New planar trace humidity sensor

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The trace humidity of gases covering the range of frost point temperature t_f less than -13 °C (equivalent to a range of water vapor volume fraction w_v less than 2000 ppm_v or absolute humidity d_v less than 1.45 g m^{-3}) is an important parameter of many gaseous precursors, final products and used carrier gases in chemical production, semiconductor industry and aerospace technology. There is a permanent need for on-line measurement of humidity as a quality parameter during the manufacturing process of these goods. Coulometric sensors are a very robust and cost-effective tool for determination of trace humidity. This sensor principle was first described by Keidel [1] and several commercial available advanced devices are based on this principle [2, 3]. However, for higher precision and traceability, the sensors themselves and the measurement procedures have to be improved as well as calibration systems are required.

Whereas a traditional coulometric sensors consists of bifilar wire wound on glass rod, the new developed planar sensor elements (HUMITRACE sensors) exhibit interdigital platinum electrodes on an alumina substrate. For activation, the electrodes were coated with diluted phosphoric acid. The phosphoric acid dehydrates in a dry gas to form a thin homogeneous tetra phosphorus decaoxide (P_4O_{10}) film. The planar sensor element is protected by a polymer membrane to diminish the influence of variating gas flow. Small amounts of water penetrate through the membrane and were absorbed by this hygroscopic coating. The P_4O_{10} will be hydrolyzed and the formed phosphoric acid will be electrolysed at a voltage of at least 2 V (DC) on the electrodes to form hydrogen and oxygen. The measured current I is proportional to the water content in the test gas according to the Faraday law [4, 5].

Two test gas calibration instruments were developed: one is a laboratory facility and the other is a device for on-site calibration. The laboratorial test gas preparation facility (THG11) generates gases of defined trace humidity and permits to detect the signals of six coulometric sensors simultaneously. The THG11 generates test gases by mixing dry and moist gas stream. These two gas streams are controlled by mass flow controllers. The total test gas flow is split into seven test gas streams, six going through the electrolytic sensors and one flowing into the precision dew point hygrometer (chilled mirror principle, S4000 TRS, Michell Instruments) which is used as reference device. For on-site calibration, permeation was applied for humidification of test gases and a method was developed. This method demands a constant and accurately known temperature and gas flow rate. Water vapor permeates through a polymer tube (polyamide PA 12) into the pristine carrier gas flow and raises the water concentration. For example, humidity of pristine carrier gas of t_f = -59.7 °C, corresponding to a water vapor volume fraction of w_v = 11.1 ppm_v, was increased to w_v = 46.0 ppm_v (t_f = -48.8 °C) and w_v = 87.2 ppm_v (t_f = -43.3 °C). The frost point temperature was calculated from the permeation rates and the measured values differ from each other by 0.03 °C to 0.37 °C. The wetting of the pristine gas flow of arbitrary trace humidity by two different, exactly known humidity concentrations enables functional tests and on-site calibration of sensors according standard addition method.

The signal of the HUMITRACE sensors were evaluated in the above described laboratory facility at frost point temperatures in the range of -80 °C to -30 °C (see Figure 1). The test gas' frost point temperature is measured with an uncertainty (coverage factor, k = 2) from 0.4 °C to 0.63 °C. The combined uncertainty (k = 2) of the determined current is 10.5 %. Figure 2 presents a characteristic calibration curve of a coulometric sensor where the frost point temperature depends on the current. Out of this data the frost point temperature detection limit of the measurement was calculated to be (-78.5 ± 0.63) °C corresponding to a current of (13.2 ± 1.5) µA. The dynamic behavior of the coulometric sensors can be characterized by a response time (t_{90}) in the range of 15 to 30 min and a recovery time (t_{10}) of 10 to 30 min. The sensor element can be continuously used up to 1×10^6 µA h or up to 12 month, respectively, before regeneration is advisable.

The new HUMITRACE sensors can be manufactured in a cost effective way and grant in relation to the calibration facilities a higher precision and reliability of industrial trace humidity measurements.

Figures

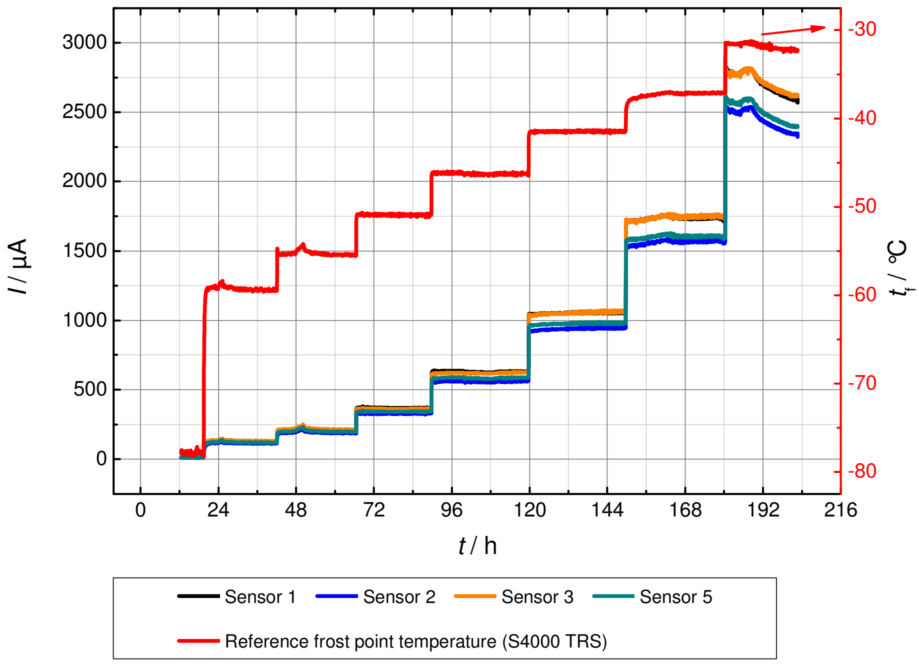

Figure 1: Signal curve of four coulometric sensors (left y-axis) and the reference humidity measurement (right y-axis) over measuring time.

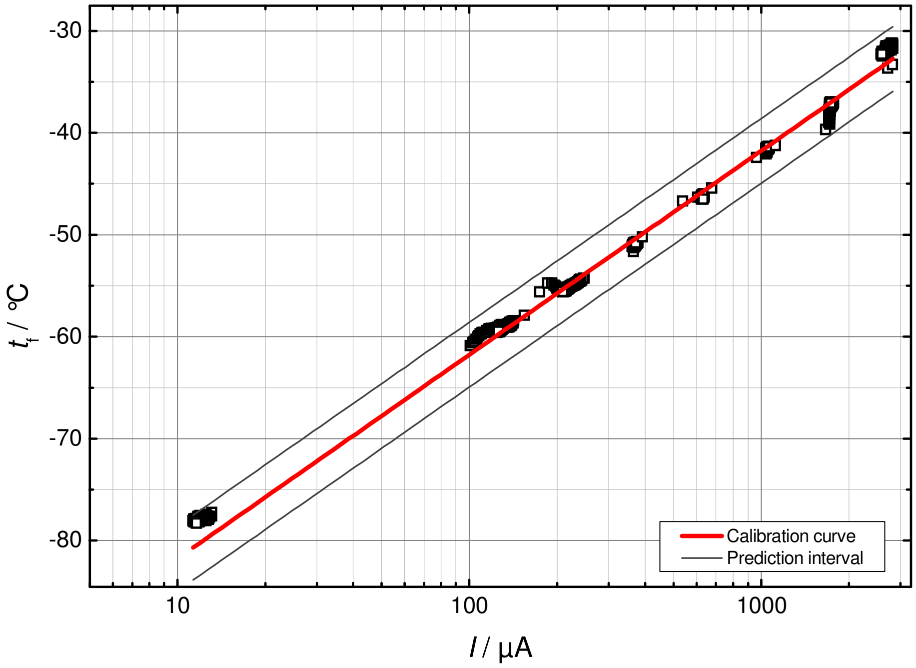

Figure 2: Calibration curve of a coulometric trace humidity sensor (sensor 1 in Fig. 1) for (20 ± 2) NL/h carrier gas flow of purified compressed air.
Uncertainties (coverage factor $k = 2$): $U(t_\mathrm{f})$ = 0.4 to 0.63 °C and $U(I)$ = 10.5 %.

References

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