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Chemosorption sampling and analysis of formaldehyde in air. Influence on recovery during the simultaneous sampling of formaldehyde, phenol, furfural and furfuryl alcohol.

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Chemosorption sampling and analysis of formaldehyde in air

Influence on recovery during the simultaneous sampling of formaldehyde, phenol, furfural and furfuryl alcohol

by Kurt Andersson, PhD, Christer Hallgren, BSc, Jan-Olof Levin, PhD, Carl-Axel Nilsson, PhD¹

ANDERSSON K, HALLGREN C, LEVIN J-O, NILSSON C-A. Chemosorption sampling and analysis of formaldehyde in air: Influence on recovery during the simultaneous sampling of formaldehyde, phenol, furfural and furfuryl alcohol. *Scand j work environ health* 7 (1981) 282—289. A method based on trapping formaldehyde on a 2,4-dinitrophenylhydrazine-coated porous polymer (Amberlite XAD-2) was evaluated for air sampling in occupational environments. The aldehyde is converted to its 2,4-dinitrophenylhydrazone on the adsorbent. The influence of some organic compounds which often occur together with formaldehyde — furfural, phenol and furfuryl alcohol — was studied. The results show that the method allows the sampling of formaldehyde in the range 0.1—10 mg/m³ of air, based on a 3-l (15 min) sample and a coating of 1 %. Furfural, phenol, and furfuryl alcohol do not interfere and may be conveniently sampled at the same time. Formaldehyde and furfural hydrazones were analyzed by high-performance liquid chromatography, phenol and furfuryl alcohol by gas chromatography.

Key terms: Amberlite XAD-2, 2,4-dinitrophenylhydrazones, gas chromatography, high-performance liquid chromatography.

Formaldehyde is extensively used in industry for various types of applications, for instance, in the production and use of phenol-formaldehyde resins and related products. These resins are used in foundries as binding agents for sand, and in the wood industry, where they are of the utmost importance as adhesives.

The irritating and allergenic action of formaldehyde has been known for a long time (21), and a very low occupational threshold limit value is recommended in most countries. The air level of formaldehyde must therefore be determined if the potential occupational health hazards involved are to be evaluated.

In most cases, formaldehyde in workplace air is associated with a variety of other organics, very often in high concentrations. Typically, phenol, resorcinol, furfural, or furfuryl alcohol occurs with formaldehyde in adhesive resins and similar products. Some of these accompanying organics are also associated with health hazards, and the levels of the substances are thus statutorily regulated. Accordingly, these compounds are very often sampled in parallel with formaldehyde. Therefore a combined sampling method for all these compounds would be very useful. Such a method should be specific and not influenced by other compounds in the sample. Furthermore, the compounds to be sampled should not cause interference.

The overall sensitivity of the method should preferably be in the range of one-tenth to five times the threshold limit value, which for most countries is in the range of 1—2 ppm. The sampling method

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should also permit easy personal sampling in the breathing zone, and therefore a solid adsorbent sampling tube is the most suitable.

To date, the method most frequently used for the sampling of volatile aldehydes involves the use of liquid absorbing solutions with bisulfite or 2,4-dinitrophenylhydrazine (DNPH) in impinger flasks (5, 7, 12, 17, 20, 22, 28).

Solid adsorbents already tested for the sampling of volatile aldehydes are alumina (31), activated charcoal (10), Tenax (11), and molecular sieves (32). In these cases the analysis has been performed by gas chromatography (GC) or gas chromatography/mass spectrometry (GC-MS). However, the relatively low sensitivity of the underivatized aldehydes requires large sampling volumes (11).

For analysis, the former optical (17, 22) and titrimetric methods have been replaced by GC and high-performance liquid chromatography (HPLC). In order to obtain higher sensitivity, the formation of hydrazones, which can be used both for GC and HPLC analysis, has been utilized. The most frequently used are the 2,4-dinitrophenylhydrazones (1, 4, 5, 7, 9, 12, 13, 16, 19, 20, 23, 24, 26, 28, 29, 30), but pentafluorophenylhydrazones (8, 15) and, for HPLC, the dansylhydrazones (6, 12) have also been demonstrated to be useful.

Since a sampling procedure involving an impinger creates practical problems, we earlier demonstrated the use of DNPH-coated Amberlite XAD-2 in solid sorbent sampling tubes for the analysis of formaldehyde in air (1).

Recently the same principle has been used by Beasley et al (2). They used DNPH-coated silica for the sampling of formaldehyde. That method shows however no advantage over the method described by us (1). In contrast our method has the advantage of permitting the simultaneous sampling of other compounds because of the adsorbing properties of XAD-2.

A method involving the sampling of formaldehyde on an oxidizing adsorbent was recently reported. It involves ion chromatographic analysis of the formate ion formed (14), and this procedure constitutes a severe limitation since ion chromatography is not as widespread an analytical method as HPLC.

In the present communication we wish to report the recovery of formaldehyde sampled from air on DNPH-coated XAD-2 in chemisorption tubes. The study includes different air levels and also the simultaneous sampling and analysis of furfural. We also demonstrate that phenol and furfuryl alcohol can be sampled at the same time. For these compounds, the previously shown adsorption properties of XAD-2 (18) have been utilized since their recovery is not affected by the DNPH coating.

Furthermore, a comparative study was performed in order to demonstrate the higher sensitivity of the chemisorption method in comparison with the widely used sampling method involving a bisulfite solution combined with spectrophotometric analysis (22).

The older method has a practical detection limit of 0.5 mg/m³ for formaldehyde (during 15-min sampling for establishing occupational exposure), while the chemisorption method is used down to 0.1 mg/m³.

Materials and methods

Chemicals

The solvents and reagents used were as follows: methanol [May & Baker, pro-lysi (pa)], diethyl ether (May & Baker, pa), and ethanol (spectrographic grade) and formaldehyde (Fisher, 37.4 % in water), furfural (EGA-chemie, 99 %, redistilled), furfuryl alcohol (Fluka, purum, redistilled), and phenol (Poly Science Corp).

DNPH (Fluka) was recrystallized twice from 4 M hydrochloric acid (HCl) as its hydrochloride.

Amberlite XAD-2 (Rohm & Haas) was purified by repeated washings with distilled water. Fines were removed by decanting. The polymer was further washed with methanol (five times) and extracted twice in a Soxhlet apparatus for 12 h with diethyl ether. Finally, the adsorbent was fractionated to produce a more uniform size distribution (20–50 mesh).

The chemisorbent was prepared as follows: 100 mg of DNPH hydrochloride was mixed with 0.5 ml of concentrated HCl, and 30 ml of ethanol (EtOH) was added. This solution was added to 10 g of purified XAD-2, which was covered with 20 ml of

Table 1. Recovery of formaldehyde. Chemosorption tube with 1 % coating of 2,4-dinitrophenylhydrazine on XAD-2.

Amount added (μg)	Recovery (%)	Relative standard deviation (%)	Number
0.4	84	7	16
4	88	5	14
20	83	4	7
64	70	4	6

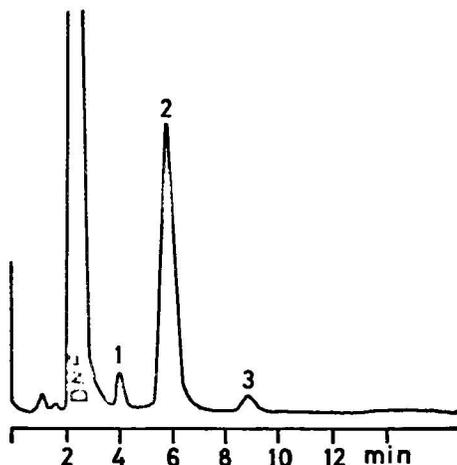


Fig 1. Chromatogram (high-performance liquid chromatography) of a chemisorbent tube blank. (1 = formaldehyde, 2 = acetaldehyde, 3 = acetone hydrazones)

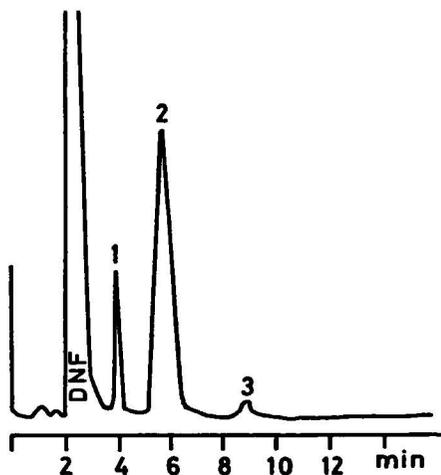


Fig 2. Chromatogram (high-performance liquid chromatography) of a formaldehyde sample. The level of formaldehyde corresponds to an air level of 0.13 mg/m^3 . (1 = formaldehyde, 2 = acetaldehyde, 3 = acetone hydrazones)

EtOH. The solvent was then removed in a rotary evaporator.

The chemisorbent tubes were 6×50 -mm (inner diameter 4 mm) glass tubes shaped like ordinary charcoal sampling tubes, and thus suitable for use with ordinary personal sampling equipment. The tubes were washed twice in methanol in an ultrasonic bath before use and dried at 200°C . One hundred and fifty milligrams of the chemisorbent was placed in the tubes, which were stoppered at both ends by silanized glasswool plugs and sealed with the same type of plastic plugs that are used in commercial charcoal tubes.

The 2,4-dinitrophenylhydrazone of formaldehyde was prepared according to Shriner et al (27) and that of furfural according to Braddock et al (3). The hydrazones were purified by recrystallization from ethanol. The two isomers (syn- and anti-) of furfuraldinitrophenylhydrazone, formed in a ratio of approximately 2:1, were used together for the recovery studies since they are formed in the same ratio during chemisorption. Standard solutions were prepared in diethyl ether.

Recovery studies

For the recovery experiments, a 6×150 -mm glass tube with a glasswool plug at the inlet end was connected in front of the chemisorption tube, and air was pumped through with a personal sampling pump at 200 ml/min . The compound to be tested was then injected into the glass tube ($10 \mu\text{l}$ of a solution with an appropriate liquid concentration for the level concerned), and 3 l of air was pumped through the tubes. During the last minute of the passage of air, the glass tubes were heated by hot air to approximately 80°C to ensure complete volatilization of the compound. This procedure ensures that a known amount of compound is sampled in a known volume of air.

For the recovery studies with the bisulfite impinger method, the same principal equipment was used except that the chemisorption tube was replaced by an impinger containing 15 ml of an aqueous 1 % sodium bisulfite solution, and a pumping rate of 1 l/min was used (22).

The chemisorbent was then placed in a

10-ml vial and extracted by shaking for 30 min with 3 ml of ether.

The aldehyde analyses were performed by HPLC, and GC was used for phenol and furfuryl alcohol. The experimental conditions are presented in the section Instruments.

The bisulfite solution was reacted with chromotropic acid, and the analysis was performed by visible spectroscopy (22).

Instruments

Gas chromatography. A Pye Unicam 204 gas chromatograph equipped with a flame ionization detector was used for the gas chromatography. The glass column (1.5 m \times 4 mm inner diameter) was filled with 10 % Reoplex 400 on Chromosorb WHP, 100–120 mesh. The column temperatures were 170°C for phenol, 150°C for furfuryl alcohol, and 120°C for furfural, which gives a retention time of approximately 5 min. The detector and injector temperature was 200°C. Recoveries were determined with the external standard method.

High-performance liquid chromatography. A Waters HPLC instrument, consisting of a WISP 710A autoinjector, two 6000A solvent delivery systems, a 660 solvent programmer, a 440 absorbance detector, and a data module, was used for the HPLC. The instrument was further equipped with a radial compression separation system with a C₁₈ reversed phase column (8 \times 100-mm, 10 μ) operating at a flow of 2 ml/min [pressure approximately 1,000 psi (6,895,000 Pa)] and methanol:water (75:25) as the solvent. The detector was operated at 365 nm. The meth-

anol (May & Baker, pa) was used without further purification. The water was purified in a Millipore Milli-R/Q water purifier. The injection volume was 25 μ l. The solvents were degassed by bubbling a slow stream of helium through them.

Results and discussion

The recovery of formaldehyde after sampling in the chemisorption tubes is shown in table 1. Levels ranging from 0.4 to 64 μ g were tested. With a sampling volume of 3 l, this range corresponds to an air level of 0.13 to 21 mg/m³ of formaldehyde, and the recovery is approximately 85 %. The liquid chromatographic chromatograms of a reagent blank and a sample of 0.4 μ g of formaldehyde are shown in fig 1 and 2, respectively. Some acetaldehyde and acetone hydrazones were also present in the blank. In the reagent blank the level of formaldehyde hydrazone corresponded to an air level of 0.03–0.1 mg/m³ of formaldehyde. However, since the standard deviation of the blank level within one batch of chemosorbent was low (table 2), the detection limit was far below the practical quantitative range for formaldehyde, which we have set at 0.1–6.5 mg/m³ based on a 3-l sample of air. This range corresponds to the levels 0.4 and 20 μ g tested (table 1). As can be seen, the recovery decreased to approximately 70 % at higher levels. The large peak of DNPH can be reduced by the removal of DNPH with an ion-exchange column prior to analysis (12, 25). Johnson et al (12) have reported that the use of capillary GC in the analysis of hydrazones requires a complete removal of DNPH to avoid de-

Table 2. Recovery of formaldehyde at a low level (0.4 μ g) and formaldehyde hydrazone in reagent blanks from the same batch of chemosorbent.

Formaldehyde added (μ g)	Actual concentration of 2,4-dinitrophenylhydrazine derivate in desorbed sample ^a (μ g/ml)	Relative standard deviation (%)	Number
0	0.36	7.0	6
0.4 ^b	1.14	5.6	17

^a Determined with external standard.

^b Corresponds to an air level of 0.13 mg/m³.

Table 3. Recovery of formaldehyde and phenol during simultaneous sampling. Chemosorption tube with 1 % coating of 2,4-dinitrophenylhydrazine on XAD-2.

Formaldehyde			Phenol			Number
Amount added (μg)	Recovery (%)	Relative standard deviation (%)	Amount added (μg)	Recovery (%)	Relative standard deviation (%)	
0.4	97	7	6	103	12	6
0.4	104	7	12	86	15	6
0.4	97	4	60	97	3	6
4	104	4	6	100	8	6
4	99	3	12	89	4	6
4	95	5	60	100	3	6
20	78	1	6	88	11	6
20	77	3	12	90	6	6
20	76	1	60	108	3	6

Table 4. Influence of furfural, phenol and furfuryl alcohol during the sampling of formaldehyde. Chemosorption tube with 1 % coating of 2-4-dinitrophenylhydrazine on XAD-2.

Formaldehyde			Furfural			Phenol			Furfuryl alcohol			Number
Amount added (μg)	Recovery (%)	Relative standard deviation (%)	Amount added (μg)	Recovery (%)	Relative standard deviation (%)	Amount added (μg)	Recovery (%)	Relative standard deviation (%)	Amount added (μg)	Recovery (%)	Relative standard deviation (%)	
0.8	88	6	6	83	9	—	—	—	—	—	—	6
0.8	82	5	30	81	4	—	—	—	—	—	—	6
0.8	86	9	60	97	4	12	NA ^a	—	60	NA	—	6
2	97	2	60	75	8	12	NA	—	60	NA	—	6
4	79	7	6	66	10	—	—	—	—	—	—	6
4	80	5	30	68	8	12	93	3	60	99	3	6
4	90	7	30	69	4	12	NA	—	60	NA	—	4

^a NA = not analyzed.

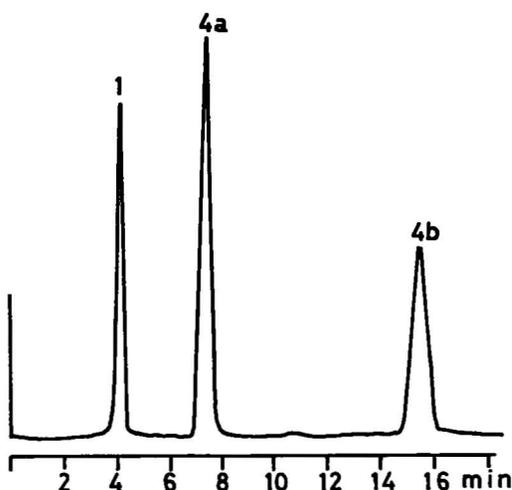


Fig 3. Chromatogram (high-performance liquid chromatography) of a standard solution of formaldehyde hydrazone (1) and the two isomers of furfural hydrazone (4a & 4b).

terioration of the column. This, however, is not necessary in the liquid chromatographic analysis.

The relatively large peak of acetaldehyde hydrazone in the blank, which corresponds to approximately 2 mg/m^3 , did not disturb the formaldehyde analysis.

Since phenol is frequently used with formaldehyde, the simultaneous sampling of phenol and formaldehyde was tested. The results showed that neither formaldehyde nor phenol recoveries are affected by the cocontaminant (table 3). The phenol recoveries were in the same range as for XAD-2 not coated with DNP, the values for which we reported in a previous paper (18). Therefore the previously used methods for sampling formaldehyde and phenol, including two different impingers, are now reduced to sampling with one solid sorbent tube.

Other common compounds used together with formaldehyde are furfural and fur-

furyl alcohol, and the influence of these compounds and phenol on formaldehyde recovery is reported in tables 4 and 5. Furfural reacts with DNPH to form the corresponding hydrazone. However, since the substrate is not symmetrical, two isomers (syn- and anti-) are formed (3). This formation is demonstrated in fig 3 for a standard solution of formaldehyde and the furfural hydrazones. The ratio between the two isomers remained relatively constant.

The recoveries were calculated from the sum of the areas for the two isomers on the assumption that there is little difference in the response between the two isomers. Phenol and furfuryl alcohol were analyzed by GC.

The recovery of formaldehyde was not affected by furfural, phenol, and furfuryl alcohol at the levels tested (table 4). However, at a level of formaldehyde corresponding to the threshold limit value, the recovery of furfural decreased to approximately 70 %. To determine whether this problem could be overcome by using a higher percentage of coating, we prepared a chemisorbent with a 2 % coating. The recoveries were tested for high levels of formaldehyde and furfural (table 5).

The results showed a significant in-

crease in the recovery of furfural, but the formaldehyde recovery decreased. For this reason it is recommended that a sampling tube containing uncoated XAD-2 be placed in front of the chemisorbent tube when a determination of furfural is required at the same time as that of formaldehyde. This XAD-2 tube will completely retain the furfural, phenol and furfuryl alcohol (18), which can then be desorbed and analyzed by GC. Formaldehyde, however, will pass through this tube and be sampled on the chemisorbent. When 4 μg of formaldehyde and 60 μg of furfural were added and sampled in this manner (together with 12 μg of phenol and 60 μg of furfuryl alcohol), the recoveries were 86 and 90 %, respectively, with a relative standard deviation of 5 % calculated for six samples. If the determination of formaldehyde is not required, phenol, furfural, and furfuryl alcohol can be sampled on XAD-2 and analyzed by GC as previously reported (18).

The influence of storage on the formaldehyde level was also studied. The results showed that there is no decrease in the formaldehyde level during 12 d of storage at room temperature in the dark. When the tubes were exposed to light, the formaldehyde recovery decreased to approximately 50 % in one week (table 6).

Table 5. Influence of furfural during sampling of formaldehyde. Chemosorption tube with 2 % coating of 2,4-dinitrophenylhydrazine on XAD-2.

Formaldehyde		Furfural		Number
Amount added (μg)	Recovery (%)	Amount added (μg)	Recovery (%)	
4	68	60	99	2
4	67	120	86	2
20	76	60	93	2
20	60	120	75 ^a	2

^a Another 7 % was found as unreacted furfural when the solution was analyzed by gas chromatography.

Table 6. Stability of formaldehyde samples during storage.

Amount of formaldehyde added (μg)	Recovery (%)	Relative standard deviation (%)	Number	Conditions
4	92	3	6	20°C, dark, 12 d
4	47	6	4	20°C, light, 7 d

Table 7. Comparison of the chemisorption and bisulfite methods for the sampling and analysis of formaldehyde.

Amount of formaldehyde given as fraction of the threshold limit value	Chemosorption method				Bisulfite impinger method			
	Amount of formaldehyde added ^a (μg)	Recovery (%)	Relative standard deviation (%)	Number	Amount of formaldehyde added ^a (μg)	Recovery (%)	Relative standard deviation (%)	Number
0.1	0.4	87	1	6	—	NA ^b	—	—
0.5	2	97	2	6	10	98	5	6
1	4	90	4	6	20	99	4	6
10	40	70	4	6	200	60	23	6

^a The study is based on a 3-l sampling volume for the chemisorption method and a 15-l sampling volume for the impinger method.

^b NA = not analyzed.

The unused chemisorption tubes are stable for several months at room temperature.

The only significant drawback of the method is that large amounts of other ketones or aldehydes can disturb the sampling, but most other methods also suffer from this drawback. The reagent can thus be consumed by the other carbonyl compounds, the consumption resulting in too low recoveries and poor analytical results.

There were differences in recoveries between different batches of chemisorbent, but, since the relative standard deviation was small, the precision of the method was good. Therefore the recovery should preferably be determined for each batch of sorbent tubes prepared. We have used the method within a quantification range of 0.1–6.5 mg of formaldehyde/m³ of air based on a 3-l sample using a 1% DNPH coating on XAD-2.

Table 7 shows the results of a comparative study of the bisulfite impinger method and the chemisorption method. The recoveries obtained by the two methods agreed. However, the bisulfite method has a less specific and less sensitive analytical step; besides, for personal sampling, it suffers from the usual inconvenience of impinger sampling. Therefore the sensitive and specific chemisorption method must be considered superior, since it also permits the simultaneous sampling of phenol, furfural, and furfuryl alcohol.

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