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ATOMIC-ABSORPTION AND ATOMIC-EMISSION WITH INDUCTIVE CONNECTED PLASMA DETECTION OF NICKEL AND ZINC IN OIL

Abstract: The effect of concentration of surfactant on the value of an analytical signal in investigation of the atomic absorption of Nickel and Zinc was determined. The use of the Triton X-100 and acetylacetone could increase Nickel detection sensitivity by 96% and Zinc by 49% by sonication of the samples. The content of analysts in the analyzed sample was determined by methods of atomic absorption and atomic emission spectroscopy with inductive coupled plasma. The correctness of results was checked by the "entered-found" method.

Changing the dose by weight, it was shown that a significant systematic error is absent. The summary of the obtained results was produced with two independent methods of F-and Student's t-criteria.

It is shown that the dispersions are homogeneous, and the average mismatch is not substantial and justified by the random spread. The atomic absorption technique has assessed the limit of the detection of analysts by the developed method. It is shown that the appeared results are below of these represented in the literature data.

Key words: atomic absorption and emission spectroscopy with the inductively coupled plasma (AES-ICP), crude oil, petroleum, Triton X-100, ultrasonic processing, analysis, metrological characteristic.

Language: English

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Introduction

Crude oil is a complex mixture of carbohydrates that occur on the Earth in a liquid state. It represents a significant portion of the original fossil fuels. Information on the concentration of microelements in crude oil is becoming more and more critical for the

geochemical characteristics of the source breeds and pools, and also requires some corrective actions during the crude oil processing [1,p.7;2, p.556;3, p.139; 4, p.38].

Generally, the traces of metals in the most significant concentrations conducive to environmental

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pollution, which have been found in different raw materials, are Nickel and Vanadium. Due to their mutagenic and cancerogenic potential, Ni and V emissions are strictly controlled in many countries [5,p.5;6,p.16;7,p.649].

Besides, Nickel, along with other metals, is a catalytic poison and causes corrosion in furnaces and boilers during oil refining. Other metals, such as Ferrum, Cooper, and Zinc, may also be present in significant quantities and can be partially transferred to fractions, reducing their quality and performance.

That is why the knowledge about the concentration of metals in raw oil provides robust information that allows to assess and regulate the further working conditions and its analysis [8, p.33].

For the analysis of crude oil on the content of analytes, the methods of atomic absorption and atomic emission with inductively coupled plasma spectroscopy are widely used. A special role is played by sample preparation, new environments, and standard samples of composition.

The purpose of work is the development of the newest methods of atomic-absorption and atomic emission with inductively coupled plasma for the definition of analytes in oil with improved metrological characteristics.

Experimental part

In this work, the Atomic Absorption Spectrometer C-115-M1 is used; Measurements were performed in the flame of propane-butane-air at optimal parameters: $\lambda_{Ni}=231,9$ nm, $\lambda_{Zn}=213,9$ nm, PhEP (photoelectric pickup) = 1kV, current = 5mA, lamps with hollow cathode for Nickel and Zinc.

Atomic emission spectrometer with inductive coupled plasma i CAP 6300 DUO (Thermo Scientific, Jewel., the USA) was used. Measurements were performed according to the instructions «Thermo SPEC/PMT software for TJA Sequential ISAP Spectrometers Getting started. Part Number 140962-00».

Optimum measuring conditions: $\lambda_{Ni}=259,940$ nm, $\lambda_{Zn}=213,6$ nm, the turnover rate of the peristaltic pump – 100 rpm., pressure of the argon stream during spraying – 30 psi, the term of integration – 2 seconds, the plasma power 1500 Watts. Ultrasonic Bath PS-20 (3.2 L; operating capacity is 120 Watts and 40 kHz of frequency). Weight-scale OHAUS PA 64 (65/0.0001 g) with external calibration/state verification. Crockery measuring, laboratory, and glass devices: cylinders, flasks (capacity of 5, 10, 50, and 100 ml), pipettes (with capacity 1, 2, 5, and 10 ml), cups in GOST (All-Union State Standard) 17770-74 and GOST 20292-74.

Nitric acid, standard sample of Nickel solution for the 022.38 -96 NSSU (National State Standard of

Ukraine), 1 mg/ml; The standard sample of the Zinc solution in the 022.47 -96, 1 mg/ml; Triton X-100 *Mr* – 646 g/mol, CCM 0.06 g/L $2,9 \cdot 10^{-4} - 1,0 \cdot 10^{-1}$ mol/l; Acetilacetone C pentane – 2,4 – dione; Nickel and Zinc acetylacetone were used although.

Results and discussion

The calibration solutions were prepared from the standard samples of the composition of metal ions solutions and acetylacetone of Nickel and Zinc with additives of surfactant and acetylacetone. The addition of the Triton X-100 reduced the surface tension of the solution but increased the dispersion of aerosol, which allowed to release the time of the retention of analyses in the atomic state and to improve the sensitivity of the atomic-absorption of Nickel and Zinc. By modifying the solutions, the sensitivity of the atomic absorption of the Nickel increased by 96%, and the Zinc by 49%.

The selection of the concentration of Triton X-100, treatment by ultrasound time, the results of the detection of analyses by two methods of the correctness of the results of atomic-absorption of Nickel and Zinc determination are given in Table 1-5.

As could be seen from the results in Table 1, the maximum of analytical signal in the detection of analytes is achieved by using Triton X-100 ($\omega = 5\%$).

We could conclude that the sample should be treated by ultrasound within 30 minutes.

By varying the mass of a dose by weight, it was established that the systematic error is not significant [9,p.1980;10,p.16;11,p.159;12,p.18;13,p.312;14,p.65;15,p.843].

Since $F < F_{table}$, $t < t_{table}$, ($F = 6,39$; $t = 3,31$), it can be assumed that the results are equivalent, the difference in reproducibility is not significant, and the discrepancy among obtained by two available methods is insignificant and justified by the spread of values.

The limit of detection of atomic-absorption of Nickel is evaluated: $C_{min} = 0,043$; $C_{lit} = 0,100$ $\mu\text{g/ml}$, and also Zinc - $C_{min} = 0,001$, $C_{lit} = 0,004$ $\mu\text{g/ml}$.

Conclusions

The use of new environments, based on surfactants can significantly increase the sensitivity of the atomic absorption of analytes. The increase of precision and accuracy of measurements is achieved by using solutions of acetylacetone of Nickel and Zinc as standard samples, which allowed to bring the composition of the calibration solutions to the analyzed ones according to chemical composition.

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Table 1. The choice of the concentration of surfactant for atomic-absorption detection of Nickel and Zinc (n=5; P=0.95).

$\omega, \%$	Ni, mg/kg		Zn, mg/kg	
	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r
3	0,44±0,01	0,01	2,28±0,03	0,01
4	0,46±0,02	0,01	2,37±0,03	0,01
5	0,54±0,02	0,01	2,50±0,03	0,01
6	0,54±0,03	0,01	2,50±0,04	0,01

Table 2. The choice of ultrasound treatment time at the atomic- absorption detection of the solutions of Nickel and Zinc (n=5; p=0,95).

Time of the treatment, min.	Ni (Triton X-100), mg/kg		Zn(Triton X-100), mg/kg	
	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r
10	0,40±0,01	0,01	2,28±0,03	0,01
15	0,45±0,02	0,01	2,35±0,03	0,01
20	0,50±0,03	0,01	2,42±0,03	0,01
25	0,53±0,01	0,01	2,47±0,04	0,01
30	0,54±0,02	0,01	2,50±0,04	0,01

Table 3. Results of the atomic absorption of Nickel and Zinc in oil using Triton X-100, stabilized by US (ultrasound) (n=5; p=0,95).

Ni, mg/kg		Zn, mg/kg	
$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r
0,54±0,02	0,01	2,50±0,10	0,01

Table 4. Check the correctness of the atomic absorption detection of Nickel and Zinc by «injected – found out» method (n=5; p=0,95).

Metal	Contain, mg/kg	Injected, mg/kg	Found out, mg/kg	S_r
Ni	0,54	0,50	1,03±0,27	0,01
Zn	2,50	3,00	5,6±0,20	0,01

Table 5. Results of the determination of Nickel and Zinc content by AES-ICP in oil with the addition of surfactants, stabilized by US (n=5; p=0,95).

Ni, mg/l		Zn, mg/l	
$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r
0,55±0,02	0,01	2,51±0,10	0,01

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Table 6. Systematic error estimation at atomic absorption detection of Nickel and Zinc by variation of the dose by weight of the sample (n=5; p=0,95).

Weight of the samples, g	Ni, mg/kg		Zn, mg/kg	
	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r	$\bar{C} \pm \frac{t_{p,f} \cdot S}{\sqrt{n}}$	S_r
0,6	0,53±0,14	0,01	2,48±0,11	0,01
0,7	0,54±0,15	0,01	2,50±0,12	0,01
1,0	0,53±0,15	0,01	2,49±0,12	0,01

Table 7. Consistency of results obtained by two independent methods of Fisher and Student's t-criteria

Metal	F	$S_{1,2}$	$t_{1,2}$
Ni	1,04	0,02	0,2
Zn	1,03	0,01	0,28

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