

## A new calibration technique for industrial gas analyzers \*

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### Abstract

The regular calibration of the zero point and span is one of the main problems in industrial gas analyzers. Due to the delicate parts in the analyzer there is a decrease (or increase) in the gas sensitivity and a drift of the zero point. The critical parts are mainly the light source and the detector in photometer based analyzers, or the sensor element in sensor based analyzers. We have developed a new calibration technique for a combined instrument (photometer and sensor element) using both an internal calibration standard and ambient air as a reliable cost free standard for a constant oxygen concentration. Furthermore the difference between a selective gas standard and a nonselective standard (e.g. beam trimmer) in a photometer based analyzer can be shown. From our research we have found that the stability of this internal standard is comparable with the standard from a gas cylinder.

**Keywords:** Infrared gas analyzer; Ultraviolet gas analyzers; Analyzer calibration/validation, Internal standard; Diagnostic

### 1. INTRODUCTION

In the past (more than 10 years ago) gas analyzers for the industrial process control were mostly based on analogue electronics. The function of these electronics was to transform the weak sensor signals into a standard output voltage, e.g. 0-10 volts, or a standard output current e.g. 4-20 mA. Due to the progress of microelectronics in the 80's microprocessor based electronics are used more and more in analytical instru-

ments. Compared to other products (e.g. laboratory equipment, domestic appliances, etc.) this application came very late. The main impediment to the implementation of microprocessor-technology was susceptibility to electromagnetic interference (EMI). This problem has now been solved so we can use the microprocessor for many tasks other than only giving a digital display. The advantage of microprocessor based analyzer-electronics is that it can be used for (i) self calibration, (ii) self check (diagnostics), (iii) error compensation, and (iv) multi component measurement.

The aim here is to reduce the maintenance costs. In this paper an instrument (gas

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analyzer) with a new calibration und self check technique based on efficient microprocessor electronics will be described.

2. PROCESS ANALYSIS SYSTEM

A complete Process Analysis System consists of a probe, cooler, pump, filter, valve, flowmeter and analyzer as illustrated in Fig. 1. The maintenance of each part is different. The least reliable parts of an analysis system are the cooler and the flowmeter-valve-unit. For these critical parts a maintenance interval of up to twice a week is necessary. Since the occurrence of the fault in these parts is statistically distributed, the operation personnel (service staff) have to look after the analysis system once a day, to prevent a fault

in the complete system. The biggest problem in the system occurs if the cooler has a fault. In this case when the wet process gas enters the sensitive analytical cell it may damage it.

Figure 2 shows a flow chart of an Autonomous Process Analysis System (APAS). Compared to the prior analysis system (Fig. 1) a central control unit (CCU) is added to check all the critical parts. The CCU gets the information from sensors which are installed in the different parts (cooler, filter etc.). If an unacceptable situation occurs (e.g. cooler fault) the information will be shown on the display, the service staff will be simultaneously informed and can attend to the problem. Due to this advantage there is in theory no need for regular maintenance intervals. In practice the APAS lengthens the maintenance interval by up to three months [1]

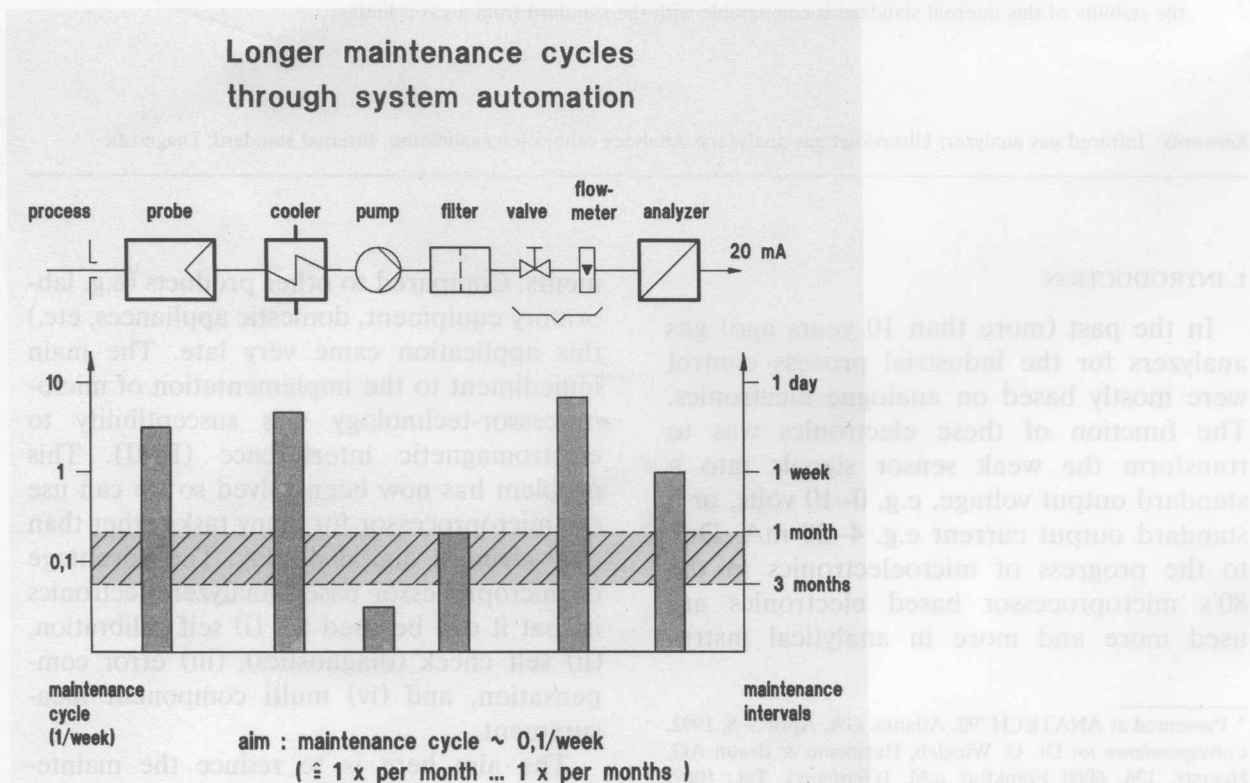


Fig. 1. Assembly of typical gas preparation components related to maintenance interval in a conventional gas analysis system [1].

**Autonomous process analysis system**

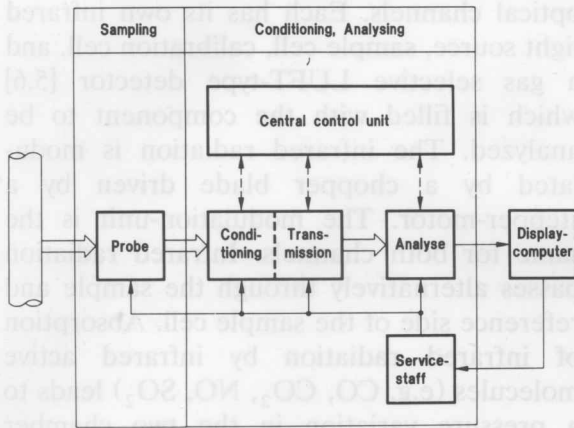


Fig. 2. Block diagram of an autonomous process analysis system.

(depending on the application). Figure 3 illustrates the arrangement of four analyzers to measure SO<sub>2</sub>, NO<sub>x</sub>, CO and O<sub>2</sub> in an exhaust gas. This expensive arrangement can be used to control a power plant (e.g. coal-fired) or an incinerator. For this task very precise instruments with a high accuracy 1% are required. Other applications include:

- combustion optimization of small burners
- monitoring of bio-processes
- control of food stores atmosphere
- monitoring of charcoal filters
- motor adjustment
- room monitoring (warehouse, garages, tunnels, etc.)

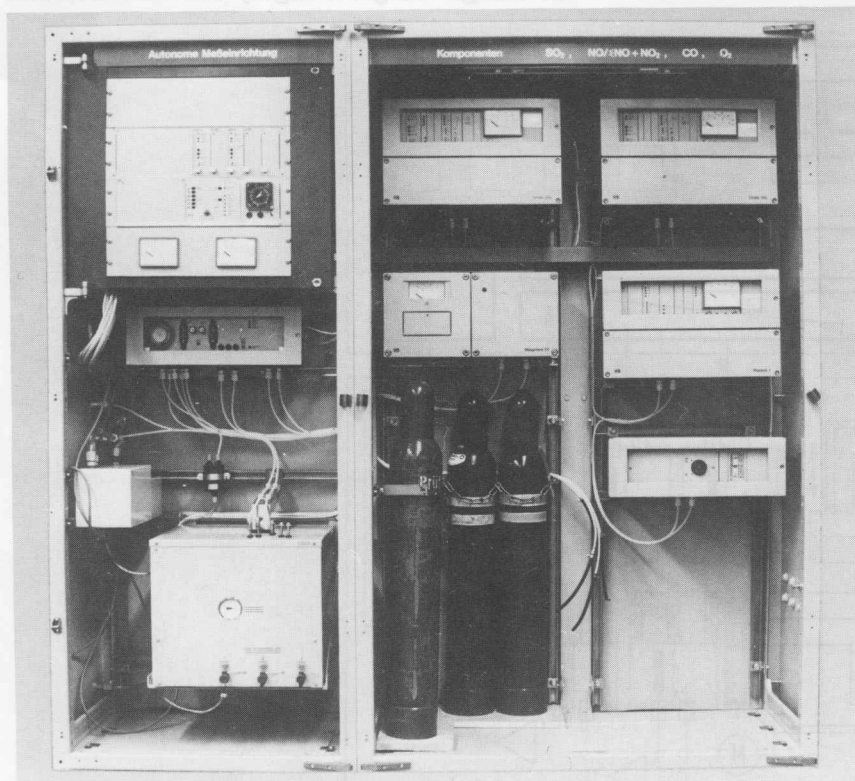


Fig. 3. Assembly of four gas analyzers (RADAS, URAS, MAGNOS) in an autonomous process analysis system including gas supply for regular calibration. (Application in a power plant.)

which require portable gas analyzers with acceptable accuracy (2-3%) and reduced cost as well. For portable analyzer systems it is necessary to integrate the sample gas conditioning parts (dust-filter, pump, flow-control/check, etc.) into the instrument.

3. INSTRUMENT DESIGN

Figure 4 shows a block diagram of the instrument assembly. In the upper part the electronics with a digital display, an operator keyboard and the information processing unit are shown. In the lower part we can see the optical arrangement of the NDIR-photometer (Non-Dispersive InfraRed) [2,3] and the gas contact parts including the sample gas preparation [4].

The photometer consists of two separate optical channels. Each has its own infrared light source, sample cell, calibration cell, and a gas selective LUFT-type detector [5,6] which is filled with the component to be analyzed. The infrared radiation is modulated by a chopper blade driven by a stepper-motor. The modulation-unit is the same for both channels. Infrared radiation passes alternatively through the sample and reference side of the sample cell. Absorption of infrared radiation by infrared active molecules (e.g. CO, CO<sub>2</sub>, NO, SO<sub>2</sub>) leads to a pressure variation in the two chamber LUFT-detector, which is measured as a capacity change. The corresponding signal change is proportional to the gas concentration in the sample cell. Due to the two chamber design we get a highly selective detector

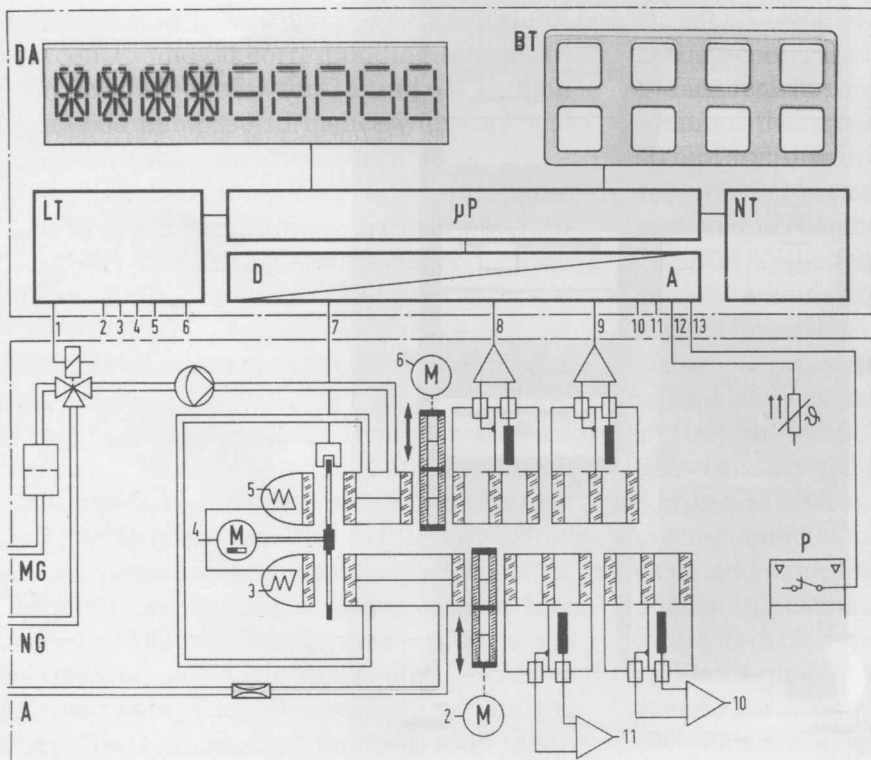


Fig. 4. Block diagram of the instrument assembly (URAS 10/Hartmann & Braun AG).

with reduced cross sensitivity to other gases [5]. The detector has an entrance and exit window so that transirradiation is possible. Hence a second detector can be installed behind the first detector. The second detector is filled with a different gas to the first detector to measure another component of interest in the gas mixture. Each channel contains up to two detectors thus it is possible to measure up to four different gases in one instrument [7]. Since the cross sensitivity of some gases is unfortunately not negligible due to the overlapping of infrared-absorption bands it has to be corrected. The levels of the gas of interest and the interfering gas are measured and from this information the microprocessor calculates the correct levels. One example of this problem is the detection of sulphur dioxide in exhaust gas with interfering water vapour present. In this case we noticed a cross sensitivity of water vapour to the  $\text{SO}_2$ -measurement in the range of 35 ppm of  $\text{SO}_2$  in the read-out. With internal compensation this error can be drastically reduced down to less than 1 ppm.

In the analyzer part a temperature sensor und pressure sensor are also installed. With these two sensors temperature und pressure compensation are feasible, and the accuracy and stability of the analyzer output are improved. A flow-check can also be carried out by the pressure sensor. The pump transports the sample from the process (probe) through the dust-filter to the sample cell. In the exit of the cell a capillary-tube is built in. Due to the flow through the capillary-tube the pressure increases proportionally to the flow. Hence the flow can be monitored by the pressure sensor [4].

In addition to the Infrared-detection of  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{NO}$ , etc. for combustion optimization or room monitoring the simultaneous detection of oxygen is required [8]. Unfortunately oxygen has no absorption bands in the

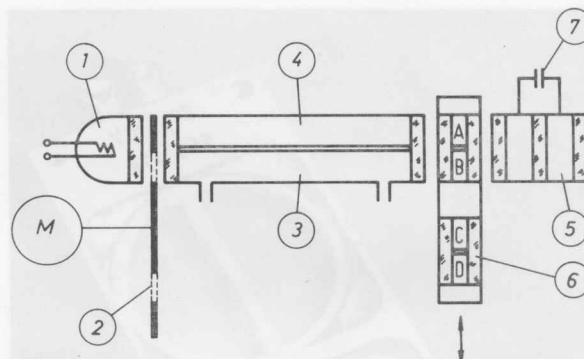


Fig. 5. Optical arrangement of the photometer. M-stepper motor, 1-infrared light source, 2-copper blade, 4-sample side, 3-reference side, 6-calibration cell, 5-two-chamber LUFT-type detector, and 7-membrane condenser.

infrared region, so detection by a photometer is not possible. For this purpose an electro-chemical sensor is installed in the instrument. The sensor is based on a HERSCH-cell [9] and generates a sensor signal proportional to the oxygen concentration.

#### 4. CALIBRATION TECHNIQUE [11]

Between the sample cell and the LUFT-Detector a calibration cell is fitted (see Figs. 5 and 6). The calibration cell is subdivided into four chambers (A-D). Figure 5 shows the position of the calibration cell during the measuring time. Chambers A and B are filled with nitrogen because nitrogen has no infrared-absorption bands (i.e. no interference). During the calibration phase the cell moves so that chambers C and D are opposite the sample cell. Chamber C corresponds to the sample side and is filled with a very stable concentration of the gas of interest. Chamber D corresponds to the reference side and is filled with nitrogen. The known gas concentration (chamber C) leads to a defined value of absorption in the sample path and simulates a gas concentration in the sample cell. Since the gas concentration in

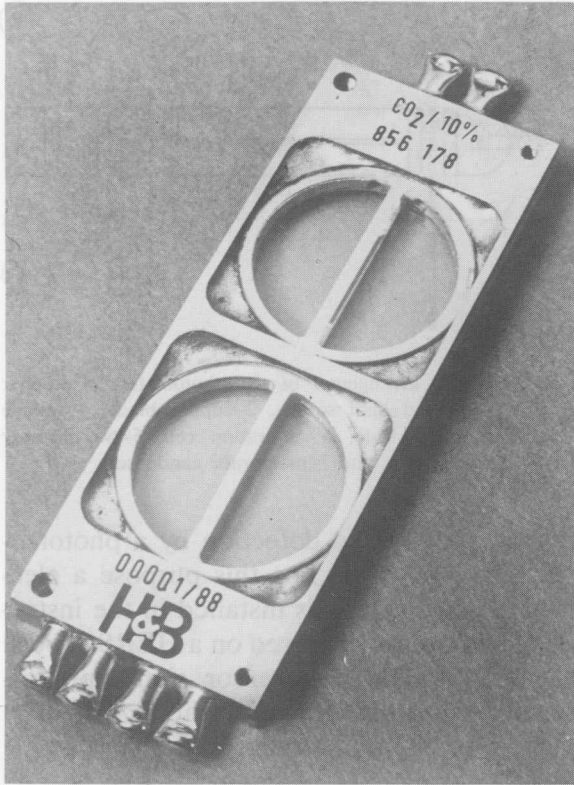


Fig. 6. Sealed calibration cell.

the calibration cell is very stable it is possible to calibrate the whole analyzer with this method. Due to aging of the infrared-source (loss of intensity) and the LUFT-detector (loss of gas) regular calibration is necessary. Many instruments use a beam trimmer to correct the photometer drift. This kind of correction is only exact for the infrared-source but not for the detector drift.

Figure 7 illustrates the sensitivity change of a LUFT-detector versus the concentration of filling gas checked by selective (gas)-and non-selective (beam trimmer)-standard. The correlation between these two curves is low and leads to an calibration error shown in Fig. 8. The calibration error with a non-selective standard is 100% if the filling concentration is reduced down to 40% due to

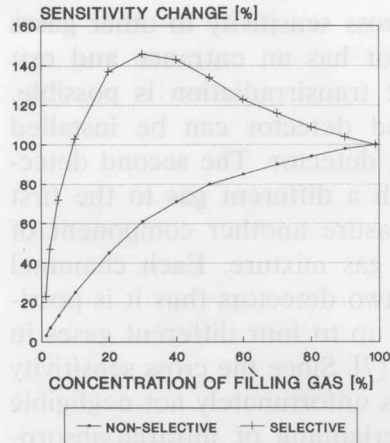


Fig. 7. Sensitivity change of the LUFT-detector versus the concentration of filling gas (carbon monoxide).

leakage or gas absorption on the walls inside the detector. The selective standard is very exact compared to the non-selective standard thus the residual error can be neglected. For a sufficient long term operation the stability of the gas concentration inside the calibration cell must be stable as well. To achieve this goal it is necessary to join the windows (CaF<sub>2</sub>) and the brass body together without adhesive.

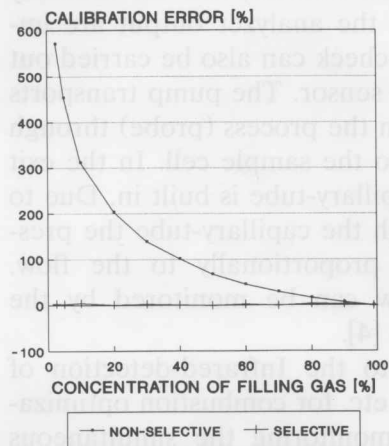


Fig. 8. Calibration error of the NDIR-photometer due to the concentration change of filling gas.

Figure 6 shows the hermetically sealed four chamber calibration cell. The calcium-fluoride-windows were metalized and soldered together with a gold coated brass body. The stability of this patented cell is very high. We noticed a weak variation in the analyzer response over more than one year in the range of 2-3% as shown in Fig. 9. The observed stability is as high as the stability of a standard sample from a gas cylinder, thus operation and calibration of the described analyzer over one year is possible without the use of test gas.

The signal output voltage of the NDIR-photometer during the calibration procedure is shown in Fig. 10. In the course of "Measurement Phase I" we sometimes get a faulty analytical result. In the case of a regular automatic or manual calibration the solenoid valve inside the instrument switches the gas flow from *sample gas* to *zero gas*. Zero gas can be unpolluted air or nitrogen. Then the signal output decreases steeply to a low level. If this "zero value" is unequal to zero, the zero-point will be corrected. Now the calibration cell slides automatically into the new position (chamber D corresponds to the sample side). Due to the calibration gas in chamber D the output voltage increases rapidly.

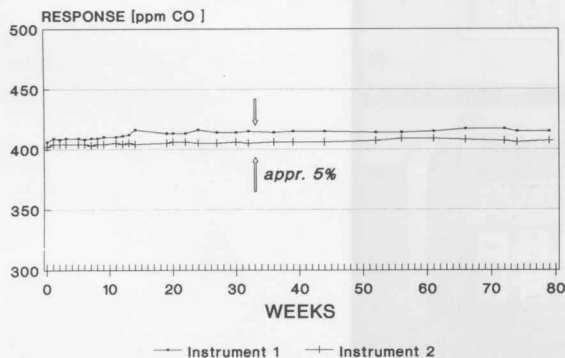


Fig. 9. Variation in drift of the calibration cell compared to the gas bottle (calibration of the analyzer by the calibration cell and checked by the test gas).

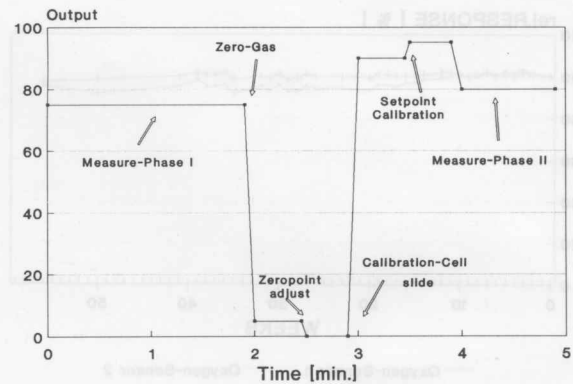


Fig. 10. Output signal of the NDIR-photometer during the calibration procedure.

The microprocessor compares this value with a stored value from the last calibration and corrects it if necessary as shown in the graph. After the calibration the calibration cell slides in to the former position and the solenoid valves switches back to sample gas and to "Measurement Phase II".

The calibration of the oxygen sensor is similar to the photometer. In this case fortunately no separate zero gas and test gas are required. The zero point of the oxygen sensor is absolutely stable, that means without oxygen we noticed no chemical reaction, thus the output voltage of the sensor must be zero. During the zero calibration of the photometer, air (containing 20.946 vol.% oxygen) flows through the instrument, so that the sensor drift (sensitivity/span) can be corrected automatically [7]. In this case the ambient air is used as a reliable cost free calibration standard for constant oxygen concentration. The long term stability of this oxygen sensor was observed over one year measuring an exhaust gas (oil-fired power plant) without automatic calibration. The result of this test including two different sensors is shown in Fig. 11. The variation of the relative response (normalized to 100%) is in the range of 5-10%.

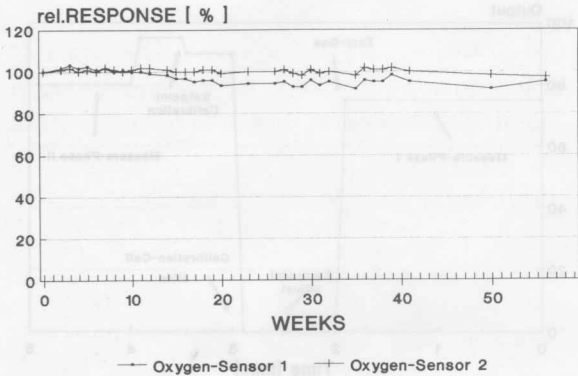


Fig. 11. Stability of two different oxygen sensors during a long term test in a oil-fired power plant.

The calibration interval can be selected between 1 h and 672 h for both methods (sensor and photometer) with the keyboard in the front panel of the instrument (see Fig. 12).

4. CONCLUSIONS

Maintaining process analyzers is an important area in the field of maximum operating efficiency and process optimization [10]. In this paper a new design of an industrial gas analyzer was presented. Computer-based electronics made a self checked and self controlled operation possible to reduce the maintenance costs. Compared to an autonomous process analysis system the presented instrument (URAS 10) makes new applications possible. The advantage of this instrument design is that for an operation no further equipment is necessary. Due to the internal calibration standard (calibration cell) no test (zero) gas supply is required.

The instrument is being tested by the German-Institute TÜV (Technischer Überwachungs Verein), the American FM (Fac-

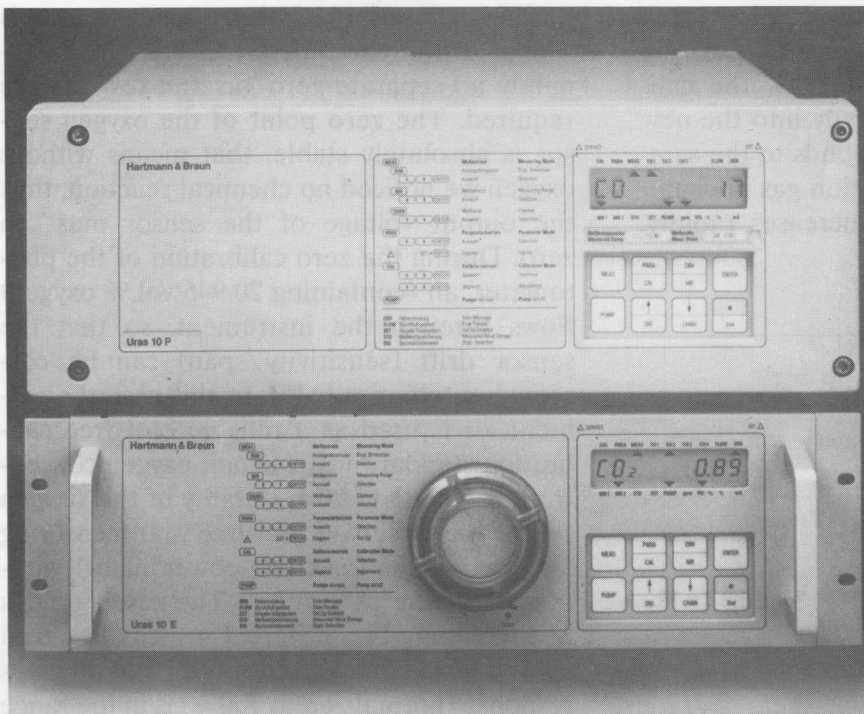


Fig. 12. Design of the URAS 10-instrument for process application (top) and rack-mounting (bottom).



tory Mutual Research) and the CSA (Canadian Standards Association). The present results of the TÜV shows a sufficient stability of the calibration cell over several months for carbon dioxide (CO<sub>2</sub>) and carbon monoxide (CO).

A similar design of the calibration cell can be successful used in other instruments as well. We have tested such a cell in an improved ultraviolet photometer (RADAS/H & B) for several years with no significant loss of the encapsulated gas (nitrogen oxide/NO) [12].

In a future paper this improved ultraviolet photometer will be described.

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