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Ecological Geochemistry of Mercury and the Methods for Demercurization the Mercury-Containing Solid Wastes under the Conditions of South Siberia (by the Example of the Industrial Area of the JSC "Novosibirsk Chemical Concentrates Plant")

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Abstract

Results are presented for hydrogeochemical monitoring the industrial area of the JSC "Novosibirsk Chemical Concentrates Plant" as well as experimental data concerning the demercurization of soils and building materials those were contaminated with mercury. A comprehensive approach to the demercurization and geological conservation of mercury-containing waste is proposed that includes three stages: 1) a centrifugal extraction of mercury from solid wastes including contaminated grounds, soils and building refuse; 2) chemical immobilizing the mercury remaining in the insoluble form (naturally occurring mineral Stchuettite); 3) geological conservation.

Key words: lithium, chemical and metallurgical industries, mercury-containing solid wastes, demercurization, geological conservation

INTRODUCTION

At all the enterprises of the industrial production of lithium metal and its compounds (the USA, UK, Russia) there is a significant mercury contamination of soil, ground and building structures observed [1].

In each case the demercurization mercury-containing solid wastes was carried out taking into account the ecogeochemical situation [2–9].

The JSC "Novosibirsk Chemical Concentrates Plant" (NCCP) is not an exception, since the problem of mercury as a superecotoxicant is brought to the forefront [7–10].

This paper is devoted to analyzing the ecogeochemical situation connected with a man-caused mercury anomaly, as well as to the methods of centrifugal demercurization and chemical immobilization of solid wastes as a basis for the elimination and geological conservation of mercury contamination.

HYDROGEOLOGICAL CHARACTERISTICS OF THE INDUSTRIAL AREA OF THE JSC "NOVOSIBIRSK CHEMICAL CONCENTRATES PLANT"

The problem of flooding the territories of large factories is urgent for the enterprises in Western Siberia. This also concerns the right-bank part of the Novosibirsk, where, according to the routine observations of the Novosibirsk Territorial Centre for the State Geological Environment Monitoring, for many years there is a rise of the groundwater level observed. Almost the entire right bank part of the Novosibirsk city, including the industrial area of the JSC NCCP, belongs to the area of flooding, wherein the groundwater level increases up to 10 cm per year.

In order to accomplish the tasks concerning the drainage of enterprises' territories, mathematical modelling is widely used. Constructing the mathematical (hydrodynamic) models allows satisfactorily describing the fluctuations of the level of groundwater in different seasons, including the flood period, calculating the efficiency of drainage work and making adjustments after performing the mentioned above [11]. In 2004–2008, these works were made within the territory of the industrial area of the JSC NCCP.

In order to correctly construct a mathematical model for the dynamic behaviour of groundwater we established a uniform monitoring network (Fig. 1) to perform the annual cycle of observing the groundwater level, with generalizing the physical and mechanical properties of soils, determining the position and the number of water-resistant and water-bearing horizons, their physical, mechanical and filtration properties. On this basis, a digital terrain model was constructed for the industrial area of the JSC NCCP, including the bottoms and tops of water-bearing and water impermeable horizons, the surfaces of groundwater level hydroisohypses were plotted for seasonal periods, together with schemes for aeration of the industrial area and surrounding areas, and with performing a lock-on with respect to man-caused mercury anomalies.

As the results obtained in the course of constructing a hydrodynamic model and solving prediction ecogeochemical problems, it should be noted that: 1) flowing the groundwater within the industrial area of JSC NCCP is free, it can be simulated by the Boussinesq equation; 2) there is no indicating any hydrodynamic interaction between groundwater and underlying aquifer; 3) there is no significant additional water supply associated with technological leaks in the territory of the industrial area of JSC NCCP; 4) with the rise of groundwater above the level of banked earth, quite rapid unloading thereof occurs, due to good filtration properties of man-caused soil layer.

The existing situational mathematical model could be used just now in the course of the calculation and choosing of the drainage systems of the industrial area of the JSC NCCP. However, progressive changing the level of groundwater that occurs from year to year on the right bank of the Novosibirsk City, requires for the fact that the annual monitoring become a basis for constructing the ecogeochemical scenarios of the behaviour of toxic anomalies in the system soil–water–atmosphere to make pre-project decisions [9].

ECOGEOCHEMICAL SITUATION

Basing on the drilling of 32 boreholes to a depth of 8–10 m, with the formation interval testing of ground in these boreholes, areal sampling the industrial area on a scale of 1 : 500 and quantitative mercury determination by means of atomic absorption method, there have been a spatial distribution of man-caused mercury anomalies in the man-caused soil layer and in grounds. The quantitative determination of mercury was carried out at the Analytical Center of the IGM of the SB RAS using modern

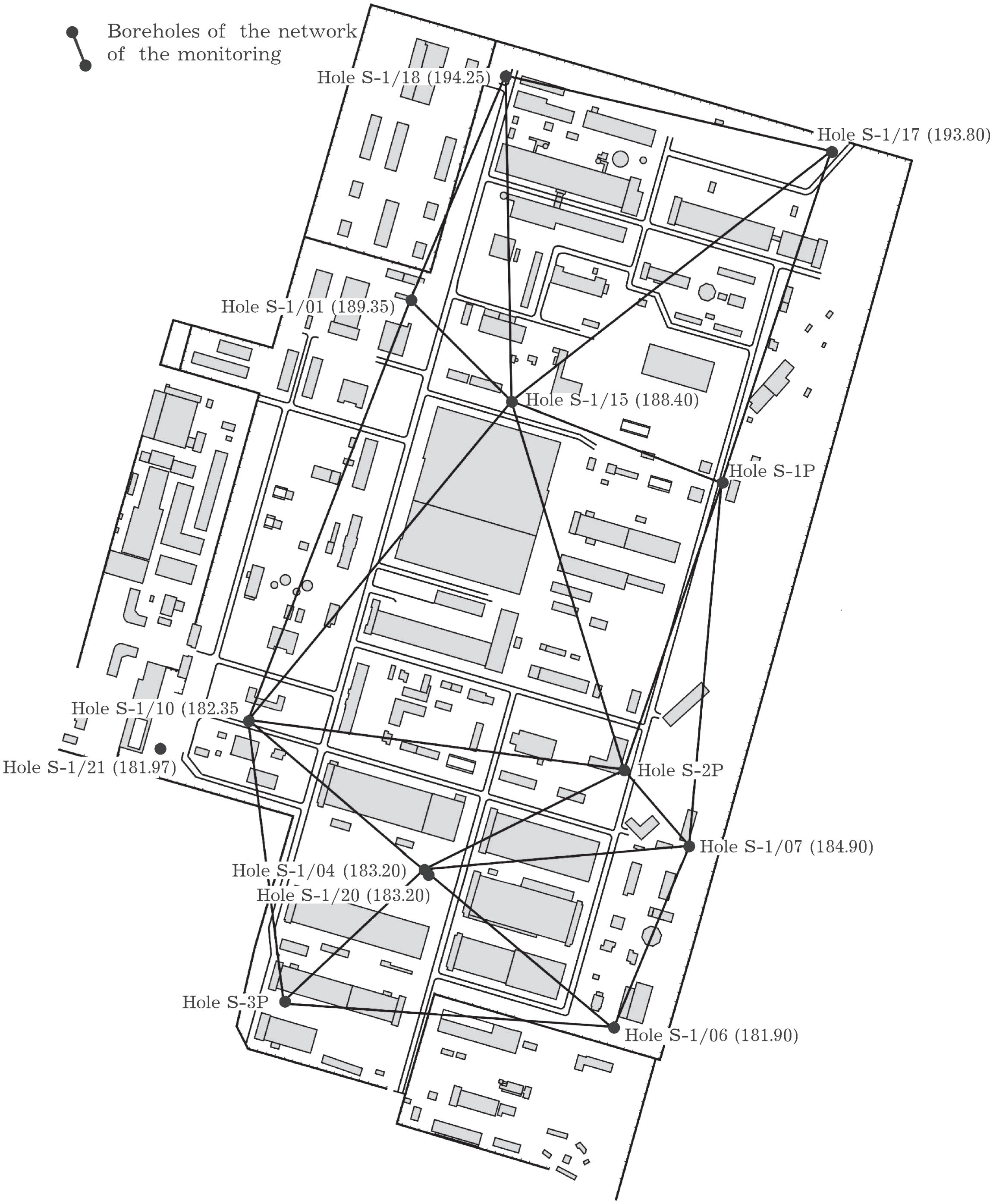

Fig. 1. Industrial area of the JSC NCCP and the network of the monitoring ecogeochemical and hydrogeological boreholes.

GIS technologies using spatial analysis as well as statistical processing and analysis of hydrogeological data (ArcView GIS © ESRI company®). It was found that the general slope of the surface of water-impermeable layer within JSC NCCP exhibit south and southeast direction (Fig. 2). Two "channels" of the underground flows of groundwater flow across the surface of water-impermeable layers were revealed those are separated by uplifted clay and loam features. At the confluence of these channels (the central part of the industrial area) there is an enhanced facility of the sandstone reservoir installed that can accumulate signifi-

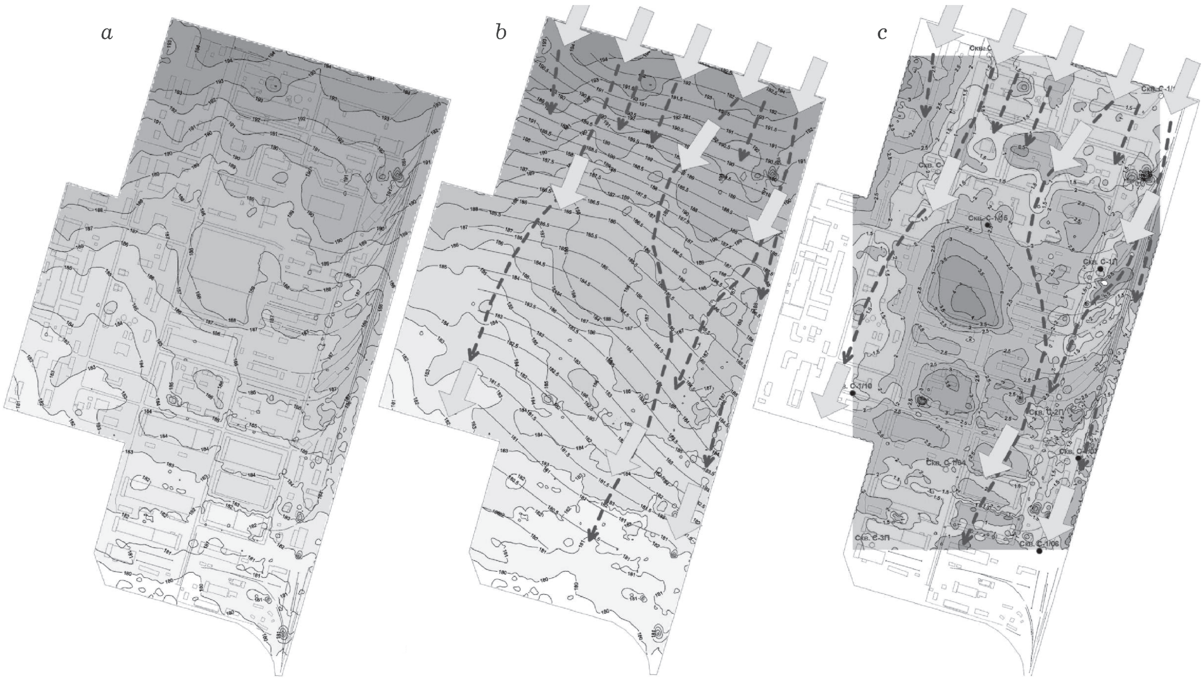

Fig. 2. Hydrogeological model of the industrial area at the JSC NCCP (1998. Compiled by V. G. Vladimirov, A. G. Vladimirov, I. V. Karmysheva): *a* – the position of buildings in the industrial area landforms, *b* – hydroisohypses, natural dissected of the industrial area relief and the directions of groundwater movement along the negative forms of the relief; *c* – a schematic diagram of the depths of the groundwater level and the directions of their in the movement thereof in the territory of the industrial area of the JSC NCCP (Novosibirsk).

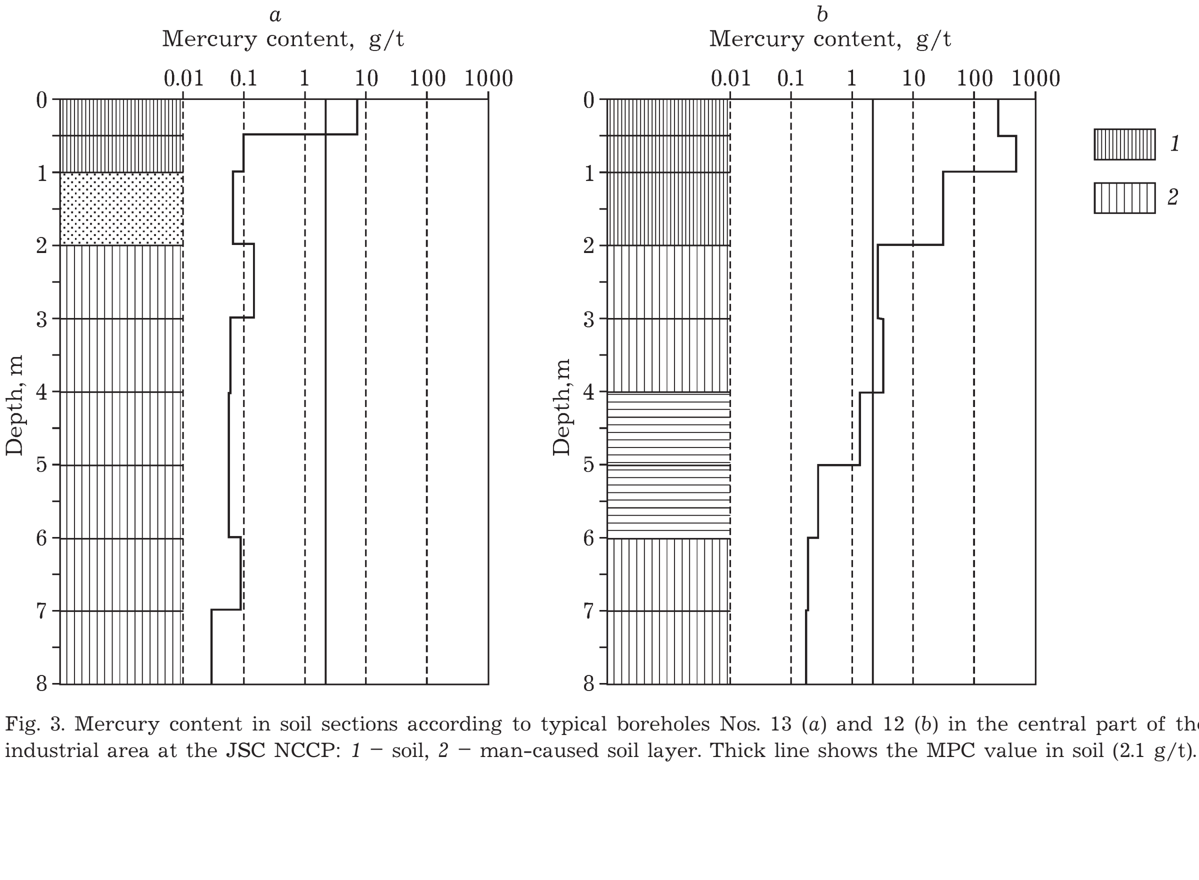

Fig. 3. Mercury content in soil sections according to typical boreholes Nos. 13 (*a*) and 12 (*b*) in the central part of the industrial area at the JSC NCCP: *1* – soil, *2* – man-caused soil layer. Thick line shows the MPC value in soil (2.1 g/t).

cant amounts of groundwater those are draining the mercury contaminated soil sites.

The information concerning the “stocks” of mercury buried within the grounds of the industrial area of the JSC NCCP was amended to a significant extent. It has been found that the vast majority of mercury is concentrated in the upper man caused soil layer of 1 m thick (Fig. 3). With the depth, the mercury amount exhibits a decrease to remain almost unchanged starting from a depth of 3–4 m, which corresponds to the surface of the water impermeable layer, so called retainer (±1 m). The total amount of mercury buried is estimated to be

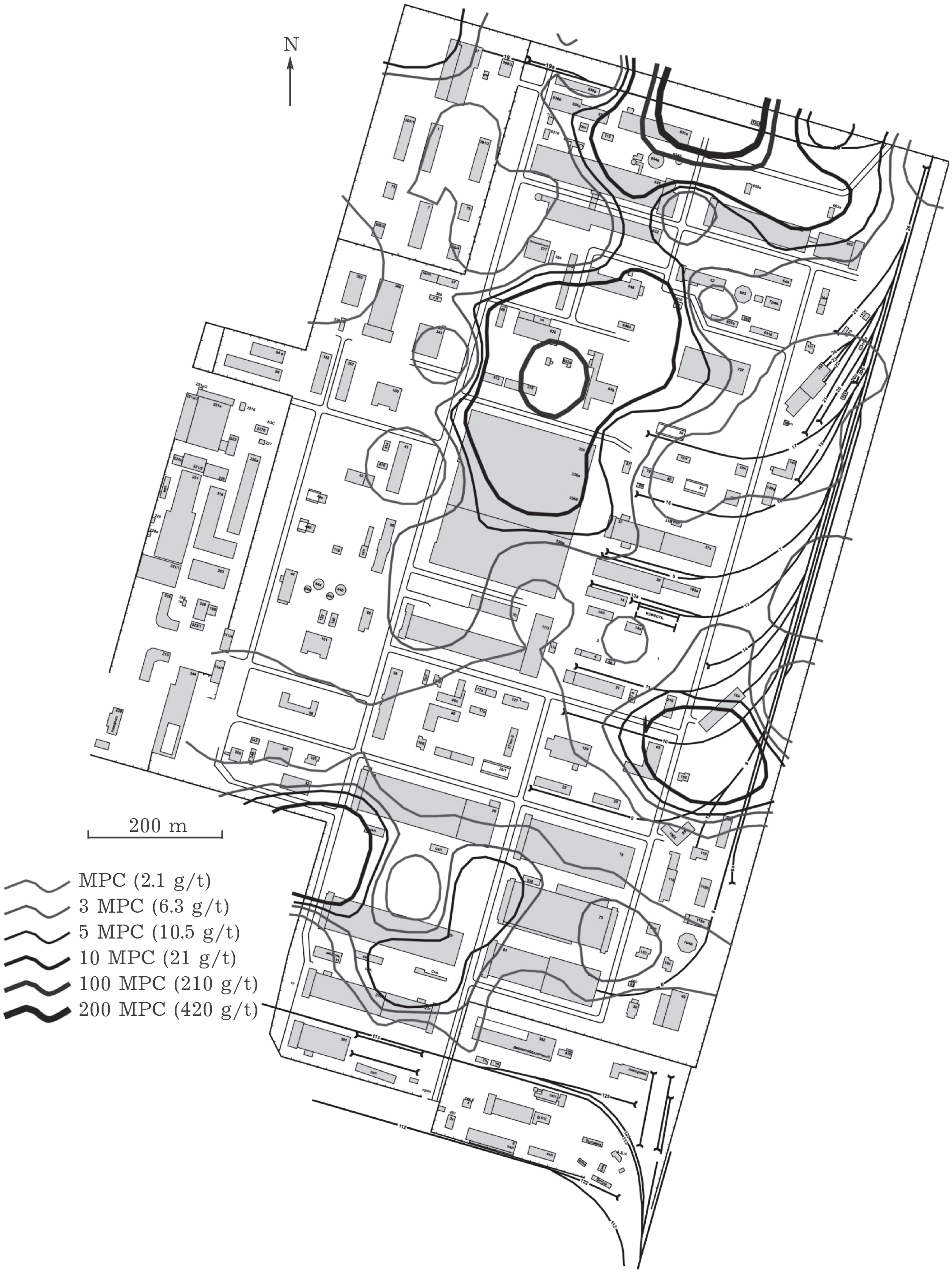

Fig. 4. Mercury anomalies in the surface man caused soil layer the industrial area at the JSC NCCP. Compiled by A. G. Vladimirov, I. M. Belozerov and S. V. Vystavnoy.

33.7 tons. Within the range of 1 m from the surface, there is 26–29 t of mercury contained; at the same time, at the territory amounting to 50 % of the industrial area the mercury content exceeds the MPC level (2.1 g/t). Six major anomalies were revealed wherein the concentration of mercury exceeds the MPC (Fig. 4). Anomaly A is located in the northern part of the industrial area; it is characterized by a high level of mercury in the soil (up to 700 g/t). Within anomaly B, the mercury content amounts up to 400 g/t, further decreasing with the depth to become lower than MPC within the depth range of 4–5 m, with remaining higher in value with respect to the surrounding background. Anomalies C–E significantly inferior to anomalies A and B in the size, depth and mercury concentration values (see Fig. 4). Here the maximum mercury content exhibited in the surface layer does not exceed 40 g/t (10–20 MPC values), whereas at a depth ranging within 2–3 m, these anomalies disappear completely.

A comparative analysis of different mechanisms of mercury transport (man-caused, water-driven, air-driven one) allowed revealing the fact that in the territory of the industrial area there occurs mainly a mechanical transport of mercury, which is caused by the production activity of the plant. The contribution of the water-driven and air-driven mercury transport factors in the territory of the industrial area is several orders of magnitude lower than the mechanical transfer. At the same time, the dynamics of the near-surface (soil-air) mercury anomalies depending on seasonal fluctuations in air temperature remains unknown (the maximum mercury transport value at the industrial area fails for the summer) and depending on floods (the maximum of flood is observed in the spring). The studies performed could be considered to be the first step in demercurization work aimed at reducing the contamination of the industrial area of the JSC NCCP with mercury under the conditions of the operating chemical and metallurgical production of lithium.

LOW TEMPERATURE METHOD FOR THE REHABILITATION OF MERCURY-CONTAINING SOILS AND BUILDING REFUSE

The metallic mercury under normal conditions exists in the liquid aggregative state, having a high ionization potential and a sufficiently low boiling point, which determined the a thermal method to be chosen for the extraction thereof from ore concentrates as the main approach. This traditional method is known as a pyrometallurgical one and consists of two basic operations: ore calcination and the condensation of mercury vapour from technological process gases.

In the course of the treatment of mercury-containing solid wastes, just the thermal method of demercurization is the most widely spread one (roasting the raw, mercury vapour condensation, processing the mercury stupp). However, in most of the cases, the mercury content of cinder sent to dumps is equal to 25–26 mg/kg, which is at least an order of magnitude higher than the MPC value (2.1 mg/kg). Moreover, as it was rightly noticed by Academician I. A. Sheka as early as in the 70ths of the last century, the use of the thermal distillation method in mercury technologies inevitably involves a significant mercury contamination of considerable specific gas volumes, and, therefore, the environment.

In order to reduce emissions it was proposed to convert the residual mercury into stable compounds. For this purpose, the final stage of processing the mercury-containing wastes consisted in blowing selenium or iodine therethrough, which results in converting the mercury into mercury selenides or iodides. The use of selenium appeared to be very expensive, in this case a good sealing of the operating chamber and selenium supply system are required. The application of iodine is more attractive, but a high corrosive activity of this chemical element does not allow using it in the operating chamber. The treatment of wastes with iodine after discharging the waste from the apparatus also results in the release of mercury vapour in the atmosphere.

Mercury readily reacts with chlorine to form mercuric chloride $HgCl_2$. This property of mercury is used in the course of performing the chemical demercurization. As the reagents there are solutions of ferric chloride, potassium permanganate, acidic solutions, sodium hypochlorite or dissolved chlorine recommended [2, 4, 6]. Good results are obtained by using sodium hypochlorite. In order to remove mercury from the solution one could use a variety of methods, including the reduction by formic acid, cementation with the use of aluminum electrolysis, extraction. The characteristics of these methods are presented in the review [4]. The chemical methods allow one, in principle, to provide a very high level of mercury extraction and an efficient demercurization of different objects. However, in each particular case, it is necessary to clarify experimentally the optimum experimental conditions required for mercury leaching to choose the most efficient ways to extract mercury from the solution. Moreover, the use in this process such corrosive reagents as chlorine, and (or) formic acid, even greater cause the negative load on the environment to increase.

The analysis of the various methods aimed at remediation of mercury-contaminated territories performed in the USA in 2007 [6], demonstrated that the most demanded technologies are presented by ones based on solidification procedure, that consists in physical binding the contaminant within a stabilizing mixture, *i.e.*, reducing the solubility, mobility and toxicity thereof. However, the bottleneck of the stabilization technologies is connected with the lack of reliable information concerning the duration of staying the toxic substance in the incapsulated state [8]. The objects of the JSC NCCP to be industrial demercurized are located within the precincts of the megapolis with more than 1.5 million inhabitants, which makes it impossible to apply thermal, chemical, and stabilization remediation methods.

In view of the above mentioned we attempted to investigate the possibility of extracting mercury from contaminated soil and building refuse by means of a low-temperature mechanical method using industrial centrifugal concentrators Knelson ITOMAC. The operation principle of such concentrators is based on the use of an increased centrifugal force (from 60*g* up to 200*g*). The cycle of enrichment begins with supplying the fluidizing water into a rotating conical concentrator. Water is pumped from a water chamber through fluidization holes under a high pressure. Then, pulp is fed through a stationary tube of the feeder. Upon reaching the reflecting plate at the bottom of the cone the pulp is directed upwards under the action of the centrifugal force to fill the cone riffles from bottom to top. The concentration environment (“bed”) appears to be formed when all the riffles are filled. When the optimal fluidization is achieved, the particles a high specific weight (density) are retained within the cone, whereas the lighter particles of waste material are discharged from the top part of the rotating cone.

The enrichment with the use of classical gravitational methods is widely used for a variety of minerals such as coal, iron, manganese, tin, tungsten and other ores as well as rare and precious metal ores, asbestos, kaolin, chalk, etc. In order to evaluate the efficiency of the Knelson ITOMAC concentrator in the course of demercurization process we performed experiments concerning mercury removal from building refuse and soils taken from the JSC NCCP [12].

The samples of grounds (man-caused soil layer) were taken from the sites of the industrial area of JSC NCCP with abnormally increased mercury content. All the samples were divided into fractions +2.0 and −2.0 mm. Quarter samples from the fine fraction without further processing were sent to a laboratory for the analysis for mercury. The coarse fraction was grinded in a jaw crusher, further with the use of a vibration grinder and then it was analyzed for Hg content.

The samples of building refuse were taken in the course of reconstructing the production building from the material of walls, floors on the ground floor of the building and ceiling at the mark +4.5 m. These samples were ground using a jaw crusher, then quarter samples with the mass of 200 g (representative weighed sample portions) were ground into powder using a vibration grinder to be analyzed for the content of mercury.

As a result, we selected soil and building refuse samples most “rich” with respect to mercury content, those underwent to separation on the Knelson concentrator.

Soil samples were taken from the areas with high mercury content. The ground represented a man-caused soil material consisting of a mixture of soil, clay loam and sandy loam with an admixture of gravel and pebbles of various rocks, pieces of construction materials, rare fragments of graphite, tar and pieces of scrap. The mercury content in the shallow (<2 mm) fraction of soils averaged 66 mg/kg, *i. e.*, 32 times higher than the MPC for soil (2.1 mg/ kg).

The mercury content in the coarse (>2 mm) material is much lower to be equal to 2.9 mg/kg, *i. e.*, close to the MPC for soil. The extraction of mercury with the help of the concentrator was made only from the fine fraction of the soil.

Before processing the samples, in the concentrator there was artificial light "bed" of silica sand created to add domestic-use surfactants, which promoted the process of high density phase separation. Basing on the material size and the volume of the sample, the primary pressure in the apparatus was preset at 12 kPa (1 kPa = 0.1 atm). The pressure change by the end of the sample processing cycle was negligible. Data concerning the content of mercury in three soil samples before and after the separation of heavy component concentrates as well as on the mercury extraction level are presented in Table 1.

In order to perform the experiment on the demercurization of building refuse we took an averaged sample with the mass of 12 kg. The sample was first of all milled by means a jaw crusher to obtain particle size of 5 mm, further with the use of a vibration grinder up to the size equal to 0.5 mm. Using a Johnson divider from the averaged sample there were taken three independent quarter samples to treat using the Knelson concentrator. Removing the heavy fraction was performed with no addition of silica and surfactants. The initial pressure of the fluidization water was preset at the level of 14–16 kPa, by the end of the process the pressure is demonstrated a somewhat increase. The enrichment results are presented in Table 1.

It can be seen that the Knelson concentrators allow one to extract from the soil up to 30 % of mercury. According to the XRD phase analysis, in the tailings there remains metallic mercury adsorbed on the surface of the clay particles, as well as mercury in the form of fine-dispersed montroydite (HgO) crystalline phase. We succeeded in extracting up to 85 % of mercury deposited in building refuse. The centrifugal separation thus could be used for the demercurization of contaminated soils and building refuse.

The proposed processing flowchart includes single-stage crushing, grinding, classification, two-stage concentration with the use of the Knelson ITOMAC concentration table to obtain the metallic mercury, as well as thickening and filtering the concentration tailings. The technological flowchart is shown in Fig. 5.

The initial building refuse and soils with the particle size of −340 mm with the help of a forklift is loaded into a hopper of a SMD-521 receiving secondary crushing unit, the crushed material with the size of −80 mm is loaded through conveyor into a receiving hopper with the volume capacity of 32.6 m^3. Further, the material is fed via a feeder to a ball mill. The grinded material is supplied for the classifica-

TABLE 1

Results of mercury extraction from soil and building refuse of the JSC NCCP industrial area using a Knelson ITOMAC concentrator

Sample No.	Mercury content, mg/kg		Mercury extraction level, %
	in the initial samples	in the tailings after extracting the heavy fraction	
		Soils	
G-11	74.0	54.9	25.8
G-14	75.7	60.4	20.2
G-15	61.0	33.2	45.6
Average			29.5
		Building refuse	
20	3160	390	87.7
21	2900	450	84.5
22	2330	420	82.0
Average			84.7

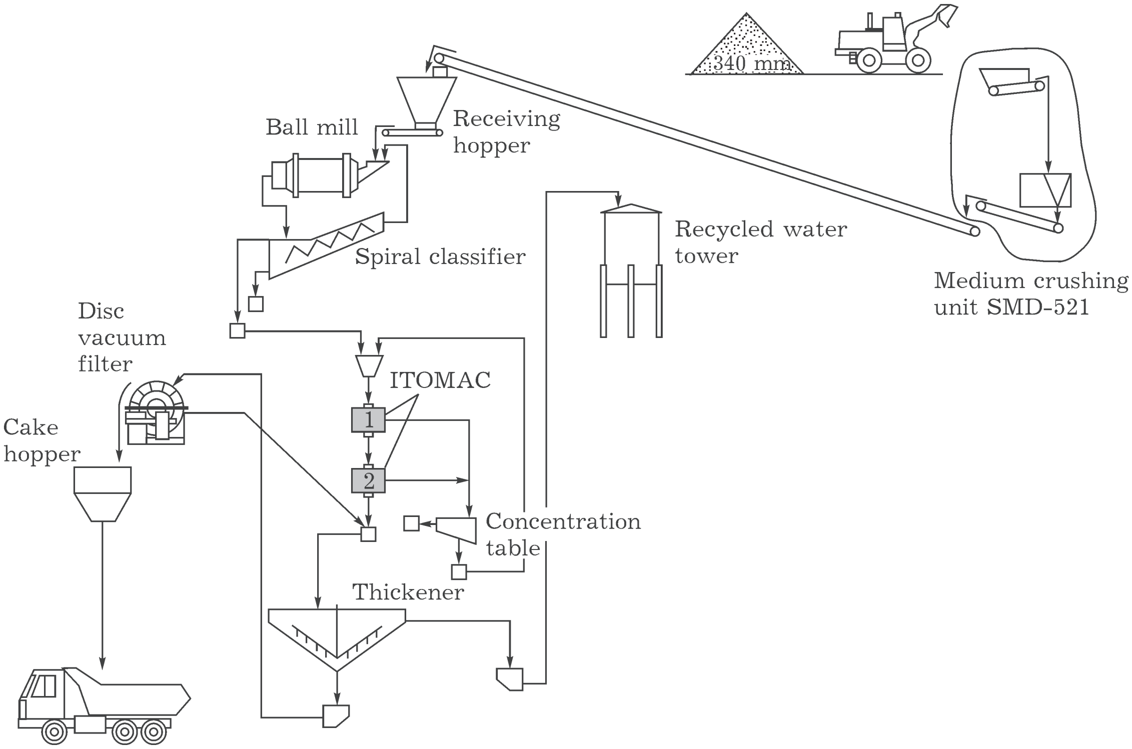

Fig. 5. Flow sheet for processing solid mercury-containing wastes and building materials at the chemical and metallurgical production sector of the NCCP. Compiled by A. V. Babushkin, I. M. Belozyorov, A. G. Vladimirov, V. A. Minin.

tion into a SPE-15 spiral classifier. Classifier sands are returned to the mill for regrinding, whereas the drain is pumped by pumps into a receiving sump located in front of the ITOMAC concentrator, which provides uniform feeding the material. The tailings from ITOMAC-1 are supplied to ITOMAC-2 for repeated purifying. The concentrates from the ITOMAC are supplied to the concentration table in order to extract the metallic mercury. Tails from the table are returned with the help of a pump to the repeated purification in the concentrators. The tails from ITOMAC-2 are pumped by a pump to a thickener. The thickened product is fed to a vacuum disc filter for the filtration. The cake from the filter is sent to the cake bunker, wherefrom it is discharged into the car to be transported to the site of semidry storage. The drains of the thickener and the filtrate from the filter are pumped into the recycled water with the volume capacity of 200 m^3. The recycled water from the tank is supplied to the technological process for grinding, classification, centrifugal concentration, and the concentration on the table. Fresh water is supplied for washing the floors, which is provided twice a day and for replenishing the water loss (water is lost with processing tails). In order to exclude felling metal objects into the mill after secondary crushing unit, an iron catcher is installed above the conveyor.

Mercury-contaminated soils and building refuse at the JSC NCCP after the quantitative extraction of mercury therefrom according to the “cold” technology should be directed to a permanent storage landfill as the materials those belong to I hazard class, because they contain the remaining amounts of mercury both in the form of metal, and oxide exceeding the MPC value. Under the conditions of the tailing pit of the JSC NCCP located in the immediate vicinity of the border of the Novosibirsk City, it is necessary to perform an additional procedure for the immobilization of residual mercury via transferring the mercury by means of chemical methods into insoluble nature-adapted chemical compounds. In practice, as a rule, for such a compound it is appropriate to choose mercury sulphide HgS almost insoluble in water, naturally occurring in the form of cinnabar (basic raw material for the preparation of mercury) or in the form of metacinnabarite. At the same time, the industrial immobilization of residual mercury for the sanitary purposes involves the use of an excess amount of extremely organoleptically undesirable chemical reagents such as sodium sulphide Na_2S or hydrogen sulphide H_2S. Moreover, the excess of any of these reagents could result in the formation of soluble mercury polysulphides.

CHEMICAL IMMOBILIZATION

At the industrial area of the JSC NCCP there are building present to be disassembled. The walls, floors and ceilings thereof contain metallic and oxidized mercury. For primary treatment of the mercury-containing wastes a centrifugal technology is used (Knelson ITOMAC concentrator), which provides up to 85 % extraction of metallic mercury [12]. The remaining 15 % of mercury are in the form of metallic mercury and its oxidized species (oxides and halides) adsorbed on the building refuse. In order to transfer all remaining metallic mercury into the oxidized species required for subsequent improving the hazard class of mercury-containing building refuse, we proposed an oxidative precipitation technology for the treatment of mercury-containing building refuse after ITOMAC basing on the RF patent [13]. It is based on the oxidation of remaining metallic mercury in the waste by means of hydrogen peroxide solution and the subsequent deposition of oxidized mercury by means of the treatment by an aqueous solution of a reagent that converts the mercury into an insoluble compound.

In the course of realizing the technology of oxidative precipitation processing the tailings of mercury-containing building refuse treated with the help of the centrifugal concentrator Knelson ITOMAC, there occurs a two-stage chemical reaction between metallic mercury and hydrogen peroxide with nitric acid added:

$$Hg + H_2O_2 \rightarrow HgO + H_2O$$

$$HgO + 2HNO_3 \rightarrow Hg(NO_3)_2 + H_2O$$

As the result of the mentioned treatment both disperse and adsorbed metallic mercury as well as HgO are converted into single chemical species such as mercury nitrate $Hg(NO_3)_2$.

In the course of the subsequent processing of tailings from mercury containing building refuse by 2 % sodium carbonate (Na_2CO_3) solution there is a formation of almost insoluble basic mercuric carbonate ($HgCO_3 \cdot 2HgO$) observed.

The reaction between mercury nitrate (II) and sodium carbonate can be described by the equation

$$Hg(NO_3)_2 + Na_2CO_3 \rightarrow NaNO_3 + HgCO_3 \cdot 2HgO\downarrow + CO_2$$

As the result of the oxidative precipitation of the tailings of mercury-containing building refuse, all the mercury contained therein is converted into a water-insoluble reddish brown precipitate of basic mercuric carbonate ($HgCO_3 \cdot 2HgO$).

In order to realize the technology of oxidative precipitation processing of the tailings of mercury-containing building refuse treated at the centrifugal concentrator Knelson ITOMAC we proposed a two-stage dual-flow technological flowchart. At the first stage, the latter comprises treating the mercury-containing building refuse by means of reagent No. 1 *i. e.* the mixture of 1.2 % hydrogen peroxide solution, and 1 % HNO_3 solution, in the course of 1 h (the time of H_2O_2 decomposition by building refuse).

After the separation of the suspension by means of filtration and condensation the solid phase is supplied to the second processing stage with the use of reagent No. 2 (2 % sodium carbonate Na_2CO_3 solution), whereas reagent No. 1 is discharged into an intermediate tank for additional concentrating by hydrogen peroxide and nitric acid, with the further supply to processing a new batch of mercury-containing building refuse. The two-staged separation of the suspension (at first *via* thickening the suspension and then *via* filtering the precipitate) allows one to use more efficiently the filtering equipment.

The obtained solid mercury-containing wastes of I hazard class are supplied for processing. After the treatment in the centrifugal field the hazard class is reduced to II class, whereas after the chemical treatment the wastes obtained belong to the hazard class III, which allows performing the geological conservation thereof.

Thus, the technological flow chart allows realizing a two-stage processing of mercury containing building refuse: first by reagent No. 1 and then by reagent No. 2 with two loops for separating the solid and liquid phases. After the treatment of the mercury-containing building refuse by reagent No. 2 and separating the slurry via filtration and subsequent condensation, the solid phase containing insoluble nature-adapted basic mercuric carbonate, with the mercury concentration therein equal to about 0.4 g/kg (III hazard class) is supplied for waste disposal, whereas reagent No. 2 is discharged into an intermediate tank for additional concentrating the sodium carbonate Na_2CO_3. The mercury content in the solid and liquid phases after processing is determined by means of chemical analysis. According to the results of chemical analysis, also the concentration of hydrogen peroxide, nitric acid, and sodium carbonate in the reagents is determined to perform additional concentrating the reagents Nos. 1 and 2.

This scheme allows one to repeatedly reuse reagents, with adjusting the compositions thereof as the content of substances consumed decreases.

SUMMARY

1. A hydrogeological model was constructed for the dynamics of flooding the industrial area of the JSC NCCP, as well as an ecogeochemical model was formed for the behaviour of mercury in the ground-soil-water system.

2. It was established that man-caused mercury anomalies located within the industrial area of the JSC NCCP are concentrated in a man caused soil layer (0–1 m) being of no hazard for the Novosibirsk.

3. A low-temperature mechanochemical (centrifugal) method has been proposed for the rehabilitation of mercury containing soil and building refuse.

4. For the industrial area of the JSC NCCP we patented [13] the method for the sanitary immobilization of residual mercury in solid wastes *via* transforming thereof into water-insoluble basic mercury salts, preferably into carbonate and (or) the sulphate occurring in the nature as mineral schuettite with the composition of $Hg_3O_2(SO_4)$.

5. The combination of the mechanochemical separation and chemical immobilization of residual mercury in a single technological cycle, according to the authors' opinion, would not only solve the environmental problems of the JSC NCCP, but also open up good prospects for using this approach in the case of mercury-containing waste demercurization at gold mines, chemical and metallurgical production enterprises, both in Russia and abroad.

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