CHEMICAL INTERFERENCE TEST RESULTS OF A NOVEL HUMIDITY SENSOR

Simo Ikonen, Lars Stormbom, Timo Ranta-aho

Vaisala Oyj, P.O. Box 26, 00421 Helsinki, Finland

Abstract: A new type of capacitive humidity sensor has been developed to cope with chemical interference. Tests were done both in laboratory conditions and in typical humidity sensor applications. The tested humidity sensors show significantly lower measurement error in most of the test environments compared to the reference sensors. The sensors perform better both in laboratory high chemical concentration tests and in long-term field stability tests.

The resistance to chemical interference has been achieved by developing a new type of chemical resistant polymer and a special new structure. The new structure slows down the migration of adverse chemicals or completely prevents them from entering the active polymer layer. The chemical resistant active polymer and the chemical filtering structure of the capacitive sensor further contribute to the stability of the sensor.

In contrast to earlier sensor versions developed to withstand demanding chemical environments, these improvements have been obtained without compromising the other key properties of the sensor such as, high humidity stability, sensitivity and hysteresis.

Key words: Humidity sensor, Capacitive sensor, Chemical interference, Chemical drift, Performance testing

1. INTRODUCTION

In most humidity measurement applications sensors are also exposed to chemicals other than water vapor. Vaporized chemicals can diffuse into the sensor and cause disturbances in the measurements. Such vapors are present in most environments, even normal office air. Chemical vapors are also inherent in many industrial applications. For example NO_x , SO_2 , disinfectants and different organic solvents are often present in ambient air. As the responses to some gases and vapors are very slow, in many cases several weeks or months, the effects are often described as 'drift' or 'aging' of the sensor, not chemical interference. [1]

The chemical interference or drift can be dealt with by setting the calibration interval according to the conditions or by improving the sensors ability to withstand chemicals. In some cases heating the sensor can either remove a chemical from the active polymer or prevent entry. The method where heating is used to remove a chemical from the sensor's active polymer will be referred to as chemical purge in this text.

2. PURPOSE

The purpose of the experiments was to study the behavior of the new humidity sensor and in particular it's susceptibility to chemical interference. The sensors were deliberately exposed to severe conditions to find their true capability.

3. METHODS

The main focus was set on testing the plain sensors' performance without any compensation from measurement electronics, heating systems or such other. The capacitance measurements were done by a LCR-meter. However, several tests were also carried out with the sensors connected to Vaisala humidity transmitters.

The sensors and devices in the tests were exposed to different environments. All the samples were removed from the test medium at certain intervals and taken to a calibration chamber at a Vaisala laboratory for measurement.

It has been assumed that the effects caused by chemical interference are unaffected by transportation and the delay formed between the measurement event and the removal of the test samples from the test medium. The test locations and transporting methods were carefully considered to eliminate or minimize all possible factors distorting the test results.

3.1. Measurement method

All relative humidity (RH) measurement points were created with a saturated salt solution system. The measurements were done with the same salt system each time. The salt systems are susceptible to temperature differences within the system. In addition there are other known sources of uncertainty. [2] In the described experiments the major source of error is believed to be the temperature differences measured between the salt column and the adjacent air. The variations in the laboratory temperature were believed to be the second most significant source of error. Taking into account these two sources of error it has been estimated that the measurements uncertainty is ± 0.3 RH% at 75%RH (sodium chloride) and ± 0.4 RH% at 97%RH (potassium sulfate).

The dry-point was created with a nitrogen atmosphere. An error estimation test showed the measurement uncertainty to be ± 0.2 RH%.

Capacitance values were measured using an HP 4284A LCR-meter. A simple linear model was used to convert the measured capacitance values to RH-readings. Accordingly the drift of a given sensor at a selected RH-condition was calculated using the derived equation 1:

$$\Delta RH = \left(\frac{C_x - C_0}{C_{75} - C_0}\right) \cdot 75\% RH - RH_x \tag{1}$$

where,

 $\Delta RH = drift of the sample sensor in a RH-point$ RH_x = relative humidity at the measurement pointC_x = sample's capacitance value at the measurement pointC₀ = sample's initial capacitance value at 0 RH%C₇₅ = sample's initial capacitance value at 75 RH%

All tests included commercially available polymer based capacitive sensors as reference sensors.

3.2. General performance test

To study the possible adverse effects of the new structure and the polymer the sensors were subjected to a general performance test. The test comprised of several different steps at different humidity levels. The initial measurement was done at 0%RH and 75%RH. It was then followed by a 97%RH step at which the sensors were kept for 16 hours. The measurement after the 16 hour exposure determines the drift which is an indication of the sensors high humidity stability and it is referred to as creep in this text. The sensors were then placed into 75%RH for one hour and 0%RH to determine the hysteresis and offset error. The sensors sensitivity was determined from the initial measurement at 0%RH and 75%RH.

3.3. Chemical tests

The chemicals for the chemical test were selected by choosing three chemicals with different molecule size and functional groups. The selected chemicals were Methyl ethyl ketone (MEK), Diethylether and Isopropyl alcohol. The tests were done with three different chemical concentrations. The chemical concentrations were carefully selected by studying earlier such experiments. The tests were run for approximately one month or until a 3%RH drift was seen at 75%RH.

The chemical exposure setup is depicted in figure 1. The set up is the same as described in the work done by Leppänen, Stormbom and Åström. [1]



Figure 1 – Chemical exposure setup

The setup includes a small bottle containing the test substance in an evaporation chamber. The chemical evaporates and mixes with nitrogen gas which flows through the evaporator. The chemical concentration can be derived from the chemical's loss of weight and the nitrogen flow rate.

3.4. Wood dryer

To test the performance of the sensor in a typical high level application the sensors were subjected to a wood dryer environment at Timberwise Oy located in Loimaa Finland. The test was carried out for over one year to get reliable long-term stability data. The temperature conditions at the dryer varied from -30°C up to almost 100°C. At the same time humidity varied over the whole scale. In a typical wood drying process the temperature is 85°C and the initial humidity level is nearly 100%RH. The chemicals that are evaporated during the process include such adverse chemicals as formic acid, acetic acid, other acids, alcohols, ketones, aldehydes, terpenes. The quantities and the exact chemical pallet vary depending on the type of wood used.

3.5. Outdoor test set-up

"The problem of outdoor measurements of humidity is that during cold intervals of the year, such as late autumn, winter and early spring the temperature often falls below the dew point, especially in the early morning. As a consequence dew sets on the surface of the thin polymeric sensor, which results at first in a non-usable signal because of a short circuit. When the dew has evaporated, some water-soluble ingredients in air such as sulphur or nitrogen compositions, which may have diffused inside the sensor layers, cause an irreversible drift of the sensor-signal." [3]

In previous experiments the sensors have been heated above the ambient temperature to keep the dew from settling on the sensors. In this study the sensors were kept unheated at all times. In addition, for comparisons sake, a heated version of the sensor was constructed and compared to other commercially available humidity devices.

The outdoor tests were done at two locations. One set of samples was placed in the Vaisala outdoor test field located at Vantaa, Finland. The other set was placed into a roadside weather station in Helsinki, Finland near a freeway junction were the amount of traffic reaches a maximum of 1500 vehicles per hour. The tests with unheated sensors were carried out for over one year. The test with heated samples was carried out for approximately 9 months.

4. RESULTS

The results show that the new sensor has less drift than the references in most of the experiments. The general performance of the sensor is essentially the same as the current version of Vaisala's Humicap® sensor. The chemical purge increases the sensors resistance to chemical interference.

4.1. General performance test

The general performance test shows that the new sensor's behavior is similar to a commercially available humidity sensor. The new structure and polymer have no adverse affects on the sensors general behavior. The results from the general performance test are shown in table 1.

Table 1 - General performance test

	Sample sensors		Reference sensors	
	average	3σ	average	Зσ
Creep (%RH)	0.3	0.1	0.2	0.2
Hysteresis (%RH)	1.2	0.1	1.0	0.2
Offset (%RH)	0.0	0.0	-0.1	0.0

The creep and offset values in both the reference sensors and the new sensors show little or no drift. The hystersis values are approximately 1%RH for both sensors. In addition, a similar sensitivity and base capacitance level were reached with no difficulty.

4.2. Chemical tests

The new sensors showed significantly less drift in all chemical tests compared to references.

The graphs in Figure 2 show the average drift of sensors exposed to three different concentration levels of Diethylether. The concentration levels were 1000ppm, 10000ppm and 28000ppm.



Figure 2 - Sensor drift in Diethylether at 0%RH

A higher chemical concentration causes more drift at 0%RH and 75%RH measurement points. Figure 3 shows the drift in 75%RH-point.



In the beginning there is a phase were the sensors withstand the chemical exposure with little or no drift. As more time passes the sensors exhibit a downward drift. The angle of the drift and the point at which this occurs depends on the concentration. This pattern was seen with the new sensors in all the tested chemicals.

The new sensors were highly resistant to Isopropyl alcohol compared to references. Drift was seen only in saturated Isopropyl alcohol vapor, see figure 4.



Figure 4 - Sensor drift in saturated Isopropyl alcohol

The test had to be extended over one month to see any noticeable drift in the new sensors while the reference sensors reacted to Isopropyl alcohol already within 24 hours exposure.

As in other chemical exposure tests, the new sensor also showed less drift than the references in MEK at 900ppm chemical concentration level, see figure 5.



Figure 5 - Sensor drift in Methyl ethyl ketone

A heating step simulating a chemical purge was done at the end of the test to see if the sensors would recover from the chemical exposure. Both the reference and the new sensor showed signs of recovery. The chemical purge was more effective for the reference sensors.

4.3. Wood dryer tests

Figure 6 shows the drift of sensors at five checkpoints during the wood dryer test. The values are averages of three sensors.



Figure 6 – Wood dryer test with reference sensors

The new sensors showed less drift than the reference sensors at all points of the experiment. In 400 days the new sensors drifted 0.5%RH while the references drifted over 1%RH at the 0%RH-measurement point and over 2.5%RH at the 75%RH-point.

A comparison test with sensors connected to a humidity transmitter with the chemical purge feature enabled was run simultaneously with the "cold" sensor exposure test. The chemical purge further reduces the new sensors drift, see figure 7.



There is essentially no drift after 400 days of exposure.

4.4. Outdoor tests

The outdoor test at the Vaisala test field was carried out for 430 days. Figure 8 shows the drift of each sensor after the exposure.





All sensors show less than 1%RH deviation from initial measurements.

The sensors placed into the roadside weather station were measured after 400 days. The test shows a similar behavior as in the test at the Vaisala test field.



Figure 9 – Outdoor test, roadside weather station. Three sensors deviation from initial measurement at 0%RH and 75%RH after 400 days exposure

The comparison experiment with heated sensors connected to a Vaisala humidity transmitter show that the new sensor (Device 1) has similar or less drift compared to reference sensors of different manufacturer's, see figure 10.



Figure 10 – Outdoor test, heated sensor at Vaisala test field. Drift from initial measurement at 0%RH and 75%RH.

Only the device with the new sensor and one comparison device show a drift of less than 1%RH in this outdoor test.

5. DISCUSSION AND CONCLUSIONS

The results from the experiments show that the new structure and the polymer make the sensor highly resistant to chemical interference. Similar results to those seen in Figure 2 and 3 are also observed for other chemicals. Using this information and setting the purge parameters accordingly the sensor can be used even in harsh chemical conditions.

The sensors perform well even without a heating system. Therefore the sensors good long-term stability can also be put to use in applications where heating or chemical purge are not possible. In less severe conditions the improved resistance to chemical interference can for example be used to lengthen the calibration interval.

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Author: Simo Ikonen, Lars Stormbom, Timo Ranta-aho . Vaisala Oyj, P.O.Box 26, 00421Helsinki, Finland, Phone: + 358 9 894 92629, Fax: + 358 9 894 92987, E-mail: simo.ikonen@vaisala.com