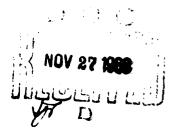
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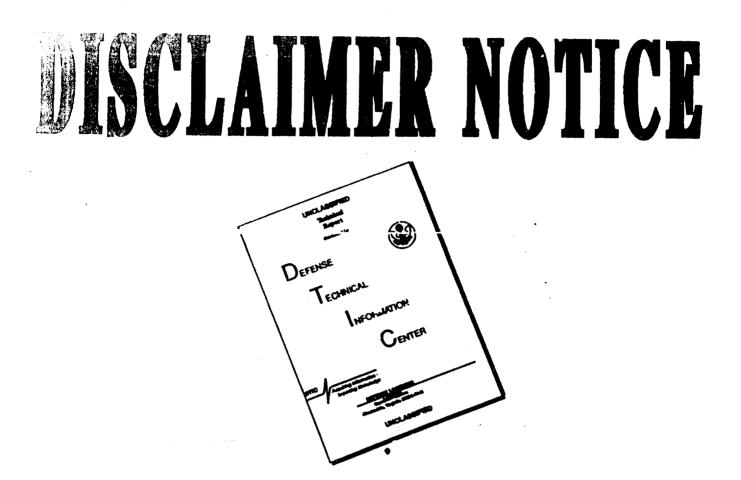
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#### 1. Direction of Scientific Research Work in Giredmet

## By I. Ya. Bashilov

(Realize Metally [Rare Metals], Moscow, No. 4, July-August 1934, pp 3-11.)

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The scientific research work in the field of rare elements is extraordinarily large with respect to its content and very diverse with respect to its forms. With respect to number, rare elements comprise a large portion of the elements that make up the earth's crust. And each of these can be studied with respect to the physical and chemical properties and the raw material in which it is encountered; it is possible to study the methods of its determination and extraction in one or another form from the ore; it is possible to find new forms of the practical application of the given element, etc. In this case when the compounds of the given element are already being obtained in a plant on a large scale, it is possible to achieve an improvement in the process, an increase in the commercial yields, and a simplification of the production systems. Thus, without considering subjects on general problems of rare elements (prospecting, geochemistry, physical chemistry), it is pos-sible to assign to each of the elements pertaining to this group a very large number of problems, the solution of which could be crective and useful for industry. Finally, the extra ordinarily diverse combinations of rare elements in their different ores, in production, and on the bench of the analyst constantly pose and will continue to pose a large number of problems, the solution of which will give rise to new possibilities and new subjects for investigations. And the is obvious that work on all of these subjects could engege an entire army of investigators, which by far exceeds those cadres which we now have in our entire rare-element industry as a whole.

For this reason, in the entire aggregate of the already existing and possible problems on rare elements, it is particularly important to have some kind of basic guiding channels along which the work is to be directed in a definite

- 1 -

sequence and order of priority. These channels cannot, obviously, be unrelated with the general direction and the total clearness of purpose of the work to create a socialist industry to which not only production but any other, including scientific activity, in the Soviet Union is subject. From this stems the first guiding instruction for the arrangement of our work - the satisfaction of the intraunion requirement for rare elements and, in the first place, of the leading branches of industry such as the metallurgical and chemical. This task was more than once underscored by the leading organs of the Soviet Union and it stems from the general position of our union industry as a whole and in the field of rare elements in particular.

The fact of the matter is that rare elements have received full recognition in wide circles of Soviet society, in essence, only during the first five-year plan. At that moment the construction of new plants in the main fields of the national economy and the concern about their normal operation required vanadium, wolfram, molybdenum, and other rare metals, while other branches of industry, which are also rapidly developing in accordance with the common industrial growth require radium, beryllium, lithium, bismuth, selenium and various other still more rare elements.

Work on rare elements was recognized as far from urgent and could not claim the same share of attention as other branches of the national economy, which dealt with elements and objects more ordinary and more known in practice and in industrial use of our country.

Because of this, when the first five-year plan created a large and steady demand for rare elements and this tended to grow rapidly, a break took place between this demand and our possibilities. This break has not been overcome to this day. It appeared, first of all, in the realm of raw-material resources which, with respect to individual elements, were insufficient and, with respect to others, were entirely lacking, when there was no thought-out method which could assure at least some kind of output of the required production. It exists even in the realm of application when the consumers who receive preparations of rare elements do not know how to utilize them for some or other goals. In 1933 the production of rubidium and cesium salts in the Odessa Affiliate of Giredmet was, for example, in such a situation. Output had to be curtailed after the production of the first batches because the customers refused to take the salts they

- 2 -

needed before they mastered the technique of their application in radio and in photo cells.

For this reason, it is quite natural that scientific research is faced with the task of making up, in the first place, at least the indicated most important gaps. Moreover, in the case of Giredmet which, because of its structure was deprived of a special raw-material sector, atten-tion was fixed on the second division - work of a technological nature. The third division - application - could also not be handled by Giredmet to the full extent because in a predominant number of cases it extended far beyond the limits of specialization of the institute and required its special expansion, additional equipment, etc. It seemed quite possible, therefore, under the given conditions, to assign the application problems to the consumer for solution and to give him only possible assistance and the necessary consultation within the limits of competence of the institute as a predominantly chemical-technological organization.

Thus, the logic of events revealed a path which, considering all the available material means of Giredmet, was the only one possible for the direction of the work - by developing the proper technological methods to solve the inquiries of our industry for those elements which are critical and which are particularly important for the national economy.

But even this path is sufficiently broad. It is possible to increase the rare-element resources of industry not only through the technological mastery of new ores and the discovery of new deposits, but also through the improvement of methods to process the already known ores as well as through the reclamation and utilization of old products not longer in use, scrap, rejects, etc. Finally, this is also possible through the rational consumption of the given products, wide utilization of non-critical substitutes for the given rare elements, and partial reduction of the use of the latter in various, less important phases of practice. For this reason, in this connection too a certain selective grouping of the work became unavoidable because the institute which actually does not have its own building would not be capable of picking up these subjects as a whole.

Hence, the outcome was a definite division of the rare elements with the plant which is also conducting investiga-

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and commences and and

tion work in its laboratories. It is perfectly clear that work on the rationalization of production and other work closely connected with production and capable of being fulfilled by means of production apparatus and parallel with production should have been given to the plant itself.

Thus, the institute was primarily left with the work of mastering new types of raw material of critical, rare elements, the replenishment of whose commercial reserves was a task of state importance. Moreover, in the case of various elements, work in this direction had to be conducted with such types of raw material, for which the ordinary methods of processing known, even though from the literature, were either not applicable at all or were only partly applicable.

Such was the first direction of scientific research work in Giredmet. It is, obviously, the first and foremost, because with a natural and understandable limitation of means it is often impossible to undertake the solution of problems connected with the possible change and improvement of the procedure of industries which are operating and giving the necessary production, if there is a shortage of efforts and means for work which will provide our industry with sharply critical rare elements. It is obvious that in case of selection in the given instance the efforts should be directed to the last section in the struggle for rare elements, all the more since most of these, including the sharply critical elements, are of defense significance.

The second direction of work in the field of investigations connected with rare elements, which is also being felt quite clearly and which exerts an influence on the nature of the subjects of Giredmet originates from the nature of the matter and from the specificity of the rare elements.

As is known, the peculiarities of the rare elements are, first of all, that, being rare, they possess various remarkable qualities which cannot be utilized because of this on a practical large scale. And along with this, the rare elements are relatively frequently encountered in the form of highly scattered aggregates which, from the ordinary point of view, lie beyond the limits of commercial recovery. Their reserves in these scattered forms, in low concentrations, are considerably greater than in other, richer forms. Thus, for example, on the entire territory of the Soviet Union, to this day the reserves of this metal in vanadium deposits proper are limited with respect to their

- 4 -

extents, whereas in those ores where this metal is present as a small admixture, the content of which is expressed in fractions of a percent, we have hundreds of thousands of tons of this valuable element. Such is also the case with molybdenum, many tens of thousands of tons of which are now present in various copper ores in which the content of molybdenum is less than 0.2 percent and drops to hundredths of a percent.

Taking into account that many of the indicated elements and minerals are sharply critical, it is natural to strive to find such new, original methods of processing, which would make it possible to utilize these reserves also for commercial recovery.

The technological systems for rare-element raw material are, as a rule, very complex. They consist of various cycles and stages and this leads to an increase in production losses, increase in cost of production, difficulties in changing to large-scale operations, complications in the mechanization of the processing, etc. At the same time, technology is already pointing out a whole series of possibilities and methods which can and should simplify these systems. Included among these methods are, for example, chlorination, sublimation or volatilization, electrolysis, cementation, and even methods of so-called "direct" reduction of the ores of the rare elements. The use of these methods will not only make it possible to reduce the number of production operations, but to reduce the most cumbersome and unpredictable ones. Proof of this can be seen in the attached exemplary systems of processing a rare-element raw material (systems 1 and 2). As regards the method of direct reduction, it appears possible to use it for the complex utilization of rare-element ores. Since there is no melting and smelting of components in this process, but a change of the material being processed, then a combination of "direct" reduction with methods of mechanical concentration and even chemical processing can give a very large effect in simplification and unification of the technological methods in processing generally complex and lean, rare-element ores.

Such a simplification of the production systems is possible also with respect to individual methods or stages of the process. Thus, mechanical concentration in one or another form is a mandatory stage of processing rare-element ores. And in a predominant number of cases during their concentration it is necessary to introduce into the process

- 5 -

an entire assortment of agents available to this method of processing useful minerals. Consequently, this also results in production systems with complex and high-cost equipment. For this reason, flotation with a good coincidence and utilization of all modern achievements of this technological method can give a tremendous simplification of the concentration process, for by means of this, the entire process can be reduced to operations of fine grinding and flotation proper. However, if single flotation does not give the final product, then by means of this method, even in case of need of repeated purifications etc., it is possible to unify the entire production process of the given concentration.

Finally, the peculiarity of the rare-element field is that very many rare elements which attract the attention of modern technology and industry, generally occur in only exceptionally scattered forms and can be extracted only from definite waste from the processing of raw material into mass-use elements, into a compound of ordinary elements.

For this reason, an examination of such types of industries, processes, and all possible waste from these, taken at different stages of production and from different fractions, is in the given case necessary and promising as regards recovery and subsequent utilization of these scattered elements. Moreover, the possibility is not excluded of a certain change in the basic process or in its apparatus, in its technical scheme in order to concentrate the given rare element more fully and in a simpler manner.

These peculiarities of the industry of rare elements require the development of an entirely new approach and a special direction of the work in this field. This direction, in the sense of its accomplishment, is more complex and difficult than the solution of problems of the first direction about which we spoke above. In that case, literature references, description of foreign experience, although very brief, make it possible to solve the rising problems rather rapidly and confidently. Here, however, we need relatively long and systematic work and scientific investigations in the direct and wide sense of this word. For this reason, it is possible to conduct work of this type provided the situation does not require urgent production. Work in this direction is, indisputably, the second and much higher stage in mastering the technology of rare elements. And it is necessary to point out that even foreign

- 6 -

technology has not yet reached this stage. The reason for this, by the way, is also that in foreign countries the requirement for rare elements is satisfied by various firms and plants and because of this, not one of these reaches a production scale which would compel the plants to change from primitive and semiprimitive forms of production to mechanized and large production. Because of this circumstance, there is no stimulus there for a radical re-examination of the technology and perhaps for a radical revision of the status of rare elements in general, but under our conditions this direction of the work has special significance and particularly favorable prerequisites. It promises great prospects and fits in completely with the basic paths of the planned development and building of socialist industry. For this reason, it should always exert a definite influence on the planning of scientific research work in our field and find more or less reflection in the make-up of the subject plan.

Because of this, the direction like that of the resultant of the above described two forces, of which the first is determined by the need to satisfy the requirement of the interests of the current day and by the urgent interests of a socialist industry and the second is connected with much deeper problems of rationalization and development of the rare-element industry and with a much higher degree of its development.

Of course, both described directions or the cross section of the work are very closely intertwined and the task of every investigator, regardless of the conditions of the time and situation, is to conduct his work on a principle and strictly scientific level. But nevertheless, practice shows that in the first stages of the development of our industry and during the moments of unfolding of the work there was a certain, sometimes also a considerable, simplification in the approach to the work. It was shown by a predominance of empiricism, in a predominance of work to find, first of all, formulations for the preparation of one or another preparation, in work which unfolds on wide theoretical foundations and enriches simultaneously theoretical data and blazes more improved technological paths.

With respect to the development of the latter work, special funds for the so-called "theoretical work," which were allocated by the TSNIS NKTP, proved of great support to the work of the institute.

- 7 -

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And so, if one is to approach an analysis of the work of Gi ednet from the point of view of what was just said, then it can be asserted that a great portion of its work consists of the solution of problems connected with the mastery of new types of raw materials, with the expansion of the production base for rare elements, i. e., work of the first direction. It is true, as it was already pointed out, that by force of events the subjects of Giredmet in this direction include work to master raw material, unusual and not coming within the usual framework of foreign practice. In almost all cases the object of the work by Giredmet in this cross section was raw material of low grade and little value. This includes vanadium-containing materials with fractions of a percent of vanadium, extraordinarily scattered radium ores and other types of radium-bearing raw material, lean ores of antimony and mercury from South Fergana, which are frequently bound with fluorspar, lovchorrites, relatively lean rare-earth minerals, eudialites, zirconium minerals with a strongly reduced content of zirconium dioxide, etc. This circumstance unavoidably retards the rates of introduction of this work into industry and Gireamet has been able during the past year to develop to an industrial scale only one work on the extraction of vanadium from the Kerch iron phosphorite ores, which was started during previous years. The other two were developed to the stage where it is possible to set up pilot-plant and semiindustrial operations.

As regards works of a methodological nature dealing with the search for new technological ways, one can name during the current year comparatively few projects and comparatively small in volume. These include projects on the investigation of the possibility of electrochemical and chemical separation of tantalum and niobium, investigations of scientific control of the production of luminescent compositions of temporary and constant action, exploratory work on the application of flotation methods to wolfram ores. Similar investigations were conducted as well of new types of radium-bearing raw material.

Although the raw-material department was abolished in Giredmet last year by a decree of higher-level organizations in connection with the organization of a special prospecting trust for rare elements; nevertheless, the institute conducted work on the site to recover slimes in the hydraulic method of gold mining in the Transbaykal. This work was organized in connection with indications of the presence of

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ing to pilot-plant verification. And as new objects of the same cross section, it is possible to designate a strengthening of the work with respect to industrial waste, which included an investigation of the waste from non-ferrous metallurgy, slimes from gold mining and alluvial deposits containing monazite and zircon from the Pamirs. There has been a strengthening of the work to utilize on a commercial scale the antimony-mercury ores from Kadam-Dzhay and Khaydarkan. A peculiarity of these deposits is, on one side, their impoverishment, and on the other - the small impregnation and non-uniformity in the distribution of valuable minerals in the containing rock.

The main difficulty in processing these ores is the concentration stage which is hindered by the indicated small impregnation of the ore and the considerable development of ochreous, oxidized compounds in interrupted, fractured zones of the deposit. This circumstance makes it difficult, because of the reduced percent of extraction, even to concentrate by flotation. Besides, it is necessary to separate the antimony and mercury at Khaydarkan.

Besides these problems, Giredmet is faced with such entirely new problems as the development of bismuth technology utilizing the Adrasman Deposit in Central Asia, which is 70 kilometers to the northeast from Khodzhent, and the extraction of cobalt from the ores of Dashkesan. The problem of bismuth extraction from the ores of Adrasman is also somewhat difficult because of the nature of the occurrence of the cres, broken up into various veins of variable thickness from 0.2 to one meter and non-uniform mineralization. The veins contain copper pyrite, bismuth and iron glance. The copper is in the amount of two to three percent and the average content of bismuth is 0.75 percent. But on individual sections the bismuth content drops to thousandths of a percent, rising sharply to two percent. The work by Giredmet should encompass here mechanical concentration of the ore mass as well as the production of metallic bismuth.

As regards Dashkesan cobalt, operations at the deposit during the past year have resulted in the discovery of new deposits of the ore material. During this year, methods for the extraction of the cobalt should be tested.

This year has seen a considerable strengthening of the methodological nature of the work or of the work of the second direction about which we spoke above. In this connection, we have a series of projects on the chlorination

- 10 -

of ores and other materials containing rare elements. These have as their aim to test the method of chlorination with respect to certain ores and, in the second place, to start the systematic investigation of this method and of different forms and ways of its application in our field.

The same set-up holds this year with respect to work for so-called cementation of rare metals, i.e., the separation thereof from solutions by means of metals that are lower in the electromotive series. This method, insufficiently studied from a theoretical viewpoint, makes it possible also to hope for a simplification of the processes connected with the production of rare-element concentrates from solutions after the decomposition of their ores.

The next expansion of work in this direction is the organization of a department of economic investigations. This department has as its task to throw light on and solve individual independent problems besides a deeper economic processing of the material which is obtained as a result of the work of the technological section. The fact of the matter is that in the economic section the field of rare elements is particularly unique. Ordinary approaches from the point of view of an economic analysis are not applicable here already because of the single fact that many rare elements are irreplaceable for various fields of the national economy and, at the same time, are consumed in small amounts so that, even with their high cost, they affect relatively little the cost of the product as a whole. Because of this, in the first stage of development of the rare-element industry, problems of cost of production receded to a second plan just as many other features of economic calculations. At the same time, this cross section has problems which can be solved exclusively by means of an economic analysis. These include, for example, problems of the establishment of the lowest concentrations of one or another element in the ores in the case of already known methods of processing these materials. In connection with the critical supply of many rare elements and sometimes chronic critical supply, it is entirely necessary to solve the problem of the rationality of one or another application of the given element and their economic evaluation as regards their comparative effectiveness.

Of the projects which are a continuation of last year's work, almost all experienced a certain modification toward a strengthening of deeper investigations. With respect to

- 11 -

the vanadium problem, for example, along with a continuation of investigation for processing different types of slag from the conversion of titanomagnetites, the investigation was strengthened to clarify the mechanism of certain reactions which take place during the conversion of vanadium slags from Kerch ore, the influence of individual factors on their course and effectiveness, etc. This work was directed toward finding means for increasing the percent utilization of vanadium which is contained in Kerch ore and which at present is very low, in particular in the metallurgical section, in the section for the production of slags from vanadium pig iron.

There was a strengthening of the work with respect to the investigation of phenomena which accompany electrolysis of tantalum and niobium compounds in order to study the possibility of combating the stoppage of the process, which takes place in the given case.

In the field of radioactive investigations, problems connected with the mechanism of the movement of radium into solution from its ores under the influence of some or other salt solutions have been set up and are being solved. This problem is not only of theoretical significance connected with the clarification of the migration of radio elements, but is of practical significance - in the direction of a possible utilization of those significant manifestations of radio-activity which, in the form of highly scattered aggregates of radioactive minerals, are now known in several sections of the USSR. To these can be added the surroundings of the Taboshar Deposit of radioactive ores and a zone which extends along the south of the Fergana Valley and the Zeryavshan Valley, in the Transcaucasus, and certain others.

Thus, the work of Giredmet, last year as well as this year, encompasses a large number of objects, united, on one side, by the amount and form of their occurrence in the earth's crust, and on the other - by the nature of the practical application. The main feature of the work in Giredmet on these objects is the task of their industrial-technological mastery in order to replenish the corresponding resources for the union industry. The work in Giredmet should have and do have as their aim the development of technological processes so as to overcome in a known manner the "rarity" of these objects and actually fully and rationally utilize those tatural resources which are in the earth's crust in the form of aggregates considered lean.

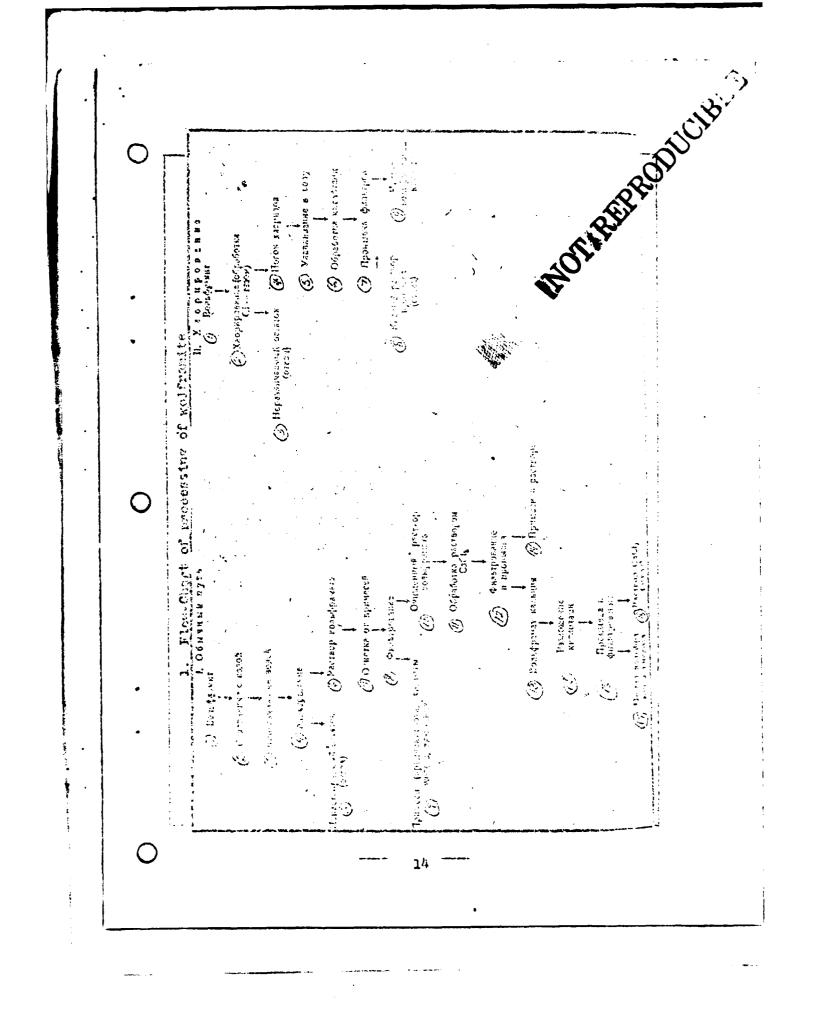
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The basic hindrance which limits the work by Giredmet in the above indicated directions are the shortage and dispersion of those buildings in which it is compelled to develop its work. The significance of the rare elements is recognized, the importance of the work by Giredmet is not disputed by any one and cannot be disputed, but the logical deduction from these positions has so far not been realized. Cirecmet is not provided with the material possibilities for the proper development of its work. Such a condition should be overcome.

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- 13 -



Key to table on page 14:

- I. Conventional method
- 1- Wolframite
- 2- Fusion with soda
- 3- Leaching with water
- 4- Filtration
  - 5- Insoluble residue (Dump)
  - 6- Solution of wolframate
  - 7- Removal of admixtures
  - 8- Filtration
  - 9- Admixtures (silicic acid, iron and aluminum hydroxides)
  - 10- Purified solution of wolframate
- O 11- Treatment with CaCl, solution
  - 12- Filtration and washing
  - 13- Calcium wolframate
  - 14- Admixtures in solution
  - 15- Decomposition with acids
  - 16- Washing and filtration
  - 17- Pure wolframic acid
  - 18- Solution of CaCl<sub>2</sub> (dump)

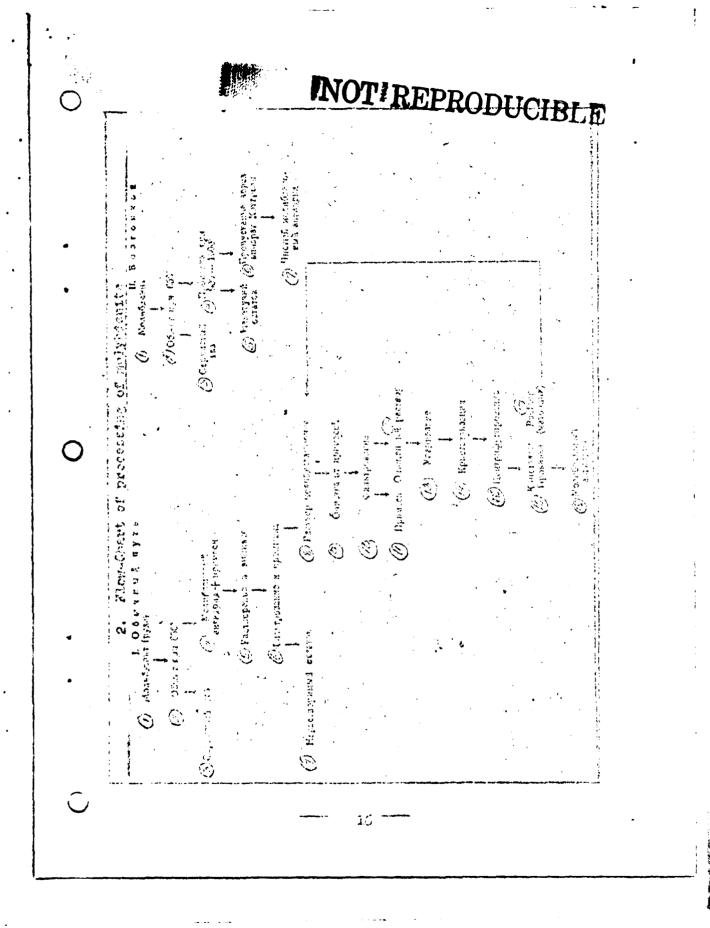
II. Chlorination

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- 1- Wolframite
- 2- Chlorination (treatment with Cl gas)
- 3- Indistinguishable residue (dump)
- 4- Distillation of chlorides
- 5- Trapping in water
- 6- Treatment with acids
- 7- Washing of Tilters
- 8- Acid solution of admixtures (dump)
- 9- Pure wolframic acid

- 15 -

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- Key to table on page 16:
- I. Conventional Method
- 1- Molybdenite (ore)
- 2- Roasting at 650°
- 3- Sulfur dioxide gas
- 4- Molybdenum trioxide + admixtures
- 5- Solution in ammonia
- 6- Filtration and washing
- 7- Incoluble residue
- 8- Solution of ammonium molybdate
- 9- Removal of admixtures
- 10- Filtration
  - 11- Admixtures
  - 12- Purified solution
  - 13- Evaporation
  - 14- Crystallization
  - 15- Centrifuging
  - 16- Crystals Calcination
- 17- Solution (mother liquor)
  - 18- Molybdenum trioxide

- II. Sublimation
- 1- Molybdenite
- 2- Roasting at 650°
- 3- Sulfur dioxide gas
- 4- Calcination at 900-1000<sup>9</sup>
- 5- Non-volatile residue
- 6- Passage through Cottrel apparatus
- 7- Pure molybdenum trioxide

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Key to table on page 18 continued:

7- Apparatus employed
8- 10 analyses per day
9- The same
10- Fuss spectrograph and Zeiss microphotometer
11- Zeiss spectrograph and Zeiss microphotometer

The above indicated methods were developed by the spectral laboratory of Giredmet in connection with work being conducted in the institute on the extraction of indium, gallium, and thallium from industrial products and waste from plants of non-ferrous metallurgy as well as silver from bismuth ores. The use of these methods makes it possible to control extraction methods being developed by technologists and, on the other side, to analyze the products themselves which are presented for examination.

Experience by the laboratory, which has completed over five thousand such analyses, has shown complete suitability of the employed methods for rapid determination of In, Ga, and T1 in solutions with the simultaneous presence of other elements.

In case of travel to the site, the examination of the plants is frequently retarded by the lack of the necessary spectral apparatus, chiefly spectrographs.

In the investigation of products and waste from the Elektrotsink Plant in the city of Ordzhonikidze for the content of indium and thallium, the spectral laboratory of Giredmet used a school spectroscope for the determination of In and T1. After a small alteration, the instrument was completely suitable for the rapid determination of In and T1 by means of the extinction method of A. K. Rusanov (A. K. Rusanov, Visual spectroscopic method of quantitative analysis of solutions. <u>Zav. labor.</u> [Plant Laboratory], 10(1934). The accuracy of the method was equal to + 10 percent.

In this case, the interval of concentrations being determined was 0.002 to 0.2 percent for In and 0.004 to 0.2 percent for T1. By means of the school spectroscope about 300 analyses were made during two months. These revealed the points of the greater concentration of indium and thallium.

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- 19 -