

Specific aspects of the heavy metals analysis in the sludge proceeding from refineries wastewater treatment plants

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Abstract. The first part of the work is a comparative study regarding the methods for heavy metals analysis in the sludge proceeding from refineries. In fact, it was applied the same spectrophotometric method of atomic absorption in flame but the preparation of the samples was different. Four different samples of sludge proceeding from the wastewater treatment plant in an oil refinery were collected and analyzed for the following metals content: nickel, copper, lead, zinc and cobalt. The work started by complying with the valid Romanian standards for the analysis (a) of the sludge. In all these standards, the sludge is treated directly with a « cocktail » of the following acids : HCl, HNO₃, H₃PO₄ and H₂SO₄. Then, the preparation method was changed, by calcinating the sludge and treating the ash with the same mixture (b). The third method was similar to that applied in case of petroleum products and residues (e.g. SR EN 13131/2001, SR ISO 10478/1991) by calcinating the sludge and treating the ash with HCl, HNO₃ and HF, and by introducing the sample in a microwaves oven in order to improve the dissolution of the metals (c). By comparing the 3 sets of analyses, the conclusion was that the national standard methods give the concentration of metal much under the actual values because of the water in the sample diluting the acid and affecting the metals dissolution; the other methods are valid and they can be used as well with a small improvement.

The second part of the work consists on a interlaboratory study in order to validate the methods (b) and (c). By processing the data with the *t* test of hypothesis for paired observations, the above conclusion was strengthened.

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