

Annotated Bibliography of the Analytical Chemistry of Niobium and Tantalum January 1935-June 1953

G E O L O G I C A L S U R V E Y B U L L E T I N 1 0 2 9 - A

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Annotated Bibliography of the Analytical Chemistry of Niobium and Tantalum January 1935-June 1953

By FRANK CUTTITTA

SELECTED BIBLIOGRAPHIES OF ANALYTICAL CHEMISTRY

G E O L O G I C A L S U R V E Y B U L L E T I N 1029-A

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the Commission*



UNITED STATES DEPARTMENT OF THE INTERIOR

FRED A. SEATON, *Secretary*

GEOLOGICAL SURVEY

Thomas B. Nolan, *Director*

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SELECTED BIBLIOGRAPHIES OF ANALYTICAL CHEMISTRY

ANNOTATED BIBLIOGRAPHY OF THE ANALYTICAL CHEMISTRY OF NIOBIUM AND TANTALUM JANUARY 1935-JUNE 1953

By FRANK CUTTITTA

ABSTRACT

This annotated bibliography includes reports of work published in the period January 1935 to June 1953 and has been compiled mainly from Chemical Abstracts and from Nuclear Science Abstracts. It refers the reader to the original publications and to abstracts and discussions (summaries) of them. Author and subject indexes are supplied, and also lists of abbreviations used for the serials to which reference is made, and of Atomic Energy Commission code designations and depository libraries where reports so designated can be examined or obtained.

INTRODUCTION

In connection with studies on the analytical chemistry of niobium and tantalum, undertaken by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission, a search of the literature has been made on this subject. Most of the references are accompanied by annotations or abstracts, many of which were copied, in part or in whole, from such standard sources as Chemical Abstracts or Nuclear Science Abstracts; they cover approximately the period January 1935 through June 53. Some annotations were obtained from other secondary sources or from the papers themselves.

The bibliography is arranged alphabetically by author. Titles of reports are in English, as given in the secondary sources.

Lists of code designations of unclassified Atomic Energy Commission reports and of cooperating libraries at which the reports are available for examination follow the general introduction. In turn they are followed by a list — in both abbreviated and full

form — of the serials to which reference is made in the bibliography. Names of serials are in the language of the country, abbreviated according to rules followed in the Geological Survey.

Author and subject indexes are provided for easy use of the bibliography. Each index is preceded by a brief introduction which describes its arrangement. The subject index is most important to the bibliography, and great care has been taken to include index reference to even small amounts of data presented in the annotations and abstracts.

A revision of this bibliography may be prepared in the future. The compiler would appreciate receiving information on errors of omission or commission which have occurred in this compilation.

ATOMIC ENERGY COMMISSION REPORTS

[Key to code designations of Atomic Energy Commission reports. Most nonclassified research and development reports of the Atomic Energy Commission and its contractors are or will be supplied to its depository libraries listed on a following page. Inquiries should be addressed to the libraries]

<i>Code</i>	<i>Installation</i>
AECD	Atomic Energy Commission, Manhattan Engineer District. Declassified documents AECD-1780 on, released after February 29, 1948. <i>See also</i> MDDC.
AECU	Atomic Energy Commission. Unclassified Reports.
ANL	Atomic Energy Commission, Argonne National Laboratory.
CRL/AE	Department of Scientific and Industrial Research, Chemical Research Laboratory, Teddington, England.
K	Union Carbide Nuclear Co., Oak Ridge Gaseous Diffusion Plant.
MDDC	Atomic Energy Commission, Manhattan Engineer District. Declassified documents MDDC 1 to 1779 released prior to March 1, 1948. <i>See also</i> AECD.
NEPA	Atomic Energy Commission, Nuclear Energy for Propulsion of Aircraft.
NP	"Non-Project." University research and development reports received from non-AEC contractors.
ORNL	Union Carbide Nuclear Co., Oak Ridge National Laboratory.
UCRL	University of California, Radiation Laboratory.

ATOMIC ENERGY COMMISSION DEPOSITORY LIBRARIES

[List of the depository libraries to which most nonclassified research and development reports of the Atomic Energy Commission and its contractors are or will be supplied. Reports may be examined at or borrowed from these libraries, and microfilm or photostat copies may be purchased from them. Inquiries should be addressed to the libraries]

<i>State</i>	<i>City and Library</i>
California	Berkeley. University of California General Library. Los Angeles. University of California Library.
Colorado	Denver. Denver Public Library.
Connecticut	New Haven. Yale University Library.

<i>State</i>	<i>City and Library</i>
District of Columbia ...	Washington. Library of Congress.
Georgia	Atlanta. Georgia Institute of Technology Library.
Illinois	Chicago. John Crerar Library. Chicago. University of Chicago Library. Urbana. University of Illinois Library.
Indiana	Lafayette. Purdue University Library.
Iowa	Ames. Iowa State College Library.
Kentucky	Lexington. University of Kentucky Library.
Louisiana	Baton Rouge. Louisiana State University Library.
Massachusetts	Cambridge. Harvard University Library. Cambridge. Massachusetts Institute of Technology Library.
Michigan	Ann Arbor. University of Michigan Library. Detroit. Detroit Public Library.
Minnesota	Minneapolis. University of Minnesota Library.
Missouri	Kansas City. Linda Hall Library. St. Louis. Washington University Library.
New Jersey	Princeton. Princeton University Library.
New Mexico	Albuquerque. University of New Mexico.
New York	Buffalo. Lockwood Memorial Library. Ithaca. Cornell University Library. New York. Columbia University Library. New York. New York Public Library. Troy. Rensselaer Polytechnic Institute.
North Carolina	Durham. Duke University Library. Raleigh. North Carolina State College Library.
Ohio	Cincinnati. University of Cincinnati Library. Cleveland. Cleveland Public Library. Columbus. Ohio State University Library.
Oklahoma	Stillwater. Oklahoma Agricultural and Mechanical College Library.
Oregon	Corvallis. Oregon State College Library.
Pennsylvania	Philadelphia. University of Pennsylvania Library. Pittsburgh. Carnegie Library of Pittsburgh.
Tennessee	Knoxville. University of Tennessee Library Nashville. Joint University Libraries.
Texas	Austin. University of Texas Library.
Utah	Salt Lake City. University of Utah Library.
Washington	Seattle. University of Washington Library.
Wisconsin	Madison. University of Wisconsin Library.

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| British Chem. Abs. | British Chemical and Physiological Abstracts. London. |
| Canadian Min. Jour. | Canadian Mining Journal. Quebec, Canada. |
| Chem. Abs. | Chemical Abstracts. Washington. |
| Chem. Eng. Min. Rev. | Chemical Engineering and Mining Review. Melbourne. |
| Chem. Fabrik | Chemische Fabrik. Berlin. |
| Chem. listy | Chemické listy pro vedu a průmysl. Prague. |
| Chem. Metall. Min. Soc. South Africa Jour. | Journal of the Chemical, Metallurgical, and Mining Society of South Africa. Johannesburg, Union of South Africa. |
| Chem. News | Chemical News. |
| Chem. Soc. Japan Jour. | Journal of the Chemical Society of Japan. |
| Chem. Soc. Japan Jour., Pure Chemistry Sec. | Journal of the Chemical Society of Japan, Pure Chemistry Section. Tokyo. |
| Chem. Soc. [London] Jour. | Journal of the Chemical Society. London. |
| Chemist Analyst | The Chemist Analyst. Phillipsburg, N.J. |
| Chimie anal. | Chimie analytique. Paris. |
| Chinese Chem. Soc. Jour. | Journal of the Chinese Chemical Society. Peiping, China. |
| Coll. Czechoslovak Chem. Commun. . | Collection of Czechoslovak Chemical Communications. Prague. |
| Comm. géol. Finlande Bull. | Bulletin de la Commission géologique de Finlande. Helsinki, Finland. |
| Electrochem. Soc. Japan Jour. | Journal of the Electrochemical Society of Japan. Tokyo. |
| Foundry Trade Jour. | Foundry Trade Journal. London, England. |
| Gos. inst. priklad. khimii Sbornik statey | Gosudarstvennyy institut prikladnoy khimii, Sbornik statey. Moscow. |
| Helvetica Chim. Acta. | Helvetica Chimica Acta. Basel, Switzerland. |
| Indian Acad. Sci. Proc. | Proceedings of the Indian Academy of Sciences. Bangalore, India. |
| Sec. A; Sec. B. | Section A., Section B. |
| Indian Chem. Soc. Jour. | Journal of the Indian Chemical Society. Calcutta, India. |
| Indian Inst. Sci. Jour. | Journal of the Indian Institute of Science. Bangalore, India. |
| Inf. química anal. | Información de química analítica. Madrid. |
| Indus. Chemist | Industrial Chemist and Chemical Manufacturer. London. |
| Indus. Eng. Chemistry, Anal. ed. . | Industrial and Engineering Chemistry, Analytical edition. |
| Ingénieur chimiste | L' Ingénieur chimiste, Bruxelles. |

Inst. Mining and Metallurgy Bull. .	Bulletin of the Institution of Mining and Metallurgy. London.
Iron Age	The Iron Age. New York, N. Y.
Iron and Steel [London]	Iron and Steel. London.
Jour. Applied Physics	Journal of Applied Physics.
Jour. chimie phys.	Journal de chimie physique et de physico-chimie biologique. Paris.
Jour. physique et radium	Le Journal de physique et Le Radium. Paris.
Kaiser-Wilhelm-Inst. Eisenforsch. Düsseldorf Mitt.	Mitteilungen aus dem Kaiser-Wilhelm-Institut für Eisenforschung zu Düsseldorf.
Khim. referat. zhur.	Khimicheskiiy referativnyy zhurnal. Moscow.
Leningrad. krasnoznamenn. khim.- tekhrol. inst. Trudy	Trudy Leningradskogo krasnoznamennago khimiko-tehnologicheskogo instituta.
Metall u. Erz	Metall und Erz, Zeitschrift für Erzbau, Metallhüttenwesen und Metallkunde. Stuttgart.
Metallurgia	Metallurgia. Manchester, England.
Mikrochemie	Mikrochemie. Internationale Zeitschrift für deren Gesamtgebiet. Vienna.
Mikrochemie ver. Mikrochim. Acta ..	Mikrochimie vereinigt mit Mikrochimica Acta. Vienna.
Mikrochimica Acta	Mikrochimica Acta, Organ für reine und angewandte Mikroskopie. Vienna.
Mineralog. Abs.	Mineralogical Abstracts. London.
Mineral. syr'ye	Mineral'noye syr're. Moscow.
Mining and Metallurgy	Mining and Metallurgy. New York, N. Y.
Natl. Bur. Standards Circ.; Jour. Research; Tech. News Bull.	National Bureau of Standards Circular; Journal of Research; Technical News Bulletin. Washington.
Natuurw. Tijdschr.	Natuurwetenschappelijk Tijdschrift. Ghent, Belgium.
Nuclear Sci. Abs.	Nuclear Science Abstracts. Oak Ridge, Tenn.
Nucleonics	Nucleonics. New York, N. Y.
Optical Soc. America Jour.	Journal of the Optical Society of America. New York, N.Y.
Physica	Utrecht, Netherlands.
Redkie metally	Redkie metally. Moscow.
Rev. Chimica Indus.	Revista de Chimica Industrial. Rio de Janeiro.
Rev. métallurgie	Revue de métallurgie. Paris.
Rev. Minera, Geología y Mineralogía	Revista Minera, Geología y Mineralogía. Buenos Aires.
Schweizerische Archiv angew. Wiss. u. Technik	Schweizerische Archiv für angewandte Wissenschaft und Technik. Solothurn, Switzerland.

- Service géol. Congo Belge et Ruanda-Urundi Bull. Bulletin du Service géologique Congo Belge et Ruanda-Urundi. Léopoldville, Belgian Congo.
- Sci. and Culture [India] Science and Culture. Calcutta, India.
- Soc. Chem. Industry [London] Jour. Journal of the Society of Chemical Industry. London.
- Soc. chim. France Bull. Bulletin de la Société chimique de France. Paris.
- Soc. sci. Bruxelles Annales Annales de la Société scientifique de Bruxelles. Louvain, Belgium.
- South African indus. Chemist The South African Industrial Chemists.
- Sovetskaya geologiya Sovetskaya geologiya. Moscow.
- Sovetskaya zolotopromyshlennost .. Sovetskaya zolotopromyshlennost.
- Spectrochim. Acta Spectrochimica Acta. London — New York.
- Staffordshire Iron Steel Inst. Proc. . Proceedings of the Staffordshire Iron and Steel Institute. Stourbridge, England.
- Steel Steel. Cleveland, Ohio.
- Sydney Tech. Coll. Chem. Soc. Jour. and Proc. Journal and Proceedings of the Sydney Technical College Chemical Society. Sydney, New South Wales.
- Tech. Mitt. Krupp Abt. A; Abt. B. Technische Mitteilungen Krupp, Abteil A, Forschungsberichte; Abteil B, Technische Berichte. Essen, Germany.
- Tidsskr. Kjemi, Bergvesen o. Metallurgi Tidsskrift for Kjemi, Bergvesen og Metallurgi. Oslo, Norway.
- Tohoku Univ., Research Inst. Sci. Repts., Ser. A.; Technology Repts. Tohoku University, Research Institute Scientific Reports, Series A, Technology Repts. Sendai, Japan.
- Univ. Nac. Litoral Rosario, Inst. Inv. Microquím. Pub. Universidad Nacional del Litoral de Rosario; Publicaciones del Instituto de Investigaciones Microquímicas. Rosario, Argentina.
- Uspekhi khimii Uspekhi khimii. Moscow.
- K. Vlaam. Acad. Wetensch. Belgie Verh. Verhandlungen van de Koninklijke Vlaamsche Academie voor Wetenschappen, Letteren end Schoone Kunsten van Belgie, Klasse der Wetenschappen. Brussels.
- Vses. konf. anal. khimii Trudy Trudy Vsesoyuznoy konferentsii 'po analyticheskoy khimii. Moscow.
- Zavodskaya lab. Zavodskaya laboratoriya. Moscow.
- Zeitschr. anal. Chemie Zeitschrift für analytische Chemie. Munich, Germany.
- Zeitschr. angew. Chemie. Zeitschrift für angewandte Chemie. Berlin — Leipzig.
- Zeitschr. anorg. allg. Chemie Zeitschrift für anorganische und allgemeine Chemie. Leipzig, Germany.

Zeitschr. Elektrochemie	Zeitschrift für Elektrochemie und angewandte physikalische Chemie. Berlin, Germany.
Zeitschr. Naturforsch. Abt. A; Abt. B.	Zeitschrift für Naturforschung, Abteilung A, Arbeiten aus Astro-physik, Physik und physikalischer Chemie; Abteilung B, Arbeiten aus Chemie, Biochemie, Biophysik, Biologie und verwandten Gebieten. Tübingen, Germany.
Zhur. anal. khimii	Zhurnal analyticheskoy khimii. Moscow.
Zhur. fiz. khimii	Zhurnal fizicheskoy khimii. Moscow — Leningrad.
Zhur. obshchey khimii	Zhurnal obshchey khimii. Moscow — Leningrad.
Zhur. priklad. khimii.	Zhurnal prikladnoy khimii. Moscow — Leningrad.
Zhur. tekhn. fiziki	Zhurnal tekhnicheskoy fiziki. Moscow — Leningrad.

BIBLIOGRAPHY

- 1 Abramov, F. I., 1938, The decomposition of tantalum-columbates, wolframites, and some rare-earth minerals by sulfuric acid: Mineral. syr'ye, no. 1, p. 61-63; cf. Khim. referat. zhur., tom 1, no. 4-5, p. 87 [Russian]; Chem. Abs., v. 33, p. 5765⁵.

An investigation of some contradictory data about the decomposition of some rare-earth minerals.

- 2 Afanas'yev, B. N. (Afanasyev) 1941, The question of the existence of organometallic compounds of tantalum: Zeitschr. anorg. allg. Chemie, Band 245, p. 381-382 [German]; Chem. Abs., v. 34, p. 7869⁸; v. 35, p. 5455.
- 3 Aleksuvskaya, N. V., and Platonov, M. S., 1937, Separation of columbium and tantalum oxides from loparite: Zhur. priklad. khimii, tom 10, p. 2139-2142 [Russian, p. 2143 German]; cf. Chem. Abs., v. 32, p. 3293².

Fusion of loparite concentrate with KOH completely separates Ta_2O_5 and Nb_2O_5 from Fe. The presence of a very small amount of Ti does not interfere with the colorimetric determination of Ta and Nb.

- 4 Alimarin, I. P., 1947, Precipitation of columbium and tantalum in the presence of hydroxy acids by means of the condensation products of formaldehyde and phenols: Zavodskaya lab., tom. 13, p. 547-548, [Russian]; cf. Chem. Abs., v. 42, p. 4488^d.

Description of technique and methods of precipitation of complex compounds of Nb and Ta. Precipitate and ignite to oxide at 900°-1000° C.

- 5 Alimarin, I. P., and Burova, T. A., 1945, Quantitative separation of columbium and tantalum with sodium hypophosphite: Zhur. priklad. khimii, tom 18, p. 289-293 [Russian, English summary]; cf. Chem. Abs., v. 40, p. 3362¹.

H_3PO_2 forms insoluble complex compounds with Ta and Nb in presence of tartaric or citric acid. Only Ta precipitates in presence of NH_4 oxalate. The reaction is specific and applicable to 1:10⁶ concentration. To effect the separation, fuse the mixed oxides with $K_2S_2O_7$, dissolve the melt in $(NH_4)_2C_2O_4$ solution, and precipitate Ta with NaH_2PO_2 as $(Ta_2O_5)H(PO_2H_2)$.

- 6 Alimarin, I. P., and Frid, B. I., 1937, New method for detecting small quantities of columbium and tantalum in minerals, rocks, and metals: Mikrochemie, v. 23, p. 17-23 [German]; cf. Zavodskaya lab., tom 6, p. 823-825 [Russian]; Chem. Abs., v. 20, p. 1574; v. 27, p. 5673; v. 32, p. 76¹.

Niobium and tantalum are precipitated from a solution strongly acid with tartaric acid. Titanium and zirconium interfere and can be

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removed by means of pyrogallol. About 2 micrograms of Nb or Ta can be detected in 1 ml of solution.

- 7 Alimarin, I. P., and Frim, B. I. 1938, Determination of columbium and tantalum by means of benzenearsonic acid: Zavodskaya lab., tom 7, p. 913-916 [Russian]; cf. Chem. Abs., v. 32, p. 762; v. 33, p. 1623¹.

A preliminary report on the application of reagent to the determination of Nb and Ta in a solution containing a large excess of tartaric acid.

- 8 ——— 1938, Separation of columbium and tantalum from titanium and zirconium with the aid of pyrogallol: Zavodskaya lab., tom 7, p. 1109-1116 [Russian]; cf. Chem. Abs., v. 32, p. 762; v. 33, 6. 1623¹, 2064³.

Full details are given for the determination of Nb and Ta in Ti- and Zr-bearing ores, minerals, and silicates by a modified method—using pyrogallol, H_2S , and benzenearsonic acid separations. Precipitate is ignited and weighed as $Nb_2O_5 + Ta_2O_5$.

- 9 ——— 1943, Determination of columbium and tantalum in minerals and ores: Vses. konf. anal. khimii Trudy, tom 2, p. 333-357 [Russian]; cf. Chem. Abs., v. 39, p. 3751⁹, 1945.

Modification of the Schoeller method (fuse Nb_2O_5 with $K_2S_2O_7$; dissolve melt in $H_2C_4H_4O_6$, hydrolyze in 30 ml of the solution in the presence of 5 ml of HNO_3) and colorimetric determination of Nb in the Ta precipitate.

- 10 Alimarin, I. P., and Podval'naya, R. L., 1946, Colorimetric determination of small quantities of columbium as thiocyanate complex: Zhur. anal. khimii, tom 1, p. 30-46; cf Chem. Abs., v. 33, p. 2064⁴; v. 39, p. 3751⁹; v. 43, p. 2543^d.

Separate Ta and Nb oxides; dissolve combined oxides in $KHSO_4 \cdot H_2SO_4$ and the melt in hot tartaric acid; determine Nb with KCNS and $SnCl_2$ in HCl; extract complex with ether (yellow).

Mo, W, U, V, Fe, Cr, Co, Cu, Au, Pt, $C_2O_4^{2-}$, F^{1-} , SO_4^{2-} , PO_4^{3-} , and AsO_4^{3-} , interfere.

- 11 American Society for Testing Materials, 1950, Am. Soc. Testing Materials methods for chemical analysis of metals: Philadelphia, Pa. 126 p.

A detailed description of the determination of Nb gravimetrically by hydrolysis with $HClO_4$ and H_2SO_3 . Nb is also determined by titration with $KMnO_4$ (tantalum by difference).

- 12 Argila, J. S., 1950, Photometric methods for the determination of elements present in steels: Inf. química anal., v. 4, p. 197-205.

Methods are given for determination of Mo, Nb, Ta, Ti, and V.

- 13 Atkinson, R. H., Steigman, J., and Hiskey, C. F., 1952, Analytical chemistry of niobium and tantalum; critical analysis of existing methods: Anal. Chemistry v. 24, p. 477-488.

A critical review of the main separative reactions employed in the analysis of Nb and Ta minerals reveals a number of serious defects. It was thought that some could be overcome by developing nonaqueous separations. For this purpose, a new atmospheric chlorination method was developed, which employs octachloropropane in large excess, heated to 300° C. These separations lay the groundwork for a new system of analysis of the niobite minerals. Ti and Sn are also separated by chlorination and distillation. Fe_2O_3 must be removed because it decomposes the chlorinating agent catalytically.

- 14 Bachelet, M., and Bouissières, G., 1947, Separation of protoactinium from its entrainers—titanium, zirconium, and tantalum: Soc. chim. France Bull., p. 281-283; cf. Chem. Abs., v. 41, p. 7247g.

The authors describe the principal methods of fractionation and of separation of Pa from its entrainers Ti, Zr, and Ta and detail more particularly the fractionation processes of crystallization of double fluorides, the precipitation of hydroxides, and some separations by chemical reactions.

- 15 Bagshawe, I. B., 1936, Modern methods of alloy steel analysis: Staffordshire Iron and Steel Inst. Proc., v. 52, p. 116-142, 1936-37; cf. Chem. Abs., v. 31, p. 52957; v. 32, p. 41024.

A review of the modern technique for analyzing alloy steels, with special consideration of the determination of Mn, Cr, Co, Ce, Ti, Cu, Mo, Al, N_2 , As, Nb, Ta, Zr, Se, and Be.

- 16 Bagshawe, I. B., and Elwell, W. T., 1947, Determination of columbium in rustless and heat-resisting steels: Soc. Chem. Industry [London] Jour., v. 66, p. 398-402; cf. Chem. Abs., v. 42, p. 2541b.

Application of classical methods to separate $(\text{Nb,Ta})_2\text{O}_5 \cdot \text{TiO}_2$, followed by colorimetric H_2O_2 determination of Ti. An examination of six methods was made with respect to Nb analysis.

- 17 Bailey, N. H., 1951, Determination of tantalum and niobium: South African Indus. Chemist, v. 5, p. 235-236; cf. British Abs., v. C, no. 1814, 1952.

The method described for determination of Ta and Nb in ores depends on the difference in stability of solutions of their oxides in aqueous NH_4 oxalate. Niobic acid and its salts are much more readily soluble than tantalic acid in aqueous oxalic acid solution. Separation of Ta and Nb is effected by treatment with tannic acid, which is added to the solution at pH 5 (buffered with NH_4 oxalate) when a yellow Ta complex is precipitated. After this has been filtered off, the filtrate is rendered ammoniacal, when the red Nb-tannic acid complex is precipitated.

- 18 Barber, H., 1947, Determination of columbium and tantalum in basic Bessemer slag: Berg. u. hüttenm. Monatsh. Montan. Hochschule Leoben, Band 92, p. 114-115; Chem. Abs., v. 42, p. 8701a.

The procedure recommended does not provide for separating Nb and Ta. Following classical separations, the final precipitate is fused with

- KHSO₄, the melt is digested with water and H₂SO₃, and the insoluble residue ignited and weighed as Nb₂O₅+Ta₂O₅.
19. Barber, E. J., Farrar, R. L., Junkins, J. H., and Bernhardt, H. A., 1950, Physical constants of niobium pentafluoride: K-652, 27 p.; cf. Nuclear Sci. Abs., v. 4, p. 961 (no. 6568).
 20. Beck, G., 1937, Detection of indium with morin and some heavy metals with cacothelin: cf. Mikrochimica Acta, Band 2, p. 287-290; cf. Chem. Abs., v. 32, p. 16067.

The formation of a colored dyestuff by the reduction of cacotheline is accomplished not only by Sn⁺⁺ but also by Ti³⁺, VO₂⁺⁺, Rh³⁺, low oxides of Mo and W, and NbCl₃. About 1 microgram of any of these elements gives the test.

21. Bhattacharya, H., 1950, Analytical separation of niobium and tantalum: Sci. and Culture [India], v. 16, p. 69-70; cf. Chem. Abs., v. 45, p. 3287b, 1951.

Study of the effect of oxalate concentration and pH on the precipitation of Ta- and Nb-tannin complexes. Control of pH effects complete separation.

22. ——— 1952, Investigations on the analysis of mixed oxides of the elements of group Va and IVa; [Pt.] 1—Preparation of pure tantalum and niobium pentoxide from Indian columbite and their analytical separation: Indian Chem. Soc. Jour., v. 29, no. 11, p. 871-879.
23. Birks, L. S., and Brooks, E. J., 1949, Quantitative analysis of Hf-Zr and Ta-Nb systems by X-ray fluorescence: NRL-3720, (AEC file no. NP-1371); cf. Nuclear Sci. Abs., v. 4, p. 692 (no. 4562), 1950.

The X-ray fluorescence analysis method was adapted to the determination of small amounts of Hf in Zr and Ta in Nb. Curves of relative intensity of spectral lines of Ta and Nb or Hf and Zr were plotted against percent composition by using prepared standard composition.

24. ——— 1950, Hafnium-zirconium and tantalum-columbium systems; Quantitative analysis by X-ray fluorescence: Anal. Chemistry, v. 22, p. 1017; Nuclear Sci. Abs., v. 4, p. 841 (no. 5646).

The X-ray fluorescence analysis method was adapted to the determination of small amounts of hafnium in zirconium and tantalum in columbium. Curves of the relative intensity of spectral lines of Ta and Nb or Hf and Zr were plotted against percentage composition by using prepared standard compositions. As an example of accuracy attainable, with a counting time of 3 to 5 minutes on a 0.5 atomic percent specimen of Ta in Nb, the probable error in Ta content due to statistical fluctuations of the Geiger counter was 0.02, or 4 percent of the amount present.

25. Bleyenheuft, L., 1936, Comparative study of different analytic methods of determining tantalum and columbium: Ingenieur chimiste, v. 20, p. 165-185 [French]; cf. Chem. Abs., v. 24, p. 1055; v. 25, p. 51; v. 31, p. 38174.

The quantitative reactions are reviewed and the results obtained on samples of pure Ta and Nb using the different methods are tabulated. Methods of separating Ta and Nb in minerals from SiO_2 , SnO_2 , TiO_2 , WO_3 , FeO , and MnO are reviewed and the results shown. A bibliography of 36 references is given.

- 26 Blinov, V. I., 1948, Determination of niobium in stainless steel with the aid of the steelscope: Zavodskaya lab., tom. 14, p. 1494-1595 [Russian]; cf. Chem. Abs., v. 46, p. 7931g.

Use of the Nb lines 5095.3 and 4573.1 Å is recommended. With the line 5095.3 Å it is advantageous to use the Fe line 5098.7 Å for comparison. For steel containing 1.5 percent Nb, the intensity of the Nb line 5095.3 Å is equal to the intensity of the Fe line, whereas the intensity of the Nb line 4573.1 Å is close to the intensity of the Cr line 4569.6 Å.

- 27 Blokhin, M. A., 1945, X-ray spectral analysis and its possibilities: Zavodskaya lab., tom 11, p. 1069-1074 [Russian]; cf. Chem. Abs. v. 40, p. 7043⁸.

A review is given of X-ray spectral analysis of elements, beginning with Mg and ending with U. Methods are discussed for the analysis for ores, minerals, and products of concentrating and metallurgical plants, and for the separation of rare elements.

- 28 Borneman-Starinkevitch, I. D., 1939, Decomposition of cassiterite by fusion with borax and some details of its complete chemical analysis: Acad. sci. U. R. S. S. Comptes rendus, v. 24, p. 353-356; cf. Chem. Abs., v. 34, p. 963⁷.

- 29 Borovskii, I. B., 1937, Analysis of minerals by the X-ray spectroscopic method: Akad. nauk SSSR Izv., ser. geol., vyp. 122, p. 929-936; cf. Mineralog. Abs., v. 7, p. 215; Chem. Abs. v. 33, p. 3718².

This method applied to various Zr and Ta silicates shows the presence in zircons of Hf, Sr, and Th, and in eudialytes of Y, Sr, and Ta.

- 30 ——— 1939, Methods of X-ray spectroscopy: Vses. konf. anal. khimii trudy, tom 1, p. 135-42; cf. Khim. referat. zhur., no. 2, p. 57, 1940. Chem. Abs. v. 28, p. 2994²; v. 26, p. 1510; v. 33, p. 1191; v. 35, p. 2089⁴; v. 36, p. 1257⁸.

X-ray spectral analysis (determinations) carried out according to the method of equalization of the intensities, and methods for determining Nb, Ta, Zr, Hf, Zn, Sr, Th, U, Ce, La, Nd, Y, and Mn were developed. The determinations differ by 5 to 20 percent from true value.

- 31 Borovskii, I. B., Blokhin, M. A., and Grzhibovskaya, L. A., 1940, X-ray-spectroscopic quantitative and qualitative analysis of the rare elements: Akad. nauk SSSR Izv., Ser. fiz., tom 4, p. 122-124; cf. Chem. Abs., v. 26, p. 1510; v. 28, p. 2994²; v. 35, p. 474, 2089⁵.

Various minerals and ores were analyzed for the rare elements with the X-ray spectrograph and with a bent crystal. For the specially

prepared mixtures of pure elements (standards) the relation between the intensity of lines and the concentration of Mn, Cr, Sr, Y, Zr, Nb, Co, Ce, La, Pr, Nd, Ta, W, Pb, Th, and U was found.

- 32 Bouissières, G., 1941, Electrolytic fractionation of tantalum oxide containing protoactinium: *Jour. physique et radium*, tome 8, no. 2, p. 72; cf. *Chem. Abs.*, v. 40, p. 17397.

- 33 Breckpot, R., and Creffier, J., 1937; Spectrographic determination of and tantalum: *Soc. sci Bruxelles Annales*, sér. 1, v. 57, p. 290-295; cf. *Chem. Abs.*, v. 32, p. 24529.

Nb and Ta content can be determined in the presence of as much as 9.1 percent Ti.

- 34 British Aluminium Co., Ltd., 1941, Analysis of aluminum and its alloys: *British Aluminium Co., Ltd.*, Pub. 399, p. 186; cf. *Chem. Abs.*, v. 36, p. 47725

The methods of sampling and analysis used in company's laboratories are described. The application of photometric methods, polarographic methods, and other classical techniques are given in detail.

- 36 Brode, Wallace R., 1943, *Chemical spectroscopy*: 2d ed., New York, John Wiley and Sons. p. 472.

Spectrum tables (principal lines of Nb).

- 37 Brubacker, C. H., and Young, R. C., 1951, Anhydrous NbBr_3 and NbCl_3 : *Am. Chem. Soc. Jour.*, v. 73, p. 4179.

Preparation and properties.

- 38 Brüning, K., Meier, K., and Wirtz, H., 1939, Determination of tantalum and columbium in ferrotantalum, ferrocolumbium, and ferrotantalocolumbium: *Metall. u. Erz.*, v. 36, p. 551-554; cf. *Chem. Abs.*, v. 34, p. 12729.

Oxides of Nb and Ta are determined first, then the Nb and Ta are separated by a crystallization method based on the work of Marignac.

- 39 Budanova, L. M., and Gavrilova, K. D., 1949, Determination of tungsten and columbium in steel: *Zavodskaya lab.*, tom 15, p. 7-11; cf. *Chem. Abs.*, v. 43, p. 5335e.

Niobium and tungsten can be separated by cupferron in presence of fluoride. Tungsten can be determined by complex formation with thiocyanate, after reduction, even in presence of 3 percent Mo. Niobium is determined as the oxide.

- 40 Burstall, F. H., Swain, P., Williams, A. F., and Wood, G. A., 1952, Inorganic chromatography on cellulose; [Pt.] 11—A study on the separation of tantalum from niobium and its application to quantitative analysis; *Chem. Soc. [London] Jour.* p. 1497-1504; cf. *Nuclear Sci. Abs.*, v. 6, p. 496 (no. 4018); *Chem. Abs.*, v. 47, p. 1537df, 3753i.

The method of separation is based on their extraction in turn as fluorides, by ethyl methyl ketone in the presence of cellulose absorbent.

Tantalum is extracted first with ethyl methyl ketone saturated with water. Niobium is then extracted with the same organic solvent containing 7.5 percent (volume per volume) of HF (40 percent aqueous solution). It has been shown that, so long as the amount of Ta present does not exceed that of Nb, a sharp and quantitative separation is obtained. The method has been applied to the separation of the earth acids from their mixtures with other metal ions, and to the determination of Nb and Ta in minerals and alloys.

- 41 Burstall, F. H., and Williams, A. F., 1952, Inorganic chromatography on cellulose; [Pt.] 11—The quantitative determination of tantalum and niobium in high- and low-grade complex minerals and ores: Analyst, v. 77, p. 983-999.

A new chromatographic method, based on cellulose as adsorbent and ethyl methyl ketone containing hydrofluoric acid as solvent, is described for the quantitative separation of tantalum from niobium and for their determination in minerals, ores, and synthetic materials.

From a solution of the sample in hydrofluoric acid containing ammonium fluoride, tantalum is extracted first in a volume of 250 ml of ethyl methyl ketone saturated with water. The column of cellulose is then washed with ethyl methyl ketone containing 1 percent volume per volume of hydrofluoric acid (40 percent weight per weight aqueous solution of hydrogen fluoride) which arrests the movements of titanium, tin, and zirconium. Niobium is then extracted with the same organic solvent containing 12.5 percent of hydrofluoric acid. After removal of solvent, the tantalum and niobium are usually determined in the appropriate fractions by ignition to pentoxides.

If phosphate ions are present in the original sample, phosphoric acid is extracted by the solvent used for extraction of niobium; hence the procedure for the final determination of niobium involves a separation of niobium from phosphate ions. Ammonium hydroxide has been successfully used for this purpose.

Owing to the large amount of sample that is necessary for the analysis of a low-grade ore, chemical treatment of such samples is made before chromatographic extraction. This treatment involves the separation of the earth acids together with small amounts of other ions by hydrolytic precipitation in acid solutions.

The procedures are simple and accurate and have been used for the analysis of a wide range of natural and synthetic materials.

- 42 Bykova, V. S., 1938, Quantitative separation of columbium and titanium: Acad. sci. U. R. S. S. Comptes rendus, v. 18, p. 655-657 [English]; cf. Chem. Abs., v. 32, p. 6172⁸.

After an initial HF attack, the sample is given a pyrosulfate-tannin treatment to remove Ti. The values obtained for Nb₂O₅ are 2-4 mg under the true value. With Schoeller's oxalate-salicylate method (Chem. Abs., v. 26, p. 1875) in separating Nb and Ti, a similar loss is obtained.

- 43 ——— 1940, Determination of columbium and tantalum in loparite ores

and concentrates: Zavodskaya lab., tom. 9, p. 1193-1196; cf. Chem. Abs., v. 35, p. 2811¹.

A new method proposed for determining Nb and Ta in loparite ores and concentrates by leaching with hydrochloric acid solution of tannin is to determine TiO_2 colorimetrically and subtract from the total weight of TiO_2 , Nb_2O_5 , and Ta_2O_5 .

- 44 ——— 1940, The separation of columbium from titanium: Akad. nauk SSSR, Trudy Kol'skaya baza, tom 5, p. 77-92; cf. Khim. referat. zhur., v. 4, no. 2, p. 61, 1941; Chem. Abs., v. 37, p. 3008⁶.

The most important old methods are reviewed and the best modern methods for the separation of the mixture Nb + Ta from Ti described in detail.

- 45 Carrière, E., and Guiter, Henri, 1943, Action of V_2O_5 and Cb_2O_5 on alkali carbonates and sulfates: Acad. sci. [Paris] Comptes rendus, v. 216, p. 568-569; cf. Chem. Abs., v. 38, p. 2281⁸, 1944.

Practically all of the compounds formed were soluble in water and fusible.

- 46 ——— 1945, Volumetric methods for the analysis of complex salts: Soc. chim France Bull., tom 12, p. 75-77; cf. Chem. Abs., v. 40, 287⁶.

A discussion of methods which have been found useful for the rapid determination of Cr, Mo, Nb, W, V, and U in their complex salts.

- 47 Chernikhov, Yu. A., and Goryushima, V. G., 1945, A review of contemporary analytical chemistry of tantalum and columbium: Zavodskaya lab., tom 11, p. 875-886; cf. Chem. Abs., v. 40, p. 7058⁴

The paper discusses the separation of earth acids from other elements and gravimetric methods of their determination, volumetric methods for the determination of Ta and Nb, color reactions for Ta and Nb and colorimetric methods of their determination, and analysis of ores, concentrates, ferroalloys, and steel for Ta and Nb.

- 48 Chernikhov, Yu. A., and Karsayevskaya, M. P., 1934, Determination of tantalum and niobium in wolframite: Zeitschr. anal. Chemie, Band 98, p. 97-98.

Application of tannin method to the determination of small amounts of Nb and Ta in wolframite.

- 48a ——— 1938, Volumetric determination of columbium: Zeitschr. anal. Chemie, Band 99, p. 398-402; cf. Chem. Abs., v. 28, p. 6387⁶; v. 29, p. 1358¹.

Careful experiments show wide variations in attempts to obtain correct Nb values by the method of Schwarz (Chem. Abs., v. 38, p. 4003⁶).

- 49 Claffy, E. W., 1949, The polarographic method for determining trace elements in rocks and minerals: Am. Jour. Sci., v. 247, p. 187-199; cf. Nuclear Sci. Abs., v. 2, p. 356 (no. 1595).

This method is applicable to about forty elements, among which are: Ra, Ti, Zr, V, Nb, U, and Ge. Range is about 0.X percent to 0.000X percent.

- 50 Colin, L. L., 1950, The use of tannin in chemical analysis: Chem. Metall. Min. Soc. South Africa Jour., p. 314-319; cf. Nuclear Sci. Abs., v. 4, p. 960 (no. 6565).

Analysis of tantaloniobates, U, separation of Ti from V, and separation of U from Ti are discussed.

- 51 Cordovi, M. A., 1948, Rapid quantitative analysis by X-ray fluorescence: Steel, v. 123, no. 25, p. 88-94; cf. Chem. Abs., v. 43, p. 959, 1949.

A progress report indicating that a few modifications in present equipment will bring the accuracy within limits required in routine determinations of Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zr, Ga, Ge, As, Se, Be, Rb, Sr, Y, Zn, Nb, Mo, Ru, Rh, Pd, Ag, Cd, In, and Sn.

- 52 Cottin, Maurice, 1950, Isotopic exchange between Co and Ta and their ions in solution: Acad. sci. [Paris] Comptes rendus, tom 231, p. 697-699 [French]; cf. Nuclear Sci. Abs., v. 5, p. 12 (no. 80), 1951.

- 53 Cottin, M., and Haissinsky, M., 1950, Electrochemical potential in hydrofluoric acid solution and electrolytic polishing of niobium: Jour. chimie physique, tome 47, p. 731-732 [French]; cf. Nuclear Sci. Abs., v. 5, p. 433 (no. 2689), 1951.

- 54 Creffier, J., 1938, Spectral analysis of columbium and tantalum: Natuurw. Tijdschr. [Belgium], Jaarg. 20, p. 63; cf. Chem. Abs., v. 33, p. 41557.

Optimum conditions are described for the spectographic determination of Nb and Ta to an accuracy of 1.5 percent with a Hilger Fd-60 spectograph.

- 55 Cropper, F. R., and Hamer, A., 1949, A review of infrared spectroscopy in analytical chemistry: Anal. Chim. Acta, v. 3, p. 169-179; cf. Chem. Abs., v. 43, p. 8297.

- 56 Cunningham, T. R., 1938, Determination of columbium and tantalum in stainless steel: Indus. Eng. Chemistry, Anal. Ed., v. 10, p. 233-235; cf. Chem. Abs., v. 32, p. 4461⁹.

Detailed directions are given for carrying out the determination by precipitation of Nb and Ta with cupferron and also by a modification of the Schoeller and Powell (Chem. Abs., v. 22, p. 2899) tannin procedure. $\text{Nb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ are precipitated, ignited, and weighed. Treatment of a solution of the oxides with H_2O_2 and succinic acid reduces Nb to trivalent state, whereas Ta remains pentavalent.

- 57 Davydov, A. L., Vaïsberg, Z. M., and Burkser, L. E., 1947, Photometric method for determining columbium in steel: Zavodskaya lab., tom 13, p. 1038-1043; cf. Chem. Abs., v. 43, p. 4177e.

The blue complex Nb with phosphomolybdate is most intense when the solution is 0.4 - 0.7 N in H_2SO_4 ; the color is independent of the

excess of SnCl_2 used; phosphate above 0.05 mg does not affect color intensity (below 0.05 mg color is weaker). Mn, Cr, Ni, Si, and C do not affect the result, which will agree within 0.01 – 0.02 percent of the true value.

- 58 Delaney, J. C., and Owen, L. E., 1950, The spectrochemical analysis of bismuth matrices employing the porous electrode technique: NEPA-1424; cf. Nuclear Sci. Abs., v. 4, p. 655 (no. 4291).

A spectrochemical procedure for the analysis of Be, Co, Cr, Fe, Mn, Mo, Nb, Ti, Ta, V, and W in bismuth matrices is described. The technique developed calls for the excitation in a porous cup electrode of solutions representing the dissolved sample and containing an added internal standard element. Analytical curves were obtained by means of standard solutions prepared and excited in a manner analogous to that of the samples.

- 59 Doan, Ursula M., and Duval, Clement, 1957, Thermogravimetry of analytical precipitates; [Pt.] 57—Determination of niobium: Anal. Chim. Acta, v. 5, p. 81–83 [French]; cf. Chem. Abs., v. 46, p. 7466b.

Pyrolysis curves of precipitates used for the gravimetric determination of Nb show that none of them can be recommended.

- 60 ————1951, Thermogravimetry of analytical precipitates; [Pt.] 58—Determination of tantalum: Anal. Chim. Acta, v. 5, p. 135–137 [French]; cf. Chem. Abs. v. 46, p. 7466c.

Pyrolysis curves show that the best method for the gravimetric determination of Ta is the one employing tartaric acid. The Ta_2O_5 precipitate is stable above 894° C.

- 61 Dobina, A. A., and Platonov, M. S., 1941, Color reactions for columbium and tantalum: Zhur. priklad. khimii, tom 14, p. 421–422; cf. Chem. Abs., v. 36, p. 2497⁹.

Authors state that the colorimetric tests for Nb and Ta proposed by Shapiro (Chem. Abs., v. 33, p. 1624¹) are unsatisfactory because of low sensitivity. Previous studies of color reactions are in Mikrochim. Acta, v. 3, p. 144–146, 1938, and in Zhur, priklad. khimii, tom 11, v. 11, p. 1029, 1938.

- 62 Dunham, Helen S., and Fred, Mark, 1951, Determination of tantalum, zirconium, and hafnium in columbium by the porous graphite method: AECD-3102; ANL-HDY-582; cf. Nuclear Sci. Abs., v. 5, p. 490 (no. 3055).

Densitometric procedure and a method for concentration of Ta in Nb are described.

- 63 Edwards, J. W., Speiser, Rudolph, and Johnston, H. L., 1951, High-temperature structure and thermal expansion of some metals as determined by X-ray diffraction data; [Pt.] 1—Platinum, tantalum, niobium, and molybdenum: Jour. Applied Physics, v. 22, p. 424–428; cf. Nuclear Sci. Abs., v. 5, p. 548 (no. 3431).

- 64 Efendiev, F. M., 1947, Combined spectral and chemical analysis of microelements: Akad. nauk SSSR Izv., Ser. fiz., tom 11, p. 313-318; cf. Chem. Abs., v. 42, p. 1843f.

A study of the determination of small amounts of elements in soils, ores, and other materials by chemical concentration and spectrographic analysis.

- 65 Eichholz, G. G., 1952, Activation assaying for tantalum ores: Nuclearonics, v. 10, no. 12, p. 58-61; cf. Chem. Abs., v. 46, p. 7468a; v. 47, p. 2638g.
- 66 Elson, Robert E. 1953, The solution chemistry of niobium and tantalum; [Pt.] 2—Spectra of some niobium and tantalum complexes: UAC-712, February; cf. Nuclear Sci. Abs., v. 7, p. 339 (no. 2785).

Qualitative and semiquantitative results obtained in the spectrophotometric study of Nb and Ta complexes (the perchlorate, chloride, tartarate, and fluoride systems) are presented.

- 67 ——— 1951, Reduction of niobium (V) and (IV) and tantalum (V) in solution: AECU-1384; UAC-357; cf. Nuclear Sci. Abs., v. 5, p. 733 (no. 4674).
- 68 Estartus, Francisco Garcia, 1944, Separation of columbium and tantalum; Treatment of columbites and tantalites: Afinidad, v. 21, p. 400-405; cf. Chem. Abs., v. 41, p. 1947i.

The history and sources of Nb and Ta are given, together with all known methods for their separation, purification, and analysis.

- 69 Evans, B. S., and Box, F. W., 1943, Analysis of WC tips: Analyst v. 68, p. 203-206; cf. Chem. Abs., v. 38, p. 5750², 1944.

Provision is made for determining Nb and Ta.

- 70 Fairbrother, F., and Frith, Wm. C., 1951, The halides of niobium and tantalum; [Pt.] 3—The vapor pressure of niobium and tantalum pentafluorides: Chem. Soc. [London] Jour. p. 3051-3056; cf. Chem. Abs., v. 44, p. 2879f; v. 46, p. 4871g.

The vapor pressures of NbF_5 and TaF_5 were measured by a static method using a Bourdon-type sickle gage and by boiling-point determinations under a number of controlled pressures. Thus for NbF_5 , mp = 80.0° and bp = 234.9°C ; and TaF_5 , mp = 95.1° and bp = 229.2°C .

- 71 Fedorov, A., 1940, Determination of titanium, columbium, and zirconium in steel alloys by means of a steelscope at the plant "electrostal": Akad. nauk. SSSR Izv., Ser. fiz., tom 4, p. 212-215; cf. Chem. Abs., v. 35, p. 1725⁸.

A semiquantitative method of analyzing steel alloys for Ti, Nb, and Zr by means of a steelscope is developed (spectroscopic method).

- 72 Fink, Colin G., 1937, Rare metals and minerals; pure electrolytic manganese produced; vacuum tubes important outlet for some metals: Mining and Metallurgy, v. 18, p. 22-25, Chem. Abs. v. 31, p. 2102⁷.

A review of the progress during 1936 in the chemistry and metallurgy of Sb, Be, Cd, Ca, Ce, Rb, Cu, Nb, Ta, Ga, Ge, In, Li, Mg, Mn, Hg, Mo, Mo, Th, Pd, Pt, metals, Po, Ra, U, Se, Te, Na, Ti, W, and Zr.

- 73 Fleischer, Michael, and Harder, J. O., 1946, The geochemistry of columbium and tantalum: MDDC-644, 28 p.
- 74 Fowler, R. M. (by mesne assignment to Union Carbide and Carbon Corp.), 1949, Separation of columbium and tantalum: U. S. patent 2,481,584, Sept. 13, 1949; cf. Chem. Abs., v. 44, p. 527i, 1950.
- 76 Freund, H., and Levitt, A. E., 1951, Colorimetric determination of niobium with thiocyanate: Anal. Chemistry, v. 23, p. 1813-1816; cf. Nuclear Sci. Abs., v. 6, p. 146 (no. 1143), 1952.

This paper describes a colorimetric method based on the yellow color of a niobium thiocyanate complex. The color system is stabilized by using high concentrations of acid, chloride, thiocyanate, and miscible solvent. The absorbancy of the complex follows the Beer-Lambert law in the concentration range up to 3.5 mg of Nb per ml. Interferences and means of minimizing them are discussed.

- 77 Fucke, H., and Daublander, J., 1939, Determination and separation of columbium and tantalum in steel by precipitation with benzenearsonic acid or sodium bicarbonate with addition of hydrogen peroxide: Tech. Mitt. Krupp Forschungsber., Band 2, p. 174-178, cf. Chem. Abs., v. 34, p. 404.

Two methods for separation of Nb and Ta are described. One is based on precipitation of Ta in H_2O_2 -containing H_2SO_4 solution with benzenearsonic acid; Nb stays in the solution, from which it is precipitated later by benzenearsonic acid after destruction of the H_2O_2 . The other method separates the acids of the earth metals in H_2O_2 -containing H_2SO_4 solution with $NaHCO_3$; Ta precipitates first and Nb is separated in the filtrate with H_2SO_4 . A third method describes the simultaneous separation of Nb and Ta in steel by determining the SiO_2 content at the same time.

- 78 Funk, H., and Baumann, W., 1937, Reactions of a few metallic chlorides with phenol and β -naphthol: Zeitschr. anorg. allg. Chemie, Band 231, p. 264-268; cf. Chem. Abs., v. 31, p. 3808^g.
- 79 Furey, J. J., and Cunningham, T. R., 1948, Analysis of simple and complex tungsten carbides: Anal. Chemistry v. 20, p. 563-570; cf. Chem. Abs., v. 42, p. 5798a.

Methods for separation and determination of Nb, Ta, Ti, W, Co, Ni, Fe, Si, Mn, and C are described. Nb and Ta are separated by precipitation with NaOH, followed by regulated hydrolysis and final precipitation with cupferron.

- 80 Gatterer, A., and others, 1945, Atlas of persistent spectra; [Pt.] 3—Spectra of rare metals and some metalloids: Vatican City, Astrophys. Lab. Vatican Observatory; 10 p. cf. Chem. Abs., v. 44, p. 1930, 1950.

Arc and spark spectra are given.

- 81 Geld, I., and Carroll, J., 1949, Colorimetric determination of columbium and tungsten in high-temperature alloys: *Anal. Chemistry*, v. 21, p. 1098-1101; cf. *Chem. Abs.* v. 43, p. 8950i.

Nb is determined as yellow pernibic acid produced by adding H_2O_2 to a solution of Nb_2O_5 in concentrated H_2SO_4 and W as the yellow thiocyanate resulting from the action of $SnCl_2$ and KCNS solutions.

- 82 Gentry, C. R., and Sherrington, L. G., 1948, The photometric determination of tungsten: *Analyst*, v. 73, p. 57, 1948.

In the colorimetric determination of W with KCNS, Nb interferes seriously. Complex is extracted with organic solvents.

- 83 Gest, H., and Glendenin, L. E., 1951, Coseparation of aqueous fission products with manganese dioxide, in Coryell, C. D., and Sugarman, Nathan, eds., *Radiochemical studies—The fission products*, Book 1 (*Natl. Nuclear Energy Ser., Div. IV*, v. 9): New York, Atomic Energy Comm.—McGraw-Hill Book Co., p. 170-175; cf. *Chem. Abs.*, v. 47, p. 5839f.

In dilute HNO_3 , MnO_2 is effective as a general scavenger for the fission-products elements Nb, Te, Zr, and possibly others. In acid concentrations above 10 M, Nb, Te, and Mo are specifically removed. The coseparation of Nb^{95} with externally formed, freshly precipitated wet MnO_2 in 10 M HNO_3 is quantitative after 30 minutes of contact.

- 84 Gibson, K. S., 1949, Spectrophotometry (200 - 1000 m μ): *Natl. Bur. Standards Circ.* 484; cf. *Chem. Abs.*, v. 44, p. 27, 1950.

A review with 127 references.

- 85 Gilbert, Paul T., Jr., 1952, Flame photometry—New precision in elemental analysis: South Pasadena, Calif., Beckman Instruments.

Determination of alkalies, alkaline earths, Nb, and other materials.

- 86 Giles, W. B., 1907, Observations on niobium, tantalum, and titanium: *Chem. News*, v. 95, p. 37.

Reduction of Nb in H_3PO_4 solution with Zn.

- 87 Gillis, J., 1944, Critical study of reagents for cations (vanadium, columbium, tantalum): *Mikrochemie ver. Mikrochim. Acta*, Band 31, p. 273-285; cf. *Chem. Abs.*, v. 38, p. 1979g.

Tests are given for the detection of Nb and Ta.

- 88 Gillis, J., and Eeckhout, J., 1947, Quantitative spectrochemical analysis of steel: *Anal. Chim. Acta*, v. 1, p. 377-389; cf. *Chem. Abs.*, v. 42, p. 4493i.

Operating conditions are described for the spectrographic analysis of Fe steel and C steel. Technique based on the Scheibe and Rivas method. Analysis line and standardizing tables are given for Ni, Mn, Cr, Co, Ti, Al, Mo, V, Zr, As, Cu, Nb, and Ta.

- 89 Gillis, J., Eeckhout, J., and Poma, K., 1944, Separation of columbium and tantalum with ferroin in hydrogen fluoride solution: *K. Vlaam.*

Acad. Wetensch. Belgie., Verh. v. 6, no. 10, p. 5-10; cf. Chem. Abs., v. 40, p. 43141.

Repeated precipitation and solution of the salts in HF solution with ferroin is a useful method for separating Ta and Nb.

- 90 Giratto, A., 1940, Scheme for analysis of samarskite: *Rev. Chimica Indus.*, ano 9, no. 104, p. 10-2 cf. Chem. Abs., v. 35, p. 35576.

Determination of rare earths, Th, U, Ca, Mg, Nb, Ta, Ti, Zr, Fe, and Mn, in samarskite is described.

- 91 Goldbersuch, E., and Young, R. C., 1949, Complex sulfates containing Nb^{3+} and Nb^{5+} and the possibility of their use in the purification of niobium: *Am. Chem. Soc. Jour.*, v. 71, p. 2402-2405.

- 92 Golubtsova, R. B., 1951, Determination of niobium in high-alloy steels free of tungsten: *Zhur. anal. khimii*, tom 6, p. 34-38.

Nb_2O_5 is determined colorimetrically with hydrogen peroxide.

- 93 ——— 1951, Determination of tungsten and niobium in high-alloyed alloys: *Zhur. anal. khimii*, tom 6, p. 357-363; cf. Chem. Abs., v. 46, p. 2957a.

Determination of Nb with tannin and gallic acid.

- 94 Gorbach, G., and Pohl, F., 1951, Concentration and spectroscopic determination of trace metals; [Pt.] 1—Extraction with organic reagents: *Mikrochemie ver. Mikrochim. Acta*, Band 38, p. 258-267; cf. British Abs., v. 1C, p. 30 (no. 232) 1952.

Errors due to precipitation and electrochemical techniques for the concentration of trace metals prior to spectroscopic determination are avoided by extraction methods. Studies with the following reagents capable of forming $CHCl_3$ -solution metallic complexes are detailed: 1-benzoinoxine, dithizone, isonitrosoacetophenone, cupferron, 2-nitroso-1-naphthol, 8-hydroxyquinoline, salicylaldoxine, and thionalide, in conjunction with the following metals: Ag, Al, As, Au, Bi, Cd, Ce, Co, Cr, Cr, Cu, Fe, Fe, Ga, Hg, In, Mn, Mo, Nb, Ni, Os, Pb, Pd, Pt, Sb, Sn, Ta, Th, Hf, Ti, V, W, Y, Zr, and Zr. Qualitative and quantitative examination is affected by sparking the extracts, precipitate, and residual solutions after each extraction. In general, successive extraction with two reagents is necessary for the collection of the above trace metals, dithizone and 8-hydroxyquinoline being preferred.

- 95 Goto, Hidehero, and Kakita, Yachiyo, 1950, Colorimetric determination of niobium and tantalum: *Tohoku Univ., Research Inst. Sci. Repts.*, Ser. A, v. 2, p. 249-254; cf. Chem. Abs., v. 45, p. 7913i, 1951.

Determination of Nb based on the yellow color formed with pyrogallol and Na_2SO_3 at pH 4.6 to 6.8, and that of Ta on the yellow color produced in a solution which is nearly 3 N H_2SO_4 and contains a little sulfite. Bi, Sb, Fe, and Ti interfere. As little as 0.4 gamma of Nb per ml or 50 gammas of Ta per ml can be determined.

- 96 Grube, G., and Grube, Hans L., 1938, Electrochemistry of columbium: *Zeitschr. Elektrochemie*, Band 44, p. 711-780; cf. Chem. Abs., v. 33, p. 20434.

An apparatus is described and illustrated which provides for electrolysis, potential measurement, and titration with KMnO_4 in an atmosphere of hydrogen.

- 97 Gul'dina, E. I., and others, 1940, Analysis of ferrocolumbium: *Zavodskaya lab.*, tom 9, 500-507; cf. *Chem. Abs.*, v. 34, p. 7212³.

On the basis of literature and experimental data, procedures are described for the analysis of Fe-Nb, including the determination of Nb, Ta, Ti, Sn, Al, and Si. Final determination is as the oxides.

- 98 Hagiwara, Hisato, 1948, Application of liquid amalgams to the volumetric determination of columbium: *Chem. Soc. Japan Jour.*, Pure Chemistry sec., v. 69, p. 129-133; cf. *Chem. Abs.*, v. 44, p. 9299ⁱ.

- 99 Hahn, R. B., 1950, Tantalum phosphate; a new compound: ORNL-841; cf. *Nuclear Sci. Abs.*, v. 5, p. 155 (no. 963), 1951.

Procedure for preparation, composition data, and X-ray data on TaOPO_4 .

- 100 Hahn, R. B., and Burros, C. L., 1951, The determination of radio-niobium in the presence of soluble orthophosphates: ORNL-831, cf. *Nuclear Sci. Abs.*, v. 5, p. 537 (no. 3362), 1951.
- 101 Haissinsky, M., and Jeng-Tsong, Yang, 1950, On the stability of certain organic complexes containing elements of the fourth and fifth groups of the periodic chart; [Pt.] 2—Oxalates, citrates, and tartrates of Nb, Ta, and Pa: *Anal. Chim. Acta*, v. 4, p. 328-331 [French]; cf. *Nuclear Sci. Abs.* v. 4, p. 898 (no. 6089).

The preparation of hydroxides and complexes of Nb, Ta, and Pa, their decomposition by alkalis, and the pH measurements were made according to methods previously used for Ti, Zr, and Th compounds (*Anal. Chim. Acta*, v. 3, p. 422, 1949). The resistance to hydrolysis of the complexes of Nb and Ta decreases in the order oxalate > citrate > tartrate. In the case of oxalates, it is Nb > Ta; in that of the citrates it is Nb < Ta.

- 102 Harpham, E. W., 1946, Chemical determination of tantalum and columbium in tungsten-bearing heat-resisting steels: *Metallurgia*, v. 33, p. 245-247; cf. *Chem. Abs.*, v. 31, p. 7364⁴; v. 40, p. 3363¹.

First determine Nb and Ta as Nb_2O_5 + Ta_2O_5 . The Ta and Nb can be separated by the fractional tannin procedure as described by Schoeller (*Chem. Abs.*, v. 31, p. 7364).

- 103 Hartmann, H., and Mässing, W., 1951, Electrolysis in phosphate melts; [Pt.] 4—Electrolysis of oxides of vanadium, niobium, and tantalum in phosphate melts: *Zeitschr. anorg. allg. Chemie*, Band 266, p. 98-104; cf. *Chem. Abs.*, v. 31, 8384⁸, v. 46, p. 3426^e.

- 104 Harvey, C. E., 1946, A method of semiquantitative spectrographic analysis: *Optical Soc. America Jour.*, v. 36, p. 719.

It is based on a direct-current arc method, whereby a weighed sample is completely arced, and selected line-to-background ratios are

evaluated densitometrically and the ratios multiplied by sensitivity factors for particular spectrum lines in a particular matrix.

- 105 Hayashi, Shigehiko, 1949, Analysis of niobium and tantalum, [Pt.] 1—Separation of tantalum from niobium with tannin and cinchonine: Chem. Soc. Japan Jour., Pure Chemistry Sec., v. 70, p. 376–379; cf. Chem. Abs., v. 45, p. 2818a, 1951.

At pH 1.6 all Ta is precipitated with tannin and cinchonine.

- 106 ——— 1950, Analysis of niobium and tantalum; [Pt.] 4—Ignition temperatures of the tannin complexes of niobium and tantalum; Chem. Soc. Japan Jour., Pure Chemistry Sec., v. 71, p. 17–18; cf. Chem. Abs. v. 45, p. 2818b, 5063c, 1951.

Optimum temperature for Nb over 532° C and for Ta over 424° C.

- 107 Hayashi, Shigehiko, and Katsura, Tetsuo, 1949, Analysis of niobium and tantalum; [Pt.] 2—Separation with tannin and strychnine: Chem. Soc. Japan Jour., Pure Chemistry Section, v. 70, p. 435–437; cf. Chem. Abs., v. 45, p. 2818a, 1951.

Tantalum is precipitated in presence of niobium.

- 108 ——— 1949, Analysis of niobium and tantalum; [Pt.] 3—Influence of brucine, antipyrine, aminopyrine, and urotropine on the pH of formation of tannin complex; separation of tantalum from niobium with tannin and brucine: Chem. Soc. Japan Jour., Pure Chemistry Sec., v. 70, p. 437–439.

Mixture of brucine and tannin is the best precipitating agent for the separation of Ta and Nb; Ta precipitates.

- 109 Haywood, F. W., 1942, Some modern methods of metallurgical analysis: Metallurgia, v. 26, p. 117–120, cf. Chem. Abs., v. 36, p. 6432⁹.

Spectroscopic determination methods are described for Mn, Cr, Ni, V, Cu, Ti, Mo, P, Mg, W, Sb, Si, Nb, Ta, and Fe.

- 110 Hecht, H., Jander, G., and Schlapmann, H., 1947, The action of thionyl chloride on oxides: Zeitschr. anorg. Chemie, Band 254, p. 255–264; cf. Chem. Abs., v. 43, p. 2886a.

Metal oxide + $\text{SOCl}_2 \rightarrow$ metal chloride + SO_2 . The following oxides can be treated: Ca, Ga, In, Ta, Tl, B, Al, Y, La, Nd, Pr, Ti, Zr, Th, Si, Ge, Zn, V, Nb, Ta, P, Mo, W, and U.

- 111 Herman, P., 1948, Spectrographic determination of columbium and tantalum: Spectrochim. Acta, v. 3, p. 389–396 [French]; cf. Nuclear Sci. Abs., v. 2, p. 272 (no. 1222); Chem. Abs., v. 43, p. 2543g.

Determination is of 1 to 99 percent Nb or Ta in presence of Ti and W.

- 112 Herman, P., and Gastellier, S., 1946, Rapid determination of the tantalum and columbium in columbite: Service géol. Congo Belge et Ruanda-Urundi Bull., v. 2, p. 73–84; cf. Chem. Abs., v. 33, p. 9186⁸; v. 42, p. 2543b.

Analyses were made by a combination of the solution and separation methods of Schoeller and Powell, and the colorimetric determination of Nb and H_2O_2 .

- 113 Hillebrand, W. F., and Lundell, G. E. F., 1929, Applied inorganic analysis: New York, John Wiley and Sons, chap. 38, p. 463.

Collected information about Nb and Ta—general considerations, attack of earth-acid minerals, methods of separation, and methods of determination.

- 114 Hilliard, J. W. (Assigned to the American Steel and Wire Co. of N. J.), 1949, Columbium in iron and steel: U. S. patent 2,482,850, September 27, 1949; cf. Chem. Abs., v. 44, p. 490d.

Nb is determined qualitatively and quantitatively in Fe and its alloys. Nb weighed as Nb_2O_5 .

- 115 Hiskey, C. F., Newman, L., and Atkinson, R. H., 1952, Analytical chemistry of niobium and tantalum—Atmospheric chlorination of hydrolyzed oxide precipitates: Anal. Chemistry, v. 24, p. 1988-1991.

The atmosphere chlorination of earth oxide mixtures by octachloropropane was investigated with the object of incorporating the reaction into an analytical scheme. Method adequate.

- 116 Hiskey, C. F., and Batik, A. L., 1953, Analytical chemistry of niobium and tantalum—Separation of iron and manganese from the earth acids: Anal. Chemistry, v. 25, p. 823-825.

Method involves a precipitation of the earth oxides while retaining the Fe and Mn in solution with ethylenediaminetetraacetic acid.

- 117 Holt, M. L., 1935, The electrodeposition of tantalum: Zeitschr. Elektrochemie, v. 41, p. 303-304; cf. Chem. Abs., v. 29, 5357^b.

An attempt to repeat the experiment (Chem. Abs., v. 28, p. 4985^a) on the deposition of Ta from aqueous solution was unsuccessful. Cathodic deposits of Pt from the Pt anode dissolved during electrolysis were obtained, but no Ta.

- 118 Hopkins, B. S., 1940, Columbium and tantalum; Chap. 15 in Chapters in the chemistry of the less familiar elements, v. 2: Champaign Ill., Stipes Publishing Co.

History, occurrence, extraction, separation, metallurgy, compounds, determination.

- 119 Huffman, E. H., and Iddings, G. M., 1952, Anion exchange of niobium in 7.0-molar hydrochloric acid: UCRL-1777, cf. Nuclear Sci. Abs., v. 6, p. 496 (no. 4014); Am. Chem. Soc. Jour., v. 74, p. 4714.

In a recent report (Brown and Rieman, 1952, Am. Chem. Soc. Jour., v. 74, p. 1278) that elution of Ti from a cation-exchange resin with citrate solution resulted in broad elution bands with several peaks, this behavior was attributed to partial separation of Ti isotopes. The present single-nuclide study on the anion exchange of carrier-free Nb^{95} resulted in elution curves of similar behavior and also not exactly

reproducible. This departure from the expected type of elution band can probably be attributed to the slow establishment of equilibrium among various ionic species that are present.

- 120 Huffman, E. H., Iddings, G. M., and Libby, R. C., 1951, Anion exchange of zirconium, hafnium, niobium, and tantalum in hydrochloric acid solutions: *Am. Chem. Soc. Jour.*, v. 73, 4474-4475; cf. *Chem. Abs.*, v. 45, p. 7908*h*; v. 46, p. 1330*c*.

Zr⁹⁵ and Nb⁹⁵ in 1 cc 6 to 7 *M* hydrochloric acid were separated by passing through a Dowex-2 column, 8 cm in length and 3 mm in inside diameter, and washing with 3 cc acid. The elutant and wash contain the Zr. The Nb is then washed from the column with about 4 cc of 1.5 to 4.0 *M* hydrochloric acid.

- 121 Hume, D. N., 1951, Removal of zirconium and niobium from Amberlite resin adsorbent, in Coryell, C. D., and Sugarman, Nathan, eds., *Radio-chemical studies—The fission products*, Book 3 (*Natl. Nuclear Energy Ser., Div. 4*, v. 9): New York, Atomic Energy Comm.—McGraw-Hill Book Co., p. 1521-1522; cf. *Chem Abs.*, v. 46, p. 6546*c*.

Hydrogen fluoride removes Zr and Nb activities adsorbed on Amberlite resin.

- 122 Indichenko, L. N., 1948, Spectral analysis of iron and manganese ores: *Akad. nauk SSSR Doklady*, tom 59, p. 1157-1159; cf. *Chem. Abs.* v. 42, p. 7112*e*.

Description of techniques employed in the spectrographic analysis of the ores, stressing Nb, Zr, and Ni.

- 123 Jaboulay, B. E., 1948, Determination of tantalum and columbium in ferrotantalum, ferrocolumbium, and steel: *Rev. métallurgie*, v. 45, p. 434-447; cf. *Chem. Abs.*, v. 43 p. 2119*b*.

Method described is to precipitate and weigh combined Ta₂O₅ and Cb₂O₅; precipitate Ta₂O₅ in H₂O₂·H₂SO₄ solution; reprecipitate Cb₂O₅ from solution by boiling filtrate with H₂SO₄ and Na₂SO₃; wash precipitate with water, calcine, and weigh.

- 124 Jean, M., 1950, Bibliographical summary of chemical analyses of metallurgical products: *Chimie anal.*, v. 32, p. 133-138, 162-167, 179-184, 213-217.

- 125 Jeffries, Z., 1941, Rare metals and minerals: *Mining and Metallurgy*, v. 22, p. 82-85; cf. *Chem. Abs.*, v. 34, p. 2749*g*; v. 35, p. 3574*f*.

A review of developments in 1940 covering Ba, Be, Ca, Co, Li, Pt group, Po, Ra, Se, Ta, W, U, V, and Zr.

- 126 Jeng-Tsong, Yang, 1950, The separation of protactinium from Ta by ion exchange: *Acad. sci. [Paris] Comptes rendus*, tome 231, p. 1059-1601 [French]; cf. *Nuclear Sci. Abs.*, v. 5, p. 292 (no. 1826), 1951.

In a plastic column filled with the anion-exchange resin Amberlite IR-4B and an eluant of 6.5 *N* HF, which are brought to a pH of

approximately 3 with concentrated NH_4OH , more than 90 percent of the Pa separates from Ta in a single operation. The Pa is eluted after the Ta.

- 127 Johnson, C. M., 1946, Determination of tungsten and columbium in stainless steel: *Iron Age*, v. 157, no. 15, p. 66-68, cf. *Chem. Abs.*, v. 40, p. 3363⁶.

A detailed procedure using known methods or modifications is given. The Ti is determined colorimetrically with hydroquinone. Color due to W and Nb fades.

- 128 Johnson, Charles M., 1946, Colorimetric determination of tungsten, titanium, and columbium: *Iron Age*, v. 157, no. 14, p. 66-69, cf. *Chem. Abs.*, v. 40, p. 3364¹.

Determination is made by the hydroquinone photometric method, which gives no color for Ta and V.

- 129 Karyakin, Yu. V., and Telezhnikova, P. M., 1946, Test for columbium and tantalum with polyphenols: *Zhur. priklad. khimii*, tom 19, p. 435-439; cf. *Chem. Abs.*, v. 41, p. 1170ⁱ.

Studies with 22 polyphenols and ions of Nb, Ta, and Ti showed that only pyrogallol, pyrocatechol, and gallic acid produce yellow colors; no other reagent was effective, in contradiction to numerous references cited. The colors were produced as follows: Nb gave the color only in alkaline solution, and Ta gave color only in acid solution, whereas Ti gave the color in either acid or alkaline solutions.

- 130 Kiba, Toshiyaso, and Ikeda, Tadasi, 1939, Studies of gravimetric analysis by means of the thermobalance; [Pt.] 21—Determination of calcium, lead, and thorium as picrolonate and of phosphorus, palladium, columbium, zirconium, gold, beryllium, tantalum, and silicon by the oxine method: *Chem. Soc. Japan Jour.*, v. 60, p. 911-914; cf. *Chem. Abs.*, v. 28, 4331⁹; v. 34, p. 1585⁵.

The proper temperatures for drying as determined by the thermobalance are: Th picrolonate, less than 35° C; anhydride, 160°-180° C; Nb_2O_5 oxine, 100°-130° C; Zr oxinate, 115°-150° C; and Ta_2O_5 , more than 450° C.

- 131 Klimenko, N. G., and Syrokonski, V. S., 1947, Determination of the pH of the beginning of precipitation of niobium and tantalum: *Zavodskaya lab.*, tom 13, p. 1029-1034; cf. *Chem. Abs.*, v. 43, p. 4083^d.

- 132 Klinger, P., 1939, Columbium and tantalum; [Pt.] 1—Introduction: *Tech. Mitt. Krupp, Forschungsber.*, Band 2, p. 171-173; cf. *Chem. Abs.*, v. 34, p. 40³.

Methods of separation of Nb and Ta are critically discussed.

- 133 Klinger, P., and Koch, W., 1939, Photometric determination and separation of columbium, tantalum, and titanium in steel and ferroalloys: *Archiv Eisenhüttenw.*, Band 13, p. 127-134; cf. *Chem. Abs.*, v. 33, p. 9186⁸.

The known methods for separating and determining Nb, Ta, and Ti are reviewed. Procedure recommended based on the coloration of Ti and Nb with H_2O_2 ; the Ti coloration is strongest in dilute H_2SO_4 , that of Nb in concentrated H_2SO_4 . By using differences in the intensity of coloration in different acid concentrations a method was developed for determining both Nb and Ti in the presence of HTaO_3 . For the determination of Ti the color reaction with chromotropic acid is recommended. Procedures are described in detail.

- 134 ——— 1939, Photometric determination and separation of columbium, tantalum, and titanium in steel and iron alloys: *Tech. Mitt. Krupp, Forschungsber.*, Band 2, p. 179–185, cf. *Chem. Abs.*, v. 33, p. 9186⁸; v. 34, p. 40⁶.

- 135 Klinger, P., Stengel, E., and Wirtz, H., 1941, Analysis of tantalum metal: *Metall u. Erz*, v. 38, p. 124–127; cf. *Chem. Abs.*, v. 35, p. 5409⁸.

The Ta is precipitated with hydrogen peroxide (after solution in HNO_3 , HF, and $\text{K}_2\text{S}_2\text{O}_7$), ignited, and weighed as Ta_2O_5 ; Nb is determined colorimetrically with hydrogen peroxide in 100 percent H_2SO_4 ; and Ti is determined by means of color reaction of Ti^{4+} with chromotropic acid.

- 136 Klinkenberg, P. F. A., and Van Den Berg, G. J., 1950, Structure and Zeeman effect in the spectrum of the tantalum atom Ta I: *Physica*; v. 16, p. 861–889;; cf. *Nuclear Sci. Abs.*, v. 5, p. 441 (no. 2747); *Chem. Abs.*, v. 45, p. 7868ⁱ, 1951.

- 137 Knowles, H. B., and Lundell, G. E. F., 1949, Volumetric determination of columbium: *Natl. Bur. Standards Jour. Research*, v. 42, p. 405–408; cf. *Chem. Abs.*, v. 44, p. 7710^d.

The determination of Nb is based on reduction to Nb^{3+} with amalgamated Zn and reoxidation to Nb^{5+} with KMnO_4 , using ferroin as the indicator.

- 138 Kohn, André, 1953, A radioactive technique for determination of tantalum in the ferroniobiums and niobium minerals: *Acad. sci. [Paris] Comptes rendus*, tome 236, p. 1419–1421 [French]; cf. *Nuclear Sci. Abs.*, v. 7, p. 411 (no. 3368).

The method permits the determination of Ta without any chemical separation of Ta and Nb. It consists in measuring the activity of samples and standards irradiated together in a slow neutron flux. The tests undertaken have permitted the exact determination of the corresponding importance of the diverse causes of error. The content of Ta can be known to less than 5 percent.

- 139 Komarovskii, A. S., and Shapiro, M. I., 1938, Sensitive, catalytic test for columbium and tantalum: *Mikrochimica Acta*, Band 3, p. 144–146, cf. *Chem. Abs.*, v. 32, p. 5329⁶.

The test depends, as Feigl has shown, upon the fact that substances capable of forming per-acids cause $\text{Na}_2\text{S}_2\text{O}_3$ to be oxidized to SO_4^{2-} instead of S, which usually forms. 0.2 γ Nb or 0.4 γ Ta suffices to give the test.

- 140 Komovskii, G. F., and Golovchiner, Ya., 1942, An application of ionic Roentgen tubes for quantitative spectrographic analysis: *Zhur. tekhn. fiziki*, tom 12, p. 579-586; cf. *Chem. Abs.*, v. 37, p. 2264³, 4301⁹.

Quantitative spectrographic analyses were made of artificial mixtures and minerals for Ta, W, Nb, Mo, Zr, Hf, Th, U, Pb, Co, and Ni by using a somewhat modified Roentgen tube.

- 141 Krasilshchikov, B. S., and Popova, N. M., 1945, Determination of niobium in steel: *Zavodskaya lab.*, tom 11, p. 512; cf. *Analyst*, v. 73, p. 176, 1948.

A determination of Nb and Ta as combined oxides is followed by a colorimetric determination of Ta with pyrogallol.

- 142 Kraus, K. A., and Moore, G. E., 1949, Separation of columbium and tantalum with anion-exchange resins: *Am. Chem. Soc. Jour.*, v. 71, p. 3855.

- 143 ——— 1950, Anion exchange studies; [Pt.] 1—Separation of zirconium and niobium in $\text{HCl} \cdot \text{HF}$ mixtures: ORNL-661; also *Am. Chem. Soc. Jour.*, v. 73, p. 9-13; 1951; cf. *Nuclear Sci. Abs.*, v. 4, p. 648 (no. 4241), 1950.

Study of the behavior of Zr^{4+} and Nb^{5+} on the anion-exchange resin Dowex-1 in a number of $\text{HF} \cdot \text{HCl}$ mixtures. Separation of the two elements is excellent under a number of conditions.

- 144 ——— 1950, Anion exchange studies; [Pt.] 2—Tantalum in some $\text{HF} \cdot \text{HCl}$ mixtures: ORNL-661; also *Am. Chem. Soc. Jour.*, v. 73, p. 13-17, 1951; cf. *Nuclear Sci. Abs.*, v. 4, p. 571 (no. 3713), 1950.

The ion-exchange behavior of Ta was studied for a number $\text{HF} \cdot \text{HCl}$ mixtures. It was found to adsorb very strongly under most conditions, even in 9 *M* hydrochloric acid.

- 145 ——— 1950, Anion-exchange studies; [Pt.] 3—Protactinium in some $\text{HCl} \cdot \text{HF}$ mixtures; separation of niobium, tantalum, and protactinium: ORNL-673; also *Am. Chem. Soc. Jour.*, v. 73, p. 2900-2905; cf. *Nuclear Sci. Abs.*, v. 5, p. 159 (no. 990).

- 146 Krasilshchikov, B. S., and Popova, N. M., 1945, Determination of columbium in steel: *Zavodskaya lab.*, tom 11, p. 512-515; cf. *Chem. Abs.*, v. 40, p. 2409⁵.

Determine $\text{Nb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$; fuse precipitate with KHSO_4 ; dissolve the melt in $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution; and determine the Ta colorimetrically with pyrogallol and the Nb by difference.

- 147 Krishnaswami, K. R., and Murthi, D. Suryanarayana, 1935, Studies in tantalum and columbium: *Indian Inst. Sci. Jour.*, v. 18A, p. 69-73; cf. *Chem. Abs.*, v. 21, p. 2624; v. 29, p. 7860⁴.

The optimum conditions are given for a proposed method. Ta and Nb are determined by heating the mixed pentoxides in a stream of hydrogen. The Nb_2O_5 is reduced to NbO_2 ; the Ta_2O_5 is unaffected.

Authors advise heating for 1 hour at 800° C and cooling in the H₂ stream.

- 148 Krivoslykov, N. F., 1939, Colorimetric determination of columbium and tantalum: Leningrad. krasnoznamenn. khim.-tekhnol. inst. Trudy, no. 7, p. 103-122; cf. Khim. referat. zhur., v. 2, no. 5, p. 59-60, 1939; Chem. Abs., v. 34, p. 2729³.

The method is based on the color produced by Nb and Ta compounds with pyrogallol in acid and alkaline solutions; 0.05 mg of Nb₂O₅ and 0.05-0.07 mg of Ta₂O₅ in 1 cc of solution can be detected. Titanium is determined with hydrogen peroxide.

- 149 Krivoslykov, N. F., and Platonov, M. S., 1937, Colorimetric determination of columbium and tantalum: Zhur. priklad. khimii, tom 1, p. 184-191 [German p. 191]; cf. Chem. Abs., v. 31, p. 4921³.

Niobium gives a stable yellow color with pyrogallol in the presence of Na₂SO₃ in alkaline solution and no color in an acid solution, whereas Ta gives the same color only in acid solution. Titanium gives the same color with pyrogallol as Nb. (NH₄)₂CrO₄ solution (5 g per 250 ml of water) can be used as a standard instead of a standard solution of Nb and Ta.

- 150 Kroll, W. J., and Bacon, F. E. (to Electro Metallurgical Co.), 1948, Separation of columbium and tantalum oxides: U. S. patent 2,443,254, June 15, 1948; cf. Chem. Abs., v. 42, p. 6500e.

The NbCl₅ is separated from the residue by distillation and the cycle repeated. NbCl₅ and Nb₂O₅ are produced according to the reaction, $5\text{Nb}_2\text{O}_4 + 5\text{Cl}_2 \rightarrow 2\text{NbCl}_5 + 4\text{Nb}_2\text{O}_5$ after the Nb₂O₅ is selectively reduced to Nb₂O₄ by dry N₂-free H₂ at 900° C. The H₂ is then replaced by Cl₂ and the temperature reduced to 400° - 600° C.

- 151 Kuznetsov, V. I., 1941, Color reactions for thorium, uranium, and other elements: Acad. sci. U. R. S. S. Comptes rendus, v. 31, p. 898-900 [English]; cf. Chem. Abs., v. 37, p. 845².

A very good study of the reaction of the following reagents with the rare earths Th, U, Ti, Zr, Sn, Nb, and Ta, in neutral or slightly acid solution: 2-(*o*-arsonophenylazo)-*p*-cresol; 1-(*o*-arsonophenylazo)-2-naphthol-3,6-disulfonic acid; 1-(*o*-arsonophenylazo)-2-naphthol-6,8-disulfonic acid; 3-(*o*-arsonophenylazo)-4,5-dihydroxy-2,7-naphthalenedisulfonic acid.

- 152 ————1945, Increase of sharpness of color tests with organic reagents: Akad. nauk SSSR Doklady, tom 50, p. 233-239; cf. Chem. Abs., v. 43, p. 4174g.

The color formation in organic reagents is reviewed on the basis of the internal ionization in molecules containing distinct dipole character; the metals Ti, U, Zr, Mo, Nb, W, Bi, Th, Sb, Th, Ag, Pb, and Cu are considered.

- 153 ———— and Vasyunina, N. A., 1940, The reactions of aminobenzenearsonic acids with 2-furaldehyde: Zhur. obshchey khimii; tom 10, p. 1203-1209; cf. Chem. Abs., v. 35, p. 2869².

The reaction can be used as a color test for Sn^{4+} , Ti, Zr, Hf, Nb, Ta, Th, Sb, Bi, Cr^{3+} , Mo, U, and W ions, in solutions stronger than 0.1 mg metal per ml.

- 154 Kvalheim, A., Rutlin, S., and Aarnseth, K. A., 1952, "Schulze-method" for the chemical determination of niobium in soevite rock: Tidsskr. Kjemi, Bergvesen o. Metallurgi, v. 12, p. 93-96, cf. British Abs., v. 1C, no. 24, 1953; Chem. Abs., v. 47, p. 2633h.

This chemical analysis is based on precipitation of Ta and Nb as tannate, weighing as oxides after ashing, and deduction of Ta, which is determined colorimetrically. When carried out with care, it yields fairly good results. The Nb figures are usually approximately 7 percent (relatively) low. This is presumably due to a slight solubility of Nb mineral in HNO_3 (in the pretreatment process), and to the incomplete decomposition of Nb minerals by H_2SO_4 (to dissolve the Nb), because of unexposed particles. The average content of the soevite rock is approximately 0.2 percent of Nb_2O_5 . The residues, precipitate, filtrates, etc., from the chemical analysis were controlled by spectrographic analysis.

- 155 Lauw-Zecha, A. B. H., Lord, S. S., and Hume, D. N., 1952, Colorimetric determination of niobium using thiocyanate: Anal. Chemistry, v. 24, p. 1169-1173.

Reaction between CNS^{1-} and Nb is basis for rapid spectrophotometric determination of microgram amounts of Nb. The sample in hydrochloric acid is treated with SnCl_2 and KCNS; the resulting yellow complex is ether-extracted and the absorbancy measured at 385 m μ . Beer's Law holds for 1 to 65 micrograms per 25 ml of ether extract.

- 156 Leddicotte, G. W., and Moore, F. L., 1952, A new solvent extraction method for the separation of niobium and tantalum: Am. Chem. Soc. Jour., v. 74, p. 1618; cf. Chem. Abs., v. 47, p. 5300f.

Niobium can be extracted quantitatively from strong hydrochloric acid with a solution of methyldioctylamine in xylene. Under these conditions extraction of Ta appears to be negligible. The Nb may then be "stripped" from the organic phase with HNO_3 , H_2SO_4 , or dilute hydrochloric acid. Authors suggest approximately 8 M hydrochloric acid concentration for the separation, using equal volume of a 5 percent solution of methyldioctylamine in xylene.

- 157 Lokka, Lauri, 1950, Photometric determination of Nb_2O_5 in mixed oxides $\text{Nb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$: Comm. géol. Finlande Bull., no. 149, p. 39.

On the chemistry of the radioactive minerals of Finland.

- 158 Lomonosova, L. S., 1950, Spectrographic determination of titanium and niobium in powdered tantalum: Izv. Akad. nauk SSSR, Ser. fiz., tom 14, p. 693-695.
- 159 Long, J. V. P., 1950, The analysis of mixtures containing Ta by measurement of radioactivity produced by neutron irradiation: CRL-AE-60; Analyst, v. 76, p. 644-646, 1951; cf. Nuclear Sci. Abs., v. 5, p. 288 (no. 1801), 1951.

A neutron-activation method is described for the determination of Ta in mixtures also containing Nb, Zr, Fe, Sn, Ti, and Si. The method depends on the pile irradiation of the sample, followed after 4 days by measurement of the filtered gamma-ray activity. An accuracy of about ± 2 percent can be achieved.

- 160 Mackay, R. A., 1951, The detection of columbite by ultraviolet light: *Inst. Mining and Metallurgy Bull.*, v. 530, p. 129-131, cf. *Chem. Abs.*, v. 45, p. 3296*h*, 1951.

Flux contained 2 percent of Na_2SO_4 , 5 percent of NaF, 46.5 percent of Na_2CO_3 , and 46.5 percent of K_2CO_3 by weight. A nickel dish was used.

- 161 Martell, A. E., and Calvin, M., 1952, *Chemistry of the metal chelate compounds*: New York, N. Y., Prentice-Hall.

A useful collection and formulation of the data existing at present about chelate compounds.

- 162 Martini, Ardoino, 1941, Course in qualitative microanalysis: *Univ. Nac. Litoral Rosario, Inst. Inv. Microquím. Pub.*, v. 5, no. 5, p. 121-144, cf. *Chem. Abs.*, v. 36, p. 6108*g*; v. 40, p. 5351*2*.

Standard tests for C, Ti, Zr, Th, Bi, Sb, As, Ta, Nb, V, and U are described and discussed.

- 163 Maurer, W. C., (to Carnegie-Illinois Steel Co.) 1948, Determination of columbium carbide in stainless steel: U. S. patent 2,447,763, August 24, 1948; cf. *Chem. Abs.*, v. 43, p. 522*h*.

Insoluble Cb carbide is separated from soluble Cb by digestion with dilute H_2SO_4 and determined gravimetrically as Nb_2O_5 .

- 164 Meimberg, E., 1913, Bestimmung der Tantalssäure und Niobsäure in Tantaliten, Columbiten, Yttrotantaliten, Fergusoniten, und colorimetrische Niobbestimmung: *Zeitschr. angew. Chemie*, v. 26, p. 83.

Niobium, unlike Ta, is reduced to Nb^{3+} by various metals such as Zn and Sn in acid solution. Brown color obtained serves for a photometric determination. Method applied in determination of Nb in tantalite. Tantalite was dissolved in $\text{HF} \cdot \text{HCl}$ and the bulk of Ta separated as K fluotantalate.

- 165 Mercer, R. A., and Williams, A. F., 1952, Inorganic chromatography on cellulose; [Pt.] 13—Determination of tantalum and niobium in low-grade phosphatic and siliceous ores; *Chem. Soc. London, Jour.*, p. 3399-3403; cf. *Chem. Abs.*, v. 46, p. 7470*i*; v. 47, p. 1537*d*.

The chromatographic method is extended to ores containing less than 0.1 percent $\text{Ta}_2\text{O}_5 + \text{Nb}_2\text{O}_5$. In the chromatographic procedures previously described, the sample is digested with $\text{HCl} + \text{NH}_4\text{F}$ and placed on a column of cellulose. Ta was extracted with methyl ethyl ketone which was saturated with water and the column was then equilibrated with methyl ethyl ketone containing 12.5 percent of 40-percent HF by volume. The procedure has given good results in determining 0.2 to 0.9 percent Ta_2O_5 and 0.28 to 1.03 percent Nb_2O_5 .

- 166 Meerson, G. A., Kats, G. A., and Khokhlova, A. V., 1940, Reduction of the oxides of metals of high melting points with calcium hydride: Zhur priklad. khimii, tom 13, p. 1770-1776 [French, p. 1776]; cf. Chem. Abs., v. 35, p. p. 4712¹.

TiO₂, Nb₂O₅, V₂O₅, and Ta₂O₅ were reduced with CaH₂ in H₂ at atmospheric pressure in electric furnaces.

- 167 Melentyev, B. N., and Terekhovko, A. S., 1940, The analysis of perovskite: Akad. nauk SSSR Trudy Kol'skaya baza, no. 5, p. 71-76; cf. Khim. referat. zhur., tom 4, no. 2, p. 60, 1941; Chem. Abs., v. 37, p. 3019³.

Decomposition with H₂SO₄; citric acid used to prevent hydrolysis; Ti, Ta, and Nb precipitated with cupferron.

- 168 Miller, C. Franklin, 1937, Tannic acid in analysis: Chemist Analyst, v. 26, p. 38-39, cf. Chem. Abs., v. 31, p. 5709⁹.

The tests for Ti, Fe, Nb, Ta, Ag, Au, U, Hg, HVO₃, H₂MoO₄, as recommended by Schoeller and his coworkers, are reviewed.

- 169 Miller, C. C., and Lowe, A. J., 1940, Qualitative semimicro analysis with reference to Noyes' and Bray's system—The tantalum and tungsten group: Chem. Soc. London Jour., p. 1258-1263; cf. Chem. Abs., v. 21, p. 3583; v. 31, p. 7364⁴; v. 35, p. 707⁸.

Application of methods proposed by Noyes and Bray followed the methods proposed by Schoeller. Very satisfactory results were reported.

- 170 Milner, G. W. C., and Wood, A. J., 1952, The analysis of uranium-tantalum and uranium-niobium alloys: AERE C/R-895; 13 p.; cf. Nuclear Sci. Abs., v. 6, p. 617 (no. 5050).

Satisfactory procedures are described for the analysis of uranium-tantalum and uranium-niobium alloys that contain up to 25 percent Ta and Nb, respectively. They are based on the separation of the Ta and Nb from U by extracting the fluorides of the former elements with methyl ethyl ketone. After the evaporation of the solvent from the combined organic extracts, the residue is ignited (800° C.) to yield the pentoxides. Absorptiometric procedures are included for the determination of trace amounts of unextracted Ta and Nb.

- 171 Moir, J., 1916, Analysis of niobium-tantalum minerals, with some new tests for niobium, tantalum, and titanium: Chem. News, v. 113, p. 256.

Thiocyanate reacts with Nb³⁺ in acid solution. The resulting solution has a yellow-orange color, the intensity of which is critically dependent on acid concentration (qualitative). Thymol also gives a color reaction (qualitative).

- 172 More, Ch., 1943, Niobium or columbium: Metallurgia, v. 75, no. 12, p. 1-2; cf. Chem. Abs., v. 40, p. 5679⁵.

A review covering natural occurrence of niobium, its production, properties, and uses.

- 173 Myers, R. H., 1943, A review of the literature on the chemistry and metallurgy of tantalum and columbium: Australasian Inst. Mining and Metallurgy Proc., no. 129, p. 55-79; cf. Chem. Abs., v. 37, p. 6611⁶.
- 174 Nachtrieb, N. H., and Tobin, William J., 1951, Spectrochemical analysis of columbium and zirconium: AECD-3103; ANL-HDY-579. cf. Nuclear Sci. Abs., v. 5, p. 490 (no. 3056).
- 175 Namoradze, Z. G., and Zvyagintsev, O. Ye, 1939, Solubility of tantalum chloride and bromide in organic solvents: Zhur. priklad. khimii, tom 12, p. 603-608 [French p. 608]; cf. Chem. Abs., v. 33, p. 8475⁵.
Data are tabulated and plotted for solubility in CCl_4 , CS_2 , CHCl_3 , EtBr , PhNO_2 , and $\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$.
- 176 Novikov, V. A., 1939, Application of film reactions and of etching to the mineralogical analysis of deposits: Sovetskaya geologiya, v. 9, no. 10-11, p. 116-120; cf. Chem. Abs., v. 35, p. 406⁹.
Author finds that Mo and Ta-columbite ores can be distinguished from one another and from other ores by the characteristic color films appearing when they are treated with concentrated H_2SO_4 at about 300° C.
- 177 Oka, Yoshinaga, and Miyamoto, Masatoshi, 1948, Volumetric determination of columbium: Chem. Soc. Japan Jour., Pure Chemistry Sec., v. 69, p. 133-137, cf. Chem. Abs., v. 44, p. 9299^h.
A method of determining Nb without use of a correction factor was developed. When Ti is present, less than 1 mg Nb_2O_5 could not be determined. In a mixture of the oxides Nb_2O_5 and Ta_2O_5 , 2 mg Nb_2O_5 can be determined in the presence of 50 mg Ta_2O_5 .
- 178 ——— 1949, Volumetric determination of Cb: Tohoku Univ., Research Inst. Sci. Repts., ser. A, v. 1, p. 115-119; cf. Chem. Abs., v. 44, p. 9299^h, 10578^e, 1950.
Reduction with Zn amalgam is followed with a KHNO_4 titration.
- 179 Osborn, G. H., 1953, Bibliography of the analytical applications of ion-exchange resins: Analyst, v. 78, p. 221-252.
- 180 Oshman, V. A., 1946, The separation of columbium and tantalum from titanium: Zavodskaya lab., tom 12, p. 154-157; cf. Chem. Abs., v. 40, p. 5660.⁸.
Separate Nb and Ta from Ti by means of reduction with amalgam; determine TiO_2 and ZrO_2 in aqueous phase (Ti by H_2O_2 and Zr by phosphate); subtract the sum of the $\text{TiO}_2 + \text{ZrO}_2$ from the original weight of the precipitate of the impure earth oxides; and determine the percent content of the sum of the earth oxides by the usual method.
- 181 Palilla, F. C., Adler, N., and Hiskey, C. F., 1953, Analytical chemistry of niobium and tantalum—Simultaneous spectrophotometric determination of tantalum and niobium: Anal. Chemistry, v. 25, p. 926-931.

The peroxy complexes of groups IV to VII of transition elements, formed in concentrated H_2SO_4 media, were investigated for application to the analysis of niobia and tantalum mixtures. One result of this study was the discovery of a peroxy-tantalate whose absorption spectrum had its peak at 285 m μ . Since the peroxy-niobate formed under identical conditions had its peak at 365 m μ , it was possible to develop a method for the simultaneous determination of Nb and Ta mixtures. This method has a precision of about 0.5 percent and is independent of Nb-Ta ratio.

- 182 Pearson, J. G., 1936, Microanalysis; [Pt.] 2—Tests for rare and common metals: Chem. Eng. Min. Rev., v. 28, p. 151-154; cf. Chem. Abs., v. 30, p. 2517⁴, 3354⁵.

This instalment includes microtests for Mn, Li, Th, Ta, Ni, Ti, W, Au, Ce, Co, As, Cu^{2+} , Cu^+ , CH_2O , water, nitrates, and F.

- 183 Pennington, M. E., 1896, KCNS as reagent in colorimetric determination of niobium: Am. Chem. Soc. Jour., v. 18, p. 51.

Thiocyanate reacts with Nb^{3+} in acid solution. The resulting solution has a yellow-orange color. A study of the analytical application of the method is presented.

- 184 Pfeiffer, P., Böttcher, H., Prätorius, W., and Kwan, Li Mou, 1936, Betainelike complex salts: Zeitschr. anorg. allg. Chemie, Band 230, p. 97-111; cf. Chem. Abs., v. 31, p. 3808⁹.

- 185 Pigott, E. C., 1942, The chemical and physico-chemical analysis of iron and steel: Metallurgia, v. 26, p. 73-74; 113-115, and 145-147, Chem. Abs., v. 36, p. 6118⁹.

In this and some subsequent articles the author gives an exhaustive and descriptive bibliography of contributions to ferrous analysis published during the period 1930-41. They reveal the developments in analytical methods which have become more direct and of wider application in meeting demands for speed combined with accuracy. Photometric, spectrographic, polarographic, and chemical determinations are given in this series of papers.

- 186 ——— 1942, Twelve years' progress; [Pt.] 2—The determination of single constituents: Metallurgia, v. 26, p. 31-33, cf. Chem. Abs., v. 36, p. 6118⁹; v. 37, p. 844⁵.

The determination of P, Se, Si, S, Ta, and Te as single constituents is considered.

- 187 ——— 1942, Qualitative chemical tests for iron, steel, and foundry materials: Iron and Steel [London], v. 15, p. 196-199, 202; cf. Chem. Abs., v. 38, p. 1702⁴, 1944.

Tests are given for Al, As, B, C, Ce, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Nb, P, Se, Si, S, Ta, Te, Sn, Ti, W, U, V, Zn, and Zr.

- 188 Platonov, M. S., and Krivoslykov, N. F., 1943, Detection and colorimetric determination of Nb and Ta: Vses. konf. anal. khimii Trudy, tom 2, p. 359-370; cf. Chem. Abs., v. 39, p. 3492⁴, 1945.

Sum of pentoxides is determined gravimetrically and one of the metals determined colorimetrically with pyrogallol. Color caused by Nb or Ti.

- 189 Platonov, M. S., Krisoshylkov, N. F., and Maraka'yev, A. A., 1936, New qualitative reactions of columbium and tantalum: *Zhur obshchey khimii*, tom 6, p. 1815-1817; cf. *Chem. Abs.*, v. 31, p. 4614⁹.

New methods for the determination of small amounts of Nb and Ta in the presence of each other and of Ti consist in fusing the sample with KHSO_4 at $450^\circ - 700^\circ \text{C}$ and leaching the melt with a concentrated solution of $(\text{NH}_4)_2\text{C}_2\text{O}_4$. The solution gives color reactions with resorcinol or pyrogallol in alkaline or acid solutions. These methods are very sensitive.

- 190 ——— 1938, New quantitative reactions of columbium and tantalum: *Zhur. obshchey khimii*, tom 8, p. 829; cf. *Chem. Abs.*, v. 33, p. 497³.

Reactions of Nb and Ta with organic reagents are given.

- 191 Poehlman, W. J., and Sarnowski, R. E., 1952, Spectrochemical determination of tantalum and niobium in 18 Cr-8 Ni stainless steel: *Optical Soc. America Jour.*, v. 42, p. 489-492; cf. *Chem. Abs.*, v. 46, p. 8570^b; *British Abs.* v. 12C, p. 3936, 1952.

A spectrographic method for determining Ta and Nb, covering the concentration ranges 0.01-0.40 percent and 0.30-1.25 percent, respectively, is described. Samples are excited by an alternating-current arc discharge from point to plane. A prism-type spectrograph is employed, and the resulting spectrograms are recorded on calibrated emulsions. With the Fe matrix as an internal standard, photographic photometer readings and intensity ratio comparisons are made for the analytical line pairs 3311.16 Ta against 3165.86 Fe, and 3299.61 Nb against 3165.01 Fe. Included in the discussion are referee analyses, comparisons, working curves, and standards. The analytical results of this method indicate adequate sensitivity, precision, accuracy, and range of analysis for this kind of material.

- 192 Pokrovskaya, T. L., 1945, Analysis of cassiterite: *Zavodskaya lab.*, tom 11, p. 363-364; cf. *Chem. Abs.*, v. 40, p. 3367⁴.

Complete details are given for determining Sn, Nb, Ta, Zr, Ti, Mg, Mn, Fe, W, and As in cassiterite by known methods.

- 193 Popova, N. M., and Platonova, A. F., 1950, Determination of niobium carbide in steels: *Zavodskaya lab.*, tom 16, p. 1182-1185; cf. *Chem. Abs.*, v. 47, p. 3181^a.

Dissolve the sample anodically by using an electrolyte containing $N \text{ HCl}$, $N \text{ KCl}$, and 1 percent H_2SO_4 for Cr-Ni austenite steels and an electrolyte containing $N \text{ KCl}$ and 0.5 percent citric acid for low-alloy steels. Wash off the carbide precipitate with water and let settle; remove residues of carbide particles from the sample with filter paper, place in filter, and decant through the filter the water from settled carbides (precipitate no. 1). Wash the carbide residue with water and then with EtOH , dry in a stream of H_2 for 1 hour at

100° C on a water bath, and for 1 hour at 200° C on an oil bath, cool, and purge for 2 hours with N_2 . To 40 to 60 mg of dry precipitate add 10 to 15 ml HF, boil for 30 minutes, dilute with cold water, and filter (precipitate no. 2). Evaporate the filtrate three times with 5-ml portions of concentrated H_2SO_4 , separate Nb by hydrolysis with 3-percent HCl, fuse the precipitate with $K_2S_2O_7$, decompose in 100 ml of 5-percent pyrogallol, add 10 ml of 25-percent NH_4OH to dissolve the precipitate, and filter. Wash to remove all coloration, acidify the filtrate with 40 ml of 6 N HCl, allow to stand for 1 to 2 hours at 70° to 80° C, filter, wash with warm water, calcine, and weigh as Nb_2O_5 . Calcine precipitates numbers 1 and 2, fuse with $K_2S_2O_7$, and determine Nb with pyrogallol as above.

- 194 Portnov, A. I., 1948, Application of aromatic arsenic compounds in chemical analysis: Zhur. obshchey khimii, tom 18, p. 594-600; cf. Chem. Abs., v. 43, p. 57c.

A study of insoluble metal salts of a number of aromatic arsonic acids. An English translation has been made by the U. S. Geological Survey and reported as TEIR-324.

- 195 Portnov, M. A., 1939, Automatic and electrometric methods of analysis: Gos. inst. priklad. khimii Sbornik statey 1919-39, p. 100-142; cf. Khim. referat. zhur., no. 3, p. 52-53, 1940; Chem. Abs., v. 36, p. 2222^g.

The paper describes methods of electrometric analysis and automatic production control. Niobium and Ta are determined polarographically.

- 196 Powell, A. R., and Schoeller, W. R., 1925, A new method for the separation of tantalum from niobium: Analyst, v. 50, p. 485.

Separation effected by the use of tannin.

- 196a ——— 1925, The detection and determination of tantalum in niobium compounds: Analyst, v. 50, p. 94.

- 196b ——— 1930, A new method for the separation of titanium from zirconium and hafnium: Analyst, v. 55, p. 605-612.

A tannic acid method is described.

- 197 ——— and Jahn, C., 1935, Investigations into the analytical chemistry of tantalum, columbium and their mineral associates; [Pt.] 29—The separation of W from Ti, Nb, Ta, and Zr: Analyst, v. 60, p. 506-514; cf. Chem. Abs., v. 18, p. 950; v. 29, p. 7219.

Zirconium can be separated from W by fusing with alkali carbonate and leaching the melt with water (Na_2WO_4 extracted). The only procedure found satisfactory by authors for separating W from Ti, Nb, Ta, and Zr, consists in fusing mixed oxides with K_2CO_3 and treating the aqueous extract of the melt with slightly ammoniacal $MgSO_4$ solution containing NH_4Cl ; the earths are precipitated and K_2WO_4 remains in solution.

- 198 Prede, A. F., 1934, Columbium: Redkie metally, v. 3, no. 6, p. 6-8; cf. Chem. Abs., v. 29, p. 3206⁴.

A review of the physical and chemical properties of Nb and its compounds, and methods of its recovery is provided.

- 199 Prodingen, W., 1940, Organic reagents used in quantitative inorganic analysis: New York, Elsevier Publishing Co., 203 p.

This monograph correlates widely scattered material on organic reagents into a concise, usable form.

- 201 Rankama, Kalervo, 1944, The geochemistry of tantalum: Comm. géol. Finlande Bull., no. 133, p. 1-78; cf. Chem. Abs., v. 5, p. 3544; v. 32, p. 762; v. 40, p. 11224.

Analyses for Ta by several methods are given for 237 mineral samples, 41 ore samples, 136 rocks, and 10 meteorites. The analyses include X-ray spectrographic method after chemical enrichment. Author employed salicylic acid for enrichment of Nb and Ta in the phenylarsonic acid precipitate containing Ti and Zr.

- 202 ——— 1948, The geochemistry of columbium: Acad. Sci. Fennicae Annales, ser. A. III. Geol-geog., no. 13, 57 p. [English]; cf. Chem. Abs., v. 44, p. 3855.

- 203 Rasmussen, S. W. and Rodden, C. J., 1950, Vanadium, niobium, and tantalum, in Rodden, C. J., ed., Analytical Chemistry of the Manhattan Proj. (Natl. Nuclear Energy Ser., Div. 8, 1. 1): New York, Atomic Energy Comm.—McGraw-Hill Book Co., p. 459-482; cf. Chem. Abs., v. 45, p. 1910g.

Presentation of various analytical methods.

- 204 Redmond, J. C., 1948, Rapid spectrographic analysis of cemented carbide compositions: Steel, v. 122, no. 26, p. 86-88; cf. Chem. Abs., v. 42, p. 5798d.

Equipment, technique, and standards are described for the quantitative spectrographic analysis of Ti, Nb, Ta, Fe, Ni, W; Co, and Mo. Results are within 3 to 10 percent of those obtained by chemical methods.

- 205 Rogers, B., 1948, Routine chemical methods for the chemical analysis of columbium- and tantalum-bearing 18/8 steels: Metallurgia, v. 37, p. 326-330; cf. Chem. Abs., v. 42, p. 6265g.

This is a discussion of the principles involved in the determination of Nb, Ta, and W, in steel, and the modifications required in Cunningham's procedure in the presence of W, Mo, and Ti. Includes 42 references.

- 206 Rohmer, R., 1941, Attack of rare earth minerals and the separation of columbium and tantalum: Acad. sci [Paris] Comptes rendus, tome 212, p. 614-616; cf. Chem. Abs., v. 36, p. 3645.

Minerals decomposed by fusion with KHSO_4 followed by solution in $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$; Nb and Ta separated by fractional crystallization of double fluorides.

- 207 Rosenthaler, L., 1938, The composition of drug ash: Mikrochemie, v. 25, p. 5-8; cf. Chem. Abs., v. 32, p. 9399⁸; v. 33, p. 1442⁹.

Methods for detecting Sn, V, U, Ga, Mn, Nb, Ta, and B are described.

- 208 Ruff, O., and Thomas, F., 1926, The effect of a carbon tetrachloride on columbium and tantalum pentoxides and a procedure for the analytical determination of columbium and tantalum: *Zeitschr. anorg. allg. Chemie*, Band 156, p. 213-225; cf. *Chem. Abs.*, v. 21, p. 2624⁵.

Nb_2O_5 is easily converted to NbCl_5 at $200^\circ - 225^\circ \text{C}$ in an atmosphere of CCl_4 vapors, while Ta_2O_5 is unaffected.

- 209 Sanderson, L., 1940, Tantalum: *Canadian Min. Jour.*, v. 61, p. 803-804; cf. *Chem. Abs.*, v. 35, p. 714³.

The peculiar physical and chemical properties of Ta meet a great variety of uses. Sources, uses, and alloys are enumerated.

- 210 Sandell, E. B., 1950, Colorimetric determination of traces of metals: 2d ed., New York, Interscience Publishers, p. 291.

Some methods of determination and separation are presented and discussed.

- 211 Sato, Ichiro, and Komatsu, Manryu, (to Nihon Elec. Co.) 1947, Tantalum metal from crude tantalic acid: Japan patent 175,161, Oct. 23, 1947; cf. *Chem. Abs.*, v. 44, p. 5303f, 1950.

- 212 Savchenko, G. S., and Tanana'yev, I. B., 1947, Investigation in the field of complex fluorides of Ta and Cb: *Zhur. priklad. khimii*, tom 20, p. 385 [Russian]; cf. NP-2191, July 1950; *Nuclear Sci. Abs.*, v. 5, p. 292 (no. 1825), 1951.

A procedure for the separation of Nb and Ta has been developed based on dissolving a mixture of the oxides in HF, formation of K double fluorides of both metals, and precipitation of K_2TaF_7 at 2 percent excess of acid.

- 213 Sawaya, Tsuguo, 1951, Analytical chemistry of complex fluoride compounds: *Tohoku Univ. Technology Repts.*, v. 16, p. 17-65; cf. *Chem. Abs.*, v. 46, p. 6989c.

The use of complex F compounds in qualitative and quantitative analysis is reviewed and the results of numerous experiments are given. The methods studied include the detection of H_3BO_3 , H_2SiO_3 , Be, Al, Ti, Zr, Nb, Ta, and F, the titrimetric determination of H_2SiO_3 , Ti, Zr, Nb, Ta, and F, and the colorimetric determination of H_3BO_3 , Al, and Zr. In some cases, the different solubilities of complex F compounds can be used for separations.

- 214 Schäfer, Harold, 1948, The purification of columbium preparations: *Zeitschr. Naturforsch. [Wiesbaden]*, Band 3-B, p. 376; cf. *Chem. Abs.*, v. 43, p. 5688h.

Titanium and Sn can be separated from Nb by heating the mixture of oxides with CCl_4 in a sealed tube. On cooling, the NbCl_5 and TaCl_5 crystallized; but TiCl_4 and SnCl_4 , together with the phosgene produced, remained in solution. Preparations containing 30 percent TiO_2 or SnO_2 yield NbCl_5 that contains less than 0.01 percent Ti and no Sn.

- 215 Schäfer, Harold; Bayer, Lisel; and Lehmann, H.; 1952, Chemistry of the elements niobium and tantalum; [Pt.] 9—Equilibrium pressure of the decomposition of niobium tetrachloride: *Zeitschr. anorg. allg. Chemie*, Band 268, p. 268–278; cf. *Chem. Abs.*, v. 47, p. 3745a, 5224g.
- 216 Schäfer, Harold; and Breil, Günther; 1952, Chemistry of niobium and tantalum; [Pt.] 8—The system $\text{Nb}_2\text{O}_5\text{--Ta}_2\text{O}_5\text{--NbO}_2\text{--TaO}_2\text{--H}_2\text{O--H}_2$: *Zeitschr. anorg. allg. Chemie*, Band 267, p. 265–276; cf. *Chem. Abs.*, v. 47, p. 3745a.
- 217 Schäfer, Harold; and Pietruck, C.; 1951, Chemistry of niobium and tantalum; [Pt.] 2—The analytical determination of niobium and tantalum: *Zeitschr. anorg. allg. Chemie*, v. 264, p. 2–16 [German]; cf. *Chem. Abs.*, v. 43, p. 5688h; v. 47, p. 3163e; *Nuclear Sci. Abs.*, v. 5, p. 695 (no. 4386).

Analysis of Nb and Ta, separate or mixed, and in the presence of Sn and Ti, is described. A 0.5-g mixture of oxides is treated with 2 ml CCl_4 at 280°C in a sealed tube for 5–10 hours. TiCl_4 and SnCl_4 are then sublimed out under vacuum at 100°C . At 200°C the NbCl_5 and TaCl_5 are sublimed into a rubber-stoppered glass container, in which they can be weighed. The chlorides are dissolved in concentrated hydrochloric acid and evaporated to dryness, treated with water and evaporated twice, with concentrated NH_3 solution and evaporated twice, and then heated at $800^\circ\text{--}900^\circ\text{C}$ to constant weight as oxides. From the weights of the pure chlorides and oxides the percentages of Nb and of Ta are calculated. Errors in the sum of the two oxides do not exceed 0.5 percent; for 1:1 mixtures of Nb and Ta, they are no more than 1 percent for each.

- 218 ——— 1951, Chemistry of niobium and tantalum; [Pt.] 3—The preparative separation of niobium from tantalum, titanium, and tungsten by the oxalic acid method; *Zeitschr. anorg. allg. Chemie*, v. 264, p. 106–119; cf. *Chem. Abs.*, v. 47, p. 3163g.

The Nb-Ta acid precipitate, with ratio of $\text{Ta}_2\text{O}_5/\text{Nb}_2\text{O}_5$ of $\frac{1}{4}$ or less, is treated with a solution of 3 to 10 g $\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ dissolved in 100 ml 2 N hydrochloric acid. After 7 hours at 60°C from a starting sample in which the $\text{Nb}_2\text{O}_5\text{--Ta}_2\text{O}_5$ mixture contains 91 percent Nb_2O_5 , the solution formed by 5 g $\text{H}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in 100 ml 2 N hydrochloric acid gives an oxide which is 99 percent Nb_2O_5 ; the oxide from the insoluble residue is 75 percent Nb_2O_5 . If the treatment is repeated on the soluble portion, the Ta_2O_5 in the dissolved part is 0.3 percent or less. For samples with a higher initial ratio of Ta, a different preliminary separation is worthwhile. From all tests on acid precipitates prepared in a number of ways and aged various times, the values of percentage of Nb_2O_5 in the oxalic acid solution versus percentage of Nb_2O_5 in the residue fall near one or the other of two straight lines, the one line breaking into the other at about 75 percent of Nb_2O_5 in residue. Two solid phases, one a tantalic acid which can hold Nb_2O_5 to about 75 percent, the other a niobic acid holding Ta_2O_5 to about 25 percent, are indicated. If the initial acid precipitate contains TiO_2 , WO_3 , and SnO_2 ,

the oxalic acid extraction removes Sn with the Nb but leaves the Ti and W largely in the residue.

- 219 Schäfer, Harold; Göser, Camill; and Bayer, Lisel; 1951, Chemistry of niobium and tantalum; [Pt.] 4—Niobium tetrachloride and its decomposition to the trichloride and the pentachloride: *Zeitschr. anorg. allg. Chemie*, v. 264-265, p. 258-272; cf. *Chem. Abs.* v. 47, p. 3163i.

Heating NbCl_3 and NbCl_5 in a sealed evacuated tube, one end at 280°C , the other $20^\circ - 100^\circ$ cooler, gives NbCl_4 . The cooler end serves as a reservoir for NbCl_5 , whose vapor represses the decomposition of NbCl_4 . Hydrogen passed over NbCl_5 (*Chem. Abs.*, v. 33, p. 8132⁹), first at $180^\circ - 190^\circ\text{C}$ and then at 450°C for 18 hours gives NbCl_3 in the hottest part of the tube and NbCl_4 in an intermediate position. Nb with excess NbCl_5 at 300°C and excess NbCl_5 vapor over Nb or Fe at 400°C for 15 hours or over Al powder or foil at 270°C from NbCl_4 . AlCl_3 can be sublimed away in excess NbCl_5 vapor. NbCl_5 is more easily reduced than TaCl_5 . NbO_2 can not be converted to NbCl_4 with CCl_4 or SOCl_2 , but anhydrous $\text{AlCl}_3 + \text{NbO}_2$ in a sealed tube at 290°C gives $\text{NbCl}_4 + \text{AlOCl}$. NbCl_5 at 290°C also converts NbO_2 to NbCl_4 and NbOCl_3 . The extinction direction for NbCl_4 crystals is parallel to the length of the needles when between crossed nicol prisms. The X-ray pattern is different from those of NbCl_3 and NbCl_5 . NbCl_4 is stable in dry air; in moist air it gives off hydrochloric acid and is oxidized, the crystals first darkening and then turning white with no change in external shape and no deliquescence. Solution in the absence of air in a little water or 2 N hydrochloric acid gives a deep-blue solution. Adding 2 N hydrochloric acid to the blue solution causes no change; a drop of NH_3 solution or dilution with water changes the color to greenish-brown and then brown. More NH_3 gives a dirty-brown flocculent precipitate. Oxidizers decolorize the blue solution. NbCl_4 heated in air is changed to white NbOCl_3 . The order of increasing volatility is NbCl_3 , NbCl_4 , NbOCl_3 , NbCl_5 . When heated in the absence of air, NbCl_4 decomposes to NbCl_3 and NbCl_5 unless the decomposition is repressed by a pressure of NbCl_5 vapor greater than its pressure from the NbCl_4 . The quadruple point for NbCl_3 solid, NbCl_4 solid, NbCl_5 liquid, NbCl_5 gas is about 400°C . The composition of NbCl_4 samples is found by determining the amount of hydrochloric acid freed by heating the NbCl_4 in a stream of moist air and weighing the ignited residue, or by determining the reducing power on dissolving in $\text{Fe}_2(\text{SO}_4)_3$ solution and the weight of AgCl obtained from this solution.

- 220 Schäfer, Harold; Bayer, Lisel; and Pietruck, Christel; 1951, The Chemistry of niobium and tantalum; [Pt.] 5—The preparation of titanium- and tin-free samples of tantalum and niobium: *Zeitschr. anorg. allg. Chemie*, v. 266, p. 140-150 [German]; cf. *Chem. Abs.*, v. 47, p. 3164e; *Nuclear Sci. Abs.*, v. 6, p. 108 (no. 847).

Samples of 250-500 mg pure Nb_2O_5 or Ta_2O_5 , 2 ml CCl_4 or SOCl_2 , and 100-300 mg TiO_2 were heated for 24-30 hours at $270^\circ - 350^\circ\text{C}$ for complete chlorination. The products were heated to 110° under

evacuation first with a water aspirator, then with an oil pump. The residue, after conversion to oxide, contained 0.05 percent or less of TiO_2 . When SnO_2 was substituted for TiO_2 , SOCl_2 was not used and the chlorination temperature was 380°C . The oxide formed from the distillation-separation residue was 0.03 percent or less of SnO_2 . Since NbCl_5 and TaCl_5 are soluble in CCl_4 only to the extent of 10 mg per ml or less, the mixed chlorides formed were also separated by 5 extractions with 5 ml portions of CCl_4 . Air need not be excluded. The residue contained twice as much Ti or Sn as after distillation. Heating $\text{COCl}_2 + \text{Nb}_2\text{O}_5$ for 20 hours at 300°C gave complete formation of NbCl_5 ; heating $\text{TiO}_2 + \text{CCl}_4$ at 250°C for 9 hours and $\text{SnO}_2 + \text{CCl}_4$ at 380°C for 10 hours gave almost no chlorination; but if the solids were 50 percent or more Nb_2O_5 , especially when coprecipitated, the chlorinations were complete. Heating Nb_2O_5 with Ta_2O_5 also lowered the temperature of chlorination of Ta_2O_5 with CCl_4 well below 320°C .

- 221 Schäfer, Harold, and Pietruck, Christel, 1951, Chemistry of niobium and tantalum; [Pt.] 6—The separation of niobium and tantalum through the reduction of niobium pentachloride: *Zeitschr. anorg. allg. Chemie*, v. 266, p. 151–160 [German]; cf. *Chem. Abs.*, v. 47, p. 3164g; *Nuclear Sci. Abs.*, v. 6, p. 108 (no. 846), 1952.

NbCl_5 or TaCl_5 is heated to 200°C in half of a sealed evacuated tube. In the other half is Nb or Ta at 400°C . The NbCl_5 is reduced to NbCl_4 by both metals; TaCl_5 is unchanged. A NbCl_4 sample exposure either to vapor or melt of a 50–50 mol. percent TaCl_5 – NbCl_5 mixture contains only 1.8–1.2 mol. percent of TaCl_4 . A fairly pure Nb preparation (analyzing no more than 1.5 percent of Ta_2O_5) can be precipitated by using no more than 80 percent of the Al needed to change all NbCl_5 to NbCl_4 . A fraction containing not more than 14.3 percent of Ta_2O_5 and a fraction containing not more than 12.5 percent of Nb_2O_5 can be obtained by using enough Al to reduce all NbCl_5 to NbCl_3 in a sealed evacuated tube at 280° – 300°C for 60 hours. Tantalum preparations containing less than 0.5 percent of Nb_2O_5 can be obtained by using 25 percent excess Al. NbCl_5 causes very little reduction of TaCl_5 . For H_2 reduction mixed chlorides are heated for 24 hours in a sealed tube with H_2 , 10–40 percent in excess of that needed to reduce the NbCl_5 to NbCl_3 . The reduced sample collects in the tube arm heated to 400°C , and the unreduced sample remains in the tube arm heated to only 200°C . A stream of H_2 (1 liter per hour through a 200-mm tube 35 mm in diameter) also brings about selective reduction at 450°C .

- 222 ——— 1951, Chemistry of Niobium and tantalum; [Pt.] 7—Note on the binary system niobium pentachloride–tantalum pentachloride; *Zeitschr. anorg. allg. Chemie*, v. 267, p. 174–80 [German]; cf. *Chem. Abs.*, v. 47, p. 3164i; *Nuclear Sci. Abs.*, v. 6, p. 254 (no. 2003), 1952.

The chlorides were prepared by treating pure and mixed Nb and Ta oxides with CCl_4 at 270° – 300°C and subliming the product into glass ampoules. Other preparations of the pure chlorides when fully purified gave the same melting point. Temperatures were read from a Hg

thermometer calibrated to 0.1°C . NbCl_5 melts at 204.7°C ; TaCl_5 melts at 216.5°C . Liquidus points for mixtures of the two fit very closely the line calculated from the Clausius-Clapeyron and Raoult equations and the heats of fusion of Alexander and Fairbrother (Chem. Abs., v. 43, p. 7851*h*); the solutions are nearly ideal. The gas phase above mixtures of the chlorides is slightly richer in TaCl_5 . The low-temperature modification of NbCl_5 of Alexander and Fairbrother is not confirmed. NbOCl_3 is soluble only to a limited amount in liquid NbCl_5 , the saturated solution melting 1°C lower than pure NbCl_5 . A solution of 1.0 mol. percent WCl_6 in NbCl_5 is bright red and has a melting point lowered no more than 0.2°C .

- 223 Schliessmann, O., 1941, The sensitivity of spectral analysis of alloy components of steel and iron: *Archiv Eisenhüttenw.*, v. 15, p. 167-174; cf. Chem. Abs., v. 47, p. 845⁸.

The limits of sensitivity were ascertained for pure solutions of the usual components of iron and steel. The determinable limits and the suitable lines for the respective metals and metalloids are given. The elements tested for were Al, As, B, Ba, Be, Ca, Co, Cr, Cu, Mg, Mn, Mo, Nb, Ni, P, Si, Sn, Sr, Ta, Ti, V, W, and Zr.

- 224 Schoeller, W. R., 1937, The analytical chemistry of tantalum and niobium; The analysis of their minerals and the application of tannin in gravimetric analysis: London, Chapman and Hall, Ltd., 198 p.; cf. Chem. Abs., v. 31, 7364⁴.

- 225 Schoeller, W. R., and others, 1921-36. Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates: pts. 1, 2—Chem. Soc. [London] Jour., v. 119, p. 1927-1935, 1921; pts. 3-33—Analyst, v. 49, p. 215, 1924; v. 50, p. 485, 494, 1925; v. 51, p. 613, 1926; v. 52, p. 504, 506, 625, 1927; v. 53, p. 258, 264, 467, 515, 1928; v. 54, p. 320, 453, 704, 709, 1929; v. 55, p. 605, 1930; v. 56, p. 304, 795, 1931; v. 57, p. 72, 284, 550, 750, 1932; v. 58, p. 143, 1933; v. 59, p. 465, 667, 1934; v. 60, p. 284, 506, 1935; v. 61, p. 235, 449, 585, 806, 1936.

The investigations were made in the laboratories of The Sir John Cass Technical Institute and are separately cited and annotated under the names of the individual authors. The following classification of their contents gives their reference numbers in this bibliography:

1. Analytical technique — 227, 235, 247
2. Tartaric acid method — 231, 240, 243, 248, 251, 252, 254
3. Separation of tungsten from the earth acids and other earths— 197, 236, 239, 250
4. Separation of tannin group A from group B — 241, 244, 245, 247a, 253
5. Separation of titania from the earth acids — 226, 233, 237, 238
6. Separation of tantalum from niobium — 196, 196a, 228, 242, 246
8. General summary — 231

- 226 Schoeller, W. R., 1929, Investigation into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 15—A

new method for the separation of tantalum and niobium from titanium and zirconium (tannic acid method): *Analyst*, v. 54, p. 455.

- 227 ——— 1931, Investigation into the analytical chemistry of tantalum, niobium, and their mineral associates; [Pt.] 19—Laboratory notes on analytical technique: *Analyst*, v. 56, p. 304–310.
- 228 ——— 1932, Investigation into the analytical chemistry of tantalum, niobium, and their mineral associates; [Pt.] 24—An improved method for the separation of tantalum from niobium: *Analyst*, v. 57, p. 750–756.
- 229 ——— 1934, Zum Nachweis kleiner Mengen von Tantal und Niob: *Zeitschr. anal. Chemie*, v. 96, p. 252.
- 231 ——— 1936, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 33—General summary and results: *Analyst*, v. 61, p. 806–811; cf. *Chem. Abs.*, v. 30, p. 74877; v. 31, p. 6275.

The chronological sequence in which the experimental results obtained in the study and development of methods for analyzing minerals containing Ta, Nb, and their associates Ti, Zr, Hf, Th, Ce, Tb, Y, etc. have been described was of necessity haphazard. The present paper, which marks the end of 17 years of painstaking work, is a classified index of the whole investigation and a brief discussion of the results.

- 232 ——— 1942, Oxidimetric determination of columbium: *Analyst*, v. 67, p. 321–322; cf. *Chem. Abs.*, v. 36, p. 40505; v. 37, p. 3239.

An electrolytic reduction of Nb^{5+} to Nb^{3+} and its titration with an oxidizing agent. Method necessitates prior removal of Ta and offers no practical advantages over the tannin method.

- 233 ——— and Deering, E. C., 1927, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 9—The precipitation of tungstic acid by tannin; the separation of tungsten from tantalum and niobium: *Analyst*, v. 52, p. 625–637.
- 235 Schoeller, W. R., and Jahn, C., 1926, Investigation into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 6—The precipitation of the earth acids by sodium compounds: *Analyst*, v. 51, p. 613–620; cf. *Chem. Abs.*, v. 21, p. 10732.

When Ta_2O_5 is fused with K_2CO_3 and the aqueous extract of the melt is treated with NaCl, a precipitate is formed of $\text{Na}_8\text{Ta}_6\text{O}_9 \cdot 25\text{H}_2\text{O}$. If Nb_2O_5 is treated similarly, $\text{Na}_{14}\text{Nb}_{12}\text{O}_{37} \cdot 32\text{H}_2\text{O}$ is obtained.

- 236 ——— 1927, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 7—The precipitation of tungstic acid by tannin; [Pt.] 8—The separation of tungsten from tantalum and columbium: *Analyst*, v. 52, p. 504–514; cf. *Chem. Abs.*, v. 21, p. 1073, 38546.

A new method of separation is proposed which depends on the fusion of the mixed oxides with K_2CO_3 , precipitation of the resulting aqueous solution with $NaCl$, recovery of the small quantity of non-precipitated earth acid by hydrolysis in bicarbonate solution and precipitation of the W in the filtrate by tannin and cinchonine.

- 237 ——— 1929, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 14—A new method for the separation of small quantities of tantalum and niobium from titanium: *Analyst*, v. 54, p. 320.
- A tannic acid method.
- 238 ——— 1932, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 21—A reliable method for the quantitative separation of titanium from tantalum and niobium: *Analyst*, v. 57, p. 72.
- 239 ——— 1934, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 26—The interference of tungsten in earth-acid determinations: *Analyst*, v. 59, p. 465.
- 240 Schoeller, W. R., and Powell, A. R., 1921, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 1—The use of tartaric acid in the analysis of natural tantalocolumbates: *Chem. Soc. Jour.*, v. 119, p. 1927-1935.
- 241 ——— 1921, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 2—The separation of zirconium from tantalum and from columbium: *Chem. Soc. Jour.*, v. 119, p. 1927-1935.
- 242 ——— 1928, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 11—The precipitation of titanium by tannin: *Analyst*, v. 53, p. 258-263.
- 243 ——— 1928, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 10—The separation of silica from the earth acids: *Analyst*, v. 53, p. 264-267.
- 245 ——— 1932, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 23—The quantitative separation of tantalum, niobium, titanium, and zirconium, and a new analytical grouping: *Analyst*, v. 57, p. 554-554.
- 246 Schoeller, W. R., and Waterhouse, E. F., 1924, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; The volumetric estimation of columbium: *Analyst*, v. 49, p. 215-220.
- 247 ——— 1928, Observations on the pyrosulphate-hydrolysis method: *Analyst*, v. 53, p. 467.
- 247a ——— 1928, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 13—A new method for the separation of zirconium and hafnium from tantalum and niobium: *Analyst*, v. 53, p. 515.

A further modification of the tannic acid method.

- 248 ——— 1932, Investigations into the analytical chemistry of tantalum, columbium, and their mineral associates; [Pt.] 22—The separation of the earth acids from metals of the hydrogen sulphide group: *Analyst*, v. 57, p. 284–290.
- 249 ——— 1935, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 28—Separation of the rare earths from the earth acids: *Analyst*, v. 60, p. 284–291; cf. *Chem. Abs.*, v. 29, p. 75⁶, 4287⁴.

The mineral is decomposed with HF, whereby the earth acids go into solution and the rare earths, ThO₂, and alkali earths are left as insoluble fluorides.

- 250 ——— 1936, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 31—Determination of tungsten in earth-acid minerals: *Analyst*, v. 61, p. 449–455; *Chem. Abs.*, v. 28, p. 5777¹; v. 29, p. 7219¹; v. 30, p. 3741⁶, 6304⁶.

The small amounts of W that are commonly present in Ta–Nb minerals are precipitated with the earth acids by tartrate hydrolysis at fairly high concentrations under standardized conditions; they are determined in the precipitate produced by hydrolysis by the magnesia method.

- 251 Schoeller, W. R., and Webb, H. W., 1929, Observations on tartaric hydrolysis and the quantitative precipitation of the earth acids and certain other oxides from tartrate solution: *Analyst*, v. 54, p. 709.

Papers 16 and 17 of the Schoeller series.

- 252 ——— 1931, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 20—The separation of tin from tantalum and niobium: *Analyst*, v. 56, p. 795–801.
- 253 ——— 1933, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 25—Separation of uranium from Ta, Nb and Ti: *Analyst*, v. 58, 143–147.

Like the earth acids and the rare earths, U is precipitated completely by the addition of tannin to a neutralized tartrate solution containing NH₄OAc and NH₄Cl. It is also precipitated from oxalate solution by tannin in the presence of a slight excess of NH₃. Uranium, like Zr, Th, Al, and Fe, can be separated from Ta, Nb, and Ti by adding tannin to the faintly acid oxalate solution half saturated with NH₄Cl. In tartaric hydrolysis, U interferes with the normal precipitation of Nb.

- 254 ——— 1934, Investigations into the analytical chemistry of tantalum, niobium, and their mineral associates; [Pt.] 27—Observations on manganese and the analysis of tantalite: *Analyst*, v. 59, p. 667–671; cf. *Chem. Abs.*, v. 28, p. 5777¹; v. 29, p. 75⁵.

A scheme of analysis is recommended for tantalite and columbite which is based on the analytical procedures already described in this series of papers.

- 255 ——— 1936, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 30—Observations on beryllium: *Analyst*, v. 61, p. 235-241; cf. *Chem. Abs.*, v. 6, p. 2725; v. 29, p. 4287⁴, 7219¹; v. 30, p. 3741⁶.

The earth-acid precipitate obtained by boiling the tartrate solution with an excess of mineral acid does not include BeO. Tannin precipitation from oxalate solutions half saturated with NH_4Cl separates Ti, Nb, and Ta from Be as well as from Zr, Th, Al, U, etc.

- 256 ——— 1936, Investigations into the analytical chemistry of tantalum, columbium [niobium], and their mineral associates; [Pt.] 32—Observations on P, V, and a tannin precipitation series: *Analyst*, v. 61, 585-590; cf. *Chem. Abs.*, v. 30, p. 6304⁶, 7487⁷.

To recover P_2O_5 from a Nb, Ta, and P precipitate with tannin, it is fused with NaOH and the melt extracted with half-saturated NaCl solution. Na_3PO_4 dissolves, but the Na salts of Ta_2O_5 and Nb_2O_5 are practically insoluble. Traces of V give tannin precipitate of earth acids a blue-black color. In oxalate solution which is half-saturated with NH_4Cl , Ta, Nb, and Ti are precipitated by tannin completely, while Zr, Th, Al, U, and Be remain in solution. The order in which precipitation with tannin alone takes place is Ta, Ti, Nb, V, Fe, Zr, Hf, Th, U, and Al. Only an elaborate system of repeated crystallization will separate two adjacent members of the series by tannin precipitation. Position of Cr in series is not yet known.

- 257 Schofield, M., 1945, Tantalum: *Indus. Chemist*, v. 21, p. 207-209; cf. *Chem. Abs.*, v. 39, p. 2592².

Past history and present uses.

- 258 Schwarz, Victor, 1947, Chemical analytical studies on niobium and tantalum—A new volumetric method for determining niobium in the presence of tantalum: *Rev. Minera, Geología y Mineralogía*, v. 18, no. 1, 2, p. 13-24; cf. *Chem. Abs.*, v. 46, p. 2445^d.

Comparison of the location of Nb with that of Ta in the periodic table is followed by a discussion of the oxidation levels of the two elements. The reactions of their oxalates in the presence of tannic acid is reviewed as is the reactions of H_2SeO_3 with Nb, Ta, Ti, and other rare elements. A new method for determining Nb is described that depends on reduction with amalgamated Zn, oxidation of the reduced Nb with a known excess of FeCl_3 , and titration with 0.1 N TiCl_3 solution with KCNS as indicator.

- 259 Scott, W. W., 1939, Standard methods of chemical analysis, v. 1: 5th ed., New York, D. Van Nostrand Co.

Collected information on analytical methods and procedures.

- 261 Shakhov, A. S., 1939, Application of polarographic method to the determination of lead and tin in potassium fluoroxy columbates and

metallic columbium: Zhur. priklad. khimii, tom 12, p. 1555 [French, p. 1559].

- 262 Shapiro, M. Ya., 1938, Sensitive test for tantalum and columbium: Zhur. priklad. khimii, v. 11, p. 1028-1032 [French, p. 1032]; cf. Chem. Abs., v. 33, p. 1624¹.

With rhodamine B, 0.2 gamma of Ta can be detected by the violet color and flaky precipitate, but Mo, W, Hg, Au, Sb, and Fe interfere. The fine-grained, grayish-blue precipitate with 0.1 percent aqueous methylene blue is sensitive to 1 gamma of Ta but W, Mo, V, Sb, Sn, and Al interfere at high concentrations. Gentian violet, in 0.1 percent aqueous solution, gives coarse violet flakes when 0.4 gamma of Ta is present, but alkali salts of W, Nb, V, and Sn interfere. Nb can be detected with pyrocatechol and strong NaOAc solution by the yellow color when 0.4 gamma of Nb is present, but mineral acids, Fe, and Ti interfere. After reduction with SnCl_2 , 1.0 gamma of Nb can be detected by the yellow color in the absence of Mo, W, V, and much mineral acid.

- 263 Shemyakin, F. M., and Pilipenko, V. A., 1938, Reactions of columbium and tantalum with resorcinol: Zhur. obshchey khimii, v. 8, p. 824-827 [English, p. 828]; cf. Chem. Abs., v. 33, p. 497³, 1235⁸.

Examination by the method of Gibbs showed that contrary to Platonov and others (Chem. Abs., v. 31, p. 4614⁹), Nb and Ta do not give characteristic color reactions with resorcinol in alkaline solutions, as distinct from the blue-green or green-yellow reactions of resorcinol alone with NH_4OH and KOH. Resorcinol with salts of Ce, Th, Pr, La, Zr, and U in NH_4OH solutions forms blue-green precipitates which differ but little in the shades. If KOH instead of NH_4OH is used, yellowish and gray precipitates are formed.

- 264 Slavin, Morris, 1946, Tantalite of the northeast [of Brazil]; its exportation and analysis; [Pt.] 2—Analysis of tantalite-columbite concentrates: Brazil, Lab. Produção Min. Bol. 21, p. 27-58; cf. Chem. Abs., v. 31, p. 7364⁴; v. 42, p. 7674^c.

This is a modification of Schoeller's tannin method. The entire procedure is diagrammed. Time required for the analysis has been cut from about 15 days to 4 to 5 days.

- 265 Smith, G. Frederick, 1938, Cupferron and neo-cupferron: Columbus, Ohio, F. Frederick Smith Chem. Co., 42 p.

Determination of niobium and tantalum with cupferron is described.

- 266 Snell, F. D., and Snell, C. T., 1949, Colorimetric methods of analysis: 3d ed., New York, D. Van Nostrand Co., v. 2, chap. 28, 29.

The colorimetric determination of Nb and Ta with pyrogallol, peroxide, reduction of fluoride, and hydroquinone are given in detail.

- 267 Société générale métallurgique de Hoboken, 1939, Separating columbium from tantalum: France patent 846,584, September 20, 1939; cf. Chem. Abs., v. 35, p. 1195².

Ores containing Ta and Nb are mixed with a base, such as CaO, and chlorinated at a temperature not above 1050° C. Nb is volatilized as the chloride.

- 268 ——— 1947, Separation of columbium, tantalum, and similar metals: Belgium patent 470,891-2, February 1947; cf. Chem. Abs., v. 43, p. 976bc.

Nb_2O_5 is reduced selectively, preferably with H_2 , to NbO_2 , without acting on the Ta, and part of the Nb is removed by chlorination to the volatile NbCl_5 .

Niobium is selectively nitrified. The nitride is treated with Cl_2 , Br_2 , I_2 , or a dissociable compound thereof, to convert it selectively into a volatile halide, which is separated from Ta_2O_5 by distillation and condensation.

- 269 ——— 1940, Treating of tantalum- and columbium-containing materials: German patent 695,788, August 1, 1940; cf. Chem. Abs., v. 35, p. 5658³; British patent 507,124 (Chem. Abs., v. 34, p. 596⁸).
- 270 Srinivasan, N. R., 1950, Studies on niobium and tantalum; [Pt.] 3—Tartratonibates of alkaline earths and other metals: Indian Acad. Sci. Proc., v. 31A, p. 381-388; cf. Nuclear Sci. Abs., v. 5, p. 11 (no. 78), 1951.
- 271 ——— 1952, Studies on niobium and tantalum; [Pt.] 5—Tartratotalates: Indian Acad. Sci. Proc., v. 36A, p. 278-283, Chem. Abs., v. 47, p. 4238a, 4777h.

Treatment with mineral acids precipitates tantalic acid. Alkali hydroxides also decompose the tartratotalates. In general, they are similar to the tartratonibates.

- 272 Stromberg, A. G., and Reinus, L. M., 1946, Polarographic study of columbium solutions in nitric acid: Zhur. fiz. khimii, v. 20, p. 693-705; cf. Chem. Abs., v. 41, p. 343h.

No polarographic wave is obtained for Nb solutions in HCl , H_2SO_4 , tartaric acid, salicylic acid, KNO_3 + pyrogallol, and NH_4 oxalate. If Nb_2O_5 is fused with K_2CO_3 and then dissolved in HNO_3 , the solution gives a wave the height of which is proportional to the concentration "X" of Nb between 0.19 and 1.12 millimol. per liter, independent of the concentration "Y" of HNO_3 between 0.2 and 2 N.

Niobium solutions in other acids do not show polarographic waves, presumably because their reduction potentials are more negative than that of H_2 .

- 273 ——— L. M., 1949, Polarographic investigation of niobium: Akad. nauk SSSR, Otdel. khim. nauk, Trudy Kom. anal. khimii, v. 2, no. 5, p. 34-53; cf. Chem. Abs., v. 44, p. 9299b.

Niobium can be determined polarographically only in acid nitrate solution.

- 274 Süe, P., 1933, Determination of niobium with 8-hydroxyquinoline: Acad. sci. [Paris] Comptes rendus, tome 196, p. 1022.

- 275 ——— 1937, Contribution to the study of the columbates: *Annales chimie*, v. 7, p. 493–592; cf. *Chem. Abs.*, v. 15, p. 2380; v. 30, p. 3308⁵; v. 31, p. 6991¹.

Preparation and properties of various niobates: Na, K, Li, Cu, Co, Ni, Zn, Al, alkaline earths, Ag, Tl, etc.

- 276 Syrokowski, V. S., and Klimenko, N. G., 1947, Determination of columbium in presence of titanium: *Zavodskaya lab.*, tom 13, p. 1035–1038; cf. *Chem. Abs.*, v. 43, p. 4177^c.

After removal of SiO_2 with $\text{HF} \cdot \text{H}_2\text{SO}_4$, fuse residue with KHSO_4 and extract melt with H_2SO_4 in presence of H_2O_2 ; reduce Ti with Zn-Hg in a separatory funnel; add NH_4OAc , adjust pH to 1.5, boil, filter off precipitate; ignite, weigh, and analyze precipitate colorimetrically for Ti.

- 277 Takagi, Katsuki; and Shimizu, Yukimi; 1950, Application of tantalum to potentiometric titrations; [Pt.] 1—Several potentiometric titrations with tantalum electrodes: *Electrochem. Soc. Japan Jour.*, v. 18, p. 150–152; cf. *Chem. Abs.*, v. 45, p. 8392^h.

Some potentiometric titrations with the Ta electrode are described, including acidimetry with Ta-Ta oxide electrodes and titration of chloride with AgNO_3 with Ta-Pt electrodes.

- 278 Taylor-Austin, E., 1940, Chemical analysis of alloy cast iron: *Foundry Trade Jour.*, v. 63, p. 201–202, 220–222, 230; cf. *Chem. Abs.*, v. 35, p. 2441⁵.

A brief description is given of the analytical procedure for determining the following elements in alloy cast iron: C, Si, Mn, S, P, Ni, Cr, Cu, Al, Ti, V, W, Zr, As, Sb, Sn, Mo, Co, Ta, and B. The use of organic reagents in inorganic analysis is discussed.

- 279 Telep, G., and Boltz, D. F., 1952, Ultraviolet spectrophotometric determination of columbium: *Anal. Chemistry*, v. 24, p. 163–165; cf. *Nuclear Sci. Abs.*, v. 6, p. 179 (no. 1405).

An investigation was undertaken of conditions under which Nb can be determined spectrophotometrically as perniobic acid. Formation of the perniobic acid complex in a concentrated acidic mixture of H_2SO_4 and H_3PO_4 results in a stable yellow hue exhibiting maximum absorbancy in the ultraviolet region. The system conforms to Beer's Law at 342 m μ . The optimum concentration using 1-cm cells is from 0 to 125 parts per million of niobium. The effects of acid concentration, H_2O_2 concentration, and diverse ions were determined. The main interferences are due to ferric, molybdate, vanadate, titanate, tungstate, nitrate, chloride, and fluoride ions.

- 280 Thanheiser, G., 1940, Method for the photometric determination of columbium and tantalum in steel, ferrous alloys, and slags: *Kaiser-Wilhelm Inst. Eisenforsch. Düsseldorf Mitt.*, v. 22, p. 255–265; cf. *Chem. Abs.*, v. 35, p. 2438⁸.

Method based on the yellow coloration of Nb with H_2O_2 in a concentrated H_2SO_4 - H_3PO_4 solution. Forty percent H_3PO_4 (ratio of 2

acids) suppresses the Ti coloration (unless more than 1 percent Ti). The basis for the photometric determination of Ta is the yellow coloration produced with pyrogallol in 3 percent NH_4 oxalate solution. Since Ti also gives a yellow coloration with pyrogallol, it must be determined independently and a correction applied (use chromotropic acid in same solution).

- 281 Thurstan, A. W., 1941, Estimation of tantalum and columbium: Sydney Tech. Coll. Chem. Soc. Jour. and Proc., v. 8, p. 36-43, 1938-40; cf. Chem. Abs., v. 35, p. 6893⁶.

A review.

- 282 Tikhomiroff, Nicolas, 1953, Separation of niobium and tantalum by chromatography on activated alumina: Acad. sci [Paris] Comptes rendus, tome 236, p. 1263-1265 [French]; cf. Nuclear Sci. Abs., v. 7, p. 371 (no. 3044).

The separation of Nb and Ta is based on the absorption of the two elements from oxalic solution on activated alumina and their elution at different pH's. Ta is only partially retained on alumina.

- 283 Tomíček, Oldrich, and Holeček, Václav, 1952, The analysis of niobium and tantalum; [Pt.] 3—Application of morin and quercetin for testing and gravimetric estimation of niobium and tantalum: Chem. listy, v. 46, p. 11-14; cf. Chem. Abs., v. 46, p. 3900^f.

Morin and quercetin form red precipitates with Nb and orange precipitates with Ta. The qualitative test is best carried out in strongly acidic solutions containing 20-25 percent volume for volume H_2SO_4 by adding alcohol solutions of the agents (15 mg in 3 ml EtOH). Ti gives a dark-red precipitate even in neutral solutions. Reprecipitation is advisable.

- 284 Tomíček, Oldrich, and Jerman, Ladislav, 1952, The analysis of niobium and tantalum; [Pt.] 4—Colorimetric determination of niobium and tantalum: Chem. listy, v. 46, p. 144-148; cf. Chem. Abs., v. 46, p. 11024^h.

Ta and Ti form with pyrocatechol a yellow coloration in $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. Nb forms a similar coloration in NH_3 solution. In a mixture of Nb or Ti with Ta, Nb or Ti can be determined colorimetrically after electroreduction which does not affect Ta. In oxalate solution Ti can be reduced, but not Nb, with amalgamated Zn. The extinction maximum for the colored complexes of Nb, Ti, and Ta are 400-440 m μ . Procedure: A sample (10 to 100 mg of oxides) is fused with 3 g KHSO_4 . The melt is dissolved in 50 ml $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution and diluted to 10 ml. An aliquot is treated with 5 ml 10 percent solution of pyrocatechol and diluted with a saturated solution of $(\text{CO}_2\text{NH}_4)_2$ to 25 ml. Determination of Nb or Ti in the presence of Ta was made after electroreduction, and determination of Ti in the presence of Nb and Ta after reduction with Zn amalgam in the Jones reductor.

- 285 Tomíček, Oldrich, and Spurný, Kvetoslav, 1952, The analysis of niobium and tantalum; [Pt.] 1—Cathodic reduction of niobium: Chem. listy, v. 46, p. 6-8; cf. Chem. Abs., v. 46, p. 3900^e.

Reduction of Nb with ZnHg proved unsuccessful. Good results were obtained by electroreduction of Nb^{5+} in 26 percent H_2SO_4 on a Hg cathode in a special apparatus. Nb^{3+} thus formed is oxidized with Fe^{3+} and the resulting Fe^{2+} is titrated with 0.01 N $\text{Ce}(\text{SO}_4)_2$ with o-phenanthroline as indicator. The presence of Ta does not interfere. Ti is also reduced quantitatively.

- 286 ——— 1952, The analysis of niobium and tantalum; [Pt.] 2—Volumetric estimation of titanium in the presence of niobium and tantalum: Chem. listy, v. 46, p. 9–11; cf. Chem. Abs., v. 46, p. 3900f.

Ti salts are reduced with ZnHg_x in 25 percent tartaric acid in a stream of H_2 , even in the presence of Nb, Ta, Y, and Fe. Ti^{3+} ions are titrated potentiometrically with 0.05 N FeCl_3 at 80°C in a stream of H_2 , or with methylene blue or NH_4CNS as indicator. The method is suitable for determining Ti, Nb, and Ta in steels and ores.

- 287 Tompkins, E. R., Khym, J. X., and Cohn, W. E., 1947, Ion exchange as a separation method; [Pt.] 1—The separation of fission-produced isotopes, including individual rare earths, by complexing elutions from Amberlite resin: Am. Chem. Soc. Jour., v. 69, p. 2769–2777; cf. Chem. Abs., v. 42, p. 1834h.

Application of ion-exchange technique to the separation of the major fission species using Amberlite IR-1. Factors affecting separations were studied with respect to rare earths, Th, Nb, Zr, and alkalies.

- 288 Touhey, W. O., and Redmond, J. C., 1948, Analysis of cemented carbide compositions: Anal. Chemistry, v. 20, p. 202–206; cf. Chem. Abs., v. 42, p. 3699f.

Determine W, Ti, Nb, Ta, Fe, Co, Ni, and Al by known methods, which have been modified slightly and require careful attention to details, such as the recovery of Ti, Nb, Ta, etc. from the first WO_3 precipitate.

- 289 Traub, K. N., 1946, Hexamethylenetetramine in separations of titanium and columbium: Indus. Eng. Chemistry, Anal. ed., v. 18, p. 122–124; Chem. Abs., v. 40, p. 1749⁵.

Precipitate Nb + Ti with hexamethylenetetramine, dry, ignite, and weigh as $\text{TiO}_2 + \text{Nb}_2\text{O}_5$. Then determine Ti colorimetrically with H_2O_2 .

- 290 Treadwell, F. P., and Hall, W. T., 1932, Analytical chemistry: 9th ed., New York, John Wiley and Sons, v. 2, p. 224–226; cf. Analyst, v. 54, p. 453, 1934; v. 57, p. 72, 1930.

Procedure is given for the separation of Nb from Ta with tannin.

- 291 Treadwell, W. D., and others, 1952, Determination of niobium and tantalum in columbite and euxenite: Helvetica Chim. Acta, v. 35, p. 2248–2259 [German]; cf. Chem. Abs., v. 47, p. 2634a; Nuclear Sci. Abs., v. 7, p. 129 (no. 1065).

The minerals were subjected to dry chlorination by heating at 500°C for 8 hours in a current of hydrochloric acid gas charged with CCl_4 . By this method TiCl_4 can easily be separated from the more volatile

chlorides of Nb, Ta, U, and Fe; and after reduction of the Fe^{3+} to Fe^{2+} , by using SO_2 , Nb and Ta are quantitatively precipitated as hydroxides. Experiments were made to reduce solutions of niobic acid in H_2SO_4 by means of Zn, and a reproducible method with good yields is described. The potentiometric titration of Nb^{3+} sulfate with KMnO_4 is discussed, and some informative spectroanalytic experiments with a mixture of Nb_2O_5 and Ta_2O_5 are mentioned.

- 292 Treadwell, W. D., and Nieriker, R., 1942, Oxidimetric determination of columbium by itself and in the presence of iron, vanadium, and titanium: *Helvetica Chim. Acta*, v. 25, p. 474-488; cf. *Chem. Abs.*, v. 36, p. 4050⁵.

Method employs an electrolytic reductor; the solution is titrated with KMnO_4 and the end point determined potentiometrically.

- 293 Tschernikov, J. A., and Karsajevskaya, M. P., *See* Chernikhov, Ya. A., and Karsayevskaya, M. P. (48a).

- 294 Uspenskaya, T. A., and Chernikhov, Yu. A., 1940, Application of the iodate method to the separation and determination of the rare elements: *Acad. sci. U. R. S. S. Comptes rendus*, tome 28, p. 800-801 [English]; cf. *Chem. Abs.*, v. 35, p. 2440⁴.

Ce and Th are precipitated as iodate and the precipitates made the basis of a gravimetric or volumetric determination of Ce and Th. Similar precipitates can be obtained with Zr and Ta. Nb is not precipitated by KIO_3 , hence reaction can be used for separating Ta and Nb. Details concerning procedure not given.

- 295 Vail, J. H., 1944, Modified rapid method for Cb: *Chemist Analyst*, v. 33, p. 14-15; cf. *Chem. Abs.*, v. 38, p. 1974¹.

A procedure is suggested for determining Nb, Si, Cr, and Ni, but no definite analytical data are given with respect to its reliability.

- 296 Van Valkenburgh, H. B., and Crawford, T. C., 1941, Detection of certain metals in mineral and ores — an ammonium hypophosphate fusion method: *Indus. Eng. Chemistry, Anal. ed.*, v. 13, p. 459-460; cf. *Chem. Abs.*, v. 35, p. 5410⁹.

Fuse 0.1 g of finely powdered material with $\text{NH}_4\text{H}_2\text{PO}_4$. The mass has a melting point of about 60° C after fusion becomes quiet. The color of the melt or the color resulting from treatment with water or hydrogen peroxide solution will serve for detecting Cr, Co, Nb, Mn, Mo, Te, Ti, U, V, and W. To test for Nb, add concentrated hydrochloric acid to melt, boil, and add a little mossy Sn, which gives an intense blue color.

- 297 Vinogradova, N. A., and Gushtyuk, E. I., 1945, A rapid method for the analysis of loparite concentrates: *Zavodskaya lab.*, tom 11, p. 223-226; cf. *Chem. Abs.*, v. 40, p. 1114².

The method proposed is rapid and gives sufficiently accurate results. Niobium and Ta are separated from Ti and Fe by tannin method; Ta is determined colorimetrically with pyrogallol in $(\text{C}_2\text{O}_4)_2\text{H}_2$ solution; and rare earths are separated from oxalic acid solution.

- 298 Von Hevesy, G., and Bohn, J., 1927, Determination of tantalum by means of X-ray spectra: *Zeitschr. anorg. allg. Chemie*, v. 164, p. 69-80; cf. *Chem. Abs.*, v. 21, p. 3172⁸.

- 299 Voznesenski, S. A., 1934, Inner-complex salts: *Uspekhi khimii*, v. 4, p. 531-562; cf. *Chem. Abs.*, v. 29, 732⁸.

A review on the metallic derivatives of a variety of substances, chiefly organic, such as glyccoll, picolines, amino, and other substituted acids, aliphatic and aromatic amines of various types, phenols, esters, ketones, and aldehydes. Among the metals considered are Cu, Zr, Th, Ta, K, and Fe. Their applications in analytical chemistry are discussed.

- 300 Wacker, R. E., and Baldwin, W. H., 1950, The separation of zirconium and niobium oxalate complexes by anionic exchange: ORNL-637; cf. *Nuclear Sci. Abs.*, v. 4, p. 469 (no. 3018), *British Abs.*, v. C, p. 244, 1951.

It was found that Zr and Nb are adsorbed on Dowex-1 resin from oxalic acid solution (0.1-0.4 M) and can be preferentially eluted (Zr being removed first) with 1 M hydrochloric acid and 0.01 M oxalic acid solution.

- 301 Waterkamp, M., 1949, The determination of columbium and tantalum in steels: *Archiv Eisenhüttenw.*, v. 20, p. 5-8; cf. *Chem. Abs.*, v. 43, p. 4597^h.

The properties of the salts of Nb and Ta which are useful in analytical chemistry are carefully reviewed, and 6 procedures are described for determining Nb and Ta.

- 302 Weiser, Bruno, 1941, Progress in the inorganic heavy-chemical industry; [Pt.] 14—Heavy-metal compounds and others: *Chem. Fabrik*, v. 14, p. 248-255, 281-286, 302-305; cf. *Chem. Abs.*, v. 35, p. 4922⁷; v. 38, p. 5369⁴.

A review of about 943 patents and papers between 1933 and December 31, 1940 on compounds of Fe, Cu, Zn, Cr, Mn, Ni, Co, V, Mo, W, U, Nb, Ta, Sn, Bi, Hg, Au, Zr, Th, Pt, Pd, Rh, Ru, Ge, Ra, Tl, and In, with many references.

- 303 Wenger, P., Duckert, R., and Rusconi, Y., 1945, Critical study of reagents for cations; [Pt.] 13—Reagents for the cations of the rare earths and yttrium: *Helvetica Chim. Acta*, v. 28, p. 274-278 [French]; cf. *Chem. Abs.*, v. 37, p. 5335^{6, 8}; v. 38, p. 6228⁶; v. 40, p. 529⁵.

A discussion of the reagents: oxalic acid, NH_4 succinate, I_2 , cacotheline, and quinalizarine. Elements discussed were the rare earths: Th, Sc, Hf, Zr, Nb, Ta, Ba, Sr, Ca, U, Al, and Fe.

- 304 Wernet, Josef, 1952, The behavior of titanium, niobium, and tantalum compounds in hydrochloric acid as a foundation for a new preparative separation method: *Zeitschr. anorg. allg. Chemie*, v. 267, p. 213-237 [German]; cf. *Nuclear Sci. Abs.*, v. 6, p. 298 (no. 2344); *Chem. Abs.*, v. 46, p. 5484^f.

With the intention of separating Ti as $(\text{NH}_4)_2\text{TiCl}_6$ from Nb and Ta, the author has studied the behavior of various compounds of these elements, particularly the hydrated oxides and chlorides, in highly concentrated hydrochloric acid, and the reaction of these solutions with NH_4Cl . The Ti precipitates in every case as $(\text{NH}_4)_2\text{TiCl}_6$. The Ti content of a mixture containing approximately 3 percent TiO_2 in Nb_2O_5 was reduced to less than 0.07 percent TiO_2 in one step by this method. In a mixture of 50 weight percent TiO_2 and Ta_2O_5 , the separation resulted in approximately 1 percent TiO_2 remaining in the Ta_2O_5 . In a Ti-Nb-Ta mixture with a Ti:Nb ratio greater than or equal to 2, the Nb precipitated with the $(\text{NH}_4)_2\text{TiCl}_6$. This coprecipitation suggests a way of separating both these elements from Ta.

- 305 Williams, A. E., 1940, Estimation of tantalum and columbium as combined oxides: *Chem. Eng. Min. Rev.*, v. 32, p. 271-272; cf. *Chem. Abs.*, v. 34, p. 4691⁵.

For determining $(\text{Ta, Nb})_2\text{O}_5$, the Schoeller method is used.

- 306 Williams, A. F., 1952, Inorganic chromatography on cellulose; [Pt.] 12—The quantitative determination of tantalum and niobium in complex minerals, ores, and synthetic materials: *Chem. Soc. [London] Jour.*, p. 3155-3163, cf. *Nuclear Sci. Abs.*, v. 6, p. 771 (no. 6308),

A new chromatographic method for the quantitative separation of Ta from Nb and for their determination in certain types of mixtures, based on cellulose as absorbent and ethyl methyl ketone containing HF as solvent, was described as Part 11 of this series [see reference 40 of this bibliography]. This method has been modified so that it is now applicable to the quantitative determination of these elements in complex materials containing appreciable amounts of Ti, Sn, and Zr. A solution of sample in HF containing ammonium fluoride being used, Ta is extracted first in ethyl methyl ketone saturated with water. The column is then washed with ketone containing 1 percent (volume per volume) of HF (40 percent weight per weight aqueous solution) which arrests the movement of Ti, Sn, and Zr. Niobium is then extracted with the ketone containing 12.5 percent of HF. After removal of solvent the Ta and Nb are determined in the appropriate fractions by ignition to the pentoxides.

The procedure is simple and accurate and has been used for the analysis of a wide range of natural and synthetic materials.

- 307 Winkler, J. E. R., 1940, Absolute colorimetry in the metal industry: *Schweizerische Archiv angew. Wiss. u. Technik*, v. 6, p. 194-201; cf. *Chem. Abs.*, v. 34, p. 7779⁸.

The author reviews fundamentals of absolute colorimetry and describes the Pulfrich photometer. He describes the range of application, method, time, and interference by other elements for the determination of each of the following elements in steel: Al, Cr, Co, Cu, Mn, Mo, Ni, Nb, P, Si, N, Ti, V, and W.

- 308 Wirtz, H., 1939, Separation of tantalum and columbium: *Zeitschr. anal. Chemie*, v. 117, p. 6-9; cf. *Chem. Abs.*, v. 33, p. 6748².

In presence of bromphenol blue indicator all Ta and very little Nb can be precipitated by means of tannin. Before this treatment, determine the sum of Nb_2O_5 and Ta_2O_5 as usual. The small quantity of Nb in the tannin precipitate can be determined by dissolving the oxide, reducing in a Jones reductor and titrating with KMnO_4 .

309. ——— 1941, Separation of columbium and tantalum: *Zeitschr. anal. Chemie*, v. 122, p. 88–94; cf. *Chem. Abs.*, v. 31, p. 6275; v. 33, p. 67482; v. 36, p. 47731.

Author recommends tannin separation of Schoeller to determine Nb and Ta. Very little Nb is precipitated with Ta at the proper pH.

- 310 Wood, G. A., 1950, The determination of total Ta and Cb in minerals and ores by solvent extraction in presence of activated cellulose: CRL-AE-62; cf. *Nuclear Sci. Abs.*, v. 5, p. 288 (no. 1802), 1951.

A chromatographic method is described for the separation of mixed Ta and Nb oxides from minerals and ores by extraction of fluorides with methyl ethyl ketone containing HF in presence of activated cellulose. NH_4F is used to inhibit extraction of a number of other metals. After removal of solvent, the Ta and Nb are precipitated with tannin and weighed as oxides.

- 311 Yager, J. C., 1937, Tantalum—its occurrence, properties, and uses: *Australian Chem. Inst. Jour. and Proc.*, v. 4, p. 59–65; cf. *Chem. Abs.*, v. 31, p. 33496.

- 313 Yatsenko, V. G., and Basse, A. P., 1937, Separating columbium and tantalum salts: Russian patent 51,125, May 31; *Chem. Abs.*, v. 33, p. 47504.

Salts of Nb and Ta are separated by hydrolysis of their fluorides. The HF separated thereby is combined with metallic Zn or similar electronegative metal. The insoluble $\text{K}_4\text{Ta}_4\text{O}_5\text{F}_{14}$ separates from solution.

- 314 Yoshida, Yukito, 1942, Quantitative study of fusions with an alkaline oxidizing flux: *Chem. Soc. Japan, Jour.*, v. 63, p. 439–451, 615–628; cf. *Chem. Abs.*, v. 41, p. 3011b.

The results obtained by fusion with a flux composed of 3 parts NaOH, 2 parts KNO_3 , and 1 part K_2CO_3 are tabulated for compounds of Cu, Ag, Ca, Ba, Zn, Hg, B, Al, Ce, La, silicates, phosphates, niobates, selenites, tungstates, and numerous other compounds.

- 315 Yu, Pe-Nien, 1948, A modified method in the analysis of uranium ore with high content of columbium and tantalum: *Chinese Chem. Soc. Jour.*, v. 15, p. 170–178; cf. *Nuclear Sci. Abs.*, v. 1, p. 547 (no. 1969).

Scott's method was modified by fusing the ore with $\text{K}_2\text{S}_2\text{O}_7$ and extracting with hot water. The U is precipitated by NH_3 and weighed as U_3O_8 .

- 316 Zeltzer, S., 1932, Polarographic studies with the dropping mercury cathode; [Pt.] 25—An investigation of solutions of gallium, titan-

ium, vanadium, niobium, and tantalum: Coll. Czechoslovak Chem. Commun., v. 4, p. 319.

- 317 Zemel, V., 1936, Chemical analysis of black and ruby sands: Soviet-skaya zolotopromyshlennost, no. 3, p. 50-51; cf. Chem. Abs., v. 30, p. 8063⁸.

This procedure makes it possible in one sample to determine W, V, and Mo and to detect Sm, Ta, Nb, Th, Zr, and rare earths.

- 318 Zvyagintsev, O. Ye., and Namoradze, Z. G., 1938, New quantitative reactions of columbium and tantalum: Zhur. obshchey khimii, v. 8, p. 829; cf. Chem. Abs., v. 31, p. 4614⁹; v. 33, p. 497³.

Reactions of Nb and Ta with resorcinol does not give a qualitative test for these elements.

AUTHOR INDEX

[This index to the authors of papers listed in the bibliography is arranged alphabetically by the anglicized form of the name. Accent marks, including the umlaut, have been disregarded in the arrangement. The numbers following each name listed in the index correspond to reference numbers that precede the citations listed in the bibliography]

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