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REPORT



Robotic direct reading device with spatial, temporal, and PID sensors for laboratory VOC exposure assessment

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ABSTRACT

This study evaluated a novel robotic direct reading method that used a real-time location system to measure the spatial-concentration distribution of volatile organic compounds (VOCs) in a chemistry laboratory. The CEMWIP II is a custom-made sensor that measures VOCs, temperature, humidity, and location, sending data wirelessly in real time to a remote location for display and storage. In this study, the CEMWIP II device was mounted on a robotic platform to create a CEMWIP II-mobile platform. The autonomous mobile platform was released from a corner of the room and allowed to travel randomly along an open floor with the goal of characterizing the spatial distribution of VOCs and identifying their sources in the laboratory. The experiment consisted of 12 runs made of permutations of four corner release sites and four beaker locations, with two beakers containing water and two containing the solvent acetone. The autonomous mobile platform was tasked with locating the two beakers of acetone. The sensor had a detection limit of 100 ppb and the confidence of detecting a source within a 1.46 m² area was $p = 0.0005$ by ANOVA. The CEMWIP II-mobile platform was able to measure the spatial distribution of VOCs within a laboratory that were associated with open solvent containers.

KEYWORDS

CEMWIP; direct reading; exposure assessment; real-time location system; robotic; VOC

Introduction

The work presented in this article was under the broad objectives of three initiatives from the National Institute for Occupational Safety and Health (NIOSH). The first was the NIOSH Center for Direct Reading and Sensor Technologies (NCDRST), which was established in 2014 to coordinate a national research agenda for direct-reading methods and sensor technologies.^[1] The Center's goals were incorporated into the NIOSH Strategic Plan. The second initiative came from the NIOSH Center for Occupational Robotics Research (CORR), which recognizes the need for research and effort in autonomous vehicles and drones.^[2] Finally, the third was from the Office of Laboratory Science and Safety (OLSS), which was established by the CDC in 2014 to advance the scientific excellence and safety of every CDC laboratory.^[3]

Volatile organic compounds (VOCs) are found in many microenvironments including classrooms, houses, and workplaces.^[4–18] One such workplace is the laboratory. Across the United States, laboratories

employ over 500,000 workers.^[5,12,19–25] Chemistry laboratories inherently present both chemical and physical occupational hazards. Laboratory VOCs commonly pose a physical threat from fire and explosions.^[28,29] For example, a researcher in Mulhouse, France was out for lunch when organic vapor concentrations reached an explosive level. A spark from an oven caused an explosion that injured 23 people and killed 1.^[26,27] A direct reading real-time VOC sensor, commonly used in refineries and other chemical process industries, in the laboratory, may have detected the VOC source and prevented this tragedy. VOCs also pose a toxicological threat to laboratory workers. The most common effect of VOC exposure is central nervous system toxicity, including headaches and dizziness.^[38] Some VOCs are also carcinogens^[30–33] and known to damage DNA after prolonged chronic exposures.^[11,33–37]

This study describes and evaluates the effectiveness of a newly developed real-time direct reading method (RTDRM) combined with a real-time location system (RTLS) on a mobile robotic platform. The RTDRM

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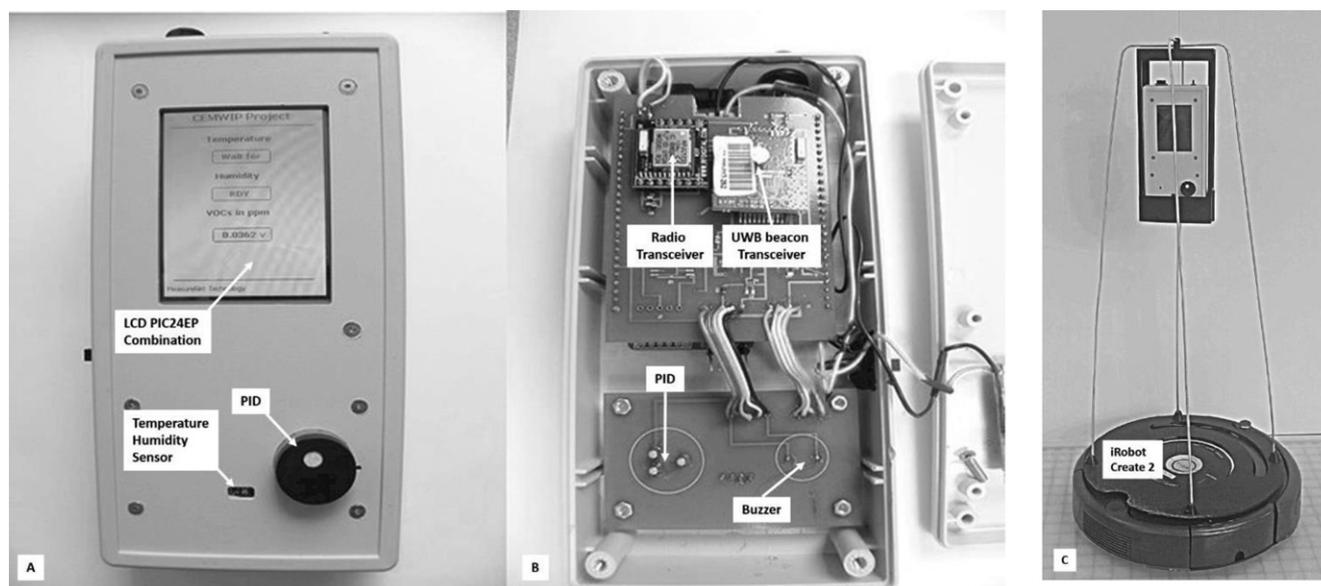


Figure 1. CEMWIP II prototype wireless sensor. (A) is the front view and (B) is the view on the right. The CEMWIP II-mobile platform consisted of a mobile iRobot Create 2 base and a CEMWIP II sensor (C).

design was based on a previous CEMWIP I (chemical exposure monitor with indoor positioning) sensor,^[39] which was upgraded with new electronics to create the CEMWIP II version. The RTLS was installed in a chemistry laboratory to measure remotely the spatial distribution of VOCs. The experiment took place in an unoccupied laboratory. A mobile robotic platform with a CEMWIP II sensor randomly traveled across the floor, collecting thousands of spatial-concentration samples in 60-min periods, covering every square meter of the open laboratory floor. The study evaluated this novel combination of a direct reading method (DRM), real-time location system (RTLS), and mobile robotic platform to evaluate the CEMWIP II for possible human exposure assessment.

There were four objectives to this study. (1) Compare the accuracy and precision of the CEMWIP II photoionization detector (PID) to the CEMWIP I PID. (2) Compare the RTLS's coverage range and density to the results in a previous study. (3) Determine if a mobile robotic platform could replace personnel in testing a personal wearable exposure device for efficacy. (4) Use a robotic RTDRM device to map VOC distribution within a room and locate the open solvent beakers on the lab benchtops.

Methods

Figures 1A and 1B show the CEMWIP II, an upgraded version of a personal chemical sensor based on the experimental design published previously.^[39] The CEMWIP I was hand-wired on a protoboard

with the RTLS beacon taped to the side. The upgrade consisted of hard wiring the circuits on a printed circuit board and embedding a UWB (ultra-wide band) RF beacon chip. (Ubisense Inc., Denver, CO). The microcontroller and LCD combination was also upgraded to a PIC24EP (MikroElektronika D.O.O, Belgrade, Serbia). However, the CEMWIP II sensors remained the same: a photoionization detector (Model# piD-TECH eVx 045-012; Baseline-Mocon, Lyons, CO) and a temperature and humidity sensor (Model # SHT11; Sensirion, Staefa, Switzerland). Radio telemetry used a RFDP8 chip (RFdigital, Irvine, CA) to communicate at 2.4 GHz using RFdigital's protocol. Finally, the data were transmitted to a base station connected to a laptop with the same RTLS monitoring software used in the original CEMWIP I study.

Figure 1C shows the CEMWIP II attached to the mobile platform made from a modified iRobot Create 2 (iRobot, Bedford, MA). Absent vacuum cleaning components, the unit was able to move about randomly, unmonitored while autonomously avoiding obstacles. A custom 3D-printed cover plate that incorporated features designed to hold the CEMWIP II sensor at approximately benchtop height (0.71 m) replaced the original cover plate of the mobile platform. This modified cover plate was affixed to the robot with Dual Lock re-closeable fasteners (3M, St. Paul, MN) to allow for easy removal and replacement. A 3-mm aluminum-rod support structure allowed the VOC sensor to hang freely at the benchtop height. The metal support structure was affixed to the cover



Figure 2. Chemistry laboratory setting in which the CEMWIP II-mobile platform was tested. The white circles show the Ubisense antennas needed to track the position of the robot in the laboratory. There was an antenna at the corner of the room that is not shown.

plate using a two-part epoxy adhesive (2-Ton Clear Epoxy, ITW Devcon, Danvers, MA) to ensure a strong connection due to the high mechanical stress experienced by the system when the robot encountered obstacles in the laboratory. For testing, a default clean cycle was used that allowed the robot to find its own path, avoid obstacles without user interaction, and survey the area autonomously.

The RTLS was installed within a chemistry laboratory for this study (Figure 2). The RTLS used four receiving antennas fixed to the periphery walls of the laboratory, three of which are circled in the figure. The antennas were cabled to each other to collect timing and location data. These data were sent directly to a remote laptop computer containing Ubisense software, while the CEMWIP II mobile device sent data to a microcomputer within the laboratory by radio telemetry. The laboratory microcomputer was in turn connected by cable to the remote laptop computer where all the data were combined, displayed, and stored. The RTLS-DRM system displayed real-time data on a computer screen and stored these data for retrospective analysis.

The antennas were able to receive the robot's radio frequency (RF) beacon signal at any point in the room. As shown in Figure 2, four antennas were mounted near the four corner ceilings of the room and tilted downward an elevation of -30° . The antennas' horizontal azimuths were tilted toward the center of the room. The moving RF beacon must be seen by at least two antennas to calculate its location. Each wall antenna was connected independently to the remote computer using Ethernet cables. The antennas

received RF pulses from the CEMWIP device, determined their angle of arrival (AOA) and the time difference of arrival between antennas (TDOA), and downloaded the data to the remote computer. The antennas were also connected to each other with shielded CAT5e cables in order to synchronize timing signals to a nanosecond. The device's location was determined using trilateration and triangulation algorithms from TDOA and AOA data at the remote computer.

The CEMWIP-mobile platform randomly traveled the laboratory floor measuring VOC concentration, location, temperature, humidity, and time. The device wirelessly transmitted data every 1.7 sec to a remote computer that stored the exposure data, provided alerts, but most significantly detected VOCs during its travels. Location and chemical concentration data were transmitted through an Ethernet cable to a remote station in real time.

Room ventilation can influence VOC distribution and, thus, sensor readings. This experiment took place in a room with general ventilation, consisting of ceiling inlet flow of 1069 CFM ($30.27 \text{ m}^3/\text{min}$) and an outlet airflow of 1400 CFM ($39.76 \text{ m}^3/\text{min}$), resulting a net negative room pressure. The upper right hood in Figure 3 had an exhaust linear velocity of 121 fpm ($36.9 \text{ m}/\text{min}$) and the upper left hood had an exhaust linear velocity of 131 fpm ($39.9 \text{ m}/\text{min}$) resulting in exhaust flow of 770 CFM ($21.9 \text{ m}^3/\text{min}$) and 630 CFM ($17.9 \text{ m}^3/\text{min}$), respectively. The room dimensions were $6.37 \times 9.05 \times 2.57 \text{ m}$, resulting in 12 air changes per hour (AC/H). If the sensor is able to detect VOCs in this setting, it will further demonstrate

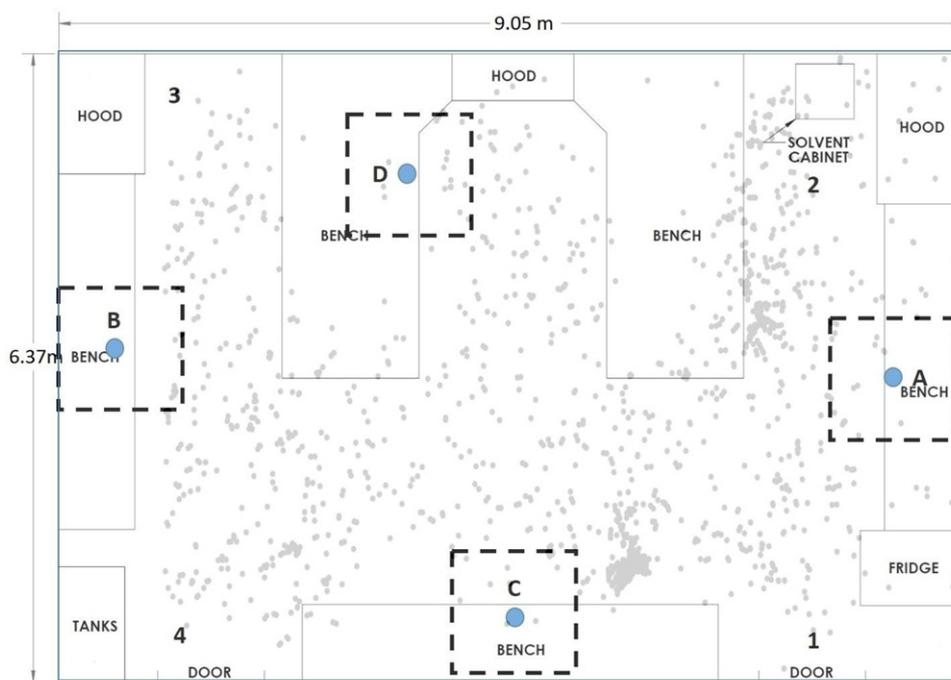


Figure 3. Floor plan of the laboratory with dots and letters denoted where the acetone and blank sources were located and the numbers denoted the location where the robot was released according to Table 1. The room was 6.37 m x 9.05 m. The squares with dotted lines represent the 1.21 m x 1.21 m areas used in data reduction. The black dots are location measurements from Run #1.

or support its efficacy in working laboratories where high levels of ventilation are common.

Calibrating the CEMWIP sensor

The CEMWIP II sensor was calibrated using a mix of nitrogen and isobutylene gas at 0, 0.106, 1.13, and 2.10 ppm. Isobutylene is an industrial standard and the response factors of isobutylene to thousands of VOCs have been documented. Amtek Baseline Co. reports a response factor of 1.2 for acetone with a 10.6 eV lamp. A PVC tube wand was constructed to deliver gases to the PID. The wand was made from 9 in. (23 cm) of $\frac{3}{4}$ in. (2 cm) ID PVC tubing with an end-cap nipple that accepted Tygon tubing from the gas cylinder. The open end of the wand fit over the PID to deliver a stream of known-concentration isobutylene. A ring of eight 1/16-in. (0.16 cm) pressure-relief holes was drilled near the open end of the wand to prevent sensor overpressure from the calibration gas cylinder.

Experimental design to map VOCs in the laboratory

Figure 3 shows the location of corners 1, 2, 3, and 4 and the benchtop locations A, B, C, and D for the

two acetone beakers (250 mL of acetone added to each) and two water beakers. The water beakers were considered blanks. Figure 3 also shows the 1.21 m x 1.21 m squares areas around the benchtop locations and the location measurement from Run #1. The upper left corner of Figure 3 was designated as the (0, 0) in the Cartesian coordinate display of location on the remote. For each 60-min run, the CEMWIP-mobile platform was able to travel randomly in every square meter of the open laboratory floor taking sensor readings. Each excursion varied based on the corner of the room the robot started from and the locations of the two acetone beakers and two blank beakers. Table 1 shows how the corner and location permutations were arranged for each of the 12 runs. The acetone was poured into a 600 mL beaker in a fume hood, and the beaker was carried to its assigned location just before the mobile platform was released (within about 5 min). Acetone vapor steady state was expected at about 6 min. Acetone was chosen because of its high PEL (permissible exposure limit) of 1,000 ppm, and its lower explosive limit (LEL) of 2.5% v/v both well above measurable levels. Using the Fleisher equation,^[40,41] calculations estimated that the evaporation of acetone during a 60-min test in a ventilated laboratory would not attain the PEL or LEL of acetone.

Table 1. Experimental protocol using locations A, B, C, and D as marked in Figure 3 with resulting concentration levels.

Run	Robot Starting Corner	Acetone Locations	Blank Locations	Average Acetone Concentration, ppm			
				A	B	C	D
1	1	C, A	D, B	0.42	0.14	0.32	0.29
2	1	C, D	B, A	0.15	0.40	0.63	1.16
3	4	D, C	B, A	0.48	0.58	0.98	1.07
4	3	B, D	A, C	0.16	0.66	0.18	0.35
5	1	D, A	C, B	0.33	0.10	0.16	0.22
6	4	D, A	B, C	0.38	0.09	0.29	0.84
7	3	A, B	C, D	0.21	0.10	0.13	0.29
8	2	A, B	D, C	0.20	0.95	0.12	0.14
9	2	B, D	C, A	0.22	0.17	0.05	0.62
10	1	A, D	C, B	0.20	0.11	0.18	0.22
11	4	B, A	C, D	0.24	0.10	0.07	0.19
12	3	C, B	A, D	0.61	0.54	0.38	0.46

Statistical method to analyze spatial-concentration data

The experimental protocol, as shown in Table 1, was such that for each run the starting corner was randomized and the order of the four apparatuses was randomized. For each robotic run, as numbered in Table 1, the mobile platform was released into the laboratory with a different permutation of release corner and locations of the sources and blanks. Each run acquired a line of spreadsheet data every 1.7 sec, containing sensor ID, temperature, humidity, PID response, time, and xyz coordinates for about 60 min. The data were sorted by x,y location data within a 4 ft × 4 ft (1.21 m × 1.21 m) area around locations A, B, C, and D and averaged for each run. Figure 3 provides a graph of these square areas. The hypothesis was that the highest concentrations of VOCs would be near or around its source location. The data within these square boundaries were averaged into a single datum for each repetition. The reduced data were converted to ppm units and analyzed with a linear mixed model appropriate for a randomized complete block design. Specifically, the two fixed factors in the model were the acetone status (present or absent) and location. Focus was limited to the effect of acetone status. Location was included in the model for consideration as a factor. The models were analyzed in SAS (Version 9.4, SAS Institute, Cary, NC) using PROC MIXED. The outcome variable was the concentration measured in the 1.21 m × 1.21 m areas.

Results

The PID was calibrated three times at three concentration levels of isobutylene. The average voltage at 2.1 ppm was 0.788V, at 1.13 ppm was 0.42V, and at 0.106 ppm was 0.06V, respectively. Linear regression of the nine calibration points gave a slope of 0.366 V/ppm and an intercept of 0.014 V. The standard error

about the y-axis, S_{ey} , was 0.0138 V and the limit of detection (LOD) was calculated as $(3 \cdot S_{ey})/\text{slope}$ was equal to 0.1 ppm. The limit of quantitation (LOQ) was 0.33 ppm. Using a correction factor from isobutylene to acetone of 1.2, and the LOD and LOQ for acetone is 0.1 and 0.40, respectively.

Figure 4 plots acetone concentration versus time from robotic Run #1 as described in Table 1. Figure 5 is a scatter plot of locations overlaid with a contour plot of acetone concentration from Run #1 data. The concentrations measurements ranged from 0.15–2.25 ppm. Run #1 lasted 56 min and 17 sec. The contour plot was made using the Matlab Curve Fitting Tool software using the biharmonic spline interpolant method to compute a surface response.

Statistical analysis of data

Figure 5 shows a 2D contour plot overlaid on the laboratory floor plans showing the data points from Run #1 and contour shades of gray proportional to concentration, with the lightest shade being downstream of the two open beakers toward the right hood. In this case, bench letters A and C were the locations of the open acetone beakers. Each black dot denotes the location at which the VOC levels were measured. Note that the location of the dots closely stayed in the area between the laboratory tables that the robot skirted.

In Table 2, the presence of acetone was statistically significant with a p-value of 0.0005. Also in Table 2, we observe the least square means estimate for the areas near acetone was 0.3902 in contrast to the areas near the blanks of 0.1993. In addition, we note that the physical locations (A, B, C, or D) did not have a significant effect.

Discussion

The first objective was to compare the accuracy and precision of the CEMWIP II to the CEMWIP I

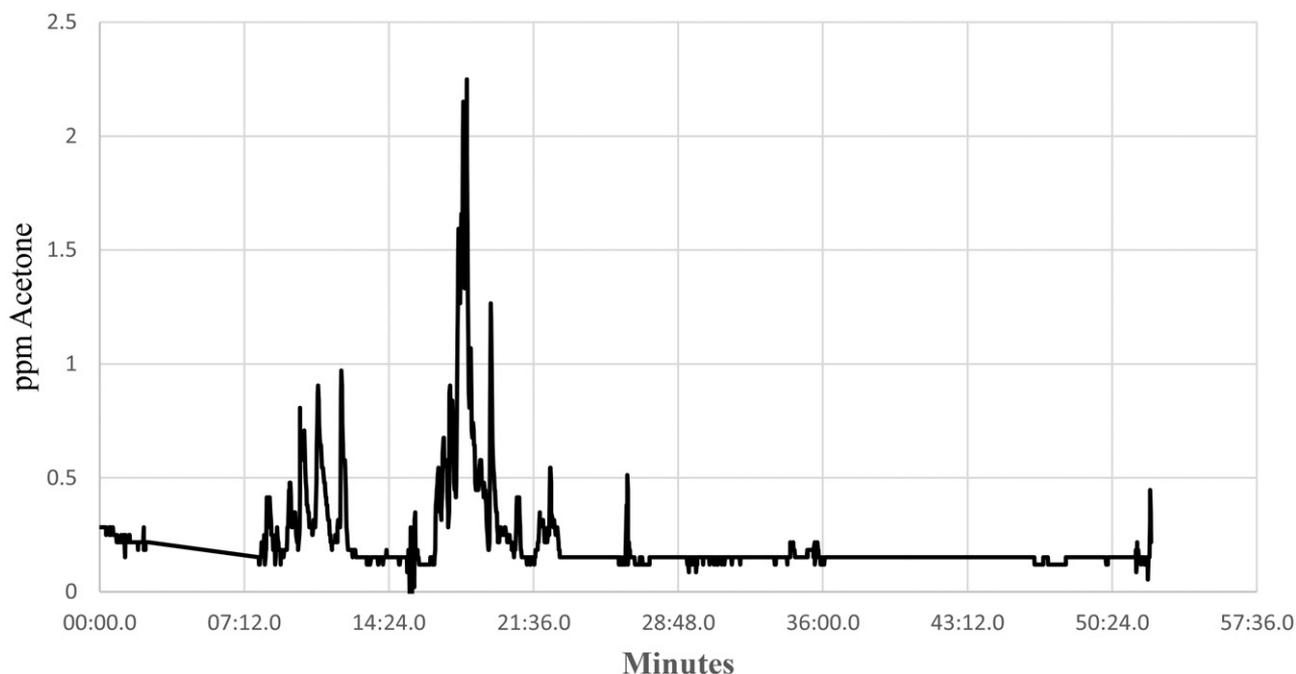


Figure 4. The concentration of acetone as seen by the CEMWIP II device plotted against time, zero being moment of platform release, for experimental Run #1 during which the CEMWIP II-mobile platform traveled the entire laboratory. Peak acetone concentration was at 18.164 min was 2.25 ppm associated with x, y location -2.11 and -1.5 m from the top right corner of Figure 3.

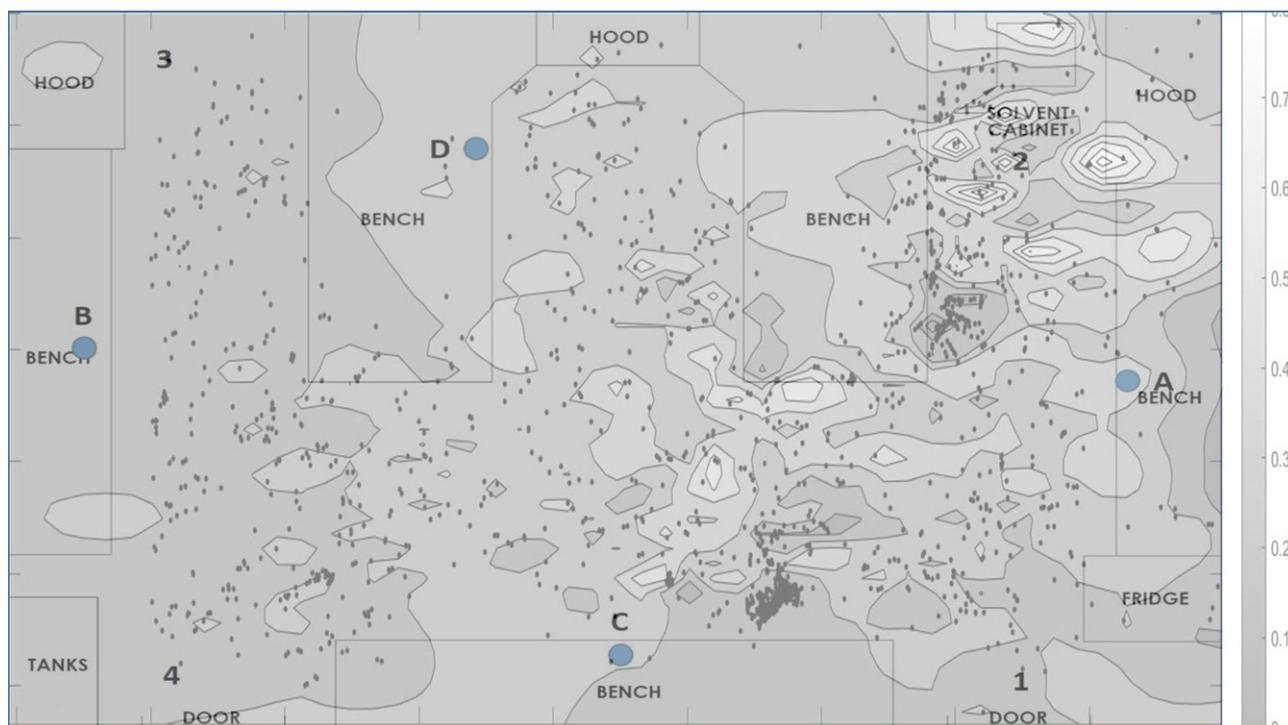


Figure 5. This is a contour plot of the data from Run #1 overlaid upon the laboratory floorplans. The grey scale bar on the right is acetone concentration in ppm. The darkest grey areas are lowest in VOC concentration and the light areas of highest concentration. The open acetone sources were located at points A and C. The black dots are the measured locations where the CEMWIP II-mobile platform traveled and took measurements.

sensor. The LOD in measuring isobutylene of the CEMWIP II sensor was 0.1 ppm and for the previous version 0.2 ppm. The LOQ was 0.33 and for the

previous version was 0.49 ppm. This PID sensor range was 0–2 ppm range PID instead of a 0–20 ppm model and it provided a slightly better LOD as determined

Table 2. Mixed model ANOVA of null hypothesis that acetone concentration within source 1.21 m x 1.21 m square area is equal to blank source 1.21 m x 1.21 m square area. "Location" refers to the physical location (A, B, C, or D) without reference as to whether the acetone was present or not.

Effect	Num. DF*	Den. DF	F Value	p-value
Acetone	1	29	15.59	<0.001
Location	3	29	1.65	0.200
Acetone x Location	3	29	0.55	0.651

Least square means from the mixed model using the 1.21 m x 1.21 m area data		
Acetone	Estimate, ppm	Standard Error, ppm
absent	0.239	0.058
present	0.468	0.058

DF = degrees of freedom in numerator and denominator for F ratio.

in a previous paper both using the calculation approach found in NIOSH SOP 018.^[42] As a point of reference, one study measuring VOCs in 126 homes found an average of 150 ppb.^[43] Thus, a sensor with an LOD of 100 ppb could measure levels slightly higher than typical indoor VOC levels.

The second objective was to measure the RTLS's range and density of the coverage within a laboratory. The sampling density in Figure 5 averaged 35 samples per cubic meter. The unobstructed line of sight between the antennas and CEMWIP II device allowed for optimal location sampling density throughout the room, which equated to its best performance compared to the previous study. The precision of the RTLS for line of sight measurements was determined in a previous study to be one standard deviation of ± 6 cm, explaining the scatter over the edges of the benchtop lines in Figure 5. Two high-density patches of location measurements can be observed in Figure 5. High-density patches were created when the mobile platform became temporarily stuck in a location. For example, in one instance the platform opened a lab bench door and became caught in a back-and-forth pattern between the door and wall, producing a higher than average density patch until the platform was able to self-correct.

The third objective of the study was to determine if a mobile robotic platform could replace personnel in testing the efficacy of personal wearable exposure devices. The mobile robotic platform tested the efficacy of the CEMWIP II sensor without exposing lab personnel to danger. It is reasonable to conclude that if lab personnel were to wear the device near their breathing zone, the sensor would be able to measure VOC concentrations from 0.4–2 ppm and provide the location and time of exposure throughout the laboratory.

The fourth objective of this study was to map VOCs in a room using the CEMWIP-mobile platform and locate beakers of solvent. When the distribution of VOCs in the room was mapped, the distribution,

measured over the timespan of an hour, added a temporal distortion. To address this distortion, research is currently being conducted using sensor arrays methods for VOC mapping. Table 2 revealed that the acetone concentration in the areas around its sources were higher than in areas with without solvent containers with statistical significance over a timespan of an hour. Retrospectively, using an area centered on the acetone and blanks might have been misleading since the robot could not access areas behind the source; however, the average sampling N in the accessible area reached 12. The sensor platform was unable to locate the beakers of solvent. The contour plot in Figure 5 shows that the highest concentrations were found at a location other than the acetone source. If the data were plotted to assume that there was a single source in the room, that source would probably be identified near the number "2" on the plot, approximately 7–8 ft from the acetone source at location A. Future studies could program the mobile platform to receive VOC sensor feedback in real-time to guide the mobile platform in sniffing out and locating the area of high VOCs in a room.

While this mobile platform prototype is not marketable, it is a harbinger of things to come when industrial hygienists can remotely survey hazardous work areas with robots that proactively patrol and monitor work space with sensors. The UWB RTLS system was capable of measuring location in the z-axis, i.e., height, at the same accuracy and precision as the x and y plane measurements. In this study, CEMWIP II device was kept at a single height on the mobile platform, and, for this reason, z data were not analyzed. Measurements in the x, y, and z plane in this laboratory system could be used in future studies to assess vapors at different heights if necessary.

Research is being conducted on other RTLSs, specifically near field electromagnetic ranging (NFER) and beacon location systems, to examine the economic feasibility of other RTLS for research to practice. This UWB RTLS was the most accurate and

precise examined so far in determining location; however, it may not be the most economically feasible for practice in the field. In addition to the future goal of making the CEMWIP device more economical is the goal of minimizing the size to a personal wearable device in a future version.

Conclusion

Figure 5 showed that the CEMWIP II could monitor instantaneous exposure to acetone at any point in the room and at concentrations greater than 0.4 ppm acetone. Thus, the device could be used for exposure assessment. Some runs showed times when the PID sensor became saturated at 2 ppm acetone. We lowered the LOD of the sensor to better measure background levels. During lab techniques using other solvents, the PID could become overloaded. If levels above 2 ppm acetone were ever sensed, the PID could be exchanged for one capable of responding to higher concentrations.

The experiment demonstrated that the CEMWIP-mobile platform was a successful novel tool in locating areas of higher VOCs in the laboratory and helping to characterize the flow dynamics in the room when sources were known. The correlation between VOC sources and areas of highest concentration would be closer in workplaces. On the contrary, the experiment showed why it is difficult to locate the source of a vapor leak with a hand-held DRM in a highly ventilated laboratory.

One objective of the project was to locate the sources of VOCs in a laboratory. While this goal was not achieved, the device was able to locate areas of higher concentrations near and downwind of the source and was able to identify the spatial distribution of vapors in the room. The experiment also demonstrated that a room could be surveyed for VOCs without putting a person in harm's way. This ability would be especially valuable in cases where vapors are highly toxic. Overall, the CEMWIP II-mobile platform was able to measure the difference between the spaces around sources versus spaces around blanks; however, in some specific location permutations, the VOC concentration differences around the source and blank were not discerned, e.g., Table 3, see runs 7, 9, 11, and 12.

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Disclaimer

The findings and conclusions in this report are those of the author(s) and do not necessarily represent the views of the Centers for Disease Control and Prevention or the National Institute for Occupational Safety and Health (NIOSH). Mention of any company or product does not constitute endorsement by NIOSH.

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