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Performance Evaluation of Real Time Formaldehyde Monitors: PTR-MS and Interscan 4160-500B Portable Monitor

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Executive Summary

Exposure to volatile organic compounds (VOCs) in buildings is a health hazard. Of particular concern is formaldehyde, a ubiquitous carcinogen emitted from furnishings and adhesives in homes and offices. Practitioners and researchers in the area of building performance are very interested in measuring formaldehyde in homes, and they need instrumentation that responds immediately. Current formaldehyde monitoring techniques are hampered by interfering substances in the sample airstream, compromising measurement accuracy and leading to drift. Many experts are now using a tabletop real-time formaldehyde instrument, the Interscan 4160-2, that LBNL researchers have found to be very sensitive to water vapor and low levels of volatile organic compounds (VOCs). Unless ways are found to remove these interferences, relying on the Interscan's readings in buildings will lead to the wrong conclusions about formaldehyde levels and could trigger subsequent unnecessary expense and/or inappropriate responses.

This report describes a solution to the challenge of monitoring formaldehyde levels accurately in real-time, LBNL's new VOC True Read device that is now ready for commercialization. Its innovative trap design strips water vapor, alcohols, and other VOCs from the sample, allowing formaldehyde — or other target gases — to pass through to a detector. The filter presents an economical answer to a long-sought method for sensitive formaldehyde measurement because it can be attached to, and can retrofit, in-place sensors to increase their detection accuracy. The filter's internal structure can be arrayed various ways to adsorb other interferents, as needed. The device answers a widely recognized need for accurate, long-term, inexpensive, real-time monitoring of building air for formaldehyde to assist exposure assessment and demand-controlled ventilation. It can be added to real-time or passive monitors for formaldehyde, such as electrochemical sensors in handheld devices or table-top monitors with sensors that respond to oxygenated VOCs as well as formaldehyde.

[*]Colorimetric sensor developed by NTT Energy and Environment Systems Lab. in Japan; marketed by Shinyei and RKI based on Maruo et. al., 2008, 2010; [#]Environmental Sensors Model Z300 measures HCHO by difference: An electrochemical sensor is periodically preceded by a formaldehyde scrubber (adapted from a NIOSH method.)

Introduction:

This report is motivated by LBNL's search for real-time instrumentation that can accurately monitor formaldehyde (HCHO) in ongoing field studies in residential and commercial buildings. While exposure to volatile organic compounds (VOCs) in buildings can adversely affect the health of occupants, formaldehyde is of particular concern because it is a ubiquitous carcinogen, and it is emitted from many materials and adhesives used indoors. Formaldehyde can cause burning sensations in the eyes and throat, nausea and difficulty in breathing when people are exposed to concentrations above 100 ppb. Higher levels can lead to asthma attacks. HCHO outgasses from urea-formaldehyde-based resins that bind pressed wood products such as plywood, veneers and particleboard. It is widely used in the manufacture of paper, textiles and paints. HCHO is also formed indoors when ozone reacts with other indoor VOCs from smoking, cooking and cleaning. Time-averaged indoor formaldehyde levels reached as high as 123 ppb in recent surveys of California residential buildings, exceeding California's recommended exposure limit of 9 ppb by at least a factor of thirteen (Mullen et al., 2013).

Conventional methods trap HCHO on a solid sorbent or into a solvent, and both approaches use chromatography for the analysis after the samples are brought back to a laboratory. Two recently commercialized methods[*] can quantify HCHO over 30-minute periods. Although these methods are accurate, they do not yield real-time data and their time resolution is limited. This study compared the accuracy, selectivity, sensitivity and portability of two commercially available instruments that measure HCHO with great time resolution. The first uses proton transfer reaction mass spectrometry (PTR-MS). The second uses electrochemical detection. Since both approaches are already known to respond to small oxygenated molecules, the objective of this study was to develop and begin validation of sampling inlets for both types of instruments that remove the main interferents for HCHO upstream of the sensing elements.

In this study the reference instrument for trace detection of multiple VOCs in real-time was the Ionicon [Model 4160-500b] PTR-MS which monitors HCHO based on the principle of chemical ionization, using water as the protonation agent. The Ionicon is an advanced research-grade instrument that costs more than $100,000. At present such instruments can be mounted on wheels but are too large to be considered portable. The Ionicon monitors HCHO by tracking its ion signal at a mass-to-charge ratio (m/z) of 31. However, the ion concentration at m/z = 31 is influenced by water vapor and other VOCs such as alcohols that also produce fragments at m/z = 31. At present these interferences severely limit the accuracy of any PTR-MS for monitoring formaldehyde in the presence of water and/or oxygenated VOCs.

Handheld sensors for volatile organics, especially formaldehyde, are desired because they produce real-time data and they are easy to use in the field. The Interscan Corporation offers a portable, real-time instrument that works on electrochemical principles that the company markets for formaldehyde detection at concentrations below 500 ppb. The manufacturer acknowledges that the instrument has external influences from water vapor and other organic compounds such as acetone and alcohols. These compounds are also electroactive substances and lead to positive artifacts for HCHO. Table 1 shows compounds that Interscan lists as interferences for its HCHO sensors.

[*]Colorimetric sensor developed by NTT Energy and Environment Systems Lab. in Japan; marketed by Shinyei and RKI based on Maruo et. al., 2008, 2010; [#]Environmental Sensors Model Z300 measures HCHO by difference: An electrochemical sensor is periodically preceded by a formaldehyde scrubber (adapted from a NIOSH method.)

Table 1. Compounds that interfere with formaldehyde measurement using the Interscan portable analyzer 4160-500b,. The chart shows the ***approximate*** concentration in ppb of the interfering gas required to cause a signal from the analyzer equivalent to a 1 ppb change in concentration of HCHO. (From manufacturer's data: http://gasdetection.com/the-tech-center/interfering-gas-data).

Compound	***Concentration, ppb***
Acetaldehyde	17
Acetone	>1000
Chlorine gas	7
Carbon monoxide	5600
Ethanol	127
Glutaraldehyde	200
Hydrogen	>10000
Hydrogen sulfide	3
Hydrochloric acid	35
Sulfur dioxide	3
Isopropanal	1000
Methanol	625
Methylethylketone	>1000
n-Butanol	3200
n-Propanol	2000
Ammonia	300
Nitrous oxide	500
Nitrogen dioxide	35
Phenol	>1000
Propionaldehyde	160

Since common oxygenated VOC and water vapor are already known to interfere with both real-time approaches for monitoring HCHO, this study included developing and testing asimple scrubber inlet (the VOC True Read) for each instrument The devices dry the incoming air and remove the oxygenated VOC without trapping the formaldehyde.

Experimental Setup and Methods:

Figure 1 presents a schematic diagram of the experimental apparatus. A constant source of formaldehyde was produced using a diffusion tube (Kin Tek, La Marque, TX, www.kin-tek.com) in a diffusion oven (AID Portable Calibration System Model 330A, Analytical Instrument Development Inc. Avondale PA). The temperature in the oven was maintained at 37 ^{o}C for a constant flow of clean dry air (~100 cc/min) to sweep a stream of formaldehyde from the diffusion oven. The emission rate was calculated by weighing the source periodically. Two separate flows of clean house air were used to dilute the formaldehyde stream to produce the targeted concentrations for evaluating the performance of the analyzers. To control the RH of the formaldehyde calibration generator one flow stream was maintained dry (<3% RH) and the other stream was saturated with water vapor using a humidifier setup. The dilution stream flow rate was adjusted using mass flow controllers (Alicat 0-50 SLPM). Formaldehyde concentration was monitored periodically by collecting 30-minute time-averaged dinitrophenyl hydrazine (DNPH) cartridges for every setting.

The mixture of VOC standards shown in Table 2 was used to evaluate the effect of selected common household VOCs on the measurements from both instruments. The concentrations of the model VOCs were maintained at levels generally observed in homes and office spaces.

Table 2. VOCs used in the 20 m^3 stainless steel chamber, along with their reported mean concentrations and physical properties

Compound	*Molecular Weight*	*Boiling Point (K)*	*Vapor Pressure (mm Hg)*	*Reported mean indoor levels (ppb[(a)])*	*Chamber concentration (ppb)*
Acetaldehyde	44	293	760	11	7-10
Benzene	78	353	100	0.3	12 – 15
1-Butanol	74	391	8.8	13	14 - 20
Ethanol	46	352	6	456	20-30
Methanol	32	338	6.7	n.d.[b]	4-8
Limonene	136	449	20	6	2-10

([a]Logue et al 2011; [b]n.d.: no data available

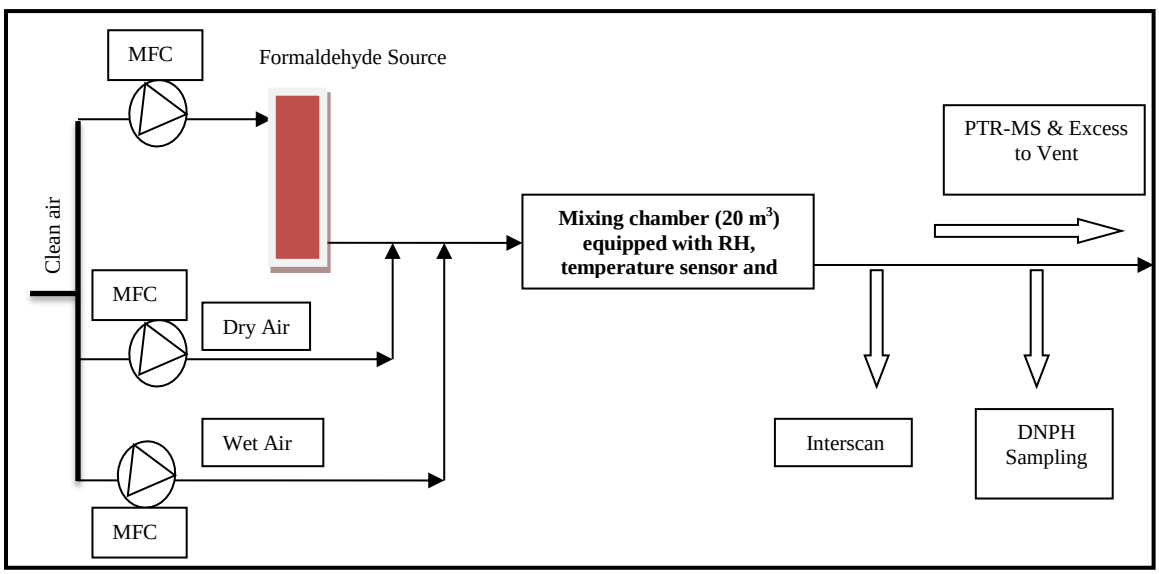

Figure 1. Experimental setup for evaluating performance of PTR-MS/Interscan formaldehyde handheld monitor (MFC- Mass flow controller).

Reference method for formaldehyde

Silica gel cartridges impregnated with 2,4-dinitrophenylhydrazine (DNPH, (XpoSure, #WAT047205, Waters Corporation, USA) were used to collect volatile aldehyde samples. The

flow through the samplers was measured immediately before and after each sampling period. The experimental uncertainty of the sample volume was less than ±3%. The samplers were extracted with 2-mL aliquots of acetonitrile and analyzed by HPLC with UV detection (Agilent 1200). Formaldehyde was quantified with a calibration curve prepared with authentic standards of the DNPH hydrazone derivatives (Sigma-Aldrich, USA). The average formaldehyde concentration in reactant blanks was equivalent to < 0.02µg m^{-3}.

Measurement using proton transfer reaction-mass spectrometry

The PTR-MS (Ionicon Model 4160-500b, Innsbruck, Austria) was operated in water mode to produce H_3O^+ ions to protonate the organic molecules. A reaction constant of $2x10^{-9}$ cm^3s^{-1}, reaction time of $9.5x10^{-5}$ s and a dwell time of 100 µs were used to monitor the concentration of formaldehyde. The voltage in the drift chamber was maintained at 600 V, and the pressure in the drift chamber was maintained at 2.2 mbar. The readings were obtained in MID mode. The signal at m/z = 31 was followed to monitor formaldehyde. The inlet of the PTR-MS was maintained at 70° C and the reaction chamber was maintained at 80° C. The air flow into the PTR-MS was 70-80 cm^3 min^{-1}. The concentration of the primary ion 31 was monitored continuously and the signal level was maintained above 10^7 counts-per-second. The real time output from the PTRMS was calibrated using the DNPH method. HCHO concentrations were derived from replicate calibrations.

Measurement via an electrochemical sensor in a portable real-time monitor

The Interscan 4160-2 (Interscan Corporation, Chatworth, CA) is a real-time portable (2 kg) formaldehyde monitor with an electrochemical sensor that has a low range of 0-500 ppb. Since the device was received calibrated from the manufacturer, no further calibration was performed before testing began, but this proved necessary during the testing, as described below. The reading from the Interscan was recorded using a digital data logger, along with ambient temperature and relative humidity. The mass of the electrochemical sensor was measured periodically, following suggestions from the manufacturer, and water was added to maintain the weight at 35g because the weight of the sensor should never fall below 25 g.

Humidity and VOC scrubbers to remove interferences

To remove interferences from water vapor and VOCs two-stage scrubber assemblies were designed and built for retrofitting the inlets of each real-time inlet. One stage of each scrubber used Nafion tubing (1 mm internal diameter, ~1 m length, Perma Pure TT-050) to remove moisture, and the other stage contained an adsorbent-coated denuder that removed interfering VOCs.

The Nafion tube was soaked in 20% (v/v) formalin solution for 4 hours and dried at 57 ^oC for 2 hours before it was used to strip humidity in the retrofit. The Nafion tube was surrounded by Drierite (dehumidifier) to remove water from the sample air stream. The denuder coating consisted of a 2:1:1 mixture of XAD-4 (Sigma Aldrich), Tenax^{TA} (Sigma Aldrich) and activated carbon, by weight in methanol. The coating was carried out using methods developed by Gundel et al. (1995, 2001). Inlets were scaled to fit each instrument to accommodate their different

intake flow rates. The denuder for the PTR-MS retrofit was a stainless steel tube (length 8.9 cm, internal diameter 0.5 cm; Perkin Elmer). After coating the denuder was dried at 200 ^{o}C for 1 hour to remove solvent residues. The net mass of coating was roughly 500 μg per tube.

For the Interscan monitor, a ¼ inch Teflon tube (0.3 cm i.d. length 60 cm) was coated with the sorbent mixture using the protocol described above and then dried at 75 ^{o}C for 12 hours to remove excess solvent and impurities. A longer tube with higher sorbent loading was used with the Interscan monitor to accommodate the higher sample flow rate of 1 L min^{-1}.

Results:

Proton Transfer Reaction – Mass Spectrometer

Calibration Curve

Initial calibration curves for formaldehyde with PTR-MS were done under dry conditions (RH<2%). The PTR-MS quantifies formaldehyde by monitoring the ion signal at *m/z* 31 corresponding to $H{\cdot}HCHO^+$ that is formed via Reaction (R1). This proton transfer is favorable thermodynamically, since the proton affinity of HCHO (170.4 kcal/mol) is higher than that of water (165.2 kcal/mol).

$$HCHO + H_3O^+ \rightarrow H{\cdot}HCHO^+ + H_2O \qquad \text{(R1)}$$
$$H{\cdot}HCHO^+ + H_2O \rightarrow HCHO + H_3O^+ \qquad \text{(R1a)}$$

However, since the proton affinity difference is sufficiently small <30 kJ, the reverse reaction of protonated HCHO with water (R1a) becomes relevant and reduces the sensitivity of detection (Vlasenko et al., 2010). Figure 2 shows the plot of PTR-MS data vs. DNPH data for dry conditions (RH<2%) while Figure 3 shows the calibration curve for 45% RH. The correction factor, estimated from the calibration curve, when incorporated with the PTR-MS readings gives a one to one correlation with the DNPH data. The slopes of the calibration curves were 3.1 and 3.3 for dry and 45% RH conditions, respectively. The small difference was not statistically significant for readings under the two conditions noticed. However, no tests were conducted at RH higher than 45%.

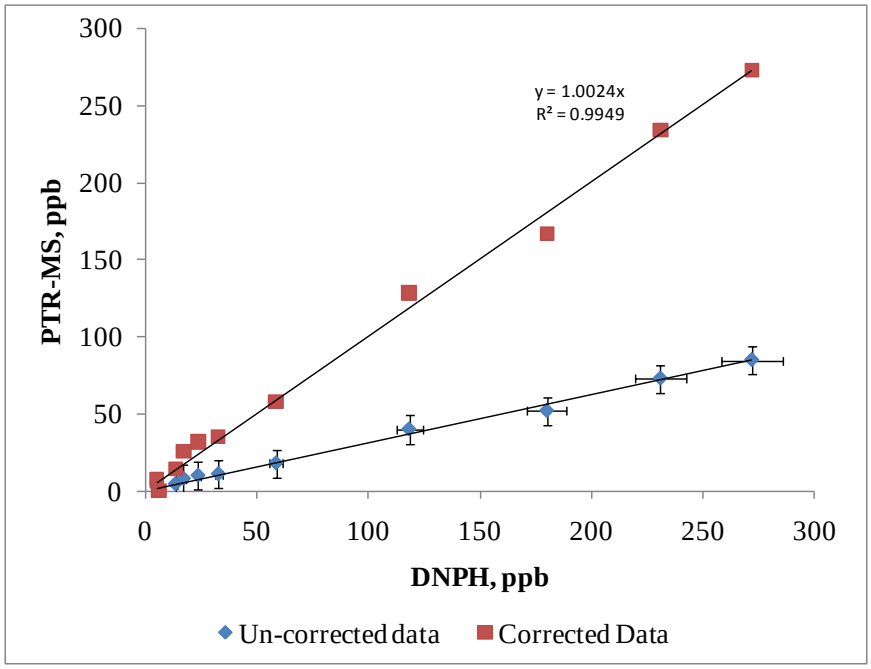

Figure 2. The circles show the PTR-MS readings vs. the with DNPH data. The squares show the how the data compared when the calibration factor of 3.1 (from equation 1) was applied to the PTR-MS measurements at RH<2% (equation 1).

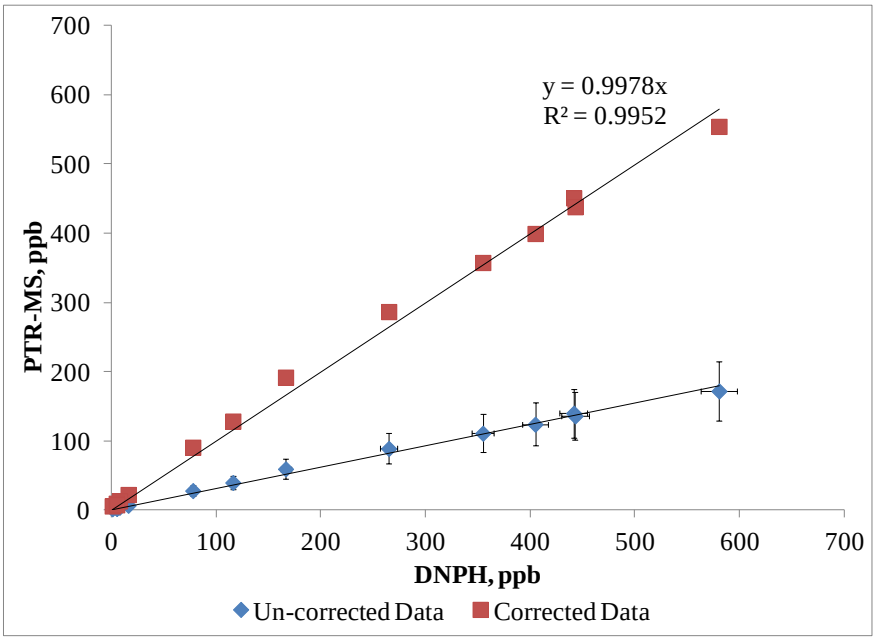

Figure 3. The circles show the PTR-MS readings vs the with DNPH data. The squares show the how the data compared when the calibration factor of 3.3 was applied to the PTR-MS measurements at RH~45% (equation 2).

Figure 4 shows the formaldehyde concentration levels observed using the PTR-MS for experiments carried out in the presence of the mixture of VOCs given in Table 2. When alcohols

such as ethanol, methanol and butanol were present in the sample stream, the apparent HCHO concentration increased by about 10%. This could be attributed to the protonation reaction of alcohols leading to the formation of a CH_2OH^+ ion which has the same protonated mass of 31 as that of the $HCHO^+$ ion. The ratios of the m/z 31 to m/z 47 (ethanol), m/z 31 to m/z 33 (methanol) and m/z31 to m/z 75 (butanol) from blank measurements were estimated to be ~0.05, 0.02 and 0.001 respectively. These ratios are in agreement with the values reported by Innomata et al., 2008 and Schripp et al., 2010. When the VOC – RH scrubber was utilized the m/z 31 signal from the other VOCs disappeared and concentrations derived from the m/z 31 ion measurement (corrected using the slopes in equations 1 and 2) agreed with the formaldehyde concentration determined with the DNPH method.

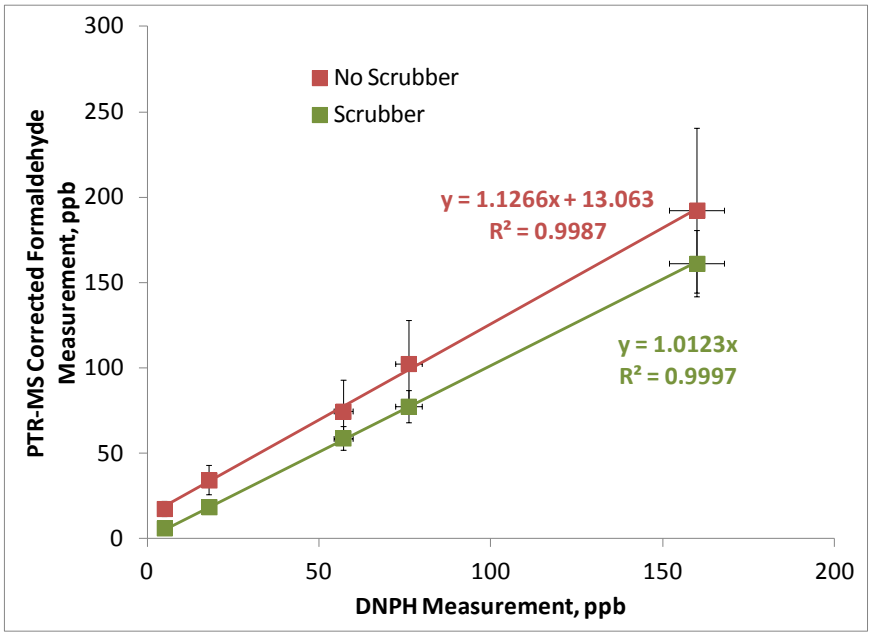

Figure 4. Formaldehyde concentrations estimated by DNPH and PTR-MS in the presence of VOCs and 45-50% RH with and without VOC/RH scrubber

Interscan Formaldehyde Monitor

Initial readings

When the Interscan monitor was turned on, room air was drawn through the inlet at 1 L min^{-1}. The formaldehyde concentration in the laboratory was less than 3 ± 1 ppb when measured using the DNPH method. However, the portable analyzer recorded a formaldehyde concentration of 300 ppb for the same time period, over which the temperature and RH averaged 25±3 ^oC and 45%, respectively. The instrument was then connected to the flow system (Figure 1) that delivered constant concentrations of formaldehyde at 2% RH. Since the output signal from the Interscan remained at 300 ppb whatever the HCHO concentration was in the inlet stream, the instrument was then re-calibrated in the laboratory using the calibrated PTR-MS (as a secondary standard) before further evaluation of its performance.

Calibration

The instrument was zeroed using formaldehyde-free air, and then the instrument was calibrated using 5 different formaldehyde concentrations ranging from 10 ppb to 400 ppb. The formaldehyde source stream was maintained dry (RH < 2 %) and simultaneous DNPH, and PTR-MS measurements were taken to ensure accuracy. The Interscan monitor had to be calibrated after every 10-hr experiment because of substantial zero drift, and the difference in calibration factors exceeded 40% before and after each experiment. The electrochemical sensor element was also weighed before after each experiment, and water was added using the manufacturer's instructions to prevent damage to the sensor.

One hour averages were calculated from the real time data for the periods during which DNPH cartridges were used. Figure 5 shows that with no VOC present and a dry (~2% RH) HCHO stream, the calibrated Interscan, PTR-MS and DNPH results agreed well.

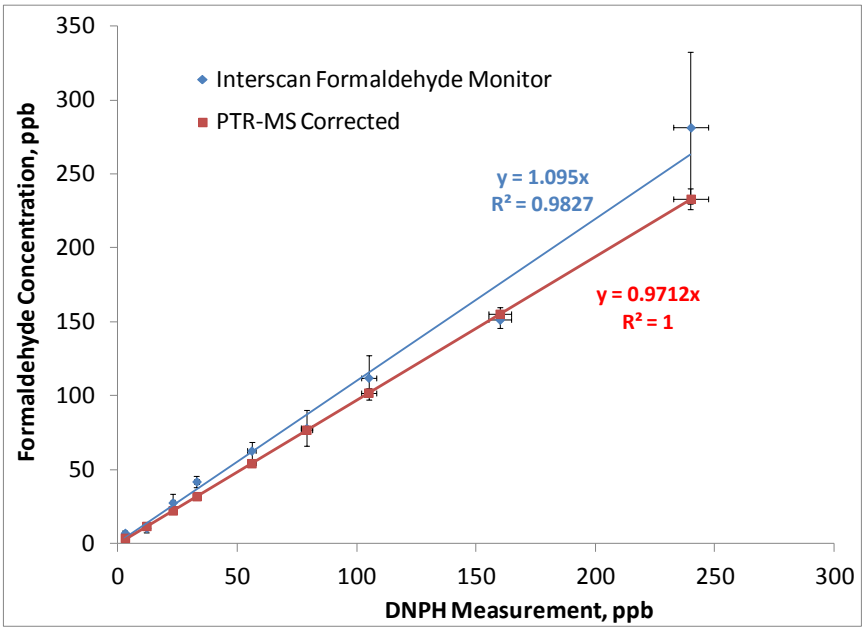

Figure 5. Formaldehyde concentrations measured by the PTR-MS and Interscan monitor compared to DNPH measurements in dry air.

Figure 6 shows the comparison of formaldehyde measurements recorded by PTR-MS and Interscan for a sample stream with RH of 45-50%. The formaldehyde readings from the Interscan plateaued at ~200 ppb without a scrubber to dry the sample stream. *These results showed that the Interscan monitor did not accurately report formaldehyde at humidity levels that are commonly found indoors.*

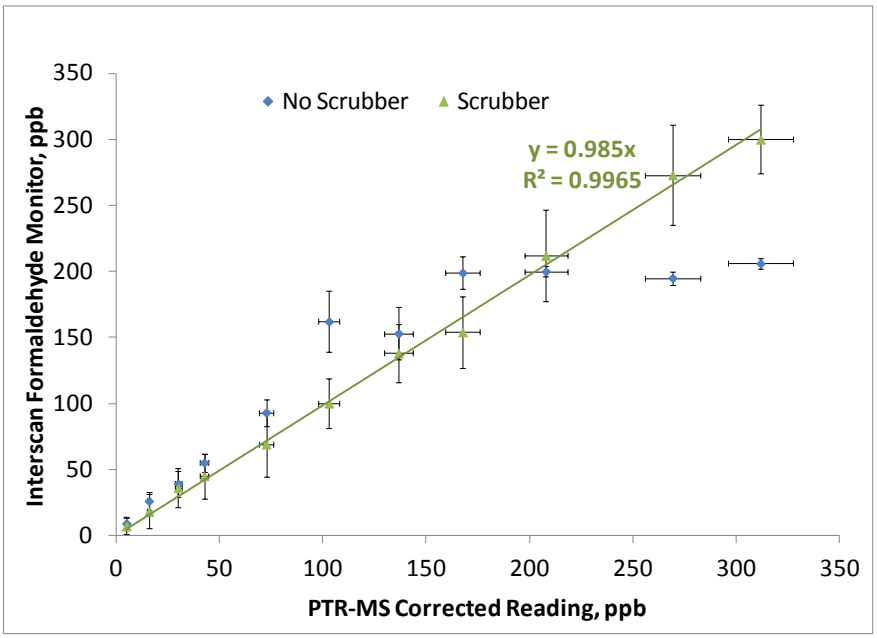

Figure 6. Formaldehyde concentrations at 45-50% RH as reported by the Interscan monitor, without the RH scrubber (diamonds) and with (triangles).

The standard VOC mixture described in Table 2 (acetaldehyde, ethanol, methanol, butanol, benzene and limonene) was used to find out how the electrochemical sensor in the Interscan monitor responded to formaldehyde in the presence of a mixture of VOCs that are often found in homes. At RH of 45-50 % and concentrations of 20-30 ppb for each VOC in the mixture, the instrument reported concentrations of formaldehyde that were roughly twice those obtained with the DNPH method, as shown in Figure 7 (blue diamonds). Replacing the electrochemical sensor in the Interscan did not improve its sensitivity to VOCs. *The level of interference observed was much higher than predicted based on the manufacturer's information in Table 1 and the VOC concentrations in used in this study (Table 2). These results showed that the Interscan monitor did not accurately report formaldehyde in the presence of low levels of common indoor oxygenated VOCs and limonene.*

How scrubbers influenced measurements of HCHO at ambient RH in the presence of VOCs

When the scrubbers described in the *Experimental Setup and Methods* section were placed upstream of the inlet of the monitor, the Nafion (surrounded by Drierite), removed water vapor, and the readings from the Interscan agreed with the (calibrated) PTR-MS readings. No

DNPH samples were collected during this test because the PTR-MS readings agreed well with the DNPH values, as shown in Figures 2 and 3.

With the scrubber in place, the Interscan reported only about 6 % more formaldehyde than the DNPH method (green data points and the line in Figure 7) when challenged with the VOC mixture. The efficiency of VOC removal was estimated by sampling onto Tenax upstream and downstream of the instrument's inlet; moisture removal efficiency was calculated from measured with RH probes. The net VOC removal was greater than 95% (from duplicate measurements), and the formaldehyde levels agreed very well with those from the DNPH method, within experimental error (~3%). *The scrubbers successfully stripped the incoming airstream of both water vapor and VOCs, thereby retrofitting the Interscan monitor to report accurate formaldehyde measurements under realistic conditions.*

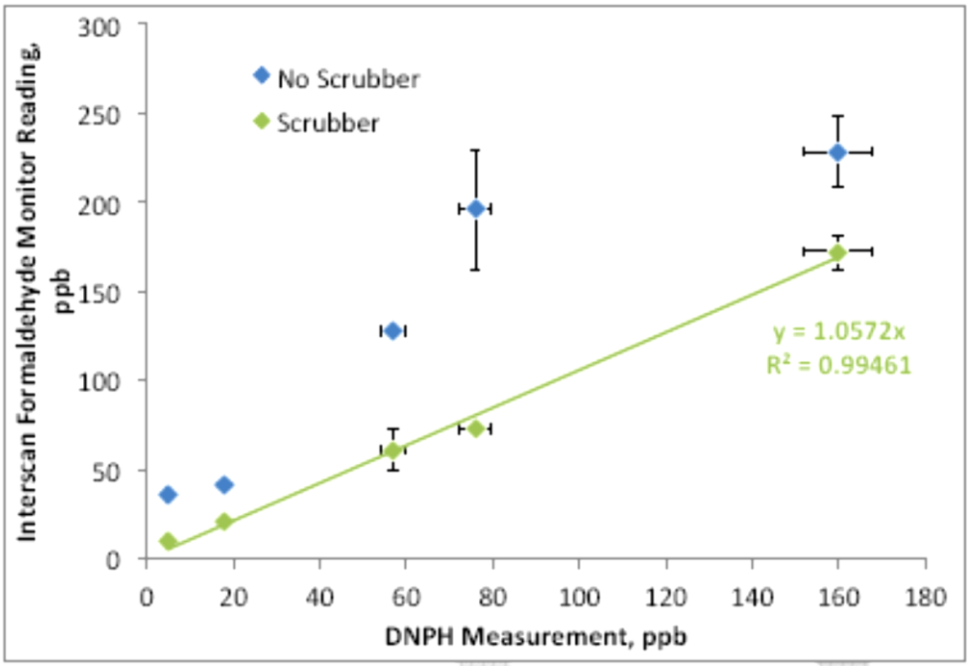

Figure 7. Formaldehyde concentrations reported by the Interscan monitor and DNPH method in the presence of VOCs at45-50% RH, with and without the RH/VOC scrubber.

Summary and Recommendations

For accurate monitoring of formaldehyde levels by the PTR-MS the instrument needs to be calibrated by making concurrent measurements by the DNPH method. The calibration factor is dependent on instrument parameters and settings such as the voltage across the drift tube, the dwell time, transmission constants and the primary ion concentration. The calibration should be checked periodically with DNPH cartridges. Calibration must also be repeated after the PTR-MS ion source has been cleaned and/or after the instrument has been moved from one location to

another. Similar procedures apply to PTR-MS measurements of other VOCs. The RH/VOC scrubber removed the positive artifact of about 10% that was observed in measurements of formaldehyde in the presence of 45-50% RH and low concentrations of oxygenated VOCs and terpenes.

To report formaldehyde concentrations accurately under typical indoor conditions the Interscan monitor must be retrofitted with a scrubber to dry the incoming air and remove VOCs because the electrochemical sensor is very responsive to ambient humidity and the presence of oxygenated VOCs and terpenes. The Interscan also requires frequent calibration (after ~ 10 hours of operation) for reliable concentration output. The sensor element requires constant monitoring for water levels and will deteriorate if the water weight in the sensor is not maintained. Field tests with the scrubber need to be conducted to verify that this monitor can obtain reproducible and reliable data in indoor environments. Caution should be exercised when measurements from this instrument are used for reporting formaldehyde concentrations in air.

Currently, available real-time formaldehyde sensors work well in environments contaminated only with formaldehyde. However, field work funded by DOE identified inaccuracy and drift-prone readings in environments with interfering compounds such as water vapor and polar VOCs like methanol and ethanol - typical conditions in most indoor work environments and homes. This study shows that the LBNL scrubber inlet (recently described as the VOC True Read), when attached to two different commercially available formaldehyde sensors, efficiently and reliably removed interfering compounds and allowed the air sample to flow on to a detector. Such inlets could be retrofitted onto commercially available sensors for formaldehyde.

In order for this measurement technology to be widely adopted for use in inexpensive mitigation applications such as demand-controlled ventilation, full validation and commercialization are necessary. The results of this study have established proof-of-concept, and LBNL has filed a patent application for the VOC True Read device. LBNL also nominated the VOC True Read for an R&D 100 Award for 2013.

Acknowledgments

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