Determination of organochlorine compounds in oil according to GOST R 52247-2004 (method B)

The standard is modified in relation to ASTM D 4929-04 "Standard Test Methods for Determination of Organic Chloride Content in Crude Oil".
The standard establishes three methods for the determination of organochlorine compounds (over 1 μg / g organically bound chlorine) **in oil** . To methods A and B, method C was added - X-ray fluorescence wave dispersive spectrometry.
All methods according to GOST R 52247-2004 include oil sampling, distillation of the naphtha fraction (204ºC), washing of naphtha from hydrogen sulfide and inorganic chlorides.

DESCRIPTION OF THE METHOD:

The presence of organochlorine compounds is potentially dangerous for oil refining processes and is detected during the cleaning of technological equipment, pipelines or tanks. Hydrochloric acid formed in hydrotreating or reforming reactors leads to corrosion of equipment. For this reason, the determination of organochlorine compounds in oil is a mandatory procedure.

The X-ray fluorescence analysis procedure is as follows:

Into the washed naphtha fraction isolated from oil, an internal standard is introduced - a bismuth solution in a non-polar solvent with a mass fraction of bismuth 5000 ppm.

The sample is poured into two cuvettes, covered with a film and, sequentially placing the cuvettes in the device, measurements are taken.

The mass fraction of organochlorine compounds is determined by the previously constructed calibration characteristic.

There are no interfering factors for method B.

MEASURING RANGE:

The X-ray fluorescence method using the SPECTROSCAN MAK-GVM spectrometer or the SPECTROSCAN CLSW analyzer allows the determination of the chlorine content **in naphtha** in the range from 2 μg / g to 50 μg / g. The presence of other elements in the product under test does not interfere with the test.

Precision parameters The precision of the
method is determined by statistical examination of the results of interlaboratory tests. The precision indices of the method are established for the chlorine content in the oil fraction boiling up to 204 ° C, in the range from 5 to 50 μg / g.

Repeatability
The discrepancy between successive results of determinations obtained by the same operator on the same apparatus under constant operating conditions on an identical test material for a long time under normal and correct performance of the test method can exceed 1.3 μg / g in only one case. out of twenty.

Reproducibility The
discrepancy between two single and independent test results obtained by different operators working

in different laboratories on an identical test material for a long time, if the test method is performed correctly and correctly, can exceed 2.0 μg / g in only one case in twenty

ADDITIONAL EQUIPMENT:

Preparation of a naphtha sample for analysis involves the introduction of a weighed portion of the internal standard; this requires an analytical balance.
The actual distillation and washing of naphtha requires equipment in accordance with GOST R 52247, clause 6.