

## Sorption Preconcentration and Determination of Nickel in Wastes of Heat Power Industry by Diffuse Reflection Spectroscopy

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The present work is focused on the preconcentration of nickel and its determination by means of diffuse reflection spectroscopy. The preconcentration of nickel was carried out by sorption on macroporous aminocarboxylic amphoteric resin ANKB-35. Based on this collector, a method to determine nickel in wastes of heat power industry was worked out using solid-phase spectroscopy. The colored surface compound to be determined was obtained by a preceding nickel sorption on the resin and by subsequent treatment of the concentrate obtained with definite amounts of 1-(2-pyridilazo)-2-naphtol (PAN). The Ni calibration curve is linear in the concentration range of 0.5-20.0 mg/L (sample volume is 200.0 mL) and the detection limit is 0.05 mg/L. The presence of  $\text{Cu}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$  ions as well as macrocomponents of natural water ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ) do not hinder the solid-phase spectroscopy determination of nickel. The nickel determination by diffuse reflection spectroscopy was carried out in model solutions as well as in solutions obtained after the dissolution of wastes of heat power industry.

**Key Words :** Nickel. Sorption preconcentration. Wastes of heat power industry. Diffuse reflection spectroscopy

### Introduction

An irrational and ecologically dangerous handling of wastes is an urgent environmental problem. More than 25 billion tons of solid wastes are furnished annually in the world, including about 7 billion tons in Russia.<sup>1</sup> During the burning of coal, complex chemical and phase conversions occur in its mineral components.<sup>2</sup> As a result, substances with new properties are formed—ash and slag. Many microelements (nickel, cobalt, copper, iron etc.) contained in natural coals are concentrated in ashes and slags, and so their content in these wastes increases several times over. That is why waste piles are one of the most active sources of environmental pollution. Their dispersion leads to the fouling of the atmosphere and of natural ground water. The latter is a result of hydraulic disposal of ash and slag wastes.<sup>3</sup>

It should be noted that nickel produces a general toxic effect on the human organism, causing nasopharynx and lung diseases, malignant tumors and dermatological diseases as well.<sup>4</sup> Nickel-containing sewage are harmful after ingress into water. This fact explains the importance of the monitoring of nickel concentrations in natural water and sewage. Therefore, it is necessary to analyze nickel concentrations in natural water and sewage on a level of parts of the maximum permissible concentration, which is for Russia 0.1 mg/L.<sup>5</sup> An analytical sorption technique can be applied here.<sup>6-8</sup>

The solid-phase spectroscopy method combines the sorption preconcentration of metal ions and their determination on a surface of different sorbents. This method has already been shown to be advantageous for analysis of different

metals as well as for their content control in electrolytes, sewage and natural water.<sup>9-13</sup> Moreover, this method is cheap and applicable under field conditions.<sup>14</sup>

In a recent study we investigated the ion exchange recovery of nickel from manganese nitrate solutions obtained after the processing of manganese ores.<sup>15</sup> It was shown that the aminocarboxylic amphoteric ion exchangers possess the selective sorption properties to nickel ions. The present paper is devoted to the nickel preconcentration on macroporous amphoteric resin ANKB-35 and to the Ni(II) determination by diffuse reflection spectroscopy.

### Experimental Section

The macroporous amphoteric ion exchanger ANKB-35 synthesized on the basis of acrylonitrile and divinylbenzene was investigated. The manufacturer of this ion exchanger is TOKEM-Company (Kemerovo, Russia).

After the standard preparation, the resin was converted into the  $\text{Na}^+$ -form. The specific swelling volume and the static exchange capacity of ANKB-35 are 2.8  $\text{cm}^3/\text{g}$  and 3.6  $\text{mmol/g}$ , respectively.

The nickel sorption was investigated under batch experiment conditions. The nickel chloride stock solution (500 mg/L) was prepared from an accurately weighed sample of pure supplier (analytical grade). This solution was standardized by 0.1 M EDTA solution. The operating nickel solutions were prepared by diluting the stock solution.

Before sorption on the resin, samples (0.2 g of dry substance) were swollen for 4 h in distilled water at the desired pH value (2.5-5.5). After swelling, the resins were stirred with  $\text{NiCl}_2$  solutions in a thermostat at  $(20 \pm 1)^\circ\text{C}$ .

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The stirring time intervals were from 10 min to 24 h. The mass ratio of resins to solutions was 1 : 50.

Apart from the individual NiCl<sub>2</sub> solutions, model solutions were also investigated. These solutions contained Ni(II) ions as well as competing ions such as cobalt(II), copper(II), iron(III), manganese(II), sodium, calcium, magnesium and fluoride. The desired pH values of the sample solutions were adjusted by diluted HCl or NaOH, controlled by a pH-meter.

Wastes of the heat power industry of Krasnoyarsk (ash and slag pulps) were also used for the determination of nickel ion. These wastes were dissolved according to the procedure described in our paper.<sup>16</sup>

After sorption, the metal-loaded resins were separated from solutions. Then analyses of both solid and liquid phases were carried out. The concentration of nickel in solutions was determined by a photometrical method<sup>17,18</sup> (PAN as a reagent) using a photocolormeter (Model KFK-3, Russia) or by flame atomic absorption spectroscopy using an atomic absorption spectrophotometer (Model Saturn-2, Russia).

The diffuse reflection spectra of the sorbent investigated were registered by means of the transportable colorimeter (Model Spectron, Russia). For this purpose, the wet samples (0.2 g) were placed into a cell and the spectra were recorded at a wavelength of 380-720 nm. The cell is a fluoroplastic disk (height is 10 mm, diameter 80 mm) with a cylindrical hole of 5 mm depth and 16 mm diameter).

The desired analyte compound was obtained on the resin surface according to the following procedures:

**Formation of nickel chelate with PAN in the resin phase.** 0.2 g of ANKB-35 and nickel solutions contained 0.34-3.4 mmol Ni<sup>2+</sup>/L were placed into graduated flasks (V = 25.0 mL) and the suspensions were stirred over a period of 30 min. Then the resins were filtered and 0.2 mL of 0.1 % aqueous PAN solution were added drop by drop to the exchangers. After 30 min, the diffusion reflection coefficients were measured.

**Sorption of nickel on the modified ion exchanger.** 0.2 g of ANKB-35 modified by 0.1% aqueous PAN solution (modification of sorbent is the preceding reagent sorption on this resin) and nickel solutions contained 0.34-3.4 mmol Ni<sup>2+</sup>/L were placed into graduated flasks (V = 25.0 mL) and distilled water was added up to the flask's mark. After the required period of time, the resins were filtered and then the diffusion reflection coefficients were measured.

In the solid-phase spectroscopy method, the change of Gurevich-Kubelka-Munk function  $\Delta F(R)$  is used as an analytical signal:

$$\Delta R(R) = \frac{(1-R)^2}{2R} - \frac{(1-R_0)^2}{2R_0} \quad (1)$$

where  $R$  and  $R_0$  are diffusion reflection coefficients (quantities of the dimension 1) of chelate of Ni<sup>2+</sup> with PAN in the exchanger phase and of the ion exchanger, respectively.<sup>19-21</sup>

The determination of the composition of Ni(II) complex compound with PAN in the resin phase was carried out by the method of isomolar series according to the following

procedures:

(1) 1, 2, 3, 4, 5, 6, 7, 8 and 9 mL of Ni(II) solution ( $1 \times 10^{-3}$  mol/L) and 9, 8, 7, 6, 5, 4, 3, 2 and 1 mL of PAN solution ( $1 \times 10^{-3}$  mol/L) were placed into graduated flasks (V = 25.0 mL). Then distilled water was added up to the flask's mark. After that, 0.2 g of ANKB-35 was added and the suspensions were stirred over a 1 h period. Then the resins were filtered and the diffusion reflection coefficients were measured at  $\lambda = 560$  nm.

(2) 1, 2, 3, 4, 5, 6, 7, 8 and 9 mL of Ni(II) solutions ( $1 \times 10^{-3}$  mol/L) were placed into graduated flasks (V = 25.0 mL) and distilled water was added up to the flask's mark. After that, 0.2 g of ANKB-35 was added and the suspensions were stirred over a 1 h period. Then the resins were filtered and placed into the PAN solutions with different concentrations. These solutions were obtained by the following way: 9, 8, 7, 6, 5, 4, 3, 2 and 1 mL of PAN ( $1 \times 10^{-3}$  mol/L) were placed into graduated flasks. Then distilled water was added up to the flask's mark. After 1 h, the resins were filtered again and the diffusion reflection coefficients were measured at  $\lambda = 560$  nm.

Using the results obtained, the isomolar diagram was plotted in the coordinates  $\Delta F(R)$ - $X$ , where

$$X = \frac{C_R}{C_M + C_R} \quad (2)$$

( $C_M$  and  $C_R$  are the molar concentrations of the metal ions and reagent solutions, respectively).

The composition ( $n$ ) of the complex compound was determined according to the following formula:

$$n = \frac{1 - X_{\max}}{X_{\max}} \quad (3)$$

where  $X_{\max}$  is the value of  $X$ , which corresponds to maximum on the isomolar diagram.

All results were statistically processed by standard methods<sup>22</sup>; the average for 3-4 parallel tests was measured, then the variance, standard deviation and confidence intervals were calculated using Student's  $t$  at  $P = 0.95$ . The standard deviation of the total analytical procedure was not more than 8%.

## Results and Discussion

In the previous work,<sup>14</sup> the optimum pH values for nickel sorption on amphoteric ion exchangers were determined. The pH increase from 2.5 to 5.5 leads to the rise in exchange capacity in the Na<sup>+</sup>-form because the concentration of coordination-active (non-protonated) functional groups decreases with the increase in the solutions' acidity.<sup>23,24</sup> It was found that pH > 3.5 is suitable for selective Ni(II) recovery.

The sorption isotherm obtained under the optimum conditions is convex (*i.e.* the selective Ni(II) sorption) (Figure 1).

We have found that the IR-spectra of ANKB-35 vary after the Ni(II) sorption (Figure 2). The intensity of absorption bands at 1800-1600 cm<sup>-1</sup> and at 300-250 cm<sup>-1</sup> has the following changes: disappearance of absorption band at 1730-1720 cm<sup>-1</sup> and the widening of the band at 1400-1370

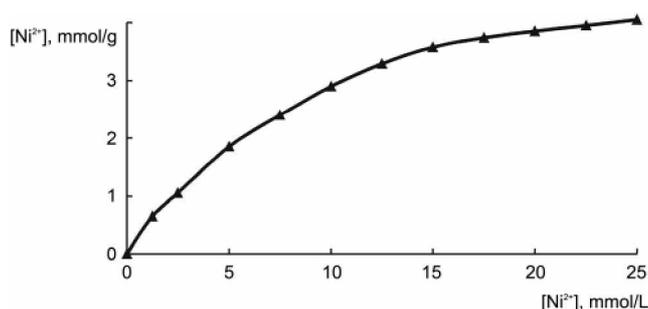


Figure 1. Isotherm of Ni(II) sorption on amphoteric ion exchanger ANKB-35.

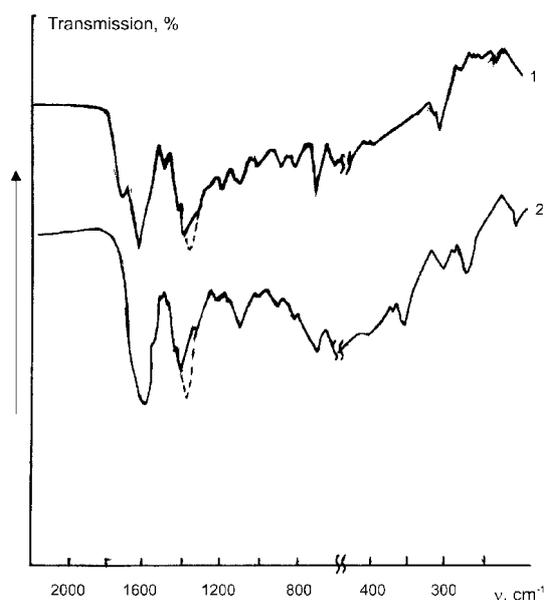
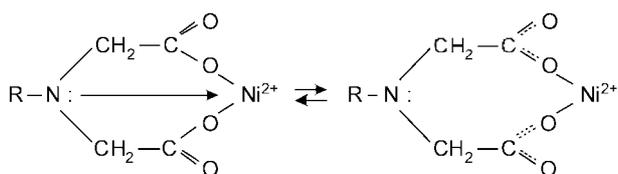


Figure 2. IR spectra fragments of amphoteric ion exchanger ANKB-35. 1-Na<sup>+</sup>, Cl<sup>-</sup>-form, pH = 3.3-3.5. 2-Ni<sup>2+</sup>-form, pH = 3.5.

cm<sup>-1</sup>. These bands are in conformity with asymmetric and symmetric vibrations of ionized carboxylic groups. The location of frequencies  $\nu_s$  and  $\nu_{as}$  changes after the Ni<sup>2+</sup> sorption ( $\Delta\nu = 210$  cm<sup>-1</sup>). It can be assumed that the complex compounds of Ni<sup>2+</sup> with the sorbent's functional groups are formed in the ion exchanger phase. Moreover, the bands, which appear at 320 and 380 cm<sup>-1</sup>, can be attributed to the complex bonds of nickel with nitrogen and oxygen atoms of functional groups of the resin.<sup>15,25</sup> The data obtained allows one to assume that the following complexes are formed in the phase of ANKB-35:



Metal reagent PAN (1-(2-pyridylazo)-2-naphthol) is highly selective for determination of nickel by the photometrical method.<sup>17</sup> Therefore, this reagent was also used for sorption-spectroscopic nickel determination in wet resin phase. For

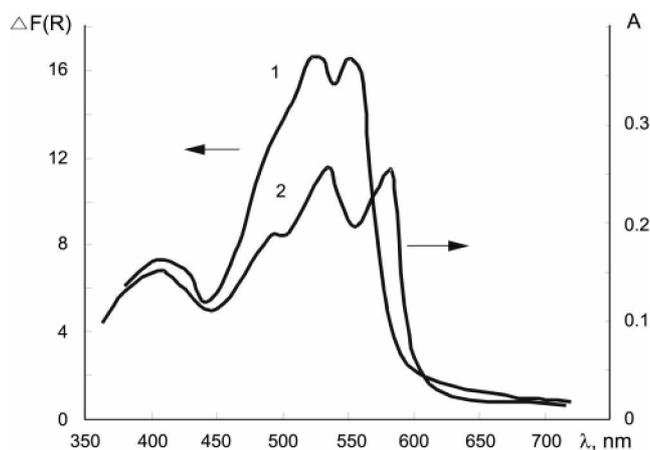


Figure 3. Diffuse reflection spectra of the nickel chelate with PAN in the phase of ANKB-35 (1) and of the nickel chelate with PAN in the solution (2). Content of Ni(II) in the resin phase is 40 μg (1);  $C_{\text{Ni}} = 3.4$  mmol/L (2).

this purpose, diffuse reflection spectra were recorded for the nickel chelate with PAN in the resin phase and in the solution (Figure 3, curves 1 and 2, respectively). It can be seen from this Figure that both spectra are similar to each other, but the maxima in diffuse reflection spectrum (Figure 3, spectrum 1) are shifted to a range of short wavelength in comparison with these maxima in spectrum of Ni-PAN-complex in a homogeneous medium (Figure 3, spectrum 2). Possibly, a matrix effect takes place.

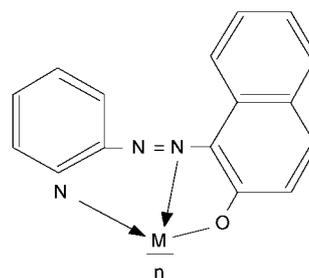
It is known<sup>21</sup> that the colored surface compound can be obtained by a preceding nickel sorption on the resin with the subsequent treatment of the concentrate obtained with a definite amount of PAN or by a preceding sorption of PAN on the resin, with the subsequent nickel sorption on the modified sorbent.

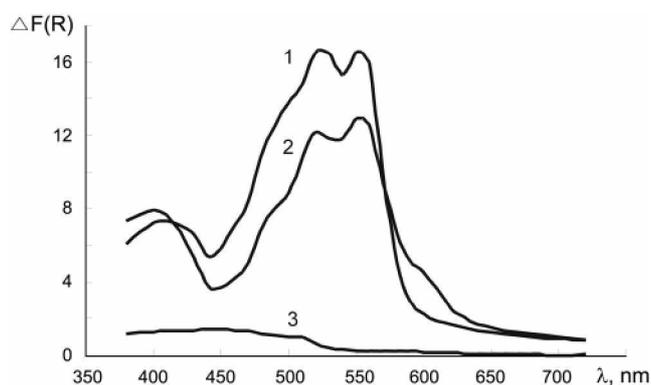
In the first case, nickel sorption on ANKB-35 proceeds in accordance with the ion-exchange mechanism



followed by a treatment with a definite amount of PAN. In the second case, a physical adsorption of PAN is probable between the macroporous resin and reagent.

The question regarding the binding form of the Ni-PAN-complex to the ion exchanger requires additional study. It is known<sup>26</sup> that PAN forms complexes with metals in solution by means of hydroxyl group oxygen atoms of as well as by nitrogen atoms of pyridyl and azogroups:





**Figure 4.** Diffuse reflection spectra of sorbates of the nickel chelate with PAN (1, 2) and of PAN (3). 1-PAN is added to the sorbent in the  $\text{Ni}^{2+}$ -form. 2-Sorption of  $\text{Ni}(\text{II})$  by the modified resin. Content of  $\text{Ni}(\text{II})$  in the resin phase is  $40 \mu\text{g}$ .

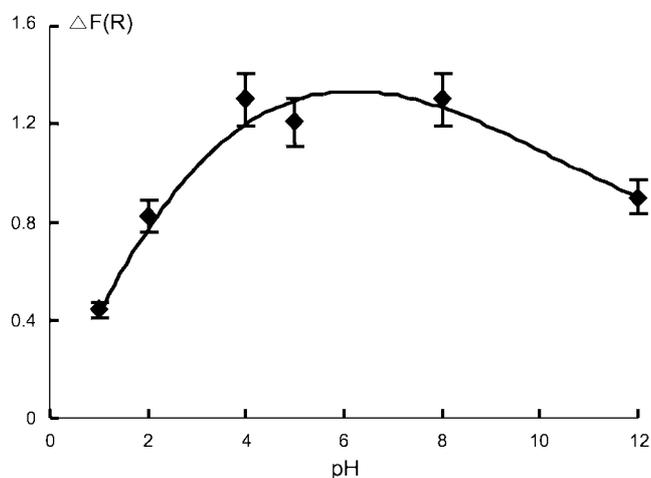
It is probable that ion-pair binding appears between the  $\text{Ni}$ -PAN-complex and carboxylic groups of the exchanger or an unspecific sorption interaction can take place.

The diffuse reflection spectra of sorbates of the  $\text{Ni}$ -PAN-complexes and of PAN are shown in Figure 4. It should be noted that the procedure of synthesis of the colored compound in the sorbent phase has no effect on the location of maxima in diffuse reflection spectra (Figure 4, spectra 1 and 2), but the analytical signal is more pronounced when the nickel-PAN-complex is formed in the sorbent phase (see Experimental procedure 1).

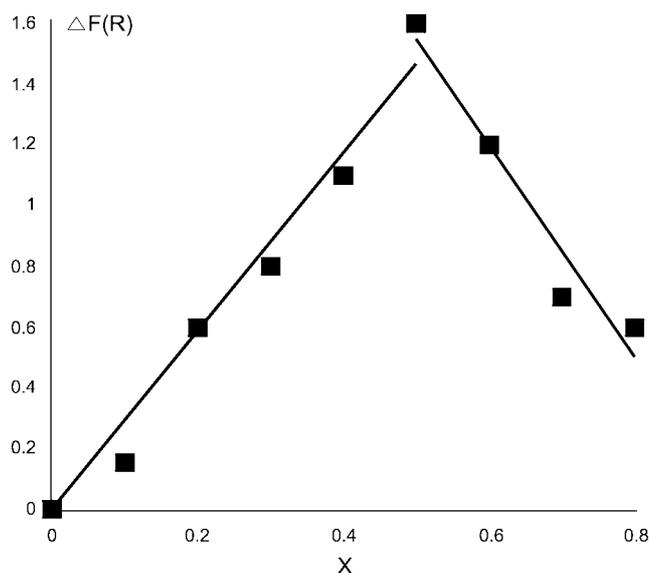
Furthermore the dependence of analytical signal on time and on pH was investigated at the wavelength 560 nm. It was found out that the equilibrium state was attained during 30 min. The peak of analytical signal was observed at  $\text{pH} = 4.5$ - $8.0$  (Figure 5).

The ratio  $\text{Ni}^{2+}$ :PAN in the sorbent phase is 1:1 based on method of isomolar series (Figure 6).

It is known<sup>17</sup> that  $\text{Cu}^{2+}$ ,  $\text{Fe}^{3+}$  and  $\text{Co}^{2+}$  ions can interfere in the spectrophotometric analysis of nickel. These ions were masked by fluoride and thioglycol acid. The results of nickel determination in the presence of these components are



**Figure 5.** Effect of pH on formation of analytical signal.  $\lambda = 560 \text{ nm}$ ; nickel content in the exchanger phase is  $5 \mu\text{g}$ .



**Figure 6.** Isomolar diagram of  $\text{Ni}(\text{II})$ -PAN-complex in the phase of ANKB-35.  $C_{\text{Ni}} = 1 \times 10^{-3} \text{ mol/L}$ ;  $C_{\text{PAN}} = 1 \times 10^{-3} \text{ mol/L}$ .

summarized in Table 1. It can be seen from Table 1 that the presence of the above-mentioned ions as well as of macro-components of natural water ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ) do not hinder the solid-phase spectroscopy determination of nickel.

Based on the results obtained, a number of sorption-spectroscopic determinations of nickel were carried out in a series of water samples. For that purpose, standard nickel solutions ( $0.1$ - $20.0 \text{ mg/L}$ ) were placed into graduated flasks ( $V = 250.0 \text{ mL}$ ). Then  $0.2 \text{ g}$  of resin, preswollen at a definite pH value and  $10 \text{ mL}$  of  $4\%$   $\text{NaF}$  solutions were added and the suspensions were stirred over a period of  $30 \text{ min}$ . After that the resins were filtered and  $0.2 \text{ mL}$  of  $0.1\%$  PAN solution were added drop by drop to the exchangers. Then  $10 \text{ mL}$  of  $0.1 \text{ M}$  thioglycol acid solution was added to the colored resins and the suspensions were stirred over a period of  $10 \text{ min}$ . After that the ion exchangers were filtered again and the diffusion reflection coefficients were measured at  $560 \text{ nm}$  and the calibration curve of  $\Delta F(R)$  was plotted as a function of the  $\text{Ni}^{2+}$  concentration.

**Table 1.** Effect of foreign ions on the determination of nickel ion by solid-phase spectroscopy

Foreign ion <sup>a</sup>	Ratio of foreign ion to $\text{Ni}(\text{II})$	Found Ni, $\mu\text{g/mL}$	Relative error, %
$\text{Co}(\text{II})$	1:1	0.98	-2
$\text{Cu}(\text{II})$	10:1	1.04	+4
	100:1	1.38	+38
$\text{Ca}(\text{II})$	10:1	1.09	+9
$\text{Fe}(\text{III})$	10:1	1.08	+9
	100:1	1.35	+35
$\text{Mn}(\text{II})$	10:1	0.92	-8
$\text{Mg}(\text{II})$	10:1	0.98	-2
$\text{NaCl}$	100:1	1.02	+2
	1000:1	0.94	-6
$\text{F}^-$	1000:1	0.96	-4

<sup>a</sup> $C_{\text{Ni}} = 1 \text{ mg/mL}$ .

**Table 2.** Sorption-spectroscopic nickel determination in model solutions

Analysis object	Nickel content, $\mu\text{g/mL}$		$S_r$	$\pm \frac{t_p \cdot S}{\sqrt{n}}$	$\epsilon, \%$
	Added	found			
MS 1 <sup>a</sup>	1.0	1.19	0.05	0.09	+19
MS 2	5.0	5.18	0.06	0.44	+4
MS 3	10.0	10.22	0.03	0.40	+22

<sup>a</sup>MS-model solution.  $n = 4$ ;  $P = 0.95$ .

**Table 3.** Sorption-spectroscopic nickel determination in wastes of heat power industry<sup>a</sup>

Material	Ni, found $w \pm \delta$ , mg/kg	
	by sorption-spectroscopic method	by flame AAS method
Slag pulp 1	85.8 $\pm$ 3.4	85.1 $\pm$ 2.1
Slag pulp 2	109.7 $\pm$ 6.3	97.8 $\pm$ 4.2
Slag	74.8 $\pm$ 5.2	72.9 $\pm$ 3.3
Ash 1	78.0 $\pm$ 5.4	71.4 $\pm$ 4.9
Ash 2	60.7 $\pm$ 5.1	68.6 $\pm$ 4.3

<sup>a</sup>The slag and slag pulps were selected from different boilers of heat power station and the ash samples were taken from electric filters.  $n = 3$ ;  $P = 0.95$ .

This calibration curve is linear at the  $\text{Ni}^{2+}$  concentration range of 0.5–20.0 mg/L and by sample volume of 200.0 mL. The detection limit determined by  $3\sigma$  method is 0.05 mg/L. The calibration curve follows the equation

$$\Delta F(R) = 0.1664 C_{\text{Ni}} + 0.0852. \quad (5)$$

Moreover, the sample volumes of 1000.0 mL were used for determinations of lower nickel concentrations in water.

The detection limit is 0.02 mg/L in this case and the calibration curve follows the equation

$$\Delta F(R) = 0.3526 C_{\text{Ni}} + 0.0995. \quad (6)$$

Based on the calibration curves obtained, a number of nickel ion determinations were carried out by sorption spectroscopy. The results are shown in Table 2.

Moreover, the nickel determination was carried out by solid-phase spectroscopy method in solutions obtained after the dissolution of wastes of heat power industry. We also compared the accuracy of the proposed method and the flame atomic absorption method. The results of comparison are given in Table 3.

The results obtained allow us to recommend the analytical systems, on the basis of macroporous amphoteric ion exchanger ANKB-35, for the ion-exchange nickel recovery from the solid wastes and for its determination directly in the sorbent phase.

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