Automatic Calibration of the METREX Analyzer

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The implemented system of the analyzer automatic calibration is computer-controlled. It enables automatic measurement and evaluation of analyzer data and testing the dynamic behaviour of the analyzer. The automatic device for preparing of a gas mixture with desired volume fraction was constructed. The influence of external conditions on the static and dynamic behaviour of the device and of the analyzer was measured during the calibration.

The analyzer METREX DH [1] has been used for volume fraction measurement of combustible gases and vapours in the atmosphere of such space, where explosive mixtures can arise. METREX DH is usable for the volume fraction measurement of hydrogen, earth gases, volatile gases, organic solvents, *etc.*

The analyzer works on the principle of the catalytic combustion of the followed material on the electric measuring thread, which has catalytic properties and is connected to the balanced Wheatston bridge [2, 3].

The apparatus has five independent scales, which are calibrated for C_2H_2, ethanol, hexane, benzene, and n-heptane. The maximal extent of each scale is determined by the lower explosion boundary of the given matter in the atmosphere. For the ethanol—air mixture, it is $\varphi = 3.3$ %. The analyzer voltage extent is 0—10 V and analyzer has an automatic compensation from 20 °C to 250 °C with the possibility to adjust the signalization of overstepping the desired volume fraction value. The measurer is made as nonexplosive Ex 3 HD. The precision is 5 % of its range. There are several ways how to calibrate the analyzer [3]. For the calibration of the mentioned analyzer, the microcomputer-controlled system of automatic calibration was applied. The advantage of this system is the total automation of the calibration process, the possibility of checking in several points and directly in the technological process. If the analyzer is used for production process control, the periodic analyzer calibration ensures the higher accuracy and reliability. In such a case, it is necessary to double the analyzer. In that time, when the first analyzer measures, the second one is calibrated and *vice versa*.

EXPERIMENTAL

For the preparation of gas mixtures with definite volume fraction of vapour of volatile liquids, the bubbling of the air through the thermostated vessel full of liquid was used (Fig. 1). In the vessel, the con-stant level and temperature at the constant pressure of air are hold. The dosing device works on the principle of the frequency dosing of the constant amount of an analyzer gas into the carrier gas stream [4]. The dosing device was constructed from the pneumatic system DRELOBA (Fig. 2) [5] and consists of four switches, which are controlled by rectangular oscillating. The volume fraction of the gas mixture is proportionate to dosing frequency. The block scheme of the device is shown in Fig. 1. The switches of DRELOBA system are two-positional (open — close) and based on the so-called two-membrane relay. For the ensuring of correct functioning of the switches, they have to be controlled simultaneously by the control signal and by its negation. The dosing device was constructed of two members TOR-Baustein type 0.000023 : 18. These members were fed by constant pressure 130 kPa. This pressure is necessary for supplying of the negator and the electro-pneumatic transformer which is used for the transformation of the two-value electric signal with adjustable frequency to the pneumatic signal used for the control of the switches. It is necessary to separate galvanically the computer control signal with TTL level and to amplify it to the level 0—24 V by the amplifier (Fig. 2). The block scheme of the automatic calibration analyzer with the help of the microcomputer is in Fig. 2. The operator communicates with the computer, defines the working regime, the volume fraction extents in which the calibration will be done, the sampling period for the volume fraction measurement by the analyzer and the conditions for statistic evaluation (numbers of measurements, correlation interval, *etc.*). The analyzer signal with the extent 0—10 V was filtered by a hardware filter and transformed by analog-digital transformer (disc ADC-1 in the microcomputer SAPI-1). The control signal was transmitted from the computer by programable timer on the disc RTC-1. The information from the analyzer is computer-evaluated by the program with the block-diagram in Fig. 3.
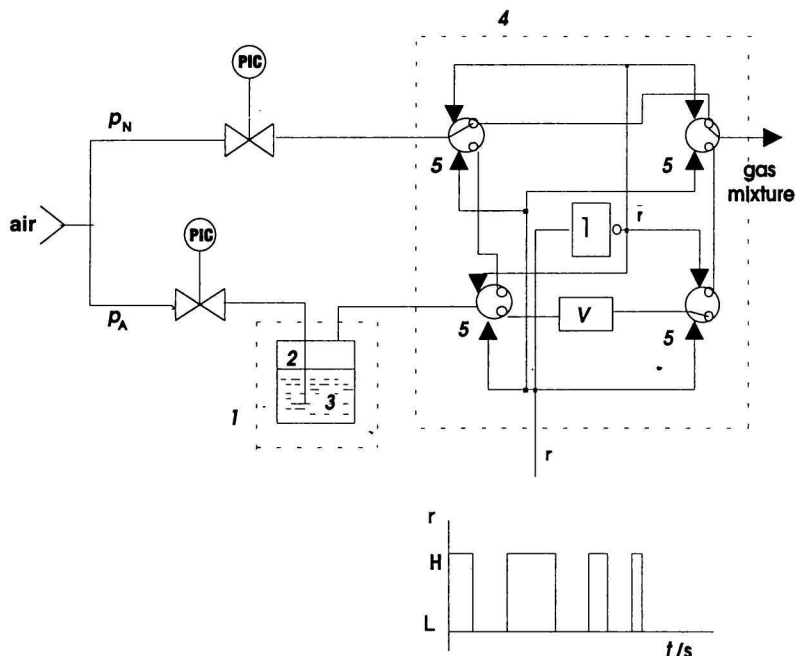
Fig. 1. Block scheme of the device for automatic preparation of the gas mixture with desired volume fraction. *1.* Thermostat; *2.* stocking of ethanol with constant height level; *3.* frit; *4.* automatic dosing device; *5.* switches system DRELOBA with two positions; r — control signal with two values; $\bar{r}$ — negation of control signal; L — low level of control signal; H — high level of control signal; PIC — regulation of pressure with indication of adjusting level; p_N — pressure in the first branch of dosing device; p_A — pressure in the second branch of dosing device; V — volume of dosing capacity.

The program enables to choose the regime 1 and 2, *i.e.* to obtain static or dynamic behaviour of the analyzer. The static behaviour gives the information about steady states, when the mixture with definite volume fraction obtained in the dosing device enters into the analyzer. Then, the steady output of the analyzer is measured and compared with the zero value which was obtained at the beginning of calibration, when only the air was supplied into the analyzer. The mean zero value is subtracted from each measured value and so the influence of shifting of zero value is eliminated.

$$X = X_{meas} - \frac{1}{n}\sum_{i=1}^{n} x_{i0} \qquad (1)$$

The sets for each volume fraction (max. 100 measurements) are then statistically evaluated. These quantities are then followed: the mean value in the chosen time interval, dispersion, arithmetical mean average error, excess coefficient, autocorrelation function for the given correlation interval [6, 7]. From these characteristics, it is possible to find out whether the error exists with the Gaussian distribution, or whether the oscillated part exists in the spectrum (autocorrelation function), what the standard deviation is and what the maximal deviation of mean is and whether the analyzer has the time-varying mistake.

The measurements with big mistakes were excluded *via* the Grubbs test. The whole procedure is done in such a way that the measurements are ranked in the increasing direction and the lowest and highest values are calculated according to the following equations
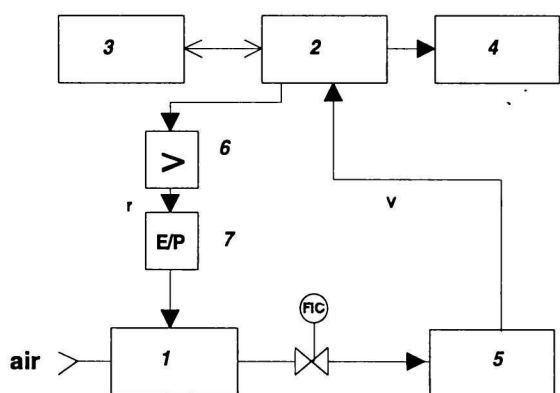

Fig. 2. Block scheme of connection for automatic calibration analyzer METREX DH. *1.* Device for the automatic preparing of the gas mixture; *2.* microcomputer SAPI-1; *3.* operator; *4.* printing device; *5.* analyzer METREX DH; *6.* efficient amplifier of control signal; *7.* electropneumatic transducer. r — control signal; v — output signal from analyzer (1–10 V).
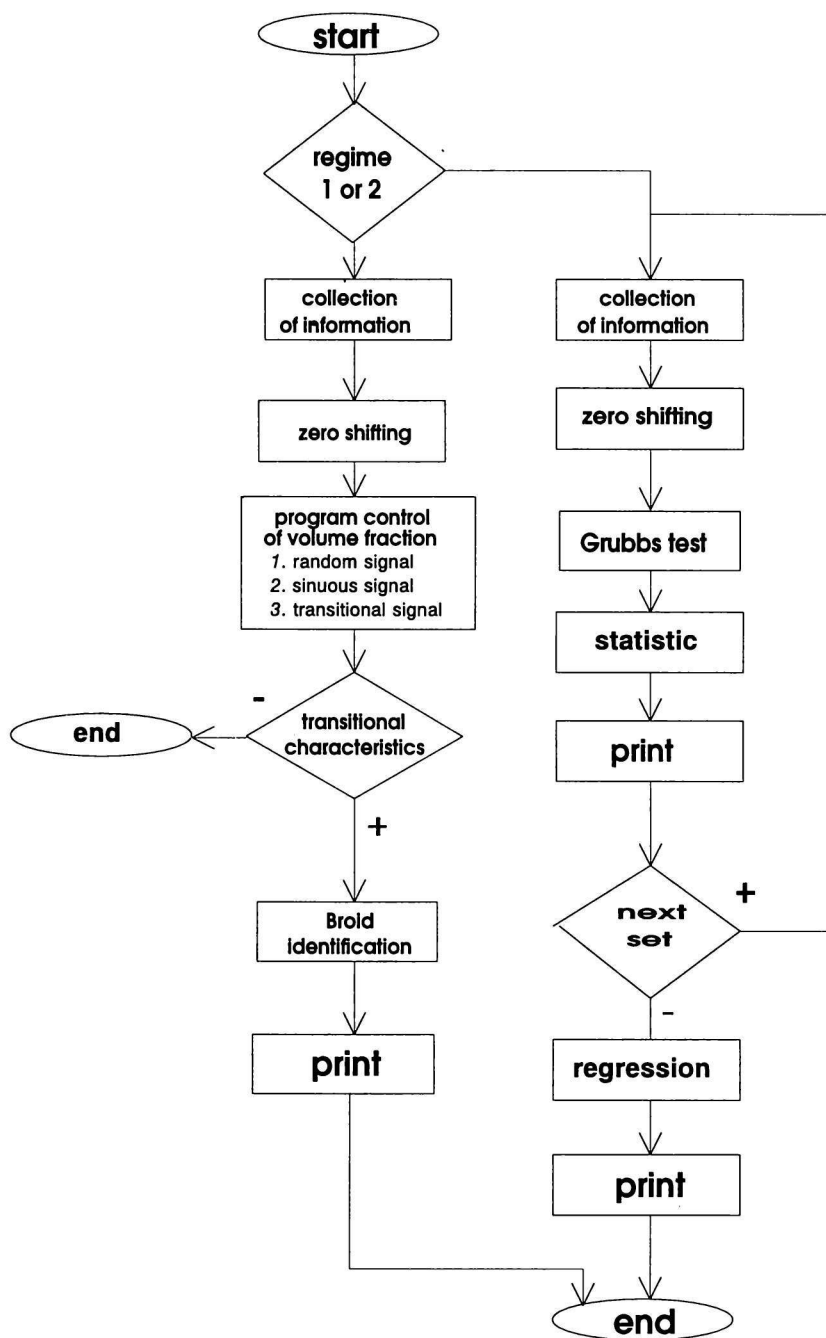
$$G_{min} = \frac{\bar{x} - x_{min}}{\sigma\sqrt{(n-1)^{1/n}}} \qquad (2)$$

Fig. 3. Evolutionary diagram.

$$G_{max} = \frac{x_{max} - \bar{x}}{\sigma\sqrt{(n-1)^{1/n}}} \qquad (3)$$

where $\bar{x}$ is the average value of measurements, σ is the standard deviation, and n is the number of measurements. If G_{max} and $G_{min} \gtrless G\ (n,\ \alpha)$ are valid, then x_{max} and x_{min} can be excluded from the set (α = 0.05 is the level of significance), which is usually used for testing.

During calibration the regression curve is calculated by the method of the least squares in the equations

$$y = a + bx \qquad (4)$$

$$y = kx \qquad (5)$$

$$y = c + dx^2 + ex \qquad (6)$$

where x is the real value of volume fraction of the gas mixture which goes into the analyzer in amount

0—100 % and y is the determined value of volume fraction (entrance signal of analyzer). Standard deviations from the regression curve are then calculated. For determination of the calibration curve from the determined values the relationship with the lowest standard deviation is utilized which is used for the real volume fraction calculation from the obtained members. In regime 1 dynamic characteristics of the analyzer as well as of the equipment for taking and adjustment of samples are investigated. This regime allows the programmed control of the volume fraction. The output from the analyzer in dependence on time when the mixture is prepared according to the program is illustrated in Fig. 4, curve 2 (see eqn (7))

$$\varphi = \varphi_0 \sin \omega t \qquad (7)$$

The prepared volume fraction can be used for the measurement of the frequency characteristic. Further the volume fraction of the prepared mixture is oscillating accidentally around φ_0 (Fig. 4, curve 1). The step response is shown in Fig. 4, curve 3. From the step response the transfer function can be calculated, which is characterized by its mathematical expression in the shape

$$F(s) = \frac{Ke^{-s\tau_d}}{(\tau s - 1)^n} \qquad (8)$$

where K is the gain of the system, τ_d time delay, τ time constant of the system, n order of the system, s operator.

The Broid method [7, 8] was used for the identification of the transfer function from the step response. The program for microcomputer SAPI-1 was prepared in the Basic G.
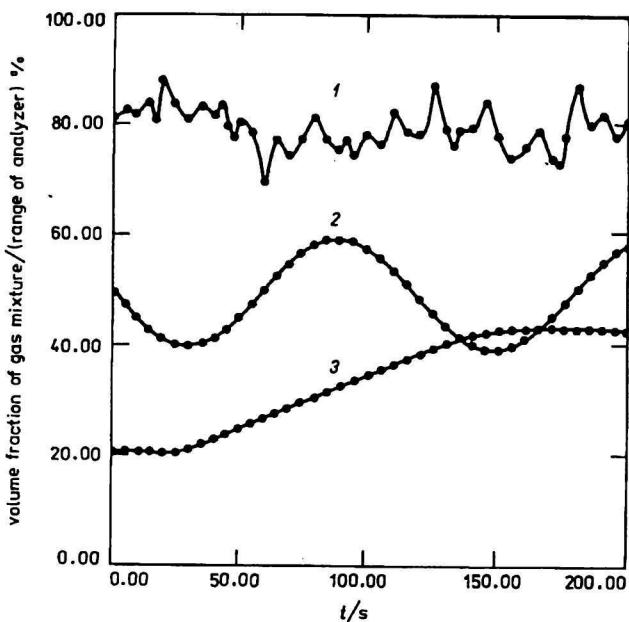
RESULTS AND DISCUSSION

The results of measurement using analyzer METREX DH in dependence on the dose frequency are illustrated in Fig. 5 and given in Table 1.

The data from analyzer METREX DH were taken every 2 s and the number of measurements for each frequency value was $n = 20$. Normative autocorrelation function for each value of frequency for the interval of correlation $t_c = 10$ s was calculated together with smoothing curve of the autocorrelation function.

$$y = e^{\beta t} \qquad (9)$$

By frequency 2 Hz the highest relative standard deviation 4.23 was measured and the data distribution had not Gaussian distribution function. This fact can be caused by the highest mistake of the analyzer at the beginning of the scale and also by insufficient mixing of the mixture at the low frequency. Partial means, calculated in certain time intervals do not differ significantly and they are not included in Table 1. This indicates that the prepared gas mixture was not changed with the time.

As seen in Fig. 5 the course of the volume fraction as a function of frequency was linear in the region 2—7 Hz. The effect of followed parameters during preparation of the gas mixture was also tested: the temperature and the height of ethanol level in the storage container and the influence of the pressure p_N on the volume fraction of the prepared mixture. The obtained results are shown in Figs. 6—8. The change of the height level by about 10 % causes the proportionality error of 10 % (Fig. 6). The pressure change about 20 % results in an additive mistake of about 10 % (Fig. 8). The temperature of ethanol has the highest effect on the volume fraction

Fig. 4. Testing signals at program control of volume fraction *via* dosing device. *1.* Random signal; *2.* sinuous signal; *3.* transitional characteristic.
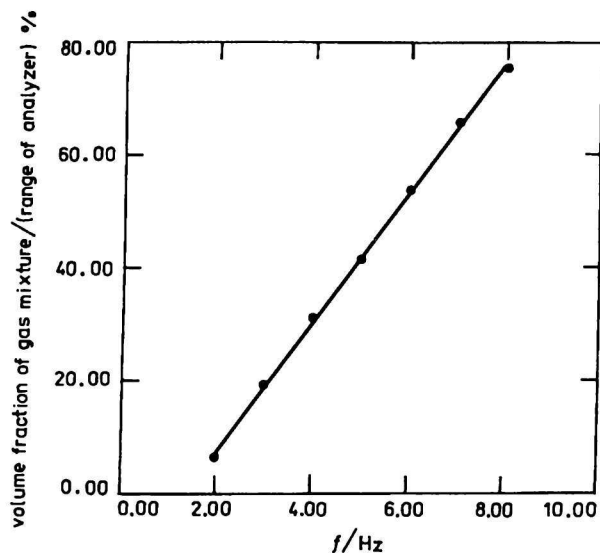

Fig. 5. Dosing device characteristics. Common conditions of measurement: carrier gas: $p_N = 2.4$ kPa; analyzed gas: $p_A = 8.7$ kPa; temperature: $\theta = 22$ °C; level: $h = 10$ cm.

Table 1. Characterization of the Prepared Mixture in Dependence on the Dose Frequency

f/Hz	Relative standard deviation %	Coefficient of asymmetry	Coefficient of excess	Dispersion %	β
2	4.23	2.57	6.36	0.08	− 0.43
3	0.96	− 0.58	− 1.74	0.03	− 0.06
4	0.505	1.38	− 0.06	0.02	− 0.14
5	0.697	0.219	− 1.27	0.08	− 0.04
6	0.465	− 0.216	− 0.85	0.06	− 0.037
7	0.910	0.31	− 1.5	0.35	− 0.144
8	0.914	− 0.11	− 1.65	0.47	**

β is the coefficient of smoothing autocorrelation function. ** For frequency 8 Hz smoothing autocorrelation function was not calculated; there was an oscillated part in the spectrum.

of the prepared mixture. As it is evident from Fig. 7 the influence of the volume fraction of gas mixture at constant frequency 3 Hz, p_N = 2.4 kPa, p_A = 8.7 kPa, and h = 10 cm is not linear. It can be concluded that for the proper function of dosing equipment it is necessary to stabilize pressures p_N, p_A and level height h as well as to thermostate the storage container with ethanol to the temperature 22 °C.

The dynamics of the system, which is important for feedback control system was also investigated. Transfer function was identified from the step response. This characteristic was compared with the real characteristic in Fig. 9. The analyzer works with time delay due to the construction of the sensor and capacity of feed line.

The dynamics of the system with sufficient accuracy was described by the transfer function with the first-order dynamics and time delay in the following equation

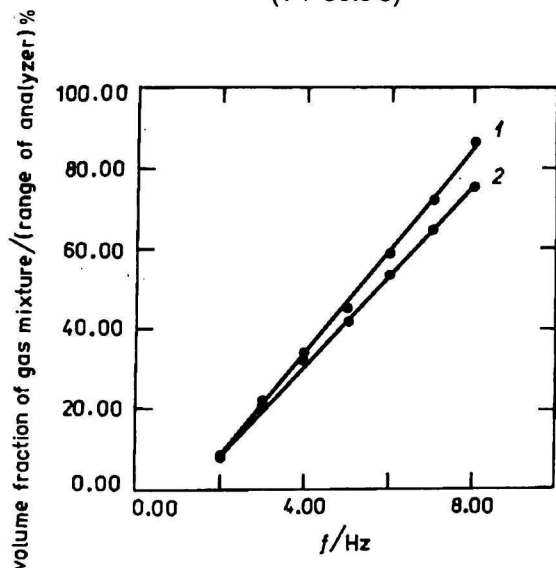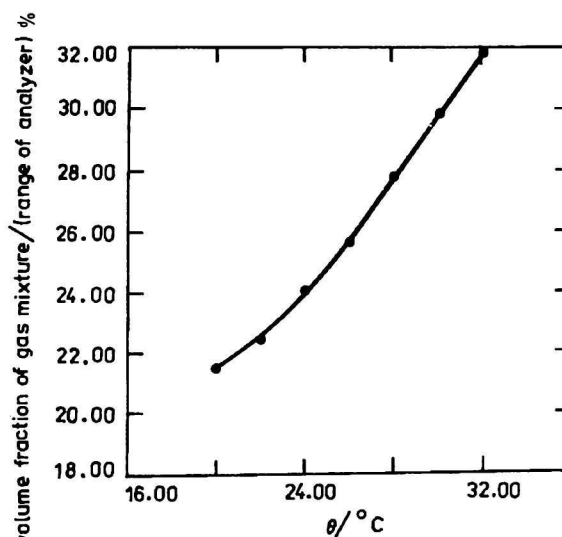$$F(s) = \frac{e^{-13s}}{(1 + 30.6\,s)} \qquad (10)$$

Fig. 7. Dependence of the volume fraction of the gas mixture on the temperature at the constant frequency. f = 3 Hz.

Fig. 6. Dependence of the volume fraction of the gas mixture on the frequency at the height level change. 1. h = 10 cm; 2. h = 9 cm.
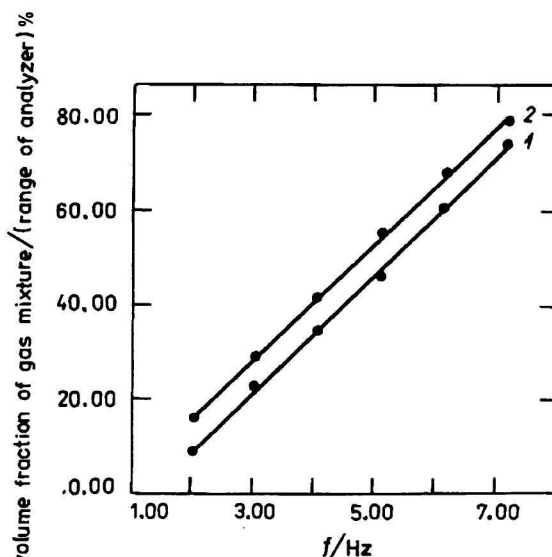
Fig. 8. Dependence of the volume fraction of the gas mixture on the frequency at the change of gas pressure. 1. p_N = 2.1 kPa; 2. p_N = 2.9 kPa.
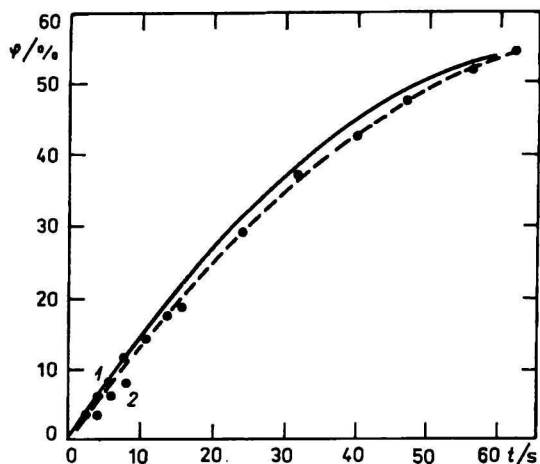
Fig. 9. Comparing of transitional characteristics. Measuring conditions: carrier gas: p_N = 2.4 kPa; analyzed gas: p_A = 8.7 kPa; temperature: θ = 22 °C; level: h = 10 cm; frequency: f = 7 Hz. *1*. Actual transient characteristic; *2*. identified transient characteristic.

The modelled system had the transfer function for verification of the method in the shape

$$F(s) = \frac{e^{-10s}}{(1 + 30s)} \qquad (11)$$

and real time estimation in the equation

$$F(s) = \frac{e^{-9.99s}}{(1 + 31s)} \qquad (12)$$

The system of automatic calibration is very important from the viewpoint of its commercial utilization. The characteristics of the analyzer are changed during measurement and therefore they have to be tested and calibrated. The calibration is carried out by the microprocessor with the automatic device for preparing of a gas mixture with a desired volume fraction. This equipment allows calibration in different points of the analyzer, which is important in the case when its characteristics are not linear. The temperature, pressure, and height level of ethanol in the storage container must be kept constant. If these conditions are not fulfilled, it is necessary to use control with feedback loop with analyzer which has by one class better accuracy than the calibrated analyzer. When using pressure regulators (Festo) [9] and storage container thermostated with accuracy 0.1 °C and constant height of level, the accuracy of the calibrated analyzer was improved about 1/6 in comparison with the data of the producer of the analyzer METREX DH.

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