

Add 2.00 ml of DI water to each sample, vortex for 5 seconds, cover with plastic and let sit for 30 minutes.

Add 0.10 ml of chromotropic acid.

Add 3.00 ml of sulfuric acid slowly and with care, using appropriate protective measures, vortex for 5 seconds, cap with vented caps and place in the hot water bath for 15 minutes.

Cool the samples for 30 minutes in air or more quickly in a cold water bath, and vortex for 5 seconds and decant into clean 13 x 100 mm test tubes (centrifuge tubes).

Centrifuge decanted solutions for 10 minutes and read values at 580 nm.

Formaldehyde Samplers, Catalog No. 526-200 (STEL) and 526-201 (PEL)

Cut the samplers open to expose the filter discs.

Using clean tweezers extract the absorbent disc and the 2 polypropylene discs on either side of the absorbent disc.

Place the 3 discs at the bottom of a 13 x 100 mm test tube.

Add 2.00 ml of DI water, vortex 5 seconds, cover with plastic and let sit for 30 minutes.

Add 0.10 ml of chromotropic acid.

Add 3.00 ml of sulfuric acid slowly and with care, using appropriate protective measures, vortex for 5 seconds, cap with vented caps and place in the hot water bath for 15 minutes.

Cool the samples for 30 minutes in air or more quickly in a cold water bath and vortex for 5 seconds and decant into clean 13 x 100 mm test tubes (centrifuge tubes).

Centrifuge the decanted solutions for 10 minutes and read values at 580 nm.

CALCULATIONS:

Calculate the μg and ppm formaldehyde with the values given to you by SKC according to the following calculations:

$\mu\text{g} = 2 (\text{A580} - \text{Intercept}) / \text{Slope}$ (Note: The calibration curve is in terms of concentration ($\mu\text{g}/\text{ml}$), but 2 ml of each standard is used.)

$\text{ppm} = (\text{Sample } \mu\text{g} - \text{Blank } \mu\text{g}) / (\text{Diffusion Constant}) (\text{Exposure Hr})$

The diffusion constant (uptake rate) of the 526-100 is 0.310 $\mu\text{g}/\text{ppm}/\text{hr}$.

The diffusion constants (uptake rates) of the 526-200 and 526-201 are individually calibrated and the information is printed on the package. If this is lost, contact SKC Customer Service at 724-941-9701 with the serial number (not the lot number) of the sampler.

Also see www.skcinc.com for Parameters for Laboratory Analysis (diffusion constants).

Formaldehyde Samplers manufactured for SKC by Air Quality Research Inc.

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