DETERMINATION OF CORROSION-ACTIVE GASES IN THE ATMOSPHERE BY THE LINEAR COLORIMETRIC METHOD

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Out of the rapid methods of determining corrosion-active gases, the simplest and most reliable is the linear-colorimetric method [1], but the UG-2 gas analyzer which was developed on the basis of this method has a low sensitivity (lower limit 1 mg/m^3 [2]). An analysis of the literature data shows that, for corrosion purposes, it is necessary to determine the content of aggressive gases right down to 0.01 mg/m^3.

One of the most characteristic special features of gas analysis is the close connection between the lower limit of the determination and volume of the sample withdrawn. Therefore, even with small concentrations there is the possibility of accumulating for analysis a sufficient amount of the gas to be determined in the volume of a sorbent, by passing a large amount of air through it. In this connection, the authors have verified the possibility of determining small amounts of gas by the linear-colorimetric method, and determined the sensitivity of the indicator tubes. The verification was carried out using synthetic gas mixtures. The methods of obtaining and purifying the gases were the usual [3]. Withdrawal of the required amount of gas was done with a gas microburet, filled with a saturated solution of $CaCl_2$ [4]. Gas mixtures with the required concentration of the substance to be determined were prepared by consecutive dilutions of purified air or nitrogen. For purifying air the same indicator tubes were used as are used for determination of gas to be analyzed. The fabrication of the indicator tubes, their verification, and their storage were done by a well-known method [2].

There is observed a linear dependence between the content of corrosion-active gases and the length of the colored column of indicator powder (see Fig. 1). An increase in the volume of air taken for analysis, with a constant content of the aggressive gas, leads to an increase in the length of the colored column of indicator powder, which is confirmed by data for SO_2 (content $1.0 \cdot 10^{-4}$ mg /liter):

Volume of air, liters.....	0.5; 1.0; 2.0; 4.0; 8.0
Length of colored column, mm	0.3; 0.7; 1.5; 3.1; 5.8

Thus, an increase in the volume of air taken for analysis permits determining small amounts of corrosion-active gases. According to the experimental data, there were calculated the sensitivities of the indicator tubes, given in Table 1.

In connection with the fact that the volume of air which can be passed through the indicator tubes, using the air-intake device of the UG-2 gas analyzer, is only 0.35 liters, it was necessary to solve the problem of the method of withdrawing samples. Analysis and verification of various methods showed that, for these purposes, it is best of all to use an 822 electroaspirator, made by the Krasnogvardeets [Red Guard] factory, which permits simultaneous analysis of two gases.

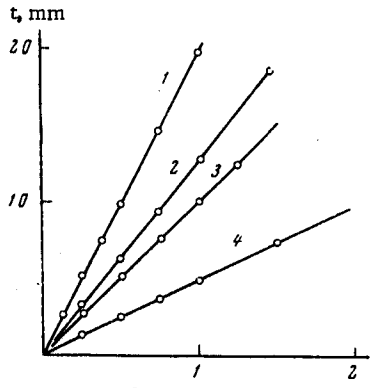

Fig. 1. Dependence of the length of the colored column of indicator powder on the content of corrosion-active gas: 1) H_2S; 2) NH_3; 3) Cl_2; 4) NO_2.

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TABLE 1. Sensitivity of Indicator Tubes for Determination of Corrosion-Active Gases

Corrosion-active gas	Sensitivity·10^{-4}, mg/mm	Corrosion-active gas	Sensitivity·10^{-4}, mg/mm
Sulfide gas	1.4	Hydrogen sulfide	2.0
Nitrogen oxides	1.65	Ammonia	1.8
		Chlorine	1.8

The results of the analyses do not depend on the suction rate of the air, right up to 1 liter/min. It is practically impossible to suck in the air at a higher rate, because of the high density of the indicator powder in the tube.

METHOD

The technique of fabricating the indicator tubes, the absorbers, and the oxidizing cartridges, as well as the verification of the filling density is set forth in detail in [2]. In place of carrying out the analysis using a scraper, the safety caps were removed from the indicator tube. During this operation, it was necessary to hold the tube with the cap downward to avoid its contamination with lumps of sealing wax. Tapping with a bar along the wall of the tube, its degree of packing is verified and, if there is a clearance between the column of powder and the wadding, it is eliminated by pressure of the rod on the wadding.

Then, one of the ends of the indicator tube is connected to the electroaspirator with a rubber hose.* After this, the aspirator is hooked up and, using a regulator and a rheometer, there is established the suction rate of the air at 0.5-1.0 liter/min, the stopwatch is connected and not less than 10 liters of air are pumped through. Depending on the content of corrosion-active gases in the atmosphere, the color of part of the column of indicator powder changes.

In the presence of a sulfide gas, the color changes from dark gray to a whitish-rose, with nitrogen oxides to red, with hydrogen sulfide to brown, with chlorine to reddish yellow, and with ammonia to bluish yellow.

After drawing air through, the timer was stopped, the tube disconnected, and its length measured correct to 0.5 mm.

The volume of air passed through the indicator tube, is calculated by multiplying the suction rate by the time, during the course of which air is sucked through the tube. The volume obtained, V_t, is expressed in liters and, under normal conditions, leads to the formula:

$$V_0 = \frac{V_t \cdot 273.2 \cdot P}{(273.2+t) \cdot 760},$$

where V_0 is the volume of air under normal conditions; P is the atmospheric pressure at the moment of the analysis, mm Hg; t is the air temperature at the point of the analysis; V_t is the volume of air taken for analysis.

The concentration, for each gas observed, is calculated by the formula:

$$C = \frac{l \cdot \alpha}{V_0} \cdot 1000,$$

where C is the concentration of the corrosion-active gas in mg/m^3; l is the length of the colored column, mm; α is the sensitivity of the indicator tube; V_0 is the volume of air taken for analysis, reduced to normal conditions.

The method described can be carried out with a relative moisture content of the air not greater than 80% and a temperature of 10-30°, but in the determination of a sulfide gas, not lower than 15° [2]. In case it is necessary to carry out the analyses at a lower temperature, air samples must be taken using a bicycle pump with rubber barrels. The barrels with the air are put in a hot location and are held for 1-2 h, until they are heated up to room temperature. Then, as usual, they are connected to the indicator tube to the aspirator, while the free end, through a transitional glass tube is connected with the barrel. After this, the

* In carrying out analysis with sulfide gases, to the free end of the indicator tube a filtering cartridge must be attached, and, with nitrogen oxides, an oxidizing cartridge.

aspirator is connected and, gradually opening the valve, there is established a suction rate of 0.3-0.5 liter /min. The calculations are carried out using the same formulas, assuming room temperature.

The method developed has been tested for a half-year in the Moscow Corrosion Station of the Institute of Physical Chemistry of the Academy of Sciences of the USSR, and has yielded results coinciding with the results of ordinary colorimetric methods.

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