21(7)

AUTHORS: Zolotov, Yu. A., Alimarin, I. P. SOV/89-6-1-11/33

TITLE: Separation of Np^{239} in Radiochemically Pure State by Using the Recoil of the Nuclei of Fission Products (Vydeleniye Np^{239} v radiokhimicheski chistom sostoyanii s ispol'zovaniyem otdachi yader produktov deleniya)

PERIODICAL: Atomnaya energiya, 1959, Vol 6, Nr 1, pp 70 - 71 (USSR)

ABSTRACT:

UO_2 is crushed in an agate bowl, and by means of elutriation a fraction of all those particles is produced which precipitate with a velocity of $\leq 2.16 \cdot 10^{-3}$ cm/sec. The concentration of the suspension is determined by weighing. A certain quantity of the suspension is emptied into a glass, diluted, and saturated with calcium nitrate. The solution is very thoroughly stirred and the calcium carbonate is precipitated with concentrated carbonate of ammonia. The precipitate is filtered, washed, and dried. The weight ratio between UO_2 and $CaCO_3$ fluctuated in the various experiments between 1:100 and 1:500.
The ready mixture is then irradiated in a reactor in the

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course of 12 to 48 hours with a neutron flux of 7.10^{12} to 2.10^{13} $n/cm^2.sec$. The irradiated sample is dissolved in a small quantity of cold 1.5 n HCl, centrifuged, and washed 2 to 3 times with 1.5 n hydrochloric acid and 3 times with water. The UO_2 washed in this way was dissolved in ~ 2ml hot concentrated nitric acid, and potassium bromide is added to the solution for neptunium oxidation. The solution (~0.1 molar $KBrO_3$) is then heated for 15 minutes at 90 to 100°C.

After cooling aluminum nitrate is added, and the solution is diluted up to 1.5 mol for $Al(NO_3)_3$ and 1 mol for HNO_3, after which it is filled into a measuring bowl for ether extraction. Extraction was carried out 3 to 4 times. Neptunium is recovered from the extracts.

The purity of the Np^{239} thus separated was checked on the basis of the half-life. The method described makes it possible to separate Np^{239} in the course of 1 to 2 hours, the

Separation of Np^{239} in Radiochemically Pure State by Using the Recoil of the Nuclei of Fission Products SOV/89-6-1-11/33

active fission products being separated already during the first two minutes.
The mixture of UO_2 and the carrier substance can be produced quickly and can be easily used for a long time. Instead of $CaCO_3$ it is possible to use also other material, which is easily able to stand the high temperature in the reactor. There are 1 figure and 5 references.

SUBMITTED: March 14, 1958

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5(2,3)

AUTHORS: Alimarin, I. P., Corresponding Member, Academy of Sciences, USSR, Zolotov, Yu. A., Pal'shin, Ye. S. SOV/ 20-124-2-24/71

TITLE: Extraction of Pentavalent Neptunium (Ekstraktsionnoye izvlecheniye pyativalentnogo neptuniya)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 124, Nr 2, pp 328-330 (USSR)

ABSTRACT: Reliable data on the extraction of pentavalent neptunium have hitherto not been available. In the present paper, the authors prove that this extraction is possible by using 1-nitroso-2-naphthol solution in n-butyl and isoamyl alcohol at pH 9-10. Indicator quantities of Np^{239} served for this purpose. The controls were performed with weighable quantities of Np^{237}. The isotope Np^{239} was isolated from uranium which had been irradiated with neutrons (Ref 3). The initial solutions of neptunium-(V) were obtained by reduction of neptunium-(VI) by means of hydrazine in the cold. The results obtained are presented in figure 1. As can be seen from it the neptunium compound cannot be extracted up to pH 6 . The percentage rate of extraction is dependent to a considerable extent on the nature

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of the extracting agent; with n-butyl alcohol 90-95% are extractable at one single extraction, with isoamyl alcohol 80-85%, whereas methyl-ethyl ketone, chloroform and amyl acetate are far less effective. Benzene and diethyl ether sparingly extract neptunium-(V). Higher amounts of fluorides, carbonates, phosphates, acetates and citrates interfere with the extraction. Nitrates, chlorides and sulfates exert no negative influence. The said extraction is first of all indicative of the interaction of the NpO_2 ion with 1-nitroso-2-naphthol. Reactions of the neptunium-(V) with organic reagents are unknown with a few exceptions only (complex formation Refs 5, 6). Analytical reactions for Np(V) are missing. The mentioned interaction is in agreement with earlier observed spectroscopic data (Ref 6). The results obtained can be utilized in the separation of neptunium from plutonium and uranium. The extraction of tetravalent plutonium (proved by the authors together with D. Nishanov) took place already at pH 1.0 - 1.5. The investigations are being carried on. There are 1 figure and 6 references, 5 of which are Soviet.

ASSOCIATION: Institut geokhimii i analiticheskoy khimii im. V. I. Vernadskogo Akademii nauk SSSR
(Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy of the Academy of Sciences, USSR)

SUBMITTED: October 16, 1958

C/005/60/000/007/004/004
F031/F004

AUTHOR: Yu. A. Zolotov

TITLE: Application of supersonic waves in analytic chemistry

PERIODICAL: Hua Hsüeh T'ung Pao, no. 7, 1960, 28-32

TEXT: The chemical characteristics of supersonic waves, the effect of supersonic waves on electrolytic processes and the application of photo-sonic effects in chemistry are described. In solutions, the speed of a sonic wave depends on the concentration of the solution and obeys the formula

$$C^2 = (1 - V) C_0^2 + V \times 2.32 \times 10^7 \phi_0$$

where V = weight of solute; C_0 = sonic speed in solvent or solution; ϕ_0 = crystallizing energy of solutions. Supersonic waves have an effect in many aspects of electrochemistry. The depolarization characteristic will lower electrolytic voltage, cause a change in the properties and composition of the electrolytic precipitate, and

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Application of supersonic ...

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affect electrolytic current and production rates. Supersonic waves can even out the density of ions around electrodes, reduce polarization of electrodes, and eliminate formation of gases. The fact that when infrared rays absorb gases, a pulse sound may occur can be utilized as a means for photo-sonic analysis of gases. Supersonic oscillation has been used for dispersing, condensing, degasing, and agitating in chemical analysis or for changing oxidation conditions of solutions. Supersonic waves are also significant in lighting sensitive photographic films and have been used to increase the speed of multiphase reactions. Research in hydrosynthesis is significant for research in analytic chemistry. Equation for calculating the magnitude of hydrosynthesis are

$$C = \sqrt{\frac{1}{\beta\phi \cdot \rho}}$$

where C = sonic speed; $\beta\phi$ = adiabatic compressibility; ρ = density;

$$h = \frac{\left(1 - \frac{\beta}{\beta_0}\right)(100 - g)}{g}$$

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Application of supersonic ...

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where h = number of grams of water entering a solute; g = number of gram of solute in 100 gm of solution β and β_0 = compression coefficients of solution and solvent (water) respectively, and

$$R = C^{1/2}\frac{M}{\rho}$$

where M = molecular weight; ρ = density; C = sonic speed; R = sonic speed not dependent on temperature. The author conludes that supersonic waves are being used in solving both theoretical and practical problems of analytic chemistry. The article was translated by Yüan Ta-wen (7086/1129/2455). There are 6 figures. Important English-language references are: Freyer, E. B. J. Am. Chem. Soc. 53, 1313 (1931); Wada J., Shimbo S., J. Acoust. Soc. Amer. 24, 199 (1952) and Chem. Abstrs. 48 9136 (1954); Sasaki T., Yasumuago T. Ball. Chem. Soc. Japan 26, 175 (1953) and 28, 269 (1955).

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S/186/60/002/001/021/022
A057/A129

AUTHOR: Zolotov, Yu.A.

TITLE: Problems concerning the chemistry of actinides at the XVII International Congress on Pure and Applied Chemistry

PERIODICAL: Radiokhimiya, v. 2, no. 1, 1960, 127 - 128

TEXT: The present paper gives short descriptions of some reports at the XVII International Congress on Pure and Applied Chemistry which took place in August - September 1959 in Munich (Western Germany). The present selected reports concern problems of actinide chemistry. Several authors from non-Soviet-bloc countries are quoted, and the following lecturers from Soviet-bloc countries are cited: Erzowska-Tszebiatovskaja, Barteky and Chmylovskaja (Poland) reported on the structure of some uranyl compounds in aqueous solutions, the dependence of solvation of the solution on the dielectric constan., dipole moment and structure of the solvent; Starý (Czechoslovakia) investigated benzoyl acetone complexes in his report: "Determination of the composition and constants of complex formation of some complexes by means of the extraction method"; Grdenić (Yugoslavia) determined the coordination number of U(IV) in acetyl acetone and dialkyl phosphate

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Problems concerning the chemistry of actinides....

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A057/A129

compounds.

SUBMITTED: October 21, 1959

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ZOLOTOV, Yu.A.

Symposium on the theory of extraction. Zhur.neorg.khim. 5 no.6: 1391-1392 Je '60. (MIRA 13:7)
(Extraction (Chemistry)--Congresses)

24081
S/186/60/002/006/001/026
A051/A129

21.4200

AUTHORS: Alimarin, I. P.; Zolotov, Yu. A.; Pal'shin, Ye. S.

TITLE: The extraction of 1-nitroso-2-naphtholate of pentavalent neptunium

PERIODICAL: Radiokhimiya, v. 2, no. 6, 1960, 637 - 642

TEXT: Pentavalent neptunium was extracted at a pH = 9 - 10 using solutions of 1-nitroso-2-naphthol in n-butyl and isoamyl alcohols and the optimum conditions of the extraction were established. It is thought that the NpO$_2^+$ ion is capable of forming intra-complex compounds, which can be extracted with organic solvents with a reagent correspondingly selected. The 1-nitroso-2-naphthol was chosen as reagent in this work. It was shown that extraction can be used for purifying neptunium from plutonium and uranium. Indicator quantities of Np^{239} separated from uranium irradiated with neutrons according to the method based on the nuclear emission of the decay products (Ref. 15: Y. A. Zolotov; I. P. Alimarin Atomnaya energiya, 6, 1, 70, 1959) and in some cases according to the extraction method by Pal'shin (Ref. 16: E. S. Pal'shin, Y. A. Zolotov, Radiokhimiya, 1, 4, 482, 1959) were used. The effect of a series of factors on the extraction degree

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was studied. The results confirmed that neptunium extraction takes place at a pH over 6. The optimum pH value for each solvent depends in addition to other factors - on the solubility of the reagent in the solvent. Seven solvents were studied: benzene, chloroform, isoamyl alcohol, n-butyl alcohol, diethyl ether, amylacetate, methylethyl ketone. The best solvents for the extraction of 1-nitroso-2-naphtholate proved to be n-butyl and isoamyl alcohol; It is pointed out that uranyl 1-nitroso-2-naphtholate is well extracted with alcohols. The extraction of macroquantities of Np^{237} (0.6 mg/ml) showed that macro-quantities are extracted in the same manner as the indicator quantities. Since the extraction takes place within a pH range where neptunium (V) is quite hydrolyzed, the concentration of the element should be as low as possible to avoid the formation of a hydroxide precipitate. It was seen that large quantities of fluorides, phosphates, carbonates, oxalates and nitrates hinder the extraction of $Np^{(IV)}$ 1-nitroso-2-naphtholate with n-butyl or isoamyl alcohol at a pH = 9 - 10. Ethylenedimaninetetraacetic acid has a significant negative effect on the extraction. Small quantities of fluorides, carbonates and hydrogene peroxide have little effect. Nitrates, chlorides and sulfates have no effect at all. The presence of borax (buffer solution, concentration 0.05 M) does not impair the extraction, but uranium (VI)

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The extraction of 1-nitroso-2-naphtholate ....

and plutonium (IV) not bound in the complexes and being highly hydrolyzed have a great negative effect. When extracting with a 0.25 % solution of the reagent in isoamyl alcohol from a 0.05 molar solution of borax (pH = 9.24) a complete extraction of $Np^{(IV)}$ is reached as a result of four extractions. Neptunium (V) can be easily extracted from accumulated organic fractions by double washing with a solution of a pH less than 6. In order to produce pure $Np^{239}(V)$, it is suggested using the extraction of nitroso-naphtholate with subsequent reextraction of neptunium in hydrochloric or nitric acid of a given concentration. The following method for $Np^{(V)}$ purification without a carrier is recommended: the initial solution of neptunium not containing interfering $Np^{(V)}$-ions is processed for the purpose of transferring it to the pentavalent state with a 0.1 M solution of hydrazine-nitrate in 1 M HNO_3 at room temperature. The solution is neutralized by a universal indicator and an equal volume of 0.1M borax solution is added. Neptunium is extracted 4 times with equal volumes of a 0.25 % solution of 1-nitroso-2-naphthol in n-butyl or isoamyl alcohol, shaking the funnel each time for 4 minutes. The organic fractions collected (3-minute shaking) are processed twice with small volumes of 0.1 M nitric acid. The combined water fractions are washed with chloroform until the water solution becomes colorless. The coexistence of $Np^{(V)}$, $U^{(VI)}$ and $Pu^{(IV)}$ in solution is accomplished in the easi-

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The extraction of 1-nitroso-2-naphtholate ....

est way by processing the element mixture with sodium nitrite in nitric acid, heating it for a long time. The authors investigated the extraction of Pu$^{(IV)}$ using various solvents (methylethylketone, amylacetate, isoamyl alcohol, n-butyl alcohol, chloroform). It was found that extraction starts at pH = 0.5 - 1.0; n-butyl alcohol extracts 1-nitroso-2-naphtholate of Pu$^{(IV)}$ better than isoamyl alcohol. The separation of the elements was found possible in certain cases only. The purification from small quantities of plutonium was accomplished in the following manner: plutonium was bound with a small excess of ammonium sulfate and Np$^{(V)}$ was extracted with a solution of 1-nitroso-2-naphthol in isoamyl alcohol. The main plutonium mass remains non-extracted. If the organic fractions are then washed with an aqueous solution at pH = 3, neptunium (V) is re-extracted and partially extracted plutonium remains in the organic phase. The washing is performed twice. There are 2 tables, 2 figures and 17 references: 8 Soviet-bloc and 9 non-Soviet-bloc. The references to the four most recent English language publications read as follows: H. A. C. McKay, Ind. Chem., 33, 297, 1957; J. Kooi, Tracer experiments on the solvent extraction of neptunium and plutonium. Amsterdam, 1956; G. Gibson, D. M. Gruen, J. J. Katz, J. Am.

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S/186/60/002/006/001/026
A051/A129

Chem. Soc., 74, 2103, 1952; D.M. Gruen, J. J. Katz, J. Am. Chem. Soc., 75, 3773, 1953.

SUBMITTED: July 15, 1959.

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ZOLOTOV, Yu. A.

report to be submitted for the IUPAC 21st Conference and 18th Intl. Congress of Pure and Applied Chemistry, Montreal, Canada, 2-12 August 1961

ALIMARIN, I. P., and ZOLOTOV, Yu. A., Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy, Academy of Sciences USSR - "Extraction of metal chelate compounds as affected by the nature of the solvent" (To be presented in Russian) (Section C.2 - 11 Aug 61, morning)

BAGDASAR'YAN, Kh. S., and KRONGAUZ, V. A., Scientific Research Physico-Chemical Institute imeni L. Ya. Karpov, Moscow - "Some aspects of energy transfer in radiation chemistry" (Section A.1, Session II - 7 Aug 61, morning)

DELIMARSKIY, Yu. K., Institute of General and Inorganic Chemistry, Academy of Sciences UkSSR, Kiev - "The kinetics of the electrode processes in the electrolysis of melted salts" (Section B.3 - 10 Aug 61, morning)

DELIMARSKIY, Yu. K., ANDREYA, V. N., BOYKA, K. M. (Possibly VOYKA, K. M.), NASARENKO, G. D., and KAPSOVA, T. N., Institute of General and Inorganic Chemistry, Academy of Sciences UkSSR, Kiev - "Electrochemical experiment with melted borate and phosphate" (Section A.3, c, (2), Session I - 11 Aug 61, morning)

DELIMARSKIY, Yu. K., PANCHENKO, I. D., and SHILINA, G. V., Institute of General and Inorganic Chemistry, Academy of Sciences UkSSR, Kiev - "On the convection diffusion in melted salts" (Section B.3 - 9 Aug 61, afternoon)

GERASIMOV, Ya I., Moscow State University imeni M. V. Lomonosov, (Co-Chairman, Section A.3,c,(2), Session I(3), 11 Aug 61, afternoon)

GERASIMOV, Y. I. LAVRENT'YEV, V. I., KUZNETSOV, F. A., and REZUKHINA, T. N., Moscow State University imeni M. V. Lomonosov - "The thermodynamic properties of columbium and cerium oxides" (Section A.3,c,(3), Session II(A), 11 Aug 61, morning)

GOL'DANSKIY, V. I., Institute of Chemical Physics, Academy of Sciences USSR - "Two-proton radioactivity - a new kind of radioactive decay of nuclei" (Section A.4 - 7 Aug 61, morning)

PETRUKHIN, O.M.[translator]; RODIN, S.S.[translator]; ZOLOTOV, Yu.A., kand. khim. nauk, red.; ARNOL'DOV, V.V., red.; GRIBOVA, M.P., tekhn. red.

[Extraction in analytical chemistry and radiochemistry] Ekstraktsiia v analiticheskoi khimii i radiokhimii. Moskva, Izd-vo inostr. lit-ry, 1961. 350 p. (MIRA 15:4)
Translated articles.
(Extraction (Chemistry)) (Radiochemistry)

S/078/61/006/005/003/015
B121/B208

AUTHORS: Zolotov, Yu. A., Marov, I. N., and Moskvin, A. I.

TITLE: Complex compounds of pentavalent neptunium in solutions of oxalic acid and ethylene diamine tetraacetic acid

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 5, 1961, 1055 - 1062

TEXT: The authors studied the complex formation of the NpO_2^+ ion with oxalic acid and ethylene diamine tetraacetic acid by ion exchange. The study was carried out statically by observing the distribution of Np^V used as indicator between a 0,05 M solution of NH_4ClO_4 and the КУ-2 (KU-2) cation exchanger in its ammonium form in the presence and in the absence of complexing agents. The experiments were performed at 20 ± 2°C. The grain size of the cation exchanger was 50 - 70 mesh. The pH of the solution was measured with a glass electrode and the ЛП-5 (LP-5) pH-meter. In a pH-range of 2 - 5 Np^V was found to form complex ions of the following

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Complex compounds of ...

composition with oxalic acid $NpO_2HC_2O_4$, $NpO_2C_2O_4^-$ and $NpO_2(C_2O_4)_2^{3-}$. The corresponding stability constants were: $5{,}0 \cdot 10^2$, $1{,}1 \cdot 10^4$, and $2{,}3 \cdot 10^7$. At a pH below 1,6 no neptunium oxalate complexes were obtained. In the pH-range 2,0 - 3,2 $NpO_2HC_2O_4$ and $NpO_2C_2O_4^-$ are formed, and at a pH above 4 $NpO_2(C_2O_4)_2^{3-}$. The complex formation is expressed by the following equations:

$$NpO_2^+ + HC_2O_4^- \rightleftharpoons NpO_2HC_2O_4 \quad (11),$$

$$NpO_2^+ + C_2O_4^{2-} \rightleftharpoons NpO_2C_2O_4^- \quad (12),$$

$$NpO_2^+ + 2\,C_2O_4^{2-} \rightleftharpoons NpO_2(C_2O_4)_2^{3-} \quad (13).$$

The thermodynamic stability constants of the NpO_2^+ oxalate complexes were compared with published data (Ref. 5: D. M. Gruen, J. J. Katz, J. Amer. Chem. Soc., 75, 3772 (1953)); the results are summarized in Table 3. In the pH-range 4,9 - 6,2 neptunium forms with ethylene diamine tetraacetic acid only one complex of the composition NpO_2Y^{3-} and a stability constant

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S/078/61/006/005/003/015
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Complex compounds of ...

$(4,9 \pm 1,1) \cdot 10^9$. There are 1 figure, 4 tables, and 21 references: 12 Soviet-bloc and 9 non-Soviet-bloc. The four most recent references to English language publications read as follows: 4. G. Gibson, D. M. Gruen, J. J. Katz, J. Amer. Chem. Soc., 74, 2103 (1952); 8. J. C. Hindman, L. B. Magnusson, T. J. La Chapelle, The Transuranium Elements, Nat. Nucl. En. Sev., Div. IV, 14B, paper 15,2, New York 1949; 9. R. Sjoblom, J. C. Hindman, J. Amer. Chem. Soc., 73, 1744 (1951); 10. R. M. Diamond, K. Street, G. T. Seaborg, J. Amer. Chem. Soc., 76, 146 (1954).

ASSOCIATION: Institut geokhimii i analiticheskoy khimii im. V. I. Vernadskogo Akademii nauk SSSR
(Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy of the Academy of Sciences USSR)

SUBMITTED: April 8, 1960

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Complex compounds of ...

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Table 3 Термодинамические константы устойчивости оксалатных комплексных ионов NpO_2^+ $(pK_{д})$

① Состав комплекса	② Данные [5]	③ Наши данные
$NpO_2HC_2O_4$	—	2,70
$NpO_2C_2O_4^-$	3,93	4,39
$NpO_2(C_2O_4)_2^{3-}$	7,06	7,36

Legend to Table 3: Thermodynamic stability constants of the oxalate complex ions NpO_2^+ (pK_a): (1) Composition of the complexes; (2) data (Ref. 5); (3) data of the authors.

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25506

S/078/61/006/008/005/018
B121/B203

5.3700

AUTHORS: Moskvin, A. I., Marov, I. N., and Zolotov, Yu. A.

TITLE: Complex compounds of pentavalent neptunium with citric and tartaric acid

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 8, 1961, 1813-1820

TEXT: The complex compounds of neptunium with oxy-acids have been little studied. Only Yu. A. Zolotov and Yu. P. Novikov (Ref. 1: Zh. neorgan. khimii 6, 1055, 1961) detected a change in the absorption spectra of neptunium solutions in the presence of complex-forming substances. The complex formation at different pH values was studied by cation exchange between pentavalent neptunium in 0.05 molar NH_4ClO_4 solution and the cation exchanger КУ-2 (KU-2) in ammonium form in the presence of citric and tartaric acid. The pH value was measured with an ЛП-5 (LP-5) pH meter with glass electrodes. In the pH range 4.3 - 5.2, neptunium (V) was found to form the following complexes with citric acid: NpO_2Cit^{2-} and NpO_2HCit^- with the stability constants $(4.7 \pm 1.0) \cdot 10^5$ (pK = 3.67) and

Complex compounds of... 25506 S/078/61/006/008/005/018
B121/B203

$(4.9 \pm 1.4) \cdot 10^2$ ($pK = 2.69$). The absorption spectra of neptunium (V) solutions in the presence of citric acid at pH = 0.9, 3.5, 5.1, 5.7, and 6.1 were recorded. The absorption maximum at 983 mμ was found to shift due to complex formation to the side of increased complex formation. The complex formation in the system neptunium (V) - tartaric acid was determined by a change in absorption spectra of neptunium (V) solutions in the presence of tartaric acid at different pH values of the solutions. In the pH range 3.2 - 6.2, neptunium - tartaric acid complexes of the following compositions were determined by cation exchange: NpO_2HTar, NpO_2Tar^-, $NpO_2Tar_2^{3-}$, and $NpO_2Tar_3^{5-}$. These complexes have the following stability constants: $2.3 \cdot 10^2$ ($pK = 2.36$), $2.1 \cdot 10^2$ ($pK = 2.32$), $2.0 \cdot 10^4$ ($pK = 4.30$), and $1.5 \cdot 10^6$ ($pK = 6.18$). There are 7 figures, 3 tables, and 4 Soviet-bloc references.

ASSOCIATION: Institut geokhimii i analiticheskoy khimii im. V. I. Vernadskogo AN SSSR (Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy AS USSR)

SUBMITTED: July 8, 1960

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ZOLOTOV, Yu.A.; ALIMARIN, I.P.

Some aspects of the theory of extraction of inner-complex compounds.
Dokl. AN SSR 136 no. 3:603-606 Ja '61. (MIRA 14:2)

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo
AN SSSR. 2. Chlen-korrespondent AN SSSR (for Alimarin).
(Extraction (Chemistry)) (Complex compounds)

S/186/62/004/002/010/010
E075/E136

21 4200

AUTHORS: Zolotov, Yu.A., and Nishanov, D.

TITLE: Chromatographic separation of neptunium from uranium, plutonium and fission products

PERIODICAL: Radiokhimiya, v.4, no.2, 1962, 241-244

TEXT: The authors describe a method of separation of ^{239}Np from uranium irradiated with neutrons and demonstrated a possibility of separation of Np, Pu and U. Cation exchanger КУ-2 (KU-2) in its H form was used as a sorbent, and nitric acid as eluent. The study of elution of pentavalent Np indicated that with 1M nitric acid as eluent Np is eluted very quickly and gives a sharp peak in the elution curve. The main part of the fission products is sorbed on the column and is not eluted by nitric acid. Certain fission elements such as Ru, Te and I are eluted together with Np. The final purification of Np is achieved by extraction with ether after its oxidation to hexavalent state. Experiments were carried out to discover

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Chromatographic separation of ... S/186/62/004/002/010/010
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whether Np(VI) is reduced by cation-exchanger KU-2. It was found that the resin reduced Np(VI) to Np(V) in 1M nitric acid. It was shown that Np, Pu and U can be separated easily on the cation-exchanger. Np and U are eluted separately with 1M nitric acid followed by elution of Pu with 3M nitric acid. There are 4 figures.

SUBMITTED: April 27, 1961

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S/186/62/004/003/004/022
E075/E436

AUTHORS: Zolotov, Yu.A., Alimarin, I.P.

TITLE: Influence of the nature of the solvent on the extraction of internal complex compounds

PERIODICAL: Radiokhimiya, v.4, no.3, 1962, 272-279

TEXT: The authors investigated the influence of the chemical nature of the solvent on the extraction of a number of internal complex compounds of Np(V), V (VI), Co (II), Ce, Fe and some other elements. The solvents used were alcohols, ketones, complex esters, simple esters, halogen substituted hydrocarbons and hydrocarbons. For each solvent the dependence of the extraction on the pH of the aqueous phase was studied and the comparison of the various solvents was obtained at the optimum pH values. It was shown that Np (V) and Co (II) are extracted readily with solutions of tenoyl trifluoroacetone in butyl alcohol. The internal complex compounds in which the coordination forces are not saturated are easier to extract with active oxygen-containing solvents, in particular alcohols, and are usually more difficult to extract with other compounds, such as hydrocarbons and their

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Influence of the nature ...

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E075/E436

halogen substituted derivatives. The extraction of the complex compounds can be made easier by their combination with the excess reagent. In this connection the application of polydentate reagents is considered as possible in the future. The internal complex compounds with saturated coordination valencies are extracted with different types of solvents, including nonpolar oxygen free solvents, such as chlorinated hydrocarbons. A scheme is given for the separation of the complex compounds with saturated and unsaturated coordination valencies. The "saturated" compounds are extracted with the reagent solution in a non-reactive solvent such as benzene; the compounds with the unsaturated coordination often remain in the aqueous phase. Subsequently, the second element is extracted with an active solvent such as butyl alcohol. There are 4 figures and 2 tables.

SUBMITTED: March 25, 1961

Card 2/2

ZOLOTOV, Yu.A.

Moscow Seminar on Analytical Chemistry. Zhur.anal.khim. 19 no.9:1171-1172 '64. (MIRA 17:10)

MILICH, N. [Milic,N.]; PETRUKHIN, O.M.; ZOLOTOV, Yu.A.

Extraction of uranyl thenoyltrifluoroacetonate. Zhur. neorg. khim. 9 no.11:2664-2667 N '64 (MIRA 18:1)

1. Institut geokhimii i analiticheskoy khimii imeni V.V. Vernadskogo AN SSSR i Institut "Boris Kidrich", Belgrad, Yogoslaviya.

CHMUTOVA, M.K.; ZOLOTOV, Yu.A.

Extraction and complex formation of plutonium (IV) with N-benzoylphenylhydroxylamine. Radiokhimiia 6 no.6:640-646 '64. (MIRA 18:2)

RYABCHIKOV, D.I., otv. red.; ALIMARIN, I.P., red.; PALEY, P.N., red.; BORISOVA, L.V., red.; ZOLOTOV, Yu.A., red.; SENYAVIN, M.M., red.; KARYAKIN, A.V., red.; VOLYNETS, M.P., re [illegible]

[Modern methods of analysis; methods of studying the chemical composition and structure of substances. On the seventieth birthday of Academician A.P.Vinogrado] Sovremennye metody analiza; metody issledovaniia khimicheskogo sostava i stroeniia veshchestv. K semidesiatiletiiu akademika A.P.Vinogradova. Moskva, Nauka, 1965. 333 p. (MIRA 18:7)

1. Akademiya nauk SSSR. Institut geokhimii i analiticheskoy khimii. 2. Chlen-korrespondent AN SSSR (for Ryabchikov).

ZOLOTOV, Yu.A.

Mechanism of the elementary act of extraction of inner-complex compounds. Dokl. AN SSSR 162 no.3:577-580 My '65. (MIRA 18:5)

1. Institut geokhimii i analiticheskoy khimii im. V.I.Vernadskogo AN SSSR. Submitted November 14, 1964.

ZOLOTOV, Yu.A.; PETRUKHIN, O.M.; ALIMARIN, I.P.

Extraction of ethylenediaminetetraacetates (as exemplified by iron and thorium complexes). Zhur. anal. khim. 20 no.3:347-350 '65. (MIRA 18:5)

1. Institut geokhimii i analiticheskoy khimii imeni Vernadskogo AN SSSR, Moskva.

ZOLOTOV, Yu.A.; ALIMARIN, I.P.; BAGREYEV, V.V.

Extraction of inner-complex compounds in the presence of salts. Part 2: Uranyl 1-(2-pyridylazo)-2-naphtholate. Trudy Kom. anal. khim. 15:59-63 '65. (MIRA 18:7)

ZOLOTOV, Yu.A.; KUZ'MIN, N.M.; LAMBREV, V.G.

Extraction of inner-complex compounds in the presence of salts. Part I: Extraction of hydroxyquinolinates from solution having the high content of potassium chloride and sodium iodide. Trudy Kom. anal. khim. 15:51-58

ZOLOTOV, Yu.A.

Extraction as a method of concentration in analytical chemistry. Trudy Kom. anal. khim. 15:3-29 '65. (MIRA 18:7)

SHKHANOVSKAYA, A.T.; ALIMARIN, I.P.; ZOLOTOV, Yu.A.

Extraction of thallium (III) from chloride solutions. Zhur. neorg. khim. 10 no.3:707-711 Mr '65. (MIRA 18:7)

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo AN SSSR.

L 23364-66 EWT(m)/EWP(j)/EWP(t) IJP(c) RM/JD

ACC NR: AP6008694 SOURCE CODE: UR/0075/65/020/011/1153/1159

30

28

AUTHOR: Zolotov, Yu. A.; Lambrev, V. G.

B

ORG: Institute of Geochemistry and Analytical Chemistry im. V. I. Vernadskiy, AN SSSR, Moscow (Institut geokhimii i analiticheskoy khimii AN SSSR)

TITLE: Extraction of indium 8-hydroxyquinolinate

SOURCE: Zhurnal analiticheskoy khimii, v. 20, no. 11, 1965, 1153-1159

TOPIC TAGS: indium compound, metal extracting, acid base equilibrium, hydroxyl group

ABSTRACT: The extraction of indium 8-hydroxyquinolinate with chloroform was studied as a function of pH at 8-hydroxyquinoline concentrations of 0.001, 0.01, and 0.05 M, the concentration of indium being $5\cdot10^{-7}$ g-ion/l. 0.1 M solutions of 8-hydroxyquinoline in benzene and isoamyl alcohol were also used. The chloroform extraction was carried out by the direct and reverse methods of reaching equilibrium, and the direct method was shown to give low results probably because of hydrolysis of indium. pH values for 50% extraction of indium were determined, and the extraction constant for choloroform was found from the equation

$$In^{3+} + 3HA_{(o)} \rightleftarrows InA_{3(o)} + 3H^{+}.$$

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to be log K_{ex} = 1.35 ± 0.01. Other constants obtained were the distribution constant of the indium 8-hydroxyquinolinate complex in the $CHCl_3$-H_2O system (log P_c = 3.09 ± 0.02) and the stability constants of indium complexes with 8-hydroxyquinoline (log β_1 = 12.00, log β_2 = 23.99; log β_3 = [illegible]). All the constants were obtained at an ionic strength of 0.1 (perchlorate solution) and 25°C. The authors thank I. P. Alimarin and L. N. Yakimenko for their constant attention and suggestions. Orig. art. has: 5 figures, 2 tables, 8 formulas.

SUB CODE: 07/ SUBM DATE: 03Nov64/ ORIG REF: 001/ OTH REF: 011

Card 2/2

ALIMARIN, I.P.; ZOLOTOV, Yu.A.; KARYAKIN, A.V.; PETROV, A.V.; SUKHANOVSKAYA, A.I.

Extraction of thallium (III) compounds from chloride solutions. Zhur. neorg. khim. 10 no.2:524-530 F '65. (MIRA 18:11)

1. Institut geokhimii i analiticheskoy khimii imeni Vernadskogo AN SSSR i Volgogradskiy politekhnicheskiy institut. Submitted May 5, 1964.

BAGREYEV, V.V.; ZOLOTOV, Yu.A.

Extraction of chelate compounds in the presence of salts. Report 3: Cobalt (II) and uranium (VI) acetyl acetonates. Zhur. anal. khim. 20 no.8:867-869 '65. (MIRA 18:10)

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo AN SSSR, Moskva.

ZOLOTOV, Yu.A.; LAMBREV, V.G.; CHMUTOVA, M.K.; SIZONENKO, N.T.

Extraction of inner-complex compounds of elements with 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone. Dokl. AN SSSR 165 no.1:117-120 N '65. (MIRA 18:10)

1. Institut geokhimii i analiticheskoy khimii im. V.I.Vernadskogo AN SSSR. Submitted March 29, 1965.

ZOLOTOV, Yu.A.

"Solvent extraction of metal chelates" by J. Stary. Zhur. anal. khim. 20 no.9:1026 '65. (MIRA 18:9)

ZOLOTOV, Yu.A.; LAMBREV, V.G.

Extraction of inner-complex compounds of elements by 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone. Extraction of calcium. Zhur. anal. khim. 20 no.6:659-664 '65. (MIRA 18:7)

1. Institut geokhimii i analiticheskoy khimii imeni Vernadskogo AN SSSR, Moskva.

ZOLOTOV, Yu.A.

Analytical Chemistry at the Twentieth International Congress on Theoretical and Applied Chemistry. Zhur. anal. khim. 21 no. 1:137-141 '66 (MIRA 19:1)

ZOLOTOVITCH, G. [Zolotovich, G.]; DECEVA, R. [Decheva, R.]

Respiratory intensity of the flower of Rosa damascena Mill.
Doklady BAN 17 no.7:657-660 '64.

1. Experiment Station for Aromatic Plants, Kazanluk.

ANTIPOVA-KARATAYEVA, I.I.; ZOLOTOV, Yu.A.; SERYAKOVA, I.V.

Spectrophotometric study of chloride complexes of iron (III) in relation to the extraction of iron by oxygen-containing solvents. Zhur. neorg. khim. 9 no.7:1712-1719 Jl '64. (MIRA 17:9)

1. Institut geokhimii i analiticheskoy khimii imeni Vernadskogo AN SSSR.

ZOLOTOV, Yu.A., kand.khimich. nauk; PETRUKHIN, O.M., kand.khimich.nauk

Theory and practice of extraction. Zhur. VKHO 9 no. 2:145-156 '64.
(MIRA 17:9)

ZOLOTOV, Yu. A.; ALIMARIN, I.P.; BODNYA, V.A.

Kinetics of extraction; a survey. Zhur. anal. khim. 19 no. 1:
28-36 '64. (MIRA 17:5)

1. Institut geokhimii i analiticheskoy khimii imeni Vernadskogo
i Moskovskiy gosudarstvennyy universitet imeni Lomonosova, Moskva.

SHAKHOVA, N.V.; ALIMARIN, I.P.; ZOLOTOV, Yu.A.

Coextraction of calcium and strontium with hydroxyquinolinates of certain elements. Dokl. AN SSSR 152 no.4:884-887 O '63. (MIRA 16:11)

1. Institut geokhimii i analiticheskoy khimii im. V.I. Vernadskogo AN SSSR. 2. Chlen-korrespondent AN SSSR (for Alimarin).

PALEY, P.N.; RYABCHIKOV, D.I.; DEDKOV, Yu.M.; ZOLOTOV, Yu.A.

Methods of concentration in analytical chemistry. Zav.lab. 29
no.11:1279-1280 '63. (MIRA 16:12)

CHMUTOVA, M.K.; PETRUKHIN, O.M.; ZOLOTOV, Yu.A.

Extraction of inner-complex compounds of plutonium and other elements with N-benzoylphenylhydroxylamine. Zhur. anal. khim. 18 no.5:588-595 My'63. (MIRA 17:2)

SERYAKOVA, I.V.; ZOLOTOV, Yu.A.

Extraction of iron from solutions of chlorides of various metals. Zhur. neorg.khim. 9 no.1:187-189 Ja '64.

Extraction of a chloride complex of iron from solutions of perchloric and sulfuric acids. Ibid.:190-195 (MIRA 17:2)

1. Institut geokhimii i analiticheskoy khimii imeni V.I.Vernadskogo AN SSSR.

ALIMARIN, I.P.; ZOLOTOV, Yu.A.; SHAKHOVA, N.V.

On coextraction phenomenon. Trudy Kom.anal.khim. 14:24-30 '63.

SERYAKOVA, I.V.; ZOLOTOV, Yu.A.; KARYAKIN, A.V.; GRIBOV, L.A.; ZUBRILINA, M.Ye.

Possibility of the solvation of a tetrachloroferrate ion in the extraction of iron from chloride solutions. Zhur. neorg. khim. 7 no.8:2013-2018 Ag '62. (MIRA 16:6)

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo AN SSSR.
(Ferrates) (Solvation) (Chlorides)

ALIMARIN, I.P.; PETRUKHIN, O.M.; ZOLOTOV, Yu.A.

Extractibility on inner complex compounds as dependent on the pH of the aqueous phase. Minima on the extraction curves. Zhur.-anal.khim. 17 no.5:544-550 Ag '62. (MIRA 16:3)

1. V.I.Vernadskiy Institute of Geochemistry and Analytical Chemistry Academy of Sciences, U.S.S.R., Moscow.
(Chelates) (Extraction (Chemistry))
(Hydrogen-ion concentration)

SERYAKOVA, I.V.; ZOLOTOV, Yu.A.; KARYAKIN, A.V.; GRIBOV, L.A.

Hydration and solvation of strong acids during their extraction.
Zhur.neorg.khim. 8 no.2:474-480 F '63. (MIRA 16:5)

1. Institut geokhimii i analiticheskoy khimii imeni V.I.Vernadskogo
AN SSSR.
(Acids--Spectra) (Extraction (Chemistry)) (Solvation)

BAGREYEV, V.V.; ZOLOTOV, Yu.A.

Effect of the nature of the organic solvent on the extraction of inner-complex compounds of thallium. Zhur.anal.khim. 17 no.7:852-857 O '62. (MIRA 15:12)

1. Vernadskiy Institute of Geochemistry and Analytical Chemistry, Academy of Sciences, U.S.S.R., Moscow.
(Thallium compounds) (Extraction (Chemistry)(Solvents)

ZOLOTOV, Yu.A.; SERYAKOVA, I.V.; KARYAKIN, A.V.; GRIBOV, L.A.; ZUBRILINA, M.Ye.

Infrared spectra of some strong acids extracted with oxygen-containing solvents. Zhur.neorg.khim. 8 no.2:481-486 F '63. (MIRA 16:5)

1. Institut geokhimii i analiticheskoy khimii imeni V.I.Vernadskogo AN SSSR.

(Acids—Absorption spectra) (Solvents)

BAGREYEV, V.V.; ZOLOTOV, Yu.A.

New method for increasing the selectivity during the extraction of inner-complex compounds. Zhur.anal.khim. 18 no.4:425-429 Ap '63. (MIRA 16:6)

1. V.I.Vernadsky Institute of Geochemistry and Analytical Chemistry, Academy of Sciences, U.S.S.R., Moscow.
(Chelates) (Extraction (Chemistry))

L 10617-63 EWP(q)/EWT(m)/BDS AFFTC JD

L 10617-63

ACCESSION NR AP3001020

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Card 2/2

ALIMARIN, I.P.; ZOLOTOV, YU.A.; PETRUKHIN, O.M.

"Separation of heavy metals" by A.K.De. Reviewed by I.P.Alimarin, IU.A.Zolotov, O.M.Petrukhin. Zhur.anal.khim. 17 no.6:772 S '62.
(MIRA 16:1)

(Metals—Analysis) (De, A.K.)

ZOLOTOV, Yu.A.

Periodical and monographic literature on extraction. Zav.lab. 28 no.11:1404-1408 '62. (MIRA 15:11)
(Bibliography--Extraction (Chemistry))

ZOLOTOV, Yu.A.

Hydration and solvation of acids and salts during extraction.
Usp.khim. 32 no.2:220-238 F '63. (MIRA 16:4)

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo AN SSSR.
(Acids) (Extraction (Chemistry)) (Salts)

ZOLOTOV, Yu. A.; ALIMARIN, I. P.

Effect of the nature of the solvent on the extraction of chelates.
Radiokhimiia 4 no.3:272-279 '62. (MIRA 15:10)

(Chelates) (Solvents)

ALIMARIN, I.P.; ZOLOTOV, Yu.A.

Extraction in chemical analysis. Zav.lab. 28 no.11:1285-1286 '62. (MIRA 15:11)

(Extraction (Chemistry)) (Chemistry, Analytical)

ZOLOTOV, Yu.A. (Vorobevskoye Chausse 47a, Moskva)

Extraction of chelate compounds and the coordination number of the central atom. Acta chimica Hung 32 no.3:327-335 '62.

1. Institut geokhimii i analiticheskoy khimii imeni V.I. Vernadskogo Akademii nauk SSSR.

ZOLOTOV, Yu.A.; SERYAKOVA, I.V.; KARYAKIN, A.V.; GRIBOV, L.A.;
ZUBRILINA, M.Ye.

Hydrate-solvate mechanism of extraction. Dokl.AN SSSR 145
no.1:100-103 Jl '62. (MIRA 15:7)

1. Institut geokhimii i analiticheskoy khimii imeni V.I.Vernadskogo AN SSSR. Predstavleno akademikom A.P.Vinogradovym.
(Extraction (Chemistry))

ZOLOTOV, Yu.A.; SERYAKOVA, I.V.; ANTIPOVA-KARATAYEVA, I.I.; KUTSENKO,
Yu.I.; KARYAKIN, A.V.

Effect of the organic solvent on the formation of the tetrachloroferrate ion during extraction of iron from chloride solutions.
Zhur.neorg.khim. 7 no.5:1197-1203 My '62. (MIRA 15:7)
(Iron) (Chlorides) (Extraction (Chemistry))

ALIMARIN, I.P.; ZOLOTOV, Yu.A.

Nomencloture of extraction. Zhur.anal.khim. 17 no.2:263-266 Mr-Ap '62. (MIRA 15:4)

1. Institut geokhimii i analiticheskoy khimii imeni V.I.Vernadskogo AN SSSR, Moskva.

(Extraction (Chemistry)--Nomencloture)

CHUSOV, V.G.; ZOLOTOV, Yu.I.

Testing and improving a uniflow disk-knife grinder. Trudy TSNIISP no.7:101-105 '59. (MIRA 13:9)
(Grinding machines)

GAVALOV, I.V.; ZOLOTOV, Yu.I.

KS-200 control apparatus. Spirt.prom. 26 no.2:12-14 '60.
(MIRA 13:6)
(Alcohol)

KOLOSKOV, S. P.; ZOLOTOV, Yu. I.

Mechanized washing of fermentation tanks. Spirt. prom. 29
no.3:24-26 '63. (MIRA 16:4)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut fermentnoy
i spirtovoy promyshlennosti.

(Fermentation—Equipment and supplies)

CA

A new method of synthesizing amides of carboxylic acids directly from the acids. A. V. Kirsanov and Yu. M. Zolotov (Stalin Met. Inst., Dnepropetrovsk). *J. Gen. Chem. U.S.S.R.* 19, No. 12, a675-81(1949)(Engl. translation).—See *C.A.* 44, 4416g. E. J. C.

CH

A. P. Sachs

New method of preparation of amides of carboxylic acids directly from the acids. A. V. Kirsanov and Yu. M. Zolotov (Stalin Metallurg. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 19, 2291-7 (1949).—Monobasic carboxylic acids are readily converted to the *amides* by treatment with $SO_2(NH_2)_2$ (I) in the presence of pyridine. BzOH and I in aq. soln. failed to give any $BzNH_2$ either at room temp. or on heating, with or without added bases. Heating an equimolar mixt. of I and BzOH to 125° 2 hrs. until the lower layer solidified, followed by treatment with H_2O and Et_2O, extn. of the Et_2O layer with N NaOH, and evapn. gave up to 50% $BzNH_2$ in a series of expts.; increase of the temp. to 220° gave more tarry matter, lower yields of $BzNH_2$, and some PhCN. When equimolar mixts. of BzOH and I are heated with 5-6 moles pyridine, the max. yields of $BzNH_2$ approach 50% only, since I reacts with pyridine even at 100°, yielding apparently derivs. of either $NH(SO_3H)_2$ or trisulfamide; other reactions of the by-product H_2NSO_3H also intervene, making is necessary to use an excess of I for best results. The best yields of the amide result on long heating at relatively low temps. and the optimum procedure is as follows: the well-dried reagents 1.22 g. BzOH 50-100 cc., 1.92 g. I, and 5 ml. pyridine in a flask provided with an air-cooled reflux condenser are heated on a steam bath 3 hrs., the pyridine removed in vacuo, the residue treated with 10 ml. H_2O or 10 ml. 2 N Na_2CO_3, the mixt. evapd. to dryness, taken up in 10 ml. H_2O, heated rapidly to boiling, cooled, let stand 3 hrs. in a cool place, and filtered, yielding 74.4% $BzNH_2$, m. 125-7°, after repeated washing with H_2O; extn. of the acidified aq. soln. with Et_2O gave an addnl. 9.8% $BzNH_2$, for a total yield of 84.2%. Similarly, 1.67 g. *p*-$O_2NC_6H_4CO_2H$, 1.92 g. I, and 5 ml. pyridine after 3 hrs. at 100° gave 81.5% *amide*, m. 196-7°; the *m*-isomer, m. 139-40°, is obtained in 84% yield by using 1.5 moles of I; *o*-$ClC_6H_4CONH_2$, m. 140-1°, is prepd. in 90% yield, the *p*-isomer, m. 169-71°, in 96% yield by using only 25% excess of I. Heating 1.2 g. AcOH, 2.88 g. I, and 5 ml. pyridine 3 hrs. to 100° gave after addn. of Na_2CO_3, concn., and extn. with Me_2CO, 84% *$AcNH_2$*, m. 80° (from Et_2O-EtOH); *caprylic acid* gave 80% *amide*, m. 103-4° (from H_2O); and *palmitic acid* gave 81.5% *amide*, m. 102-3° (from Me_2CO), best prepd. by heating 0.5 hr. to 130°; *$PhCH_2CO_2H$* gave 70% *amide*, m. 154-5° (from H_2O), after 3 hrs. at 100°.

G. M. Kosolapoff

CA

New method of transformation of carboxamides into nitriles. A. V. Kirsanov and Yu. M. Zolotov (Stalin Metallurg. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 384-8(1950); cf. C.A. 44, 4440g.—$RCONH_2$ are readily transformed into RCN by means of H_2NSO_3H (I), at 180-230°. Refluxing 12.1 g. $BzNH_2$ and 11.6 g. I in 50 ml. dry pyridine 9 hrs. gave after neutralization only 0.6 g. PhCN. However, heating 12.1 g. $BzNH_2$ and 14.55 g. I to 200° in 20 min. and to 230° in 20 min. gave a slow distn. of 90.6% PhCN (crude; 88% pure). Similar heating of $AcNH_2$ with a small excess of I gave in 30 min. 62% MeCN; 11.8 g. $AcNH_2$ and 9.7 g. I, however, heated to 230° over 1.5 hrs. gave 3.95 g. (95.5% based on I) MeCN and 0.8 g. AcOH. Similarly 0.02 mole $C_7H_{15}CONH_2$ and 0.04 mole I in 20 min. at 200-10° gave 1.8 g. *caprylonitrile*, b. 195-7°, d^{20} 0.8198, and a similar reaction gave 80.5% *palmitonitrile*, b_{10} 180-8°, m. 28-9°. Heating 0.83 g. *m-nitrobenzamide* and 0.61 g. I to 230° in 20 min. and keeping 5 min. gave 94% *nitrile*, m. 115-17°; the *p*-isomer, obtained similarly in 94% yield, m. 146-7°, while *o*-ClC_6H_4CN, m. 43-6°, was obtained in 91% yield, as was the *p*-isomer, m. 92-3°. $PhCH_2CONH_2$ at 200-15° gave 72.5% *nitrile*, b_{10} 105-6°. G. M. K.

CA

New method of transformation of carboxamides into nitriles. A. V. Kirsanov and Yu. M. Zolotov (I. V. Stalin Met. Inst., Dnepropetrovsk). *J. Gen. Chem. U.S.S.R.* 20, 303-7(1950)(Engl. translation). See C.A. 44, 6814. R. M. S.

CA

Amidation of dibasic carboxylic acids. A. V. Kirsanov and Yu. M. Zolotov (Stalin Met. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 1145-50(1950); cf. *C.A.* 44, 4440g, 6384i.—Heating 0.005 mole sebacic acid, 0.015 mole $SO_2(NH_2)_2$ (I), and 6 ml. dry pyridine 3 hrs. on a steam bath gave, upon concn. *in vacuo* and treatment with 2 *N* Na_2CO_3, 85% *sebacamide*, m. 208° (from EtOH); adipic acid gave 82% *adipamide*, m. 218-20° (from H_2O). Glutaric acid (1.32 g.), 1.15 g. I, and 3 ml. pyridine, treated as above, followed by extn. with C_6H_6 and concn. of the ext., gave 78% *glutarimide*, m. 152-3° (crude), m. 153-4° (from EtOH); a double amt. of I gave a moderate amt. of an *unknown substance*, m. 63-72°. Heating 3.54 g. succinic acid, 3.17 g. I, and 10 ml. pyridine 1 hr. to 120-5°, concn. *in vacuo*, and extn. with Me_2CO gave 40% *succinimide*, m. 122.5-24.0° (from C_6H_6), and 35% *succinamic acid*, m. 153-4° (from Me_2CO); a double amt. of I gave 1.1 g. *solid*, m. 80-6°, which was not identified. Similarly phthalic acid gave 88% *phthalimide;* more than an equimol. amt. of I lowers the imide yield to 80-2%, and an *unknown substance*, m. 160-72°, is isolated. Malonic acid and 0.5 mole I in pyridine in 3 hrs. on a steam bath gave 88% *Ac-NH_2*; $PhCH_2CH(CO_2H)_2$ with an equimolar amt. of I in pyridine gave *hydrocinnamamide*, m. 102-3°. $(CO_2H)_2$ failed to yield an identifiable amide; CO_2 is evolved as in the cases cited above and the mixt. yields *unidentified substances*, m. 179-83° and m. 196-225°. G. M. K.

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Mechanism of amidation by means of sulfamide and a new method of preparation of imidosulfamide. A. V. Kirsanov and Yu. M. Zolotov (I. V. Stalin Met. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (*J. Gen. Chem.*) 20, 1637–49(1950).—In the amidation of RCO_2H by $(H_2N)_2SO_2$ in pyridine, the latter is not the only amidating agent, as the NH_4 salt of imidosulfamide (cf. *C. A.* 4, 428) that forms in the action of pyridine on sulfamide also takes part. No stable intermediates form in the reaction and $RCONH_2$ form as such in the mixt. Heating 1.22 g. BzOH with 0.96 g. $SO_2(NH_2)_2$ and 5 ml. pyridine 3 hrs. to 100°, evapn. *in vacuo* at 50–100°, extn. with abs. Et_2O, and evapn. of the ext. gave 1.1 g. product, m. 88–95°, which, treated with *N* Na_2CO_3 and extd. with Et_2O, gave 70% $BzNH_2$ and 0.22 g. BzOH. Heating 4.8 g. $SO_2(NH_2)_2$ with 10 ml. dry pyridine gave in 3 hrs. a viscous, insol. oil, which, extd. with Me_2CO, gave 0.37% $(H_2N)_2SO_2$ and 4.4 g. (91.7%) sirup, having the same compn. as sulfamide, sol. in H_2O, neutral, insol. in Et_2O and Me_2CO. If traces of H_2O are present, NH_3 is evolved during the reaction; in the presence of an equiv. amt. of H_2O a theoretical amt. of NH_3 is expelled. The sirup forms in 30 min. if the reaction is run at 130°. The behavior of the sirup indicates its structure is that of the NH_4 salt of imidosulfamide, $N(NH_4)(SO_2NH_2)_2$. Heating it with p-$O_2NC_6H_4CO_2H$ on a water bath readily gave p-$O_2NC_6H_4CONH_2$ in up to 98% yield after 3 hrs.' reaction with equimolar amts. Boiling the sirup with H_2O causes development of acidity and evolution of NH_3, leaving behind sulfamide. The aq. soln. of the sirup with 2 *N* $AgNO_3$ yields cryst. $NAg(SO_2NH_2)_2.2H_2O$, softening at 100°, m. 105–7°; the *vacuum-dried product* m. 108–9°; recrystn. from hot H_2O gave serrated crystals from concd. soln., rhombic from dil. soln.; the product is sol. 1:1150 in H_2O at 20°. Possibly the salt is chelated by hydrogen-bonding across the 2 terminal NH_2 groups and by Ag forming a 2nd bridge in same positions. The NH_4 salt loses 0.25 of its N very rapidly on boiling in aq. soln., followed by slower formation of H_2NSO_3Na. G. M. Kosolapoff

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Reaction of sulfamide with alkalies and a second new method of preparation of imidosulfamide. A. V Kirsanov and Yu. M. Zolotov (I. V. Stalin Met. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (*J. Gen. Chem.*) 20, 1650-7(1950).—The probable reaction course of the sulfamide-pyridine system is discussed in detail; most likely the reaction is initiated by coordinative addn. of the N of pyridine and S of sulfamide followed by elimination of NH_3 and formation of a product analogous to the pyridine-SO_3 complex, but having an imide group instead of 1 of the O atoms of SO_3. The product may be expected to be a sulfonating agent capable of converting sulfamide into imidosulfamide and its NH_4 salt. Similarly, sulfamide in aq. medium in the alk. range may be expected to form an ion, $(H_2N)_2S(OH)(O)O^-$, which by loss of an NH_4 ion, followed by loss of OH ion, would form an ion of sulfimide, O_2SN, which, reacting with sulfamide, should yield imidosulfamide. This hypothesis was verified exptly. Boiling sulfamide in *N* NaOH caused elimination of NH_3 to the extent of 86% called for by formation of $NNa(SO_2NH_2)_2$, which can be isolated as the poorly sol. Ag salt. Addn. of 0.05 mole sulfamide to hot 0.5 *N* NaOH (49 ml.), boiling 10 min., cooling, and addn. of 12.5 ml. 2 *N* $AgNO_3$ gives 89.9% of the *Ag salt*, collected after 3 hrs.; crystn. from H_2O gives 83% pure product; if boiling is continued 40 min., only 44.7% yield is obtained. If the boiled (10 min.) soln. is concd. *in vacuo*, cooled, and warmed with 96% EtOH, the Na salt is obtained in 90% yield; crystn. from H_2O gives the sesquihydrate, which loses H_2O at 100°, and melts over a wide range (65-85°); the anhyd. salt m. 162-4°. G. M. K.

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Free imidosulfamide and its properties. A. V. Kirsanov and Yu. M. Zolotov (I. V. Stalin Metallurg. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 1790-1801(1950).—The free $NH_2SO_2NHSO_2NH_2$ (I) was obtained in 80% yield by decompn. of the Na salt in aq. soln. with 0.9 equiv. of H_2SO_4 at 0° and subsequent evapn. at 0°. Yields up to 90% with respect to the recrystd. Na salt were obtained with the use of a mixt. of 90% of the theoretical amt. of H_2SO_4 with a slight excess of NH_2SO_3H. The crude I m. 154-70°; once-recrystd. material m. 163-4°, twice-recrystd. m. 167-9°; no further change in m.p. occurred on subsequent recrystns. The product is readily sol., with evolution of heat, in H_2O and Me_2CO, sol. in EtOH and in EtOAc, practically insol. in C_6H_6. The contents of I in 100 g. of satd. soln. at 20° are: in H_2O, 29.1-30.4 g., in Me_2CO 33.6-35.9 g., in EtOH 8.9-9.7 g., in EtOAc 4.79 g., in Et_2O 0.526 g., in Me_2CO:C_6H_6 1:3, about 0.64 g. The best method of recrystn. is soln. in dry EtOAc at room temp., filtration, and evapn. to dryness *in vacuo* at room temp., soln. of the residue in a small amt. of abs. Me_2CO and diln. with 3 vols. of C_6H_6. The preliminary treatment with EtOAc is necessary for the removal of the NH_2SO_3H, which is insol. in EtOAc but is sol. in Me_2CO to the extent of 0.3 g./100 g. After a 2nd recrystn. from Me_2CO + C_6H_6, the product is snow-white needles. Slow evapn. of solns. in Me_2CO or EtOAc gives large cryst. needles, m. 1-2° lower than the fine needles pptd. from these solns. by C_6H_6. The compn. corresponds to the formula $H_5N_3S_2O_4$, but cryoscopic detn. in H_2O gives a mol. wt. roughly half that formula. Complete hydrolysis gives $I + H_2O \rightarrow NH_2SO_2NH_2 + NH_2SO_3H$, which confirms the structure formula. I is a strong acid and is accurately titrated both with phenolphthalein and with methyl orange. By colorimetry, a 0.01 *N* soln. of I has a pH of 2.25; 0.001 *N* soln., 3.03. By cryoscopy, 0.01-0.03 *N* solns. of I in H_2O are almost completely dissocd. The rate of hydrolysis of I in H_2O could be followed by analysis based on the difference of soly. between I and NH_2SO_3H (a product of the hydrolysis) in EtOAc. The 1st-order rate const. *k* of the hydrolysis in 0.25 *N* soln., at 20, 40, and 60°, was detd. to 10^4k = 0.111, 1.78, and 6.17, resp., and the half-times to 17.3 hrs., 65, and 19 min., resp. These data are accurate only to within ±5%. They definitely contradict Hantzsch's (*Ber.* 34, 3430(1901)) assertion of instantaneous hydrolysis of I in H_2O. The following salts of I were prepd.: $(NH_2SO_2)_2N.NH_4$, sol. in H_2O, sparingly sol. in cold, somewhat more in warm EtOH, transparent thin needles, nonhygroscopic, softening at 114°, m. 115-18° to a turbid liquid; the aq. soln. is neutral to Congo. The Ag salt prepd. by neutralization of I with NaOH and addn. of an equiv. amt. of $AgNO_3$ is identical with that prepd. directly from the Na salt. Likewise, the Na salt prepd. from I and NaOH is identical with the salt obtained by action of NaOH on $SO_2(NH_2)_2$. The Ba salt, $Ba(N_3H_4O_4S_2)_2$, sol. in H_2O, sparingly sol. in EtOH, decomp. at 228-33°. The Hg and the Cu salts, not further investigated, decomp. 181-3°. $C_5H_5N.H_5N_3S_2O_4$, sol. in H_2O (soln. neutral to Congo), sparingly sol. in cold EtOH and Me_2CO, softening at 130°, m. 133-4° to a turbid liquid becoming clear at 135°. The α-aminopyridine salt, $C_5H_4NNH_2.H_5N_3S_2O_4$, softening at 127-8°, m. 130-2° to a clear liquid. $PhNH_2.H_5N_3S_2O_4$, needles or prisms, m. 181-3° to a clear liquid. N. Thon

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The preparation of free imidosulfamide and its properties. A. V. Kirsanov and Yu. M. Zolotov (I. V. Stalin Inst. Met., Dnepropetrovsk). *J. Gen. Chem. U.S.S.R.* 20, 1851-63 (1950)(Engl. translation).—See *C.A.* 45, 1451b.

R. M. S.

Preparation of sulfamide. A. V. Kirsanov, Yu. M. Zolotov, and N. L. Egorova. *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 2251-4(1950).—Dry CCl_4 (3 l.) is cooled with good stirring to −8° and treated with a rapid stream of NH_3 (dry) with concurrent addn. of 500 ml. SO_2Cl_2 in 2.5 l. CCl_4 and the temp. kept below −2°. Any large lumps of product are periodically removed. A 10-15-hr. period is required for completion. After addn. of SO_2Cl_2 the NH_3 stream is continued 0.5 hr., 3 l. H_2O added, the aq. layer sepd. from the CCl_4 and added to the lumps (above), the mixt. stirred until clear soln. forms, then boiled down until its temp. reaches 108-9° (vol. 2.5 l.), let stand 12 hrs., the NH_4Cl filtered off, the filtrate concd. *in vacuo* at 60-70° (removal of 650 g. H_2O), the 2nd crop of NH_4Cl removed, and the process repeated until the residue is a sirup, which is treated with 1 l. hot H_2O contg. enough HCl to give a 0.5 *N* soln., the soln. boiled 15 min., cooled, filtered, neutralized to Congo red with 20% NH_4OH, concd. *in vacuo* (removal of 650 ml.), NH_4Cl again removed, and the filtrate concd. *in vacuo* to a sirup, which is treated hot with 1 l. Me_2CO with agitation, the residue extd. with Me_2CO and the combined ext. (or each individually) concd., giving a total of 270 g. *sulfamide*, which is purified by crystn. from hot 96% EtOH, yielding 215 g. pure product, m. 92-3°, while the mother liquor yields enough less-pure product to give a 41% over-all yield. A 2nd crystn. gives a product m. 91.5-2.0°.

G. M. Kosolapoff

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Preparation of sulfamide. A. V. Kirsanov, Yu. M. Zolotov, and N. L. Egorova. *J. Gen. Chem.* U.S.S.R. 20, 2203-13(1950)(Engl. translation).—See C.A. 45, 6047.
H. L. M.

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1951

Methylation of imidosulfamide. A. V. Kirsanov and Yu. M. Zolotov (I. V. Stalin Met. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 642-5(1951).—Best methylation conditions are as follows: Carefully dried Ag imidosulfamide (2.82 g.) is allowed to stand 24 hrs. in the dark with 2 ml. MeI, the MeI expelled *in vacuo*, the dry residue extd. 1 hr. with 100 ml. dry Et_2O, then with 3 portions (15 ml.) of fresh Et_2O, the ext. concd. *in vacuo*, and the residue extd. with 50 ml. dry Et_2O, yielding as a residue 0.4 g. imidosulfamide, while the ext. on evapn. yields the crude Me deriv., purified by boiling 0.5 hr. with 150 ml. dry C_6H_6, then again with another 150-ml. portion; on cooling, the pure *N-methylimidosulfamide*, $MeN(SO_2NH_2)_2$ (I), m. 113-14° or 114-15° (different fractions), ppts. in a total yield of 25.0%. The recovery of imidosulfamide is 35.0%. The mother liquor from I yields on evapn. an oil which appears to be crude *di-Me deriv.* Treatment of imidosulfamide with an equimolar amt. of CH_2N_2 in Et_2O results in a vigorous reaction, and evapn. of the soln. and addn. of C_6H_6 readily yields 75.7% I, m. 112-13° (from Me_2CO-C_6H_6), along with the residue probably contg. the *poly-Me deriv.* (oil). While the pure I m. 114-15°, it softens at 112°. It has bitter taste, is sol. in H_2O, MeOH, EtOH, Me_2CO, EtOAc, poorly sol. in CCl_4 or cold C_6H_6, sparingly sol. in hot C_6H_6 (best crystn. solvent); at 20° its soly. is 0.033% in C_6H_6, between 46.9 and 49.3% in Me_2CO, and 2.80% in Et_2O at 15°. G. M. Kosolapoff

ZOLOTOV, YU. M.

"The dimethylamidation of carboxylic acids." A. V. Kirsanov and Yu. M. Zolotov. (p. 1166)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1951, Vol 21, No 6.

CA

The dimethylamidation of carboxylic acids. A. V. Kirsanov and Yu. M. Zolotov. *J. Gen. Chem. U.S.S.R.* 21, 1273-6(1951)(Engl. translation).—See *C.A.* 46, 1968*h*.
B. R.

Hydrolysis of 2-methylimidosulfamide and monomethylsulfamide. A. V. Kirsanov and Yu. M. Zolotov (Dnepropetrovsk Met. Inst.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 22, 151-3(1952); cf. *C.A.* 44, 4446g.—MeN(SO_2NH_2)$_2$ (I) is 95.4% hydrolyzed in H_2O in 30 min. at 30°, and 61% in about 15 min. at 5°. I is hydrolyzed much more rapidly than is the free imidosulfamide, possibly owing to greater distance between the central N atom and the adjacent S atoms. I (1.89 g.) in 10 ml. H_2O rapidly heated to boiling, the soln. rapidly cooled and evapd. *in vacuo* at 30°, the residue extd. with abs. EtOAc, and the ext. evapd. gave 95% *MeNHSO₂NH₂*, prisms, m. 65.5-6.0° (from hot C_6H_6), sol. in the usual solvents and giving neutral solns. in H_2O; soly. in Et_2O at 20° 90%, in C_6H_6 at the b.p. 0.265%, at 25° 0.043%. G. M. Kosolapoff

238T37

USSR/Chemistry - Sulfur Organic Compounds Nov 52

"3-Ethylimidosulfamide and Ethylsulfamide," A. V. Kirsanov and Yu. M. Zolotov, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst imeni I. V. Stalin.

"Zhur Obshch Khim," Vol 22, No 11, pp 2038-2040

3-ethylimidosulfamide was obtained by the action of ethyl iodide on the silver salt of imidosulfamide and by the action of diazoethane on free imidosulfamide. Its properties were described. By the hydrolysis of 3-ethylimidosulfamide, ethylsulfamide was obtained, and its properties were also described.

238T37

ZOLOTOV, Yu. M.

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Chemical Abst.
Vol. 48 No. 9
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(3) Chem

N-Ethylimidodisulfamide and ethylsulfamide. A. V. Kirsanov and Yu. M. Zolotov. *J. Gen. Chem. U.S.S.R.* 22, 2063-4(1952)(Engl. translation).—See *C.A.* 47, 9352i.
H. L. H.

USSR/Chemistry - Reaction products

Periodical : Zhur. ob. [illegible]

Abstract : The derivat[illegible]

Submitted : June [illegible]

ZOLOTOV, IU. M.

Kirsanov, A. V., Zolotov, IU. M.- "3-Ethyl imidosulfamide and ethyl sulfamide."
(p. 2038)

SO: Journal of General Chemistry, (Zhurnal Obshchei Khimii), 1952, Vol. 22, No. 11

ZOLOTOV Yu. M.

79-2-14/64

AUTHORS: Kirsanov, A. V., Zolotov, Yu. M.

TITLE: Substituted Imido- and Monoarylsulfamides (Zameshchennyye imido- i monoarilsulfamidy)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 2, pp. 343 - 347 (USSR)

ABSTRACT: Imidosulfamide formerly was a practically unattainable substance (reference 1) and the substituted imidosulfamides were unknown. At present the imidosulfamide is absolutely attainable (reference 2) and 3-methyl- and 3-ethylimidosulfamides (reference 3) were produced from it. 1- and 1,5-substituted imidosulfamides were hitherto unknown. According to its chemical properties the free imidosulfamide is a monobasic acid which in strength is almost not inferior to sulfuric or hydrochloric acid. The method (reference 2) proved to be suitable for the production of 1,5- disubstituted and 1,1,5,5-tetrasubstituted imidosulfamides. Thus this method is a general method for the production of as well nonsubstituted as of 1,5-di- and 1,1,5,5-tetrasubstituted imidosulfamides according to the scheme $2R_2NSO_2NH_2+OH^- \longrightarrow NH_3+H_2O + (R_2NSO_2NSO_2NR_2)^-$, where R may be hydrogen, alkyls or aryls. In the case of 1,5-diphenylimidosulfamide the initially forming unstable phenylsulfaminic acid (reference 4) is at once hydrolized and forms an acid aniline-sulfate.

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79-2-14/64

Substituted Imido- and Monoarylsulfamides

The scheme suggested for the formation of the imidosulfamide by means of an alkali liquor (reference 2) is designated as false, which is proved by the formation of (I) and (II) from N,N-dimethylsulfamide and N,N'-diphenylsulfamide. The production of imidosulfamide from sulfamide and tertiary bases may take place according to the scheme $NH_2SO_2NH_2 + Py \rightleftarrows PyH^+ + NH_2SO_2NH^-$ etc. When all schemes determined by the authors should be correct, it must be possible to obtain substituted imidosulfamides from all substituted sulfamides with the exception of tetrasubstituted ones. This conclusion is at present examined. For the synthesis of other 1,5-disubstituted imidosulfamides it was necessary to obtain monosubstituted sulfamides of the type $RNHSO_2NH_2$. These compounds were obtained by Denivell, Battegay and Meybek (reference 5) in quite a complicated manner. Paken (reference 7) produced butyl-, cyclohexyl- and piperidylsulfamides in a simpler manner by the influence of sulfamide upon the corresponding amines. Paken does not say anything on the production of N-monoarylsulfamides according to this scheme. Therefore the authors decided to determine the possibility of the production of N-arylsulfamides by the influence of aromatic amines upon sulfamides. It became evident that the aromatic amines on heating easily react with sulfamide. The yield of monoarylsulfamides

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