DEVELOPMENT OF MICROCOULOMETRIC TITRATION INSTRUMENTAL METHODS FOR THE ANALYSIS OF PETROL PRODUCTS AND ENVIRONMENTAL SAMPLES

Pavlina Ivanova
Research & Development Laboratory, Analytical Department, Lukoil-Neftochim-Bourgas JSC, Bourgas 8104, BULGARIA,
E-mail: lib@neftochim.bg

The increased attention on environment protection has increased the demand for high quality assurance of the methods for determination of sulfur and chlorine levels in the motor fuels, petrochemical products and environments related to this industry. Microcoulometric titration (MCT) methods have been developed and validated for this goal in our laboratory. The MCT method possesses some benefits compared to the other methods used for sulfur and chlorine determination: relatively low price of the instrumentation, lack of calibration procedures, quick measurement and selectivity ensured by the oxidative pyrolysis in the same apparatus. The purpose of this report is to present the most important attainments in method development.

The MCT method was validated for determination of low levels of sulfur (0.1 - 100 mg.kg\(^{-1}\)) in motor fuels with relative expanded uncertainty of the reproducibility (REU\(_r\)) of 4.6 % and the method reliability was confirmed by international proficiency test. The bias of the results in respect to the standard EWXRF and WDXRF methods was ±4.4% and ±6.9% respectively. The method is also validated for analysis of petrol fractions, aromatic hydrocarbons, polymeric products and hydrocarbon gases. Low chlorine levels (less than 1.0 mg.kg\(^{-1}\)) determination in the same products was achieved by effective oxidative conversion of the chlorine compounds in the furnace and optimizing the instrument parameters. The range of the detection limits was 0.03 - 0.09 mg.kg\(^{-1}\), the REU\(_r\) was 3.3 % and the mean value of the recovery was 99 %.

Original decisions for sample treating, removing the inorganic chlorides and injection of samples in the pyrolysis furnace were proposed applying the standard method for determination of absorbable organic chlorine compounds in water. The method was validated for natural, drinking, waste and treated waters. The REU\(_r\) was 9.0 % and the mean value of the recovery was 100.3 %.

A MCT method was developed and validated for determination of total organic chlorine in sediments performing direct combustion of the samples in the pyrolysis furnace and avoiding the commonly used liquid extraction. The REU\(_r\) was 5.6 % and the mean value of the recovery was 98 %.

The methods are adopted by Lukoil-Neftochim JSC and are used in the technological and export production control.