

PHYSICOCHEMICAL MEASUREMENTS

TESTING OF THE PERMITTIVITY-TYPE

OIL-MOISTURE METERS

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The automation of technological processes in the extraction, transportation, and processing of oil requires the production of instruments for a continuous testing of moisture content in oil. The permittivity method, which is widely used in designing oil-moisture meters, is based on the definite relationship between the volume of the water content and the permittivity of the water and oil emulsion. Several theoretical and experimental investigations made it possible to discover the physical factors which produce measurement errors and to develop specific recommendations for raising the precision of oil-moisture meters [1, 2].

Permittivity-type moisture meters consist of a capacitive transducer and a measuring instrument which evaluates the transducer's incremental capacity due to the changes in the permittivity of the emulsion, which are produced by variations in its moisture content. The zero of the scale then corresponds to the capacitance of the transducer with dehydrated oil.

Research has shown that the mineral content of water and degree of its dispersion in oil emulsions does not affect permittivity, but it has a substantial effect on losses. For instance, the loss angle $\tan\delta$ of oil emulsions which have the same amount of water (4%) with different mineral contents amounts at 0.2 MHz to 0.0002-0.0053. For a larger moisture content the difference in the losses increases. Therefore, in permittivity moisture meters it is important to measure the capacitive component of the total transducer's impedance.

At the existing level of technological development the capacitive component can be measured with great precision. The application in moisture meters of temperature compensation, the limiting of their transducers' field strength, and several other measures are used to produce moisture meters with an effective error of 2.5-4.0%.

For testing a 2.5-class instrument it is necessary to have a testing method with an error not exceeding 1%. This means that for testing, for instance, marketable-oil moisture meters with a scale of 0-3%, a method is required for evaluating the content of water with an absolute error not exceeding 0.03%.

The method adopted in our standards for evaluating the content of moisture in oil, the Dean and Stark method, is considerably less precise, moreover, the All-Union State Standard (GOST) 2477-65 based on this method does not specify the precision of measurements. It only mentions the difference between paired tests which in the above moisture range should not exceed 0.2%. This figure takes into account only the random error, whereas a detailed analysis indicates that the actual error of the Dean and Stark method is in fact even higher. Therefore, this method cannot be used for checking humidity meters not only of the class 2.5, but also of the class 4.

For evaluating the feasibility of using other direct physicochemical methods in determining moisture for the above purpose, it is possible to apply the data borrowed from [3] and shown in Table 1 on the comparison of various methods for evaluating the moisture of identical samples.

TABLE 1

Karl Fischer	Brabender's furnace	Laboratory furnace	IR spectroscopic analysis of azeo- tropic mixtures
1.55	1.79	1.92	1.60
1.73	2.01	1.97	1.80
1.16	1.43	1.39	1.18
1.80	2.35	2.26	2.05
1.37	1.97	1.96	1.96
1.63	2.01	1.76	1.78

It will be seen from this table that the discrepancy in the measurement results obtained by the above modern methods is large. Therefore, it is impossible to establish the trustworthiness of the moisture content readings thus obtained, it is difficult to express preference for any of these methods as a reference one, and there are no grounds for believing that any of them is suitable for testing moisture meters.

TABLE 2

Precision class of moisture meters	Maximum error in preparing emulsions for moisture measurement ranges		
	0—3%	0—15%	0—60%
2.5	0.03	0.15	0.6
4.0	0.048	0.24	0.96

branch of the All-Union Scientific-Research Institute of Metrology (VNIIM) that it is possible by means of simple volumetric batching of completely dehydrated oil (certified as a sample with "zero moisture") and water to obtain solutions and mixtures in which the percentage content of moisture is known with a precision exceeding the required one by two orders [4]. However, in our case the problem is complicated by the fact that it is necessary to obtain an artificial emulsion with a required degree of dispersion and a uniform distribution of the dispersed water in the sample volume.

Emulsions in general consist of systems whose stability is related to the degree of dispersion. Water-in-oil emulsions should be considered in principle as unstable systems. Therefore, artificial oil emulsions for testing moisture meters cannot be prepared, transported, and stored in metrological institutions the same as standard specimens. Only oil samples certified as having "zero moisture" can be treated as standard specimens, whereas the preparations of the checking or reference samples with the required moisture content can be easily organized directly in situ according to special instructions.

In order to organize the testing of oil moisture meters by means of artificial reference samples it is necessary to take into consideration the following circumstances.

1. The preparation of "absolutely dry" thoroughly dehydrated oil certified as a "zero moisture" specimen should be centralized. Such specimens certified for residual moisture, permittivity, and density (in volumetric batching for conversion to percentage moisture with respect to mass) can be dispatched, stored, and utilized on the spot as standard specimens for checking zero points on the moisture meter scales and for preparing reference specimens with different degrees of moisture. The production of "zero" specimens can be also made in situ. The packing, storage, and utilization of "absolutely dry" oil standard specimens should be specified by appropriate standards.
2. In preparing artificial emulsions it is necessary to ensure a careful emulsification of the sample. The degree of dispersion of water particles in oil should ensure stability of the emulsion during the entire time required for testing at the given point of the scale.

The reference samples can be emulsified by means of mechanical dispurgators, centrifugal emulsifiers, or colloid mills.

The durability, i.e., stability with time, of the obtained emulsions can be evaluated by sampling their specimens' permittivity at different stages of the settling period. The permissible duration of the specimen's utilization (without a repeated emulsification) is thus established for a substantially unstratified emulsion.

3. The instruction for preparing reference specimens should take into account the possible sources of errors, in particular the selective accumulation of moisture which is due to surface affects and could occur on the walls of containers and on the components immersed in the samples (it can be eliminated by selecting and using hydrophobic materials and coatings with a quantitative should also contain the possible "ageing" of the hydrophobic coatings during their utilization, i.e., the deterioration of their water-repelling properties, a rise in the wetting coefficient, etc. The solution of the problem of preparing reference test specimens will make it possible to design a complete testing scheme (see Table 3). According to this scheme the permittivity-type moisture meters are calibrated, adjusted, and repaired as capacitance meters. For this purpose at first the working and stray capacitances of the transducer are measured, for instance, by means of an ac bridge and a reference liquid. It is recommended to use as a reference liquid the benzene which is used in cryoscopy, specified in GOST 5955-51, and has been first dehydrated with zeolites or metallic sodium. The permittivity of such benzene is known, stable, and can be assumed to equal

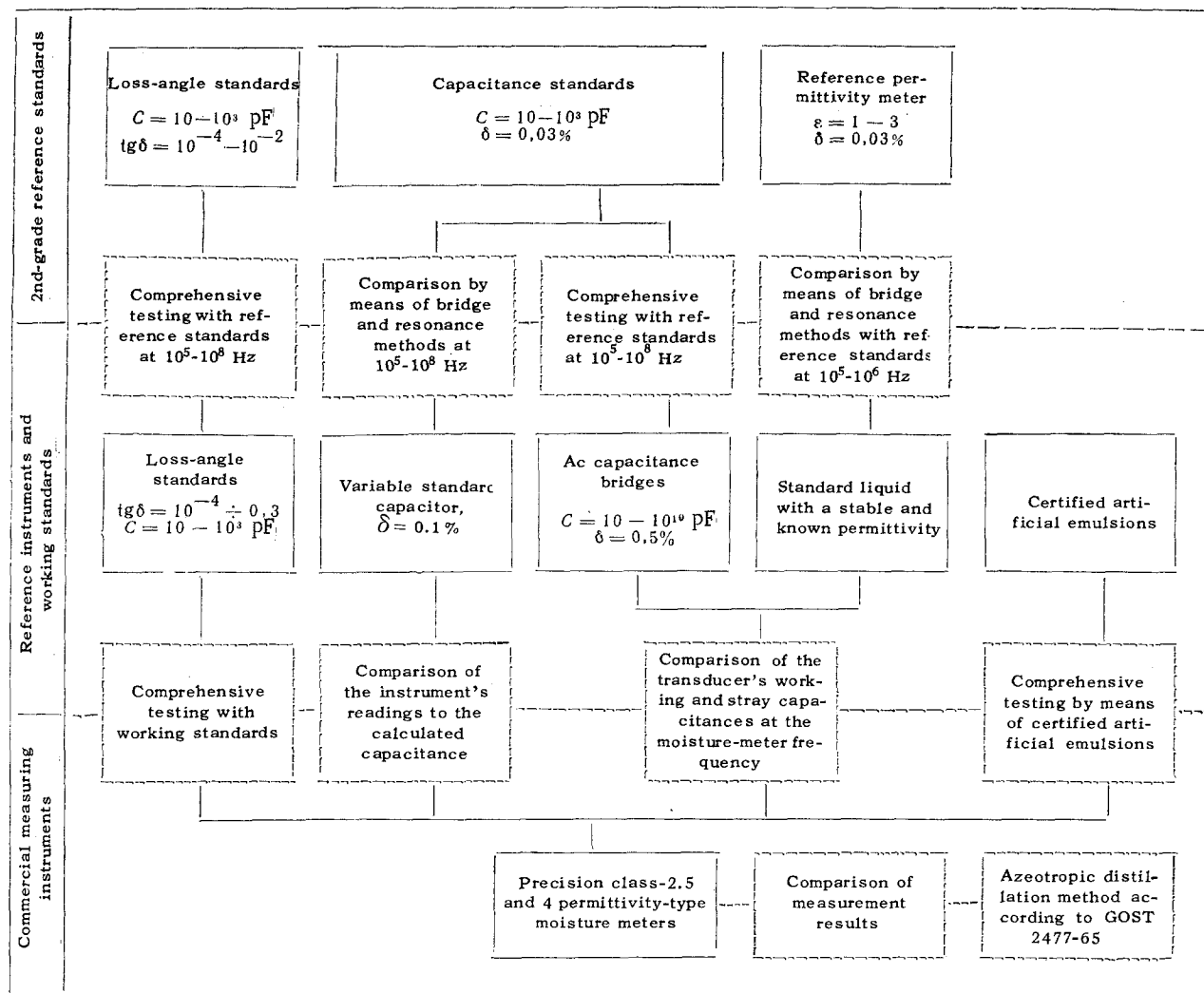
$$\epsilon_6 = 2.2825 - 0.002(t - 20),$$

where t is its temperature.

In this connection it is advisable in testing moisture meters not to compare their readings to those of a given checking (reference) method, but to use comprehensive testing by means of certified artificial emulsions. The content of water in these emulsions should then be known with the precision specified in Table 2.

It has been shown in principle at the Tbilisi

TABLE 3



From the capacitances of the transducers filled with air and benzene it is possible to calculate its working and stray capacitances. The complete testing of the moisture meters is made by means of certified emulsions, as well as capacitance and loss-angle standards.

Such a test scheme is convenient, since the measurement precision of moisture meters can be tested during their utilization by means of widely-used control and measuring instruments, and less frequently in periodic testing by means of artificial emulsions.

LITERATURE CITED

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