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Second Interregional Fertilizer Symposium

Kiev, USSR, 21 September - 1 October 1971
New Delhi, India, 2 - 13 October 1971

Agenda item III/1p

EXTRACTION AND REFINING OF POTASH ORES IN THE USSR[1]

by

R.S. Permyakov

All Union Scientific Research Institute on Halurgy
USSR

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SUMMARY

EXTRACTION AND REFINING OF POTASH ORES IN THE USSR[1]

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The Soviet Union has some of the largest reserves in the world of raw materials for the production of potash fertilizers. These reserves are estimated at 166,084 million tonnes of crude salts.

Nowadays, industrial production is concentrated in three fields - the Upper Kama field in the Urals, the Starobin field in Byelorussia and the field along the Carpathians in the Ukraine.

In 1970, a total of 33,484,000 tonnes was extracted, almost entirely from underground mines. We also have the only open-cast potash ore mine in the world: its annual output is 500,000 tonnes and it is planned to increase this to 1,250,000 tonnes per year.

In underground mining, various modifications of the room-and-pillar method are used, the resulting mineral loss being as high as 40 to 50 per cent.

In the Starobin field, industrial-scale tests have been carried out on the improvement of the room-and-pillar method of mining by progressively supporting the roof of the workings on telescopic pillars. Using this method, it has been possible to increase the rate of extraction of the deposits to 75 per cent.

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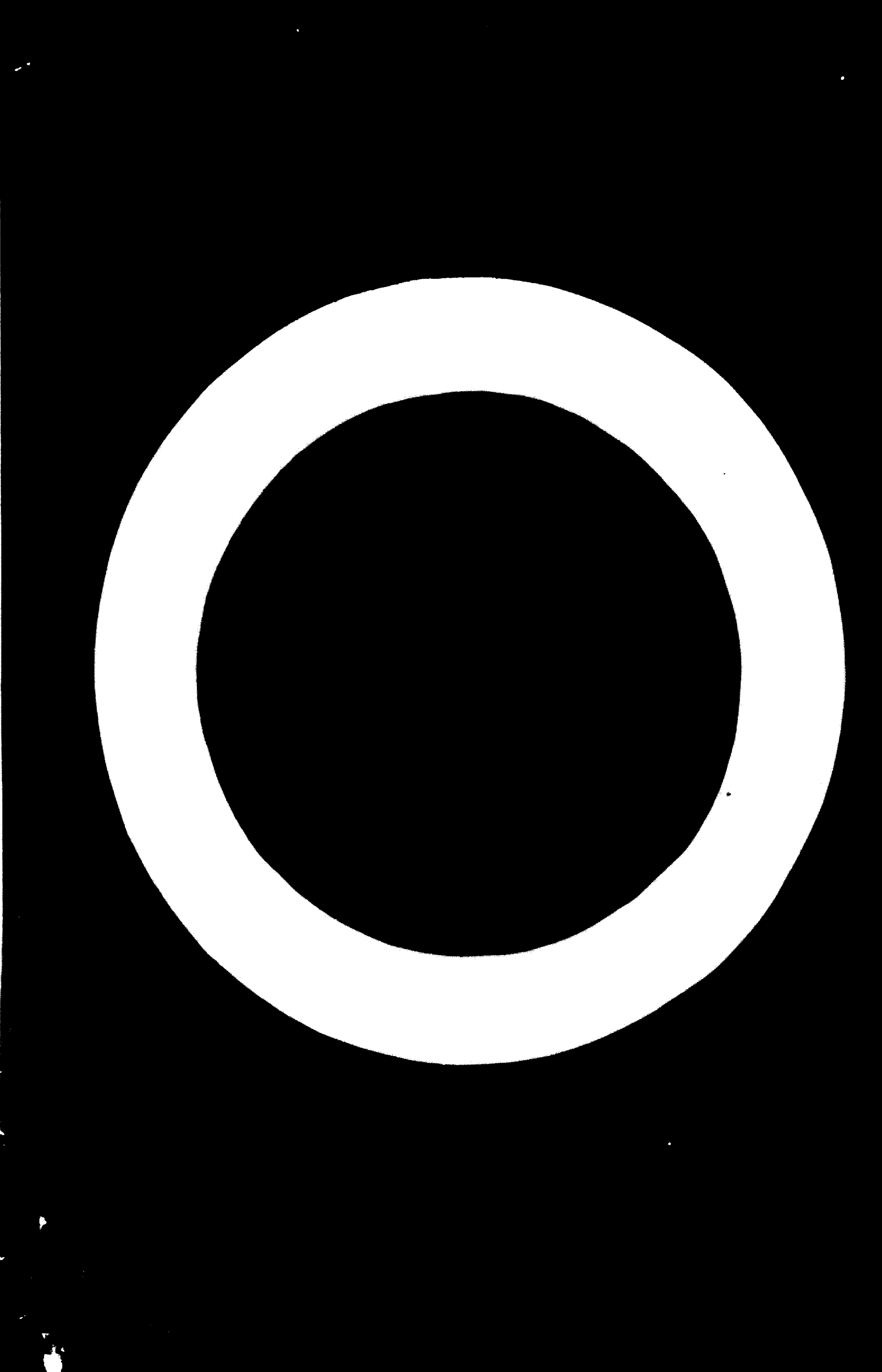

There is a trend towards replacing the blasting method of extracting potash ore by mechanized processes. Accordingly, the USSR has developed special combines, of which there are now more than 150 in operation in the mines. Self-propelled wagons and conveyor belts are used to transport the ore. The introduction of mechanized extraction and modern means of transportation has helped to improve the technical and economic indices.

In 1968, the Soviet Union became the largest producer of potash fertilizers in the world. During that year, it produced 3.1 million tonnes of potassium oxide, while in 1970 it produced 4.33 million tonnes of which 0.9 million tonnes were crystallized using the hot solution-crystallization process. In 1971 it is planned to produce 4.56 million tonnes of potassium oxide.

Eight potash complexes are currently in operation in the Soviet Union. In 1970, two complexes which refine potassium sulphate ores produced 370,000 tonnes of potassium oxide in the form of chloride-free potash fertilizers.

Another two potash complexes will be brought into operation within the next five years. By 1980 the production of potash fertilizers should reach 12.5 million tonnes of potassium oxide.

Two basic technological methods for refining crude potash are used in the USSR: flotation and hot solution-crystallization.

In the flotation method, part of the clay is separated from a slurry of the crushed ore. Potassium concentrate is added to the aerated product which is then dried after thickening and filtration. The clay which has already been separated as a clay-salt slurry is treated by hot countercurrent leaching and the hot solutions are then cooled in vacuum crystallizers. Coarse-grained potassium chloride is being produced commerciall by the flotation method at two potash complexes - the Second Berezniki and the Third Soligorsk.

Generally-known technological schemes and equipment are used in the processing of crude potash by the hot solution-crystallization process. As in the flotation process, the clay-salt slurry is treated by hot countercurrent leaching and the hot lyes are then cooled in vacuum crystallizers. The use in both refining methods of hot countercurrent leaching of the clay-salt slurry means that the final product contains a consider-ably higher proportion of potash. The degree of extraction of potash attained in flota-tion plants is 80 to 85 per cent and in hot solution-crystallization plants - 90 to 92 per cent. At flotation plants, the finished product contains as much as 95 per cent KCl and at hot solution-crystallization plants as much as 98 per cent KCl.

Plants have been planned and constructed for the extraction of various types, grades and applications of common salt from the waste products of potash plants.

A comprehensive technological scheme for commercial processing of polymineral potassium sulphate ores is being put into execution in the deposits along the Carpathians. The recently opened plant at Kalush is producing chlorine-free potash fertilizers - potassium sulphate and potassium-magnesium sulphate. Potassium-magnesium concentrates are being produced at the Stebnikovsk potash complex.

The USSR's potash industry dates from the times of the first two five-year plans of national economy development. Discovery of Verkhnekamsk potash deposit in 1925 - 1927 furnished the basis for putting the first USSR's potash production facility into operation in Solikamsk in the year of 1934. This was followed by sequential commissioning of another processing plant in Berezniki. The Great Patriotic War has subsidized the activities of potash industry which gained its further development during the postwar years. The most intensive development was marked in the last 10 - 15 years. In 1967 production of potash fertilizers (in terms of the equivalent potassium oxide) gained 2.75 million tons and in 1970 totalled 4.33 million tons, as compared to 925,000 tons in 1957.

Beginning from 1968 the USSR holds the first place in a world production of potassium salts. At present, the potash branch of USSR's geochemical industry is the main supplier of potash nutrients in this country, furnishing about 97 % of their total production. For a year of 1971 it is planned to increase the output up to [illegible].6 million tons K_2O equivalent, and it should reach not less than 12.5 million tons by 1980.

At the moment, eight potash processing facilities incorporating also mines adjacent to processing plants are operated in the USSR. Two of these handle potassium sulfate polyminerals. In 1970 their deliveries gained 370,000 tons of potassium oxide in the form of nonchloride fertilizers. The rest 6 produce potassium chloride. The range of produced potassium fertilizers has undergone, as compared to 1965, both qualitative and quantitative changes during the last five years, basically at the expense of increased production of high-grade nutrients. This relates to production of the newely introduced fertilizers, such as potassium sulfate, potassium - magnesia concentrate and potash - magnesia. Potassium chloride analyzing the highest potassium content, prevails in this range.

Its specific share reached nearly 67 % in sum total production of potassium salts in 1969.

Production of potassium fertilizers is concentrated in three areas of basic potassium salts occurences - the Urals (Berezniki and Solikamsk), Byelorussia and Ukraine. The first two deposits are represented by sylvinites used as the raw material for KCl production. Ukrainian deposits furnish potassium sulfates produced in the form of nonchloride fertilizers (potash - magnesia $K_2SO_4 \cdot MgSO_4$, potassium sulfate K_2SO_4 and potassium - magnesic concentrate).

Recovery of potassium ores is quite different as compared to the mining practices applied to other minerals deposits. These peculiarities are governed in the first place by the extremely high solubility of potassium ores, thus the basic requirements here to be followed are the prevention of water and/or brine flood into underground workings. The world-gained experience witnesses nearly 35 cases of flood-inspired loss of potash mines. In several cases it took only a few hours and in others it chews out decades of time-consuming and expensive work directed against water sippage.

Practical experience proves that the distance between water-bearing horizon and strata overlaying top potash bed (miners call this distance as the thickness of water-seal) should be not less than 50 metres to facilitate guaranteed water insulation.

At the same time, the mode of potash deposit extraction should provide for stability of water-seal strata during the entire period of mine exploitation, that means during 40 to 50 years.

All these requirements govern preferential application of the room-and-pillar method of mining, which is characterized by low extraction of the mineral. There had been noted only few cases when the ore recovery from commercial occurences was higher than 40 to 50 %, whilst the recovery from geological resources is much more lower.

It seems necessary to point out that the room-and-pillar method of mining acquires a number of advantages, such as safe, economical and efficient exploitation of a deposit.

Salty rocks, inspite of their low strength, attain high stability, which ensures extraction of large volumes of ore without propping and at a low cost. On the other hand, low strength of salt and its good stability favour mechanization of ore reduction and its haulage by means of the self-advanced and conveyer transport, i.e. it stimulates introduction of the most progressive mining technology - continuous production.

During the last few decades technical advance in the field of exploitation of potash ore deposits was carried out in two following directions.

Firstly, a great number of research programmes were fulfilled in different countries in order to increase recovery of raw materials from underground, since their production is extremely low due to a number of peculiarities, as it has been said previously.

Table 1 summarizes the most widely used parametres of the room-and-pillar method of mining of potassium ores.

Table 1

Mine-and-pillar extraction parametres for different potash mines

Mine	Overlaying strata rocks	Depth of extraction, m	Dimensions, m: room	Dimensions, m: pillar	Ore loss in pillars, %
Solikamsk	rock-salt	250	15	12	[illegible]
Kalusha	salty clay	250	12	6	[illegible]
Gluckauf (GDR)	salty clay	700-800	10	5	[illegible]
1st of Berezniki (AB bed)	rock-salt		14	12	[illegible]
Soligorsk	rock-salt		different versions		[illegible]

Investigations of recovery loss cut down were undertaken to reveal the essence of both physical and mechanical processes taking place in geological strata in the course of exploitation of beds with the help of modern machinery.

Some efforts to decrease ore losses were also undertaken in the direction of packing carved out spaces in mines, production waste being supplied both as dry and liquid media. However, the question of interaction between pillars and packing material still has not been studied exhaustively.

In the Soviet Union the most success in cutting down these losses was marked in the course of exploitation of II bed in Starebin deposit. Principle of a new method of overburden control lies in decreasing the stresses in overburden at the expense of decreasing the width of pillars to such a size, when the latters disintegrate gradually less creating critical pressures in overburden. With decreased width of pillars (down to 1.5 metre), the roof overburden acquires safe state, and pillar gradually disintegrates increasing in width.

Measurements performed with the help of remote instrumentation has truely indicated that at starting point the speed of disintegration is high enough (up to 10 mm per day), being then followed by the abrupt decrease (down to 1 mm per day).

Overburden control providing for gradual lowering of overlaying strata onto pliable pillars ensures 70%-efficiency of ore recovery from a bed.

The second trend of technical advance in recovery of potassium ores is represented by the introduction of mechanized mining instead of drill-and-blast method of recovery. For a long time now the drill-and-blast techniques and scraper haulage of ore were used as classical methods of mining. These methods have already exhausted their potential possibilities, as the production ability of recess and, consequently, the intensity of production were limited by mechanical properties of a scraper winch. That is why most countries have accepted mechanized extraction of ore. Here, the mineral is dug mechanically - by means of a miner cutter bar and then it is transported within the mine with the help of self-advanced mechanisms.

The USSR's potash industry production facilities are provided with efficient miners and means of mechanical transport. New mass-produced miners of PK-8 type possess capacity up to 100 tons per hour, and of PR-10 type - up to 150 tons per hour. Nearly 120 miner machines were used in USSR's potash mines in 1970 having extracted more than 12 million tons of ore.

Application of mechanized methods of recovery has stimulated raise of labour efficiency in potash mines. Thus, for instance, in Starebin basin mines where specific share of mechanized mining is the highest, labour efficiency during the last five years has been raising approximately by 10 per cent annually and in 1970 equalled to 3,500 ton a year per one worker.

Application of conveyor transport along main haulage roads and longwall workings (instead of previouesly used rail haulage techniques) has positively influenced increasing of the labour efficiency.

Potassium chloride is produced in the USSR with the help of two basic technological processes - method of hot dissolution of ore with further crystallization of potassium salts, and method of flotation. The specific share of flotation potassium chloride in its total production during the last five years has increased as contributed by the operation of Soligorsk potash production facilities, where it is produced by method of flotation. Production of flotation potassium chloride gained 1.24 million tons of K_2O equivalent, i.e. 59 % of its total production, in 1969. Beginning from 1970 the specific share of potassium chloride will increase even more due to commissioning of another two flotation plants of "Uralkali" and "Bielorusskali" production facilities. Potassium chloride produced with the help of dissolution - crystallization method contains up to 98 % of the basic substance, whereas flotation potassium chloride analyzes up to 95 % of this.

Basic requirements of the consumer - agriculture - for the single-component fertilizers (and potassium chloride is one of these) necessitate production of potassium chloride in granular and macrocrystalline form. This problem is solved in the USSR by introduction of the method of flotation using new, modernized flotation machinery and reaction regimes in the production of macrocrystalline concentrate. Potash fertilizers produced with the help of this methodics attain improved agrochemical properties owing to the presence of microelements in natural mineral. Another way is the production of macrocrystalline potassium chloride with the help of special method of crystallization in hot saturated solutions. The industrial scale production of macrocrystalline potassium chloride at "Uralkali" and "Bielorusskali" equals to more than 800,000 tons annually.

Methods of potassium ore beneficiation used in the USSR are analogous to these existing in the world practice. However, potassium ores of most USSR's deposits as compared to the foreign ones, have several minerals in their content (silvine, kainite, langbeinite, polyhalite, etc.) and besides that, they exhibit rather high contents of clayey admixtures (Starobin deposit ores - up to 10 % and more, Carpathian deposit ores - - 12-18 % and more). These peculiarities define the complexity of ore processing. In the process of beneficiation of such ores big quantities of insufficiently sedimented clayey slurries are formed with sequential increase in losses, consumption of reagents, etc. This explains also differing rates of recovery of potassium from an ore in production facilities operating by different technological schemes. Thus, flotation plants recover 80-85 % whereas hot dissolution-crystallization facilities recover up to 90-92 % of ore potassium content.

Increase of clayey slurries content in potassium ores of all three exploited at present basins (Starobin, Verkhnekamsk and Carpathian) has required for the further advancement of processing technology and intensified scientific research and pilot-scale and industrial trials. In order to increase recovery of potassium from the ore, there has been elaborated a new method comprising hot countercurrent washing of clay-salt slurry with the subsequent vacuum crystallization of liquors.

Let us have a look at the technology of polymineral sulfate ores processing.

Sulfate polymineral ores of Carpathian deposits are characterized by their instability in mineralogical and chemical composition which strongly complicates their processing. The basic components of salt complex are as follows: kainite, silvine, langbeinite, polyhalite, epsomite, kieserite, anhydrite, halite and clayey substances. Depending on presence and compositive ratio of minerals in ore, it is possible to apply different schemes of processing technology. Thus, production of potassium sulfate alone necessitates presence of free silvine in kainite in equimolecular quantity (or addition of potassium chloride to processed ore):

$$KCl + \underset{\text{kainite}}{KCl.MgSO_4.3H_2O} + aq \text{ -------- } K_2SO_4 + MgCl_2 + aq \qquad (1)$$

In the course of processing kainite not containing free silvine it is possible to produce only sheenite, as stipulated by the following equation:

$$KCl.MgSO_4.3H_2O + aq \text{ -------- } \underset{\text{sheenite}}{K_2SO_4.MgSO_4.6H_2O} + MgCl_2 + aq \qquad (2)$$

Raw materials occupying intermediate position between these two extreme cases permits to produce two products - potassium sulfate and sheenite in different compositive ratios.

Potassium sulfate is a highly effective nonchloride form of a potash fertilizer. According to information provided by agrochemists, it is recommended primarily for tobacco, grape, citrus, as well as for potatoe, flax, hemp, sunflower. Potassium sulfate is applicable to any kind of soil, and primarily to soils of drying zones, tending to salting. The second product of nourishing - shoenite or kalimagnesia (after drying) analyzing approximately 31 % of potassium oxide, is of the same value. This form of fertilizers is recommended for chloride-sensitive crops, as well as for those positively reacting to magnesium (woods, potatoes, winter crops). Application of kalimagnesia (potash-magnesia) seems to be feasible in areas with sandy and sandy clayey soils exhibiting simultaneous need in both potassium and magnesium.

A new technological scheme of Carpathian polimineral ores processing is introduced at Kalusha deposit at present, being subjected to extensive trials on industrial scale. Basic principles of this scheme are as follows. Natural ore containing about 9 % of potassium is subjected after preliminary reduction to a two-step hot dissolution with liquor in dissolving tanks. Resulting solution contains kainite, silvine, epsomite and other readily soluble minerals. Langbeinite, polyhalite and halite are practically insoluble under these conditions. If langbeinite and polyhalite present in mine-run are in notable quantities, then the residue obtained after hot dissolution should undergo additional treatment, e.g. separation. Hot liquor obtained after dissolution is contaminated with clayey substances, contents of which in the mine-run ore equals to 15 % and more. Clarification of this liquor is performed in Dorr settling tanks. Being cooled down, the clarified liquor produces firstly crystals of potassium chloride and sodium chloride, and those of shoenite afterwards. The given sequence of salts crystallization is unacceptable, since sedimentary chlorides will contaminate shoenite - the basic semiproduct.

The contamination is prevented by conversion of liquors, i.e. by adding 20 to 30 % by volume of conversion liquor saturated with magnesium and potassium sulfates less chlorides (sulfate liquor) to the main stream of clarified liquor. Conversion liquor is obtained in the course of shoenite production from potassium sulfate, described below in details. Conversion is illustrated by the following reaction:

$$2KCl + 2MgSO_4 + aq \longrightarrow K_2SO_4 \cdot MgSO_4 \cdot 6H_2O + MgCl_2 + aq \qquad (3)$$

Mixed liquor (clarified and sulfate) is then cooled in vacuum crystallization unit down to +20°C. Resultant shoenite pulp is filtered after preliminary thickening. Damp shoenite is then decomposed with water to obtain potassium sulfate. As a rule, only a part of shoenite obtained during the cycle is decomposed. Some quantity of it should always be removed from the cycle in the form of a finished product. Quantity of sheonite to be removed is determined depending on the composition of processed ore and mixture. The process of potassium sulfate production from sheonite is as follows:

$$K_2SO_4 \cdot MgSO_4 \cdot 6H_2O + aq \longrightarrow K_2SO_4 + MgSO_4 + aq \qquad (4)$$

Shoenite decomposition results in obtaining potassium sulfate as the finished product separated from liquors in centrifugals, and liquors recycled to the hot liquor conversion step, as mentioned above. Thickened clayey-and-salty slime obtained after clarification of hot liquor in Dorr settling tanks is then transferred to countercurrent unit for washing. Countercurrent washing minimizes loss of potassium with slimes. Washing liquor, being mixed with shoenite mother liquor, forms dissolution liquor used in ore dissolution.

The reaction of shoenite production (1) is accompanied by isolation of the equimolecular quantity of magnesium chloride, and concentration of the latter in recirculated mother liquor would increase continuously to disturb finally the proper development of a process. Thus, in order to maintain constant concentration of magnesium chloride in a system, a certained part of mother liquor is continuously recovered. Both potassium and magnesium are recovered from surplus shoenite liquor at the evaporation step, being recycled then back into the process in the form of a so-called artificial or synthetic kainite. The concentrated magnesium chloride liquor is used as raw material in the production of magnesium containing products.

This newely introduced complex technological scheme ensures processing of Carpathian polymineral ores to produce a number of rather valuable products, such as potassium sulfate, potash-magnesia, potassium chloride, sodium sulfate, commercial magnesium chloride liquors.

In parallel with some works in the improvement of potash ores processing technology, a number of scientific and research works is undertaken to elaborate dry methods of separating clayey admixtures from ores, as well as other new methods of ore beneficiation. One of the most perspective directions in desliming of ores with high contents of clayey slimes, is a selective reduction of ore with screening out of large-size fractions containing larger part of these slimes.

Obtained wastes were tested experimentally as manure salts for different soils, demonstrating positive results with some crops. Application of dry methods of desliming will simplify operations of slime processing, providing at the same time for effective solving of a problem concerned in storing the liquid slimes.

There have been also undertaken extensive investigations in the electrical beneficiation of potassium ores in two following directions. First - is a direct production of the finished product. Here, reduced silvinite ore is heated, being simultaneously treated with reagents of benzene-sulfonic acid type, and then transferred into an electric separator; in the course of heating and transportation minerals acquire opposite tribocharges. In the high voltage field these minerals deviate from the direction of vertical drop in accordance with the sign and value of a charge acquired. Semi-industrial scale beneficiation of silvinite ore containing 27 to 29 % of potassium chloride and 2 to 3 % of insoluble residue, produced the concentrate analyzing 90 to 95 % of potassium chloride. Recovery of potassium chloride into the concentrate gained 93 %. The second direction - is the electrical beneficiation of ore prior to its flotation. Here, before being transferred to electrical separation, the ore is subjected to a special kind of heat treating resulting in artificially created difference in electric conductivity between the insoluble residue and the potash minerals. After preliminary heat treatment the ore is sent into drum corona-electric separator. Carpathian polymineral ores containing 13 to 14 % of insoluble residue produced final of 3 to 6 % insoluble residue contents, being processed by the same method. Desliming of silvinite ore containing 7 to 8 % of insoluble residue and 29.5 % of potassium chloride gave beneficiated product of 2 - 2.5 % insoluble residue contents, whereas the resultant slime contained 45 to 50 % of residue.

The abovesaid methods are extensively tested at present on a pilot-industrial scale prior to their forthcoming application during reconstruction of some existing and newely projected potash production facilities.

Working out of the perspective technological schemes for both silvinite and polymineral ores with high contents of clayey slimes will provide for the more efficient complex consumption of the initial raw materials.

In accordance with these schemes the ore is reduced in a selective manner and is dry classified then with the help of high frequency screens.

Overflow is deslimed and transferred to closed cycle flotation, whereas underflow including base quantities of clayey slimes is sent to hot dissolution and crystallization.

Separate cycling of return mother liquors prevents transfer of flotation reagents into dissolution and crystallization section, which ensures production of high-grade table salt and some other products. Utilization of the combined schemes for processing of potassium ores provides for notable increase in the recovery of valuable components from ore and improves economy of technological processes at potash production facilities.

The combined scheme of ore processing (for example, processing of silvinite ores) demonstrates a number of advantages as compared both to flotation and hot dissolution and crystallization methods. The basic advantages here are as follows:

- The entire quantity of the finished product is released in macrocrystalline form. Exclusion of the fine-grained concentrate granulation provides for cutting down both the selfcost of a finished product and the capital investments for construction. Besides that, it is well known, as the experience of some foreign firms also proves, that granulation of flotation potassium chloride is characterized by lower economical merits, as compared to those involved in granulating the crystallization potassium chloride;

- The pure flotation procedure necessitates carrying out of a thorough preliminary desliming of ore with subsequent thickening of the slime product and its countercurrent washing. It still requires high consumption of a depressor reagent;

- The pure flotation procedure excludes the possibility of table salt production;

- The procedure of hot dissolution and crystallization providing for the release of the entire product in the macrocrystalline form, will require higher quantities of technological equipment to facilitate controlled vacuum crystallization;

- The specific consumption of steam per one unit of the released product decreases almost by two times, as compared to the purely halurgic procedure. This plays an important part in area with shortened fuel balance;

- The sum total recovery of potassium chloride from the ore is higher than it reaches in flotation procedure;

- The conditions of ore dissolution are improved greatly owing to the fine-grained fraction feed in hot dissolution section;

- Separation of ore at the beginning of a process into two fractions and transfer of the fine-grained fraction enriched with insoluble residue (about 80 % of the total quantity) into dissolution and crystallization section, shortens the total quantity of thickeners by several times;

- Definite refusal of costly depresser.

Besides these mentioned above ways of improvements in potash ores beneficiation technology and increase of the beneficiation efficiency, all creativeefforts of the Institute will be directed for the next five years to the improvement of the existing equipment for potash production facilities and elaboration of technical data concerning the future construction and development of crushers for selective reduction of ores, dissolving tanks, crystallizers for controlled crystallization, hydroseperators for desliming, thickeners, electric separators with a capacity of 20 tons per hour and higher, filters, sedimentation and filtering centrifugals of high capacity, high frequency screens, etc.

A number of measures for further improvement of the product quality is planned also.

Close attention will be paid to selection of the new effective home-made depresser reagents, collectors, hydrofobizators and coagulants. Development of filterless dehydration of clayey slimes with the application of corresponding floculants is one of a number of planned directions in intensifying the processes of potash production.

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