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Amidation of dibasic carboxylic acids. A. V. Kirsanov and Yu. M. Zidotov (Stalin Met. Inst., Dnepropetrovsk). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 20, 1145–50(1950); cf. *C.A.* 44, 4446g, 6384i.—Heating 0.005 mole urbanic acid, 0.015 mole $SO_2(NH_2)_2$ (I), and 5 ml. dry pyridine 3 hrs. on a steam bath gave, upon concn. *in vacuo* and treatment with 2 *N* Na_2CO_3, 88% *sebacamide*, m. 230° (from EtOH); adipic acid gave 82% *adipamide*, m. 218–21° (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. 153–3° (crude), m. 153–4° (from EtOH); a double amt. of I gave a mol ratio 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. 169–72°, is isolated. Malonic acid and 0.5 mole I in pyridine in 3 hrs. on a steam bath gave 88% *Acetamide*; $PhCH_2CH(CO_2H)_2$ 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. 196–231°. G. M. K.

CA

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The amidation of dibasic carboxylic acids. A. V. Kirsanov and Yu. M. Zolotov. *J. Gen. Chem. U.S.S.R.* 20, 1101-6(1950)(Engl. translation). See *C.A.* 45, 1517*e*. R. M. S

CA

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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, 426) 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 80–100°, 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 9.37% $(H_2N)_2SO_2$ 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 191°, m. 195–7°; the *vacuum-dried product* m. 198–9°; recrystn. from hot H_2O gave serrated crystals from concd. soln., rhombic from dil. soln.; the product is sol. 1:1151 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, 1680-7(1950).—The probable reaction course of the sulfamide-pyridine system is discussed in detail; most likely the reaction is initiated by coördinative 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, $(H_2N)S(OH)(O)O^-$, 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.; crysts. 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; crysts. from H_2O gives the sesquihydrate, which loses H_2O at 100°, and melts over a wide range (65–85°); the anhyd. salt m. 163–4°. G. M. K.

CA

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. 162–4°, 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 8.0–8.7 g., in EtOAc 4.79 g., in Et_2O 0.520 g., in Me_2CO:C_6H_6 1:3, about 0.04 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 dilu. 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 $H_4N_4S_2O_5$, but cryoscopic detn. in H_2O gives a mol. wt. roughly half that formula. Complete hydrolysis gives $I + H_2O \rightarrow NH_2SO_3NH_2 + NH_2SO_3H$, 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; a 0.001 *N* soln., 3.05. 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 50°, was detd. to $10^4K = 0.111$, 1.78, and 6.17, resp., and th. 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, 3439 (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, non-hygroscopic, softening at 114°, m. 115–18° to a turbid liquid; the aq. soln. is neutral to Congo. The Ag salt prepd. by neutralisation 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, $Ba(N_2H_3O_4S_2)_2$, sol. in H_2O, sparingly sol. in EtOH, decomp. at 228–33°. The Hg and the Cu salts, not further investigated, decomp. 181–3°. $C_5H_5N.H_2N_2S_2O_5$, sol. in H_2O (soln. neutral to Congo), sparingly sol. in cold EtOH and Me_2CO, softening at 130°, m. 132–4° to a turbid liquid becoming clear at 135°. The α-aminopyridine salt, $C_5H_4NNH_2.H_2N_2S_2O_5$, softening at 127–8°, m. 130–2° to a clear liquid. $PhNH_2.H_2N_2S_2O_5$, needles or prisms, m. 121–2° to a clear liquid. N. Thon

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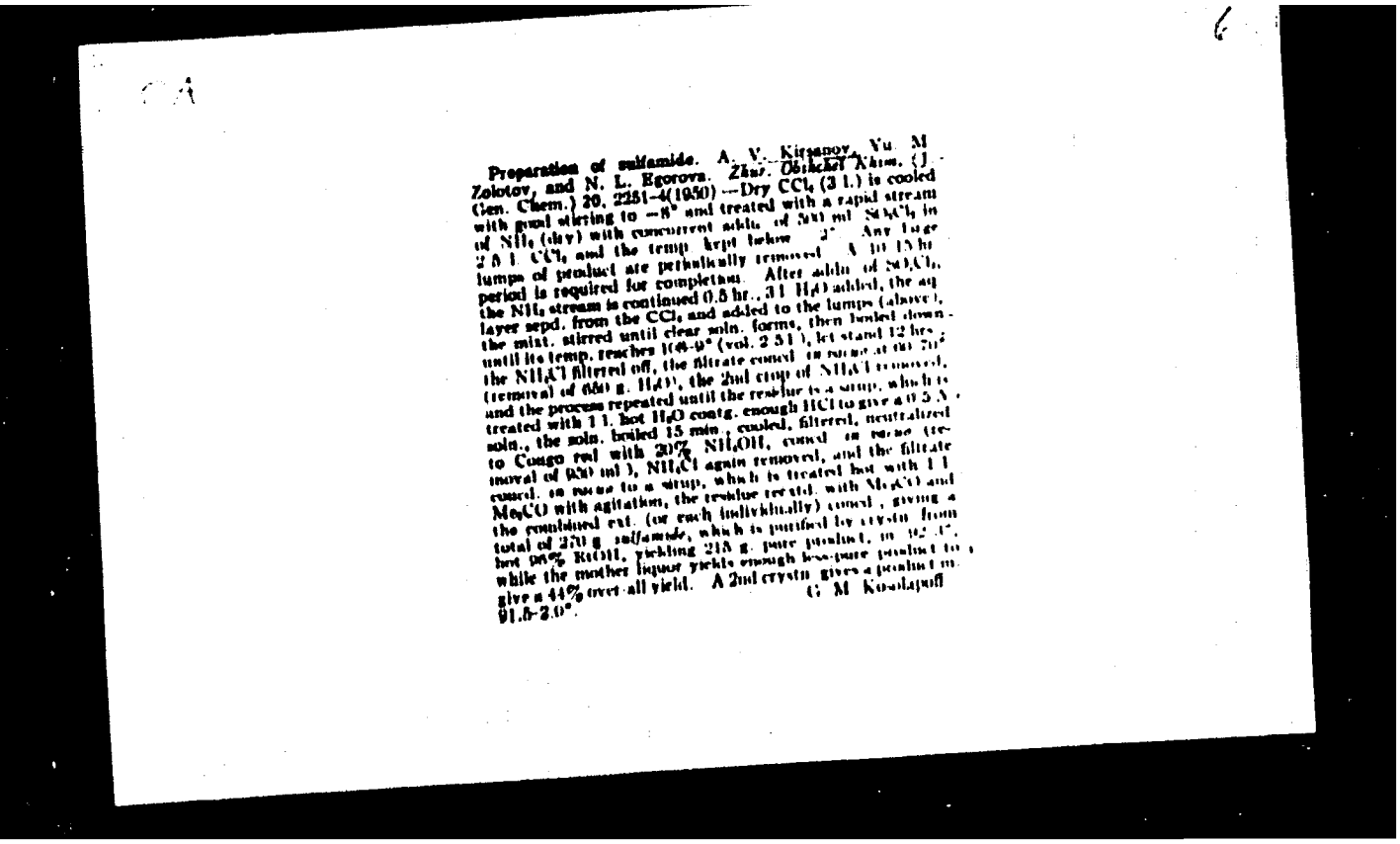
6

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, 1851-61 (1950)(Engl. translation).—See *C.A.* 45, 1451*b*.

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 300 ml SO_2Cl_2 in 2.5 l. CCl_4, and the temp. kept below 3°. 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 104–9° (vol. 2.5 l.), let stand 12 hrs.; the NH_4Cl filtered off, the filtrate concd. in vacuo at 60–70° (removal of 600 g. H_2O), the 2nd crop of NH_4Cl removed, and the process repeated until the residue is a syrup, 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 950 ml.), NH_4Cl again removed, and the filtrate concd. in vacuo to a syrup, which is treated hot with 1 l. Me_2CO with agitation, the residue treated 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. 92.5–3°, while the mother liquor yields enough less-pure product to give a 44% over-all yield. A 2nd crystn. gives a product m. 91.5–2.5°.

G. M. Kosolapoff

KIRSANOV, A. V.

USSR/Chemistry - Organic Chemistry Apr 51

"Methylation of Imidosulfamide," A. V. Kirsanov, Yu. M. Zolotov, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst

"Zhur Obshch Khim" Vol XXI, No 4, pp 642-645

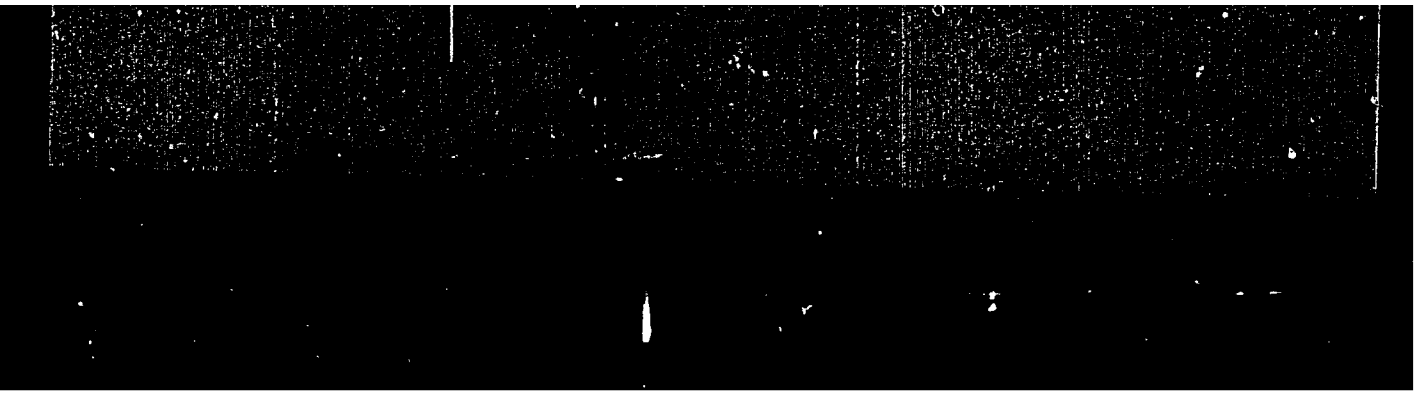
Prepd 3-monomethylimidosulfamide. Describes its properties. Notes that methylimidosulfamide reacts with NH_3 in either soln to yield binary compd with curious properties, to be described in future report.

182T16

KIRSANOV, A. V.

"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.



APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000722720004-7"

KIRSANOV, A. V.

Card 1 of 2

USSR/Chemistry - Phosphorus-Sulfur- Nitrogen Compounds Jul/Aug 52

"New Data on the Chemistry of Sulfuric Acid Amides and Trichlorophosphazosulfonaryls. I. Mechanism of the Amidation of Carboxylic Acids With Sulfuric Acid Amides," A. V. Kirsanov, Dnepropetrovsk Metallurgical Inst imeni Stalin

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 4, pp 710-720

In the reaction of phenylsulfamic on carboxylic acids in pyridine, anilides of carboxylic acids are formed. The action of amines on

(1) 229T19

(CA 47 no. 19: 9904 '53)

KIRSANOV, A. V.

Card 2 of 2

acylsulfamic acids results in acylamines. Sulfa- and p-aminobenzoic acid form polypeptides of p-aminobenzoic acid. A new method of prepg trihalo-arylsulfonamidophosphoric acids, salts of diamides of arylsulfonimidophosphoric acids, chlorides of di-anilidoarylsulfonimidophosphoric acids, dianilides

(2) 229T19

of arylsulfonamidophosphoric acids, and trianilides of arylsulfonimidophosphoric acids. Investigated the reactions of trichlorophosphazosulfonaryls with alcs and phenols. Obtained triesters of arylsulfonimidophosphoric acids and diesters of arylsulfonamidophosphoric acids.

(3) 229T19

KIRSANOV, A. V.

USSR/Chemistry - Sulfamic Acid Derivatives Jan 52
Phosphorus Compounds

"Sulfamuric Chloride," A. V. Kirsanov, Chair of Inorg Chem, Dnepropetrovsk Order of Labor Red Banner Metallurgical Inst imeni I. V. Stalin

"Zhur Obshch Khim" Vol XXII, No 1, pp 81-88

Demonstrated that thermal fission of trichlorophosphazosulfuric acid ($ClSO_2N{=}PCl_3$) yields phosphorusoxychloride and products of conversion of sulfamic acid chloride (NH_2SO_2Cl). From latter products isolated 2 trimers of $NHSO_2Cl$ and showed them to be probably the 2 theoretically possible 3-dimensional isomers of sulfamuric chloride, or 1,3,5-trichloroxy-1,3,5-trithiatriazine. Describes their properties.

207T16

USSR/Chemistry - Sulfamic Acid Derivatives Jan 52
(Contd)

207T16

CA

6

The chloride of trichlorophosphamonosulfuric acid. A. V. Kirsanov (Peterspetrovsk Met. Inst.). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 22, 898-902 (1952).—Heating a mixt. of pure sulfamic acid (9.71 g.) and 41.65 g. PCl_5 at about 110° yields HCl and $POCl_3$ in the distillate (in vacuo), while the residue, after filtration (3.3% sulfamic acid recovered), represents crude $ClSO_2NHPCl_4$, obtained in 91% yield. Only a trace (about 0.1%) of free Cl is liberated in the reaction. For best prepn. 184.3 g. dry sulfamic acid, 834 g. PCl_5, and 100 ml. CCl_4 are heated 2.5 hrs. and the cooled soln. is filtered; after removal of the volatile matter under reduced pressure from a water bath, the residue crystallizes, and after 24 hrs. the product, m. 33°, is obtained in 64% yield by careful filtration; some 11% can be recovered from the chilled filtrate. The product is purified by crystn. from petr. ether in a desiccator; pure $ClSO_2NHPCl_4$ m. 35.6°. It is best kept in the fused state, since individual crystals are rapidly attacked by moist air.

G. M. Kosolapoff

KIRSANOV, A. V.

USSR/Chemistry - Sulfamides Jan 52

"Hydrolysis of 2-Methylimidosulfamide and Monomethylsulfamide," A. V. Kirsanov, Yu. M. Zolotov, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst imeni I. V. Stalin

"Zhur Obshch Khim" Vol XXII, No 1. pp 151-153

Found that hydrolysis of 2-methylimidosulfamide (I) goes much more rapidly than hydrolysis of free imidosulfamide. Proposed that greater rate of hydrolysis of I is due to large distance between central N atom and neighboring S atoms and that stability of sulfamide ion in aq solns is due to shortening of this distance. Prepd and describes monomethylsulfamide.

207T28

KIRSANOV, A. V.

USSR/Chemistry - Organophosphorus Compounds Feb 52

"Trichlorophosphazosulfonoaryls," A. V. Kirsanov, Chair of Org Chem, Dnepropetrovsk Order of Labor Red Banner Metallurgical Inst imeni I. V. Stalin

"Zhur Obshch Khim" Vol XXII, No 2, pp 269-273

Finds that reaction of PCl_5 with arylsulfamides goes practically to completion to form trichlorophosphazosulfonoaryls ($ArSO_2N{=}PCl_3$), contrary to previous published data. Describes certain trichlorophosphazosulfonaryls.

209T23

USSR/Chemistry - Organophosphorus Compounds Feb 52
Carboxylic Acid Nitriles

"New Method for Conversion of Chlorides of Carboxylic Acid Into the Corresponding Nitriles," A. V. Kirsanov, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst imeni I. V. Stalin

"Zhur Obshch Khim" Vol XXII, No 2, pp 274-278

Discusses mech of conversion of trichlorophosphazosulfono-p-benzoylchloride into chloride of p-benzonitrilosulfonic acid. Proposes on basis of conclusions and verifies by expt new method for

20724

USSR/Chemistry - Organophosphorus Compounds (Contd) Feb 52

conversion of chlorides of carboxylic acids into their nitriles by action of trichlorophosphazosulfonoaryls ($ArSO_2N{=}PCl_3$), which can be prepd cheaply (and conveniently) by method developed previously by author.

20724

KIRSANOV, A. V.

KIRSANOV, A. V.

Chemical Abstracts
Vol. 48 No. 5
Mar. 10, 1954

Bis(trichlorophosphazo) sulfone. A. V. Kirsanov. *J. Gen. Chem. U.S.S.R.* 21, 1389-91(1951)(Engl. translation).—See *C.A.* 47, 5830e. H. L. H.

KIRSANOV, A. V.

232T26

USSR/Chemistry - Amides

Sep 52

"Trianilide of Trimesic Acid," A. V. Kirsanov, M. L. Yegorova, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst imeni I. V. Stalin

"Zhur Obshch Khim" Vol 22, No 9, pp 1614, 1615

The trianilide of trimesic acid was obtained by direct phenylamidation of trimesic acid. This product was found to melt at 320-321° and not at 118-120° as previously reported by Curtins.

232T26

Chemical Abst.
Vol. 48 No. 8
Apr. 25, 1954
Organic Chemistry

Phosphamidation of carboxylic acids. A. V. Kirsanov and N. L. Egorova. *J. Gen. Chem.* (U.S.S.R.) 22, 1063 (1952)(Engl. translation).—See *C.A.* 47, 8041h.

H. L. H.

11-11-54

KIRSANOV A. V.

238T37

USSR/Chemistry - Sulfur Organic Compounds Nov 52

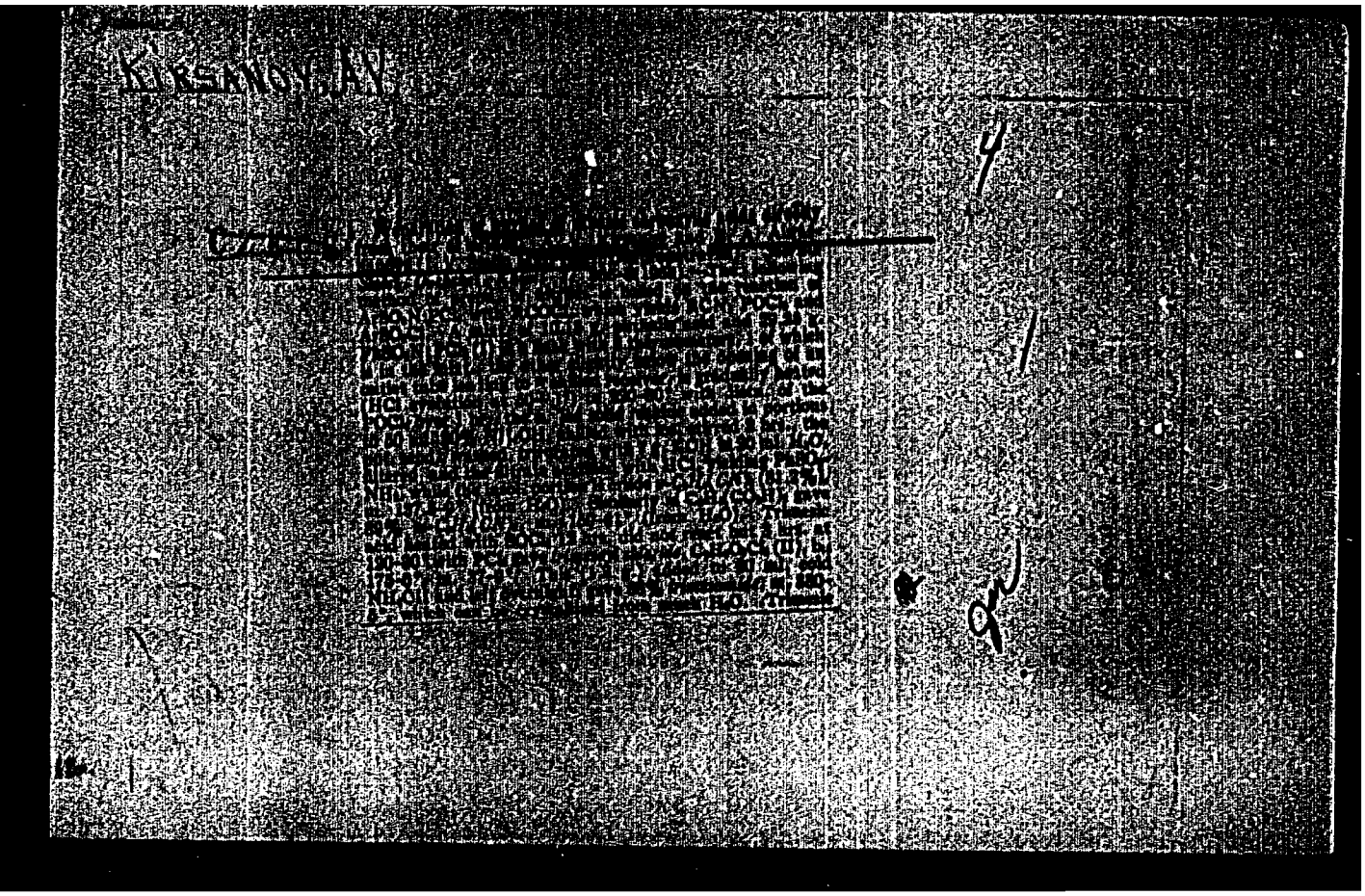
"3-Ethylimidosulfamide and Ethylsulfamide," A. V. Kirsanov and Yu. M. Zolotov, Chair of Org Chem, Dnepropetrovsk Metallurgical Inst imeni I. V. Stalin.

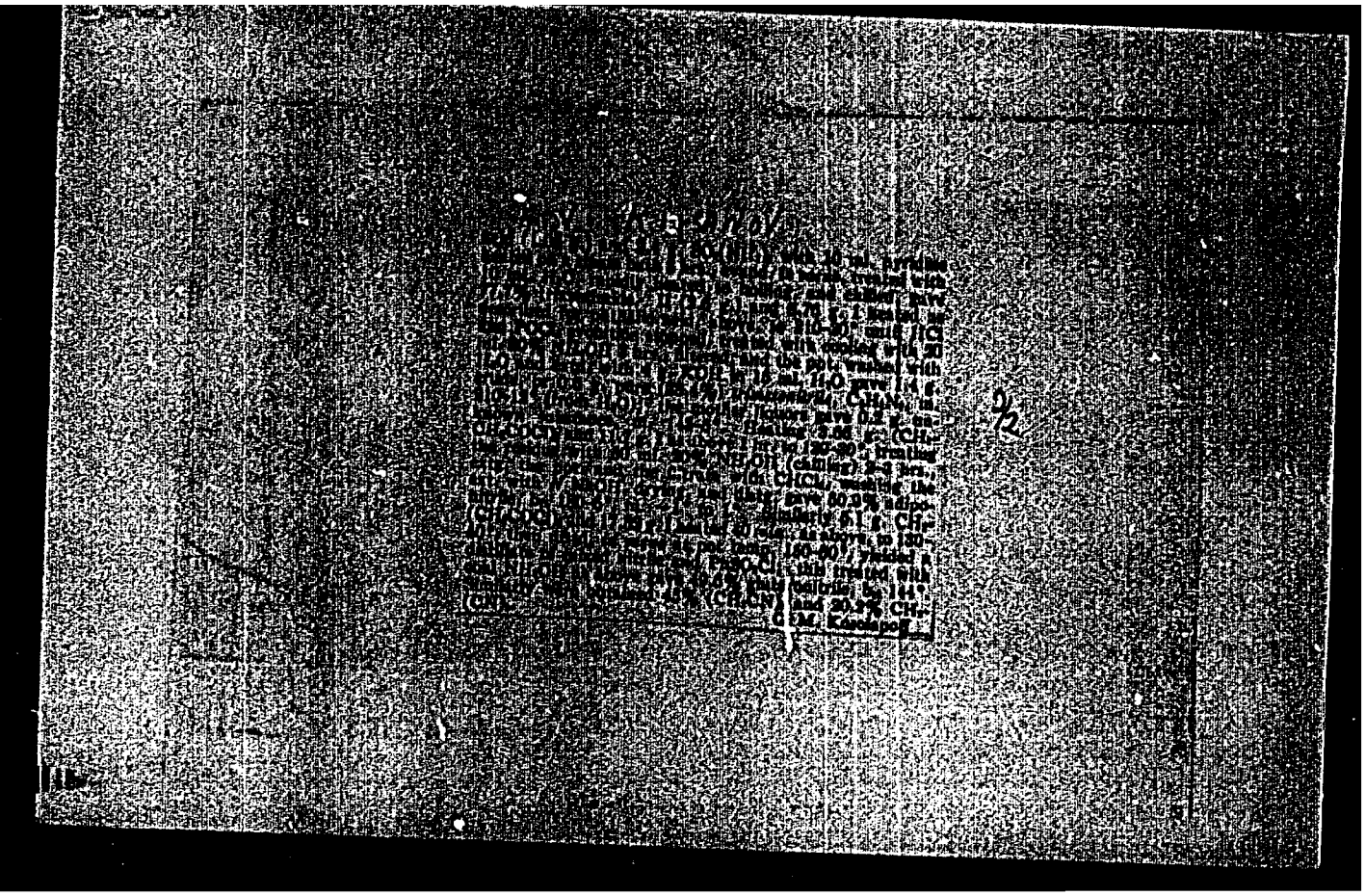
"Zhur Obshch Khim," Vol 22, No 11, pp 2038-2040

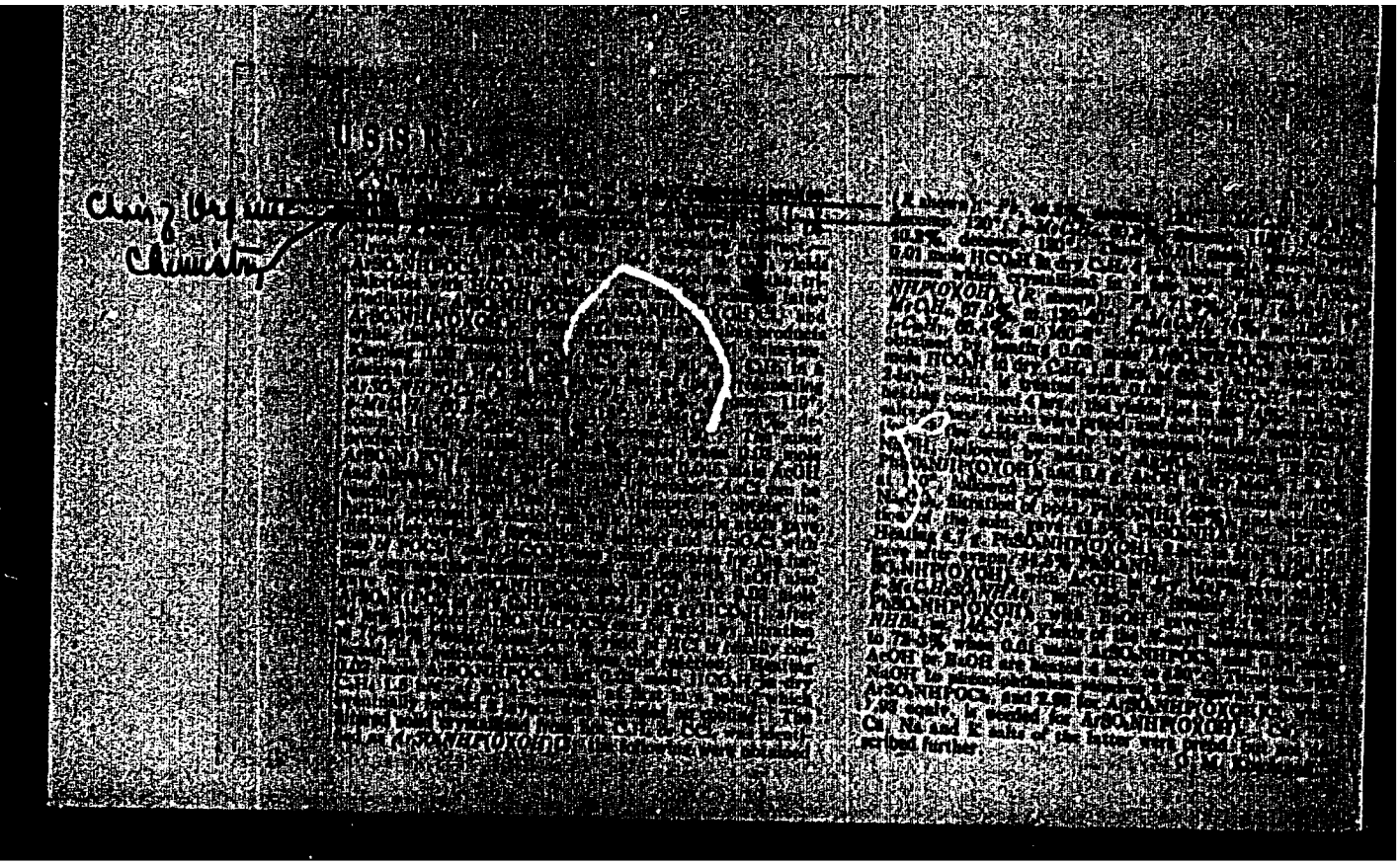
3-ethylimidosulfamide was obtained by the action of ethyl iodide on the silver salt of imidosulfamide and by the action of diazoethane on free imidosulfamide. Its properties were described. By the hydrolysis of 3-ethylimidosulfamide, ethylsulfamide was obtained, and its properties were also described.

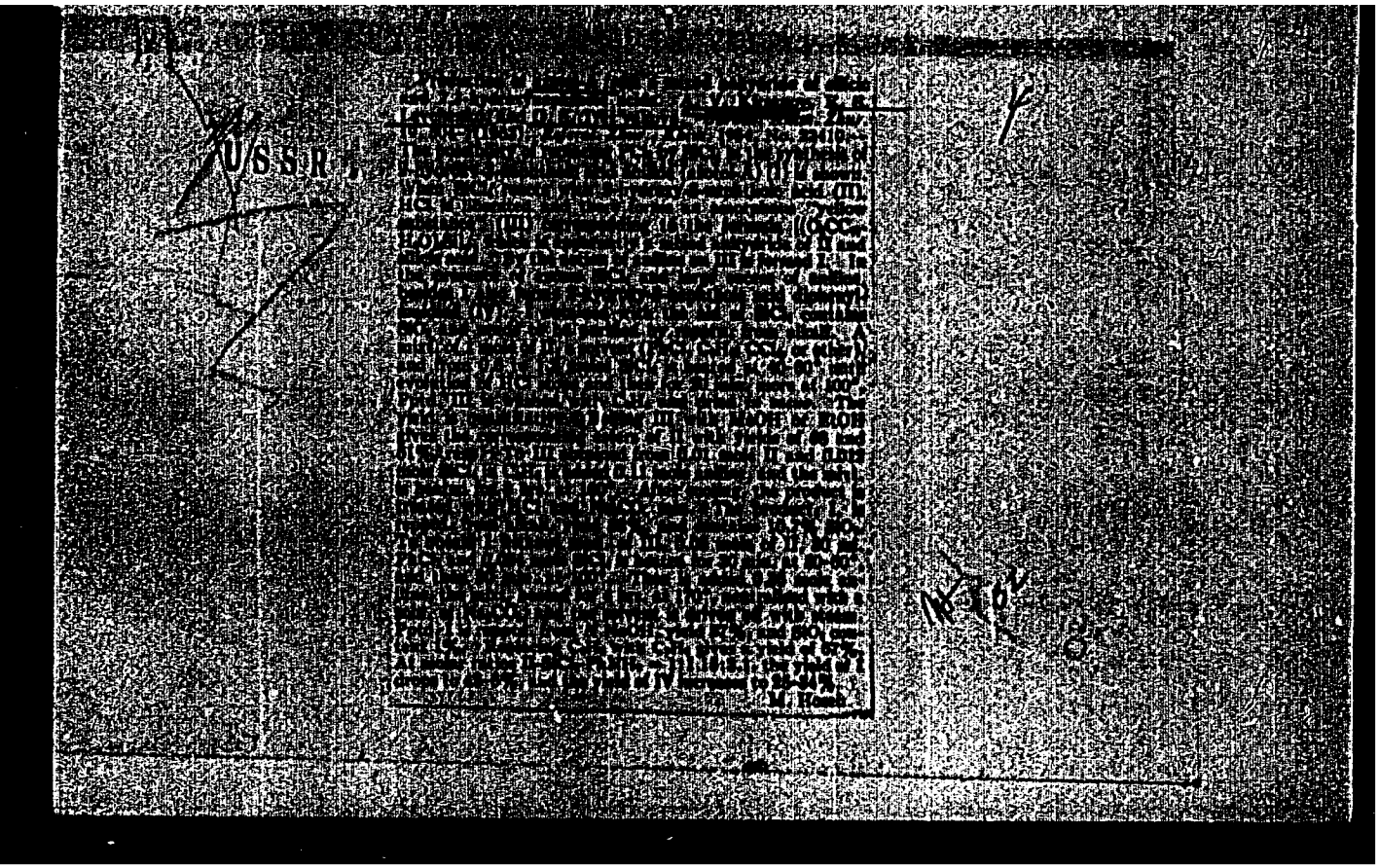
238T37

CA 47 no.18: 9252 '53)









KIRSANOV, A.V.; LEVCHENKO, Ye.S.; TRET'YAKOVA, G.S.

Diphenylamidination of carboxylic acids. Ukr.khim.zhur. 19 no.6:622-630 '53. (MLRA 8:5)

1. Institut organicheskoy khimii Akademii nauk USSR
(Amidines) (Acids, Fatty)

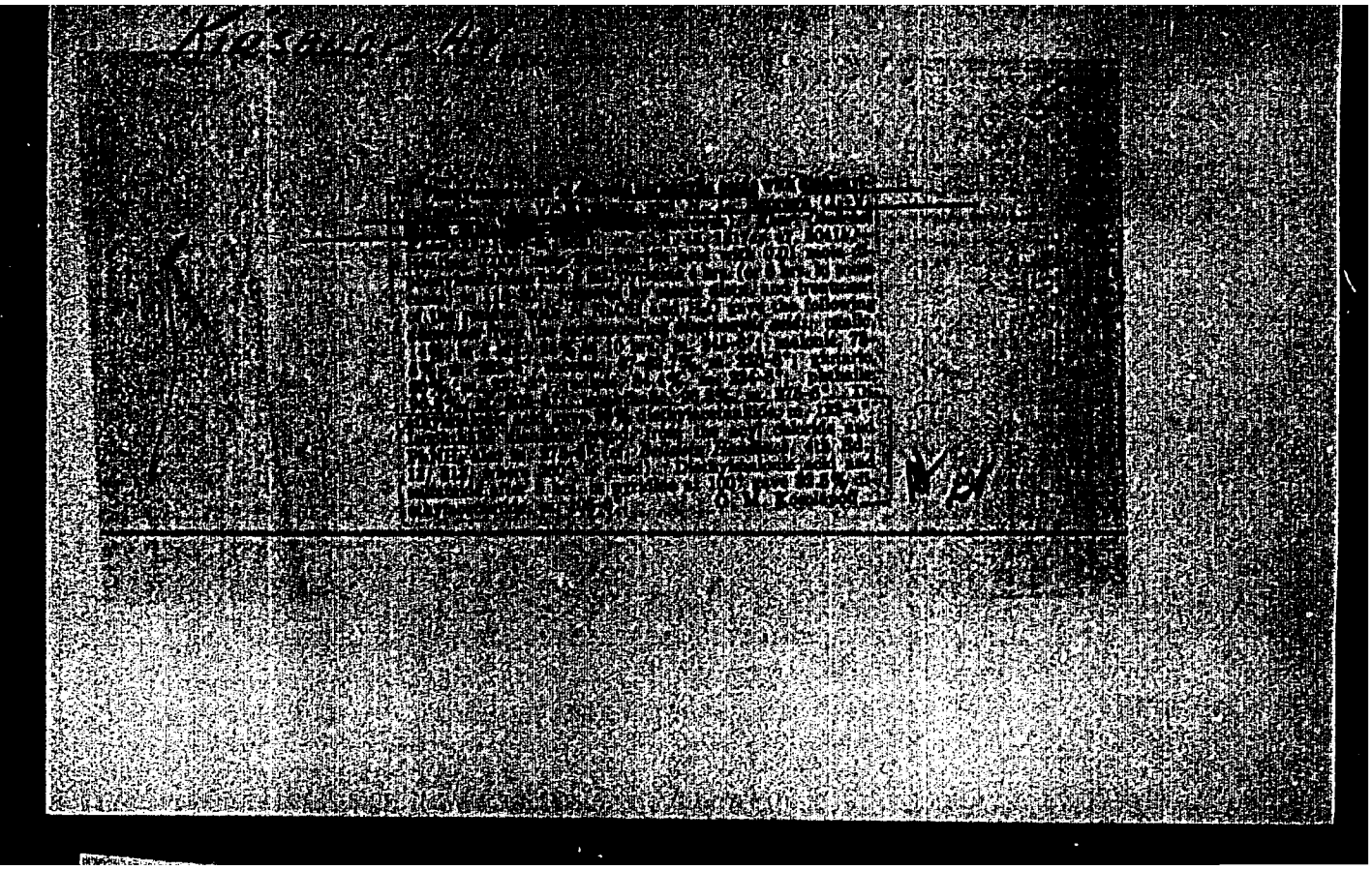
KIRSANOV, A.V.

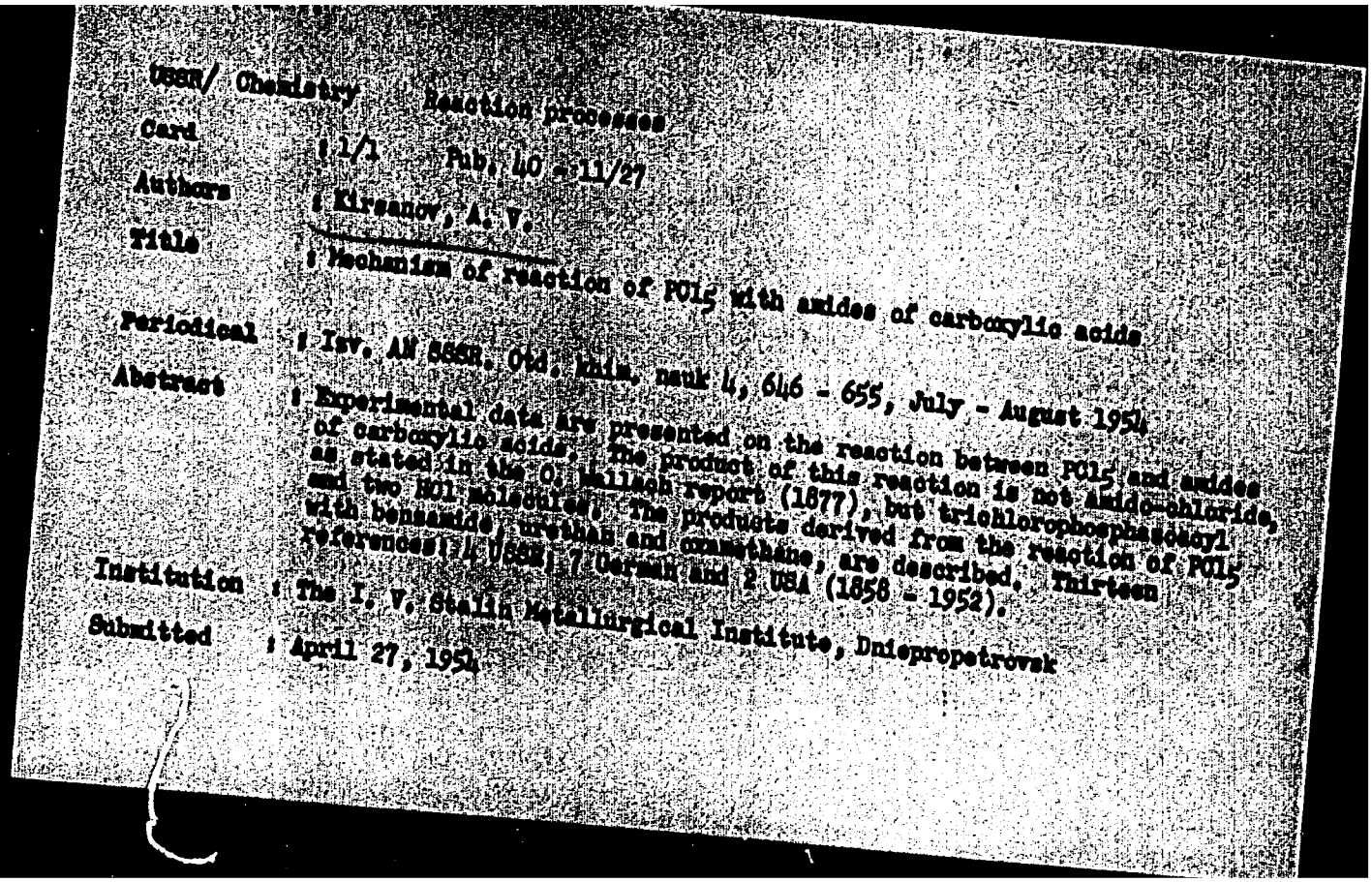
Chem

Chemical Abst.
Vol 48 No 5
Mar 10, 1954
Organic chemistry

Mechanism of amidation of carboxylic acids by amides of sulfuric acid. A. V. Kirsanov (L. Ya. Karpov Phys. Chem. Inst., Dzerzhinsk). *Zhur. Obshchei Khim.* 23, 227-9(1953); cf. *C.A.* 47, 8116.—A mechanism is proposed for the amidation of carboxylic acids with $SO_2(NH_2)_2$. Heating 2.75 g. $Me_2NSO_2NPh_2$, 1.22 g. BzOH, and 5 ml. pyridine 4 hrs. at 115°, evapg. *in vacuo*, and treating with 15 ml. *N* NaOH and Et_2O, gave 99.3% $Me_2NSO_2NPh_2$ and 98.2% BzOH. Similar reactions with AcOH, $Et_2NSO_2NPh_2$, and $SO_2(NMe_2)_2$ gave identical results. Heating 2 g. Me_2NSO_2NHPh, 1.22 g. BzOH, and 5 ml. pyridine 3 hrs. at 115°, evap. *in vacuo*, treating with Et_2O and 2*N* HCl, and sepg. the mixed amides by extn. with petr. ether gave 0.65 g. (33%) BzNHPh, m. 161-2°, and 50.2% $BzNMe_2$, m. 39-41°. A similar reaction with *p*-$O_2NC_6H_4CO_2H$ gave 28.9% *p*-$O_2NC_6H_4CONHPh$, m. 210-11°, and 61.9% *p*-$O_2NC_6H_4CONMe_2$, m. 93-5°. Heating 18.6 g. $PhNH_2$ to 170°, adding 9.7 g. H_2NSO_2H over 15 min., and heating 5 min. gave 80.4% $PhNHSO_2NH_2$. This (1.9 g.) heated 2 hrs. at 115° with 1.67 g. *p*-$O_2NC_6H_4CO_2H$ and 5 ml. pyridine gave 30.9% *p*-$O_2NC_6H_4CONHPh$, m. 210-11°. Heating 1.67 g. *p*-$O_2NC_6H_4CO_2H$ and 1.24 g. $PhNHSO_2NHPh$ in 5 ml. pyridine 6 hrs. at 115° gave 1.78 g. *p*-$O_2NC_6H_4CONHPh$ (73.6% yield assuming amidation by $PhNHSO_2H$). Heating 0.96 g. $SO_2(NH_2)_2$, 1.86 g. $PhNH_2$, 1.67 g. *p*-$O_2NC_6H_4CO_2H$, and 5 ml. pyridine 3 hrs. at 115° gave 7.2% unreacted *p*-$O_2NC_6H_4CO_2H$, 1.2 g. *p*-$O_2NC_6H_4CONHPh$, and 0.36 g. *p*-$O_2NC_6H_4CONH_2$, m. 195-7°. Heating 0.96 g. $SO_2(NH_2)_2$, 1.86 g. $PhNH_2$, and 5 ml. pyridine 6 hrs. at 115° gave 0.35 g. $SO_2(NHPh)_2$, m. 108-11°, and some H_2NSO_2NHPh. With a large excess of $PhNH_2$ somewhat better yields are obtained but not as high as suggested by Paquin (*C.A.* 43, 2573c). Heating 1.66 g. *p*-$O_2NC_6H_4CONH_2$ and 1.86 g. $PhNH_2$ in 5 ml. pyridine 3 hrs. at 115° gave 1.61 g. *p*-$O_2NC_6H_4CONH_2$. Heating 1.59 g. betainepyridinium-sulfamic acid (Baumgarten and Marggraff, *C.A.* 25, 4525) with 1.21 g. $BzNH_2$ 1 min. to 160°, adding 1.86 g. $PhNH_2$, and heating 2 hrs. at 120° gave 70.4% (based on reacted amide) BzNHPh and 75% $BzNH_2$ was recovered.

G. M. K.





USSR/ Chemistry Reaction processes

Card : 1/1 Pub. 40 - 11/27

Authors : Kirsanov, A. V.

Title : Mechanism of reaction of PCl_5 with amides of carboxylic acids

Periodical : Izv. AN SSSR. Otd. khim. nauk 4, 646 - 655, July - August 1954

Abstract : Experimental data are presented on the reaction between PCl_5 and amides of carboxylic acids. The product of this reaction is not amide-chlorids, as stated in the O. Hilbah report (1877), but trichlorophosphodiacyl and two HCl molecules. The products derived from the reaction of PCl_5 with benzamide, urethan and crotonthane, are described. Thirteen references: 4 USSR, 7 German and 2 USA (1858 - 1952).

Institution : The I. V. Stalin Metallurgical Institute, Dniepropetrovsk

Submitted : April 27, 1954

KIRSANOV, A.V.

USSR/Chemistry - Amidation

Card 1/1 Pub. 151 - 20/36

Authors : Kirsanov, A. V., and Abrazhanova, E. A.

Title : Amidation of carboxylic acids with amides of phosphoric acid

Periodical : Zhur. ob. khim. 24/1, 120-122, Jan 1954

Abstract : The reaction of p-nitrobenzoic acid with trianilide, tri-p-toluidide and dianilide of phosphoric acid, and with trianilide, dianilide, triamide and monoamide of phenylsulfonidimido phosphoric acid, was investigated. Only trianilide, among the above mentioned phosphoric acid amides, was found to be a phenyl-amidation agent. It was also established that trianilide of phosphoric acid can be successfully applied for direct phenylamidation of carboxylic acids of the fatty and aromatic series. Two USSR references (1949-1953).

Institution : The I. V. Stalin, Order of Red Banner Metallurgical Institute, Faculty of Organic Chemistry, Dnepropetrovsk

Submitted : June 20, 1953

KIRSANOV, A. V.

USSR/Chemistry - Reaction products

Card 1/1 Pub. 151 - 21/36

Authors : Kirsanov, A. V., and Zolotov, Yu. M.

Title : Tribromophosphazosulfonaryls

Periodical : Zhur. ob. khim. 24/1, 122-124, Jan 1954

Abstract : The derivation of tribromophosphazosulfonaryls from the reaction of chloramide sodium salts of sulfo acids with phosphorus tribromide, is briefly explained. The physico-chemical properties of tribromophosphazosulfonaryls, are described. The derivation and properties of tribromophosphazosulfonephenyl, o-tolyl, p-tolyl, alpha-naphthyl and beta-naphthyl are mentioned. One USSR reference (1953).

Institution : The I. V. Stalin, Order of Red Banner Metallurgical Institute, Faculty of Organic Chemistry, Dnepropetrovsk

Submitted : June 20, 1953

Kirsanov, A. V.

USSR/Chemistry - Fatty acids

Card 1/1 : Pub. 151 - 15/37

Authors : Kirsanov, A. V.; and Shevchenko, V. I.

Title : Esters of arylsulfonimidophosphoric acids

Periodical : Zhur. ob. khim. 24/3, 474-484, Mar 1954

Abstract : The reaction between trichlorophosphazosulfone aryls and sodium alcoholates of methyl, ethyl and butyl alcohols, was investigated. The derivation of numerous complete methyl, ethyl and butyl arylsulfonimidophosphates and their properties, are described. The products of acid and alkaline hydrolysis of complete Arylsulfonimidophosphates are listed. A method is introduced for the direct synthesis of dialkyl arylsulfonamidophosphates from alcoholates of alcohols and trichlorophosphazosulfonaryls. The synthesis and properties of alpha- and beta-trichlorophosphazosulfonaphthyls are described. Three references: 2-USSR and 1-Polish (1930-1953). Tables.

Institution : The I. V. Stalin Metallurgical Institute, Dnepropetrovsk

Submitted : November 3, 1953

KIRSANOV, A. V.

USSR/Chemistry

Card 1/1

Authors : Kirsanov, A. V. and Shevchenko, V. I.

Title : Dialkyl ethers of arylsulfonamidephosphoric acids

Periodical : Zhur. Ob. Khim. 24, Ed. 5, 882 - 887, May 1954

Abstract : Described is a new method for the derivation of dialkyl ethers of arylsulfonamidephosphoric acids from chloranhydrides of these acids and sodium alcoholates. The authors obtained dimethyl, diethyl and di-n-butyl ethers of arylsulfonamide acids and sodium salts of di-n-butyl ethers and described their chemical properties. The dialkyl ethers of arylsulfonamidephosphoric acids obtained from dichloroanhydrides differ in no way from the dialkyl ethers through other methods. Two USSR references. Tables.

Institution: The I. V. Stalin Order of the Red Banner-Metallurgical Institute

Submitted : December 13, 1953

USSR/Chemistry

Card : 1/1

Authors : Kirsanov, A. V.

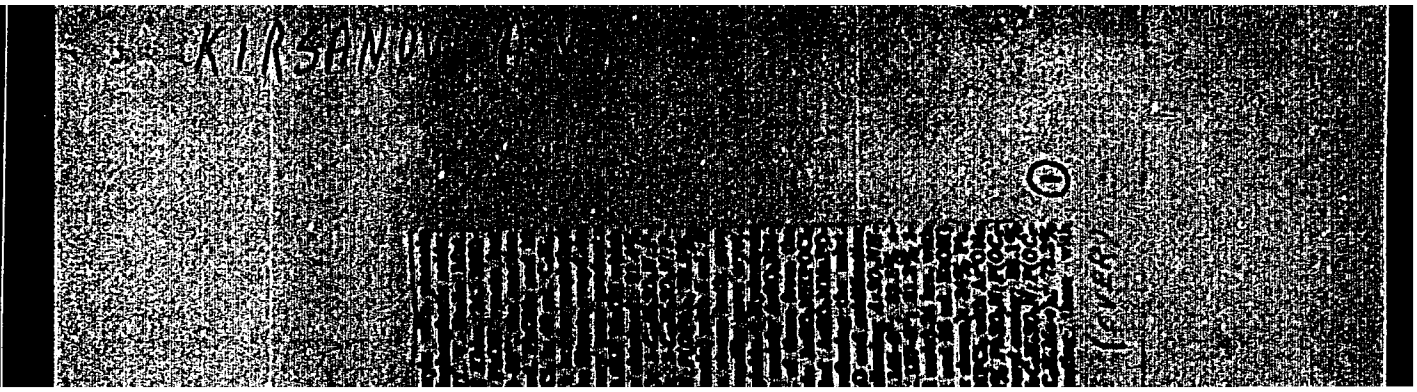
Title : Isocyanate Phosphoryl Chloride

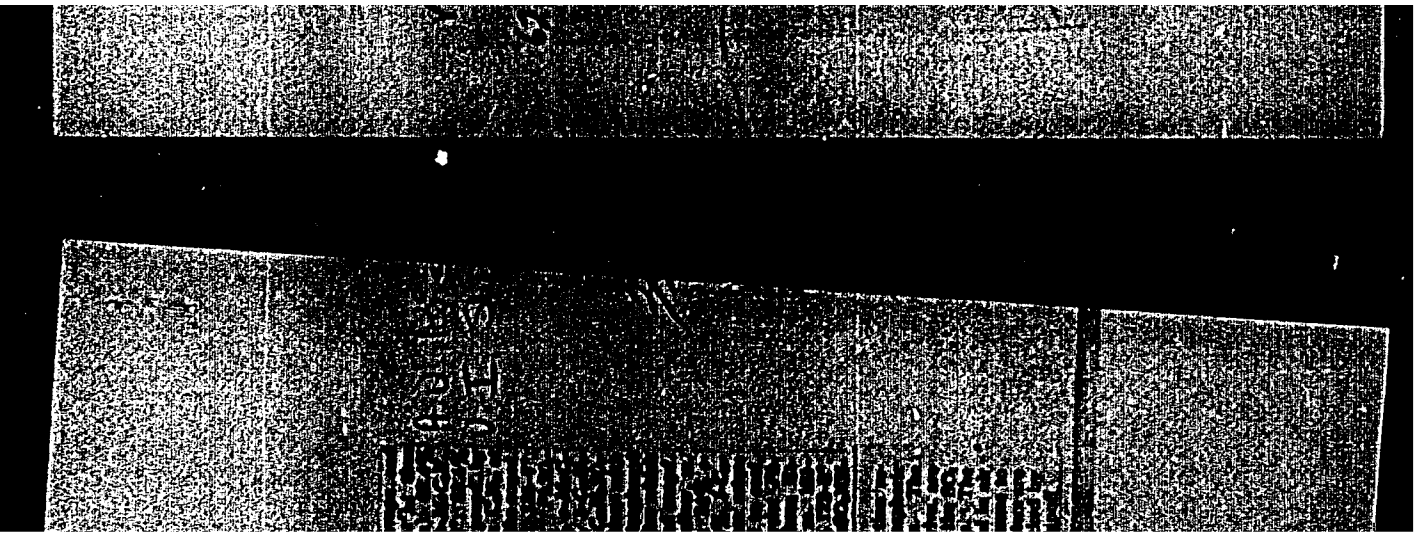
Periodical : Zhur. Ob. Khim., 24, Ed. 6, 1033 - 1038, June 1954

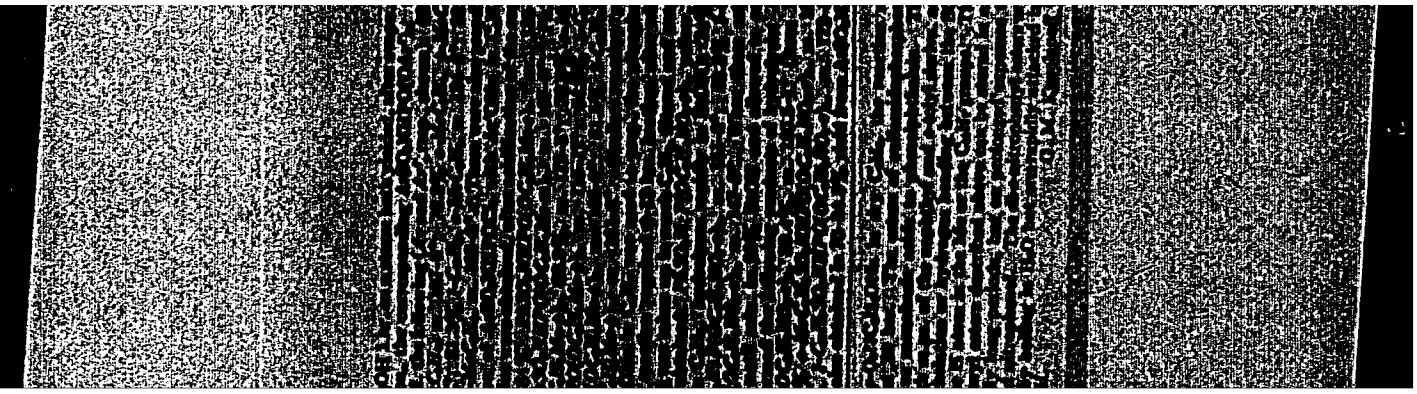
Abstract : The reaction of phosphorus pentachloride with urethan, leads to the formation of ethyl ether of trichlorophosphazocarbonic acid (ethyl trichlorophosphazocarbamate ?). Ethyl ether of trichlorophosphazocarbonic acid decomposes quantitatively at 30 - 40° into ethyl chloride and isocyanate phosphoryl chloride. The physical and chemical properties of isocyanate phosphoryl chloride and ethyl ether of trichlorophosphazocarbonic acid are described. Three references; 2 German since 1852, 1877.

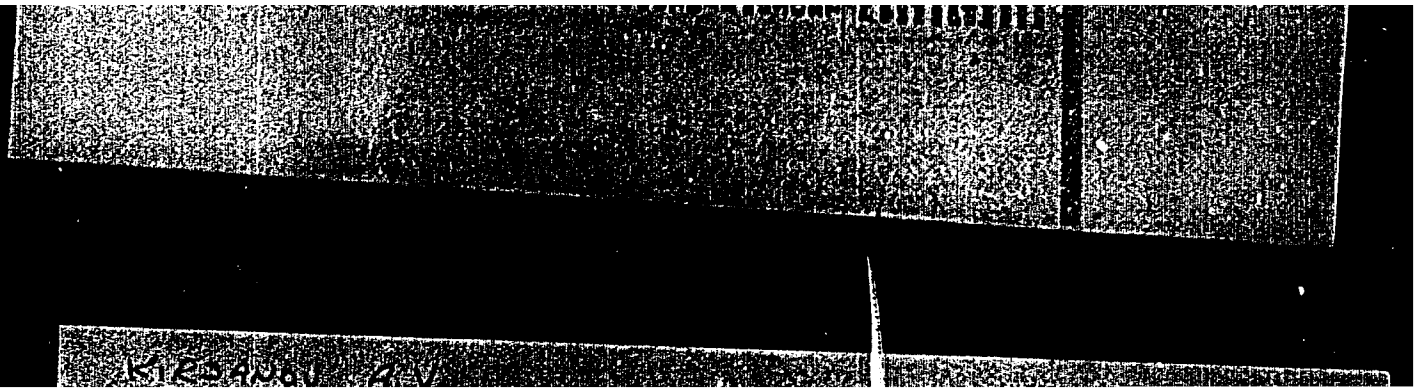
Institute : The I. V. Stalin-Order of the Red Banner Metallurgical Plant, Dniepropetrovsk

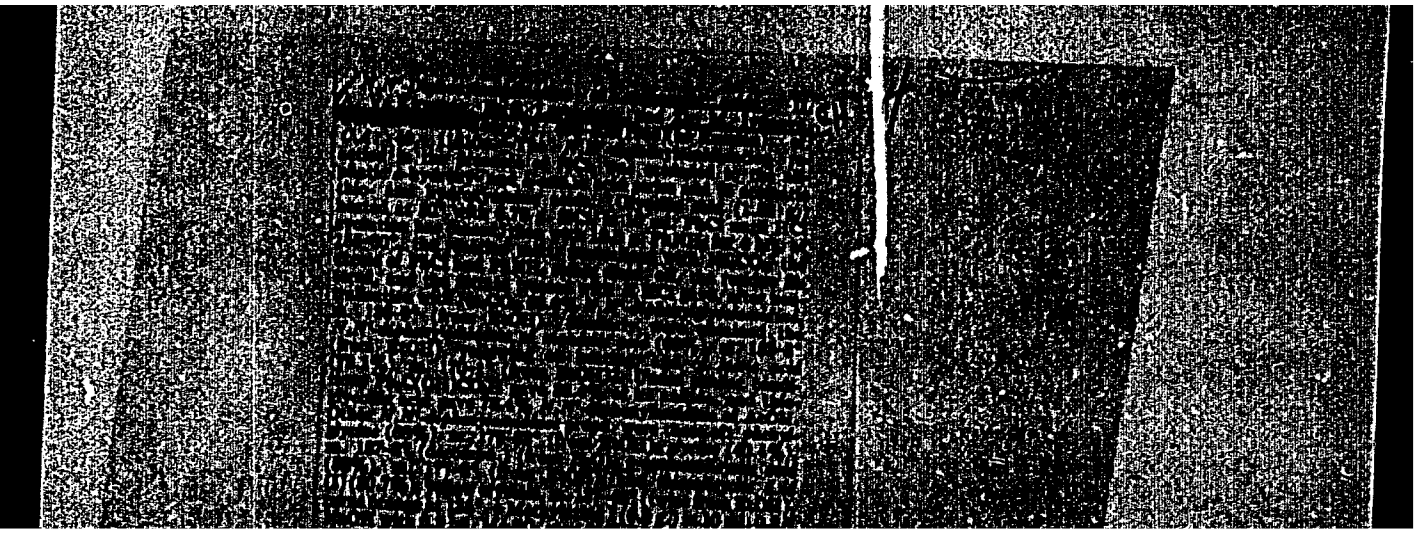
Submitted : January 3, 1954











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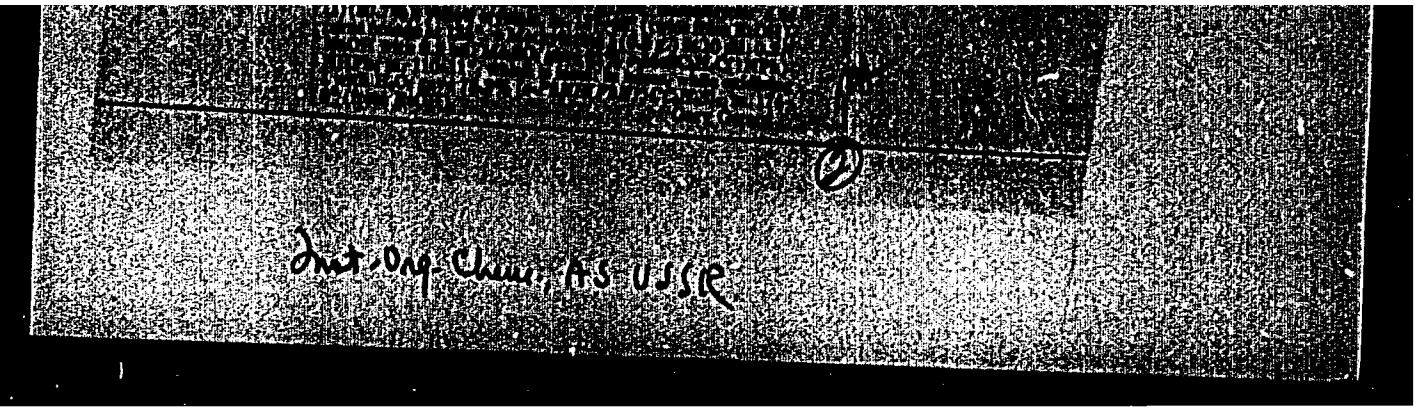

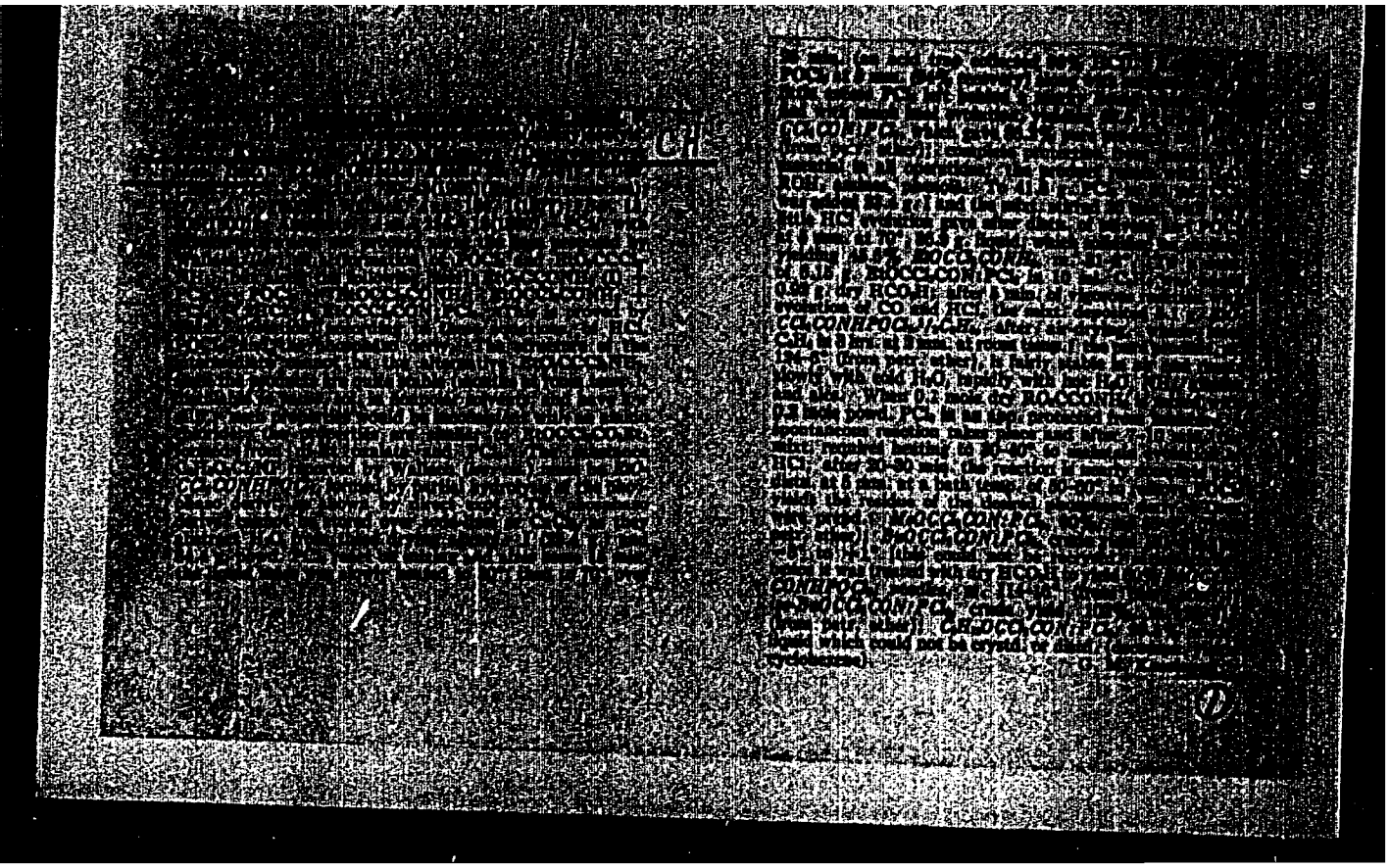

KIRSANOV, A.V.; YEGOROVA, N.L.

Dichloranhydrides of alkyl sulfonamidophosphoric acids. Zhur.ob.khim. 25 no.6:1140-1141 Je '55. (MLRA 8:12)

1. Dnepropetrovskiy metallurgicheskiy institut imeni I.V.Stalina
(Phosphoric acid) (Sulfonamides)

KIRSANOV, A. V.

"Reactions of Phosphorus Pentachloride with Acid Amides"
Paper presented at Mr First Conference on Heteroorganic Compounds, Perm., 2-10 Dec 58

Ref: B-3,324,841

KIRSANOV, A.V., kandidat tekhnicheskikh nauk.

Selecting calculated ratios to determine the coefficient of heat convection losses in cross-tube steam flow. Sudostroenie 22 no. 11:15-19 N '56. (MLRA 10:2)

(Boilers, Marine) (Heat--Transmission)

KIRSANOV, A. V.

USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61602

Author: Kirsanov, A. V., Shevchenko, V. I.

Institution: None

Title: Acid Chlorides of Aromatic Acid Esters of Arylsulfonimido-phosphoric Acids

Original Periodical: Zh. obshch. khimii, 1956, 26, No 1, 250-254

Abstract: There is described the reaction of the sodium salt of the chloramide of benzene sulfonic acid with acid chlorides and di-acid chlorides of the esters of phosphorous acid as a result of which are formed, with good yields, the following acid chlorides (or di-acid chlorides) of aromatic esters of phenyl sulfonimidophosphoric acid (I) (listing the yield in %): $C_6H_5SO_2N = PCl_2(OC_6H_5)$ (II) (oil; 92.9); $C_6H_5SO_2N = PCl(OC_6H_5)_2$ (III) (MP 66-69°; 90.1); $C_6H_5SO_2N = PCl(O\text{-}o\text{-}C_6H_4CH_3)_2$ (IV) (oil; 94.3); $C_6H_5SO_2N = PCl(O\text{-}m\text{-}C_6H_4CH_3)_2$ (V) (oil; 96.5); $C_6H_5SO_2N = PCl(OC_6H_4CH_3\text{-}p)_2$ (VI)

Card 1/2

Kirsanov, A. V.

USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 1, 1957, 960

Author: Kirsanov, A. V., and Shevchenko, V. I.

Institution: None

Title: Reaction of Trichlorophosphazosulfonearyls with Alcohols

Original Periodical: Zh. obshch. khimii, 1956, Vol 26, No 2, 504-510

Abstract: The reaction of $ArSO_2N = PCl_3$ (I) with alcohols follows a number of different mechanisms and depends on the nature of the aryl radical and on the conditions of the reaction. The first chlorine atom in I reacts faster with the alcohol than the remaining chlorine atoms; initially, an unstable molecular compound with HCl is formed which, after 60-90 minutes at 5-9°, evolves 0.8 gms-equiv HCl to form $ArSO_2N = PCl_2(OR)$ (II); the reaction rate corresponds to the first order kinetics. When the reaction is carried out without evolution of CHl, the following mechanism is observed: II ($R = CH_3$) + HCl → $CH_3Cl + ArSO_2N = PCl_2(OH)$. A large excess of alcohol leads to

Card 1/3

USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 1, 1957, 960

Abstract: CH_3ONa (prepared from 0.004 moles Na and 20 ml CH_3OH) at 3-5°, followed by heating for 30 minutes at 60° gives the following compounds IV (Ar and percent yield as indicated): α-$C_{10}H_7$, 80.5; C_6H_5, 90.2; o-$CH_3C_6H_4$, 65.6. Hydrolysis of 0.01 moles II (Ar = α-$C_{10}H_7$) with 100 ml water yields α-$C_{10}H_7SO_2NHPO(OH)(OCH_3)$; the yield is 61.4%, mp 95-99°, decomposes at 105°. The compound α-$C_{10}H_7SO_2N = PCl(OCH_3)_2$ (VI) is prepared by adding 0.01 moles II (Ar = α-$C_{10}H_7$) in 20 ml C_6H_6 to 0.01 mole II (Ar = α-$C_{10}H_7$) in 30 ml C_6H_6 and allowing the solution to stand 15 days at 20° in a dry atmosphere; the yield is 34.8%, mp 138-140° (from benzene). Hydrolysis of 0.01 moles VI in 5 ml water at 40° with 1N NaOH, followed by acidification, yields V (Ar = α-$C_{10}H_7$); yield 82%, mp 164-165°. The action of an excess of alcohol (10 ml) on 0.003 moles I (one hour at 20°) yields from 9.6 to 37.5% IV and from 21.5 to 50.3% V. C_2H_5OH under the same conditions yields from 26.4 to 34.1% IV and from 22.7 to 45.6% V. When the reaction is carried out over 24 hours at 20°, V is formed in yields of 80%.

Card 3/3

KIRSANOV, A.V.; KRASOVA, Z.D.

Triphenylphosphazosulfonaryls. Zhur.ob.khim. 26 no.3:903-904 Mr '56. (MLRA 9:8)

1. Dnepropetrovskiy ordena Trudovogo Krasnogo znameni metallurgicheskiy institut imeni I.V. Stalina.
(Sulfonaryls)

KIRSANOV, A.V.; MAKITRA, R.G.

Reaction of phosphorus pentachloride with carboxylic acid amikes. Trichlorophosphazoacyls. Zhur.ob.khim. 26 no.3:907-914 Mr '56. (MLRA 9:8)

1. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR.
(Phosphorus pentachloride) (Trichlorophosphazoacyl)

KIRSANOV, A.V.; DMRKACH, G.I.

Trichlorophosphamotrichloroacetyl and *N*-phosphoryl chloride of trichloroiminoacetyl. Zhur.ob.khim. 26 no.7:2009-2014 Jl '56. (MIRA 9:10)

1. Dnepropetrovskiy metallurgicheskiy institut.
(Acetyl chloride) (Phosphorus compounds)

KIRSANOV, A.V.

Nomenclature of compounds obtained from the action of phosphorus pentachloride on amides of carboxylic acids. Zhur.ob.khim. 26 no.7:2082 Jl '56. (MIRA 9:10)

1. Dnepropetrovskiy metallurgicheskiy institut.
(Chlorides) (Amides)

KIRSANOV, A. V.

KIRSANOV, A.V.; LEVCHENKO, Ye.S.

Chlorides and esters of arylcarbamidophosphoric acids. Zhur. ob. khim. 26 no.8:2285-2289 Ag '56. (MLRA 10:11)

1. Institut organicheskoy khimii AN USSR.
(Phosphoric acid)

KIRSANOV, A.V.; ZHMUROVA, I.N.

Acid chlorides and esters of urethanphosphoric acids. Zhur. ob.khim. 26 no.9:2642-2648 S '56. (MLRA 9:11)

1. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR.

(Chlorides) (Urethanphosphoric acid)

KIRSANOV, A. V. (Dnepropetrovsk Metallurgical Inst.)

"Reaction of Pentachloride of Phosphorus with Amides of Acids" (Reaktsiya pyatikhloristogo fosfora s amidami kislot)

Chemistry and Uses of Organophosphorous Compounds (Khimiya i primeneniye fosfororganicheskikh soyedneniy), Trudy of First Conference, 8-10 December 1955, Kazan, pp 99- Published by Kazan Affil. AS USSR, 1957

111

AUTHORS: Kirsanov, A. V., and Makitra, R. T.

TITLE: Triaroxyphosphazosulfonearyls and Diaryl Esters of Arylsulfonamidephosphoric Acids (Triaroksifosfazosul'fonarily i diarilovya efiry arilsul'fonamidofosfornykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 245-248 (U.S.S.R.)

ABSTRACT: Numerous chloro- and nitrosubstituted triaroxyphosphazosulfonaryls and diaryl esters of arylsulfonamidephosphoric acids were synthesized for the purpose of studying their insecticide properties. The o- and p-trichlorotriphenoxyphosphazosulfonaryls are described as colorless, crystalline substances (with the exception of o-trichlorotriphenoxyphosphazosulfone-p-tolyl which is in liquid state at room temperature), of neutral nature, insoluble in water, easily soluble in acetone, ethyl acetate and ethyl ethers, crystallizes in ethyl alcohol, petroleum ether, and carbon tetrachloride. The o- and p-trinitrotriphenoxyphosphazosulfonaryls represent bright-yellow crystalline substances of neutral nature, well soluble in dioxane at room temperature, crystallize in benzene, insoluble in water and in a majority of organic solvents. Dinitrodiphenyl esters of arylsulfonamidophosphoric acid are

Card 1/2

Triaroxyphosphazosulfonearyls and Diaryl Esters of Arylsulfonamidephosphoric Acids

crystalline bright-yellow substances, well soluble in alcohol and acetone, almost insoluble in water and in a majority of organic solvents, crystallize in aqueous alcohol, and have the characteristics of strong monobasic acids. When mixed with alkalis, or aryls, they yield brightly-colored well-crystallizing salts.
Two tables. There are two Slavic references.

ASSOCIATION: Academy of Sciences Ukrainian SSR, Institute of Organic Chemistry (Institut Organicheskoy Khimii, Akademii Nauk Ukrainskoy SSR)

PRESENTED BY:

SUBMITTED: Feburuary 14, 1956

AVAILABLE:

Card 2/2

79-2-37/58

AUTHORS: Kirsanov, A. V., and Makitra, R. G.

TITLE: N-Acylamidophosphoric Acids (N-atsilamidofosfornyye Kisloty)

PERIODICAL: Zhurnal Obshchey Khimii, 1957, vol 27, No 2, pp. 450-452 (U.S.S.R.)

ABSTRACT: The hydrolysis of dichloroanhydrides of acylamidophosphoric acid yielded ten (10) acylamidophosphoric acids as listed in the table. The products are described as crystalline substances, easily soluble in water and alcohol, insoluble in acetone, ether, benzene and a majority of other organic solvents. When heated to a melting point, all acylamidopnosphoric acids decompose and in some instances the decomposition is accompanied by intensive darkening and liberation of gases. They cannot be recrystallized and become useless. N-acylamidophosphoric acids are strong acids displacing carbonic and acetic acids from their salts. They submit to titration with methyl orange as monobasic and with phenolphthalein as dibasic acids.

Card 1/2 1 table. There are 4 references of which 3 are Slavic.

79-2-37/58

N-Acylamidophosphoric Acids

ASSOCIATION: Academy of Sciences of Ukrainian SSR, Institute of Organic Chemistry

PRESENTED BY:

SUBMITTED: February 14, 1956

AVAILABLE: Library of Congress

Card 2/2

KIRSANOV, A.V.; LEVCHENKO, Ye.S.

N-thiazolyl and N- pyridyl derivati es of carbamid-
N'-phosphoric acid. Zhur. ob. khim. 27 no.9:2585-2590 (MIRA 11:3)
S '57.

1.Institut organicheskoy khimii AN USSR.
(Pyridine) (Pyrro)

KIRSANOV, A.V.; PESHCHENKO, N.G.

Trichlorophosphazosulfonenitroaryls and the products of their hydrolysis. Zhur.ob.khim. 27 no.10:2817-2820 O '57. (MIRA 11:4)

1.Institut organicheskoy khimii Akademii nauk SSSR.
(Hydrolysis) (Aromatic compounds)

79-11-35/56

AUTHORS: Kirsanov, A. V.; Molosnova, V. P.

TITLE: The Aromatic Esters of Oxamine Acid (Efiry oksaminovoy kisloty aromaticheskogo ryada).

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, Nr 11, pp. 3075-3078 (USSR)

ABSTRACT: Only the phenylester synthesized by Wallach and Liebmann and obtained in small quantities was hitherto known of the aromatic esters of oxamic acid. This phenyl ester was produced by the action of phosphoric pentachloride upon the ethyl of oxamic acid with subsequent phenol treatment on the resulting raw product. Wallach's assumption that an amidochloride as intermediate product plays an important part on that occasion has not been verified. Recent investigations say that the intermediate products are no amidochlorides, but amides of dichloralkoxyacetic acids and trichlorophosphazodichloralkoxyacetyls. Thus the formula of the formation of oxamine esters can only be represented in the following manner:

Card 1/3

79-11-35/36

The Aromatic Esters of Oxamine Acid

$$NH_2COCCl_2OR + R'OH \rightarrow NH_2COC\begin{matrix}OR\\Cl\\OR'\end{matrix} \begin{matrix}\nearrow RCl + NH_2COOR'\\ \searrow R'Cl + NH_2COOR\end{matrix}$$

This process of reaction has to be further investigated, as only the phenyl ester was hitherto known. In contrast to the action of aliphatic alcohols the phenols, as tests show, act in the above-mentioned manner. By their action upon the amides of dichloralkoxyacetic acids it was possible to synthesize quite a number of aromatic esters of oxamic acid with good yields, which fact completely confirms the authors' theoretical assumption. Thus it is shown that aromatic esters of oxamic acid form in the reaction of the phenols upon the amides of dichloralkoxyacetic acids and that this reaction may serve as scheme for the production of these esters. There are 1 table, and 3 references, [illegible] of which is Slavic.

Card 2/3

79-11-35/56

The Aromatic Esters of Oxamine Acid

ASSOCIATION: Dnepropetrovsk Metallurgical Institute (Dnepropetrovskiy metallurgicheskiy institut).

SUBMITTED: October 28, 1956

AVAILABLE: Library of Congress

1. Oxamine acids - Derivatives 2. Aromatic esters - Production

Card 3/3

79-12-15/43

AUTHORS: Kirsanov, A. V., and Nekrasova, Z. D.

TITLE: Dialkamides of Triaroxyphosphorosulphuric Acids and Aromatic Esters of the N,N-dialkylsulfamic - N' - Phosphoric Acids (Dialkilamidy triaroksifosfazosernykh kislot i aromaticheskiye efiry N,N-dialkilsul'famid - N' - fosfornykh kislot).

PERIODICAL: Zhurnal Obshchey Khimii 1957, Vol. 27, Nr 12, pp. 3241-3248 (USSR)

ABSTRACT: Up to now dialkylamides of the triaroxyphosphazosulphuric acids and the esters of N,N - dialkylsulfamic - N - phosphoric acids were unknown. The present work describes their synthesis and their properties. The dimethyl-and diethylamides of the triaroxyphosphazosulphuric acids were obtained by the action of dimethylamide of the trichlorphosphazosulfuric acid and the diethylamide of the same acid on sodiumarylates according to the pattern:

$$P_2NSO_2N = PCl_3 + 3\ NaOAr \rightarrow 3\ NaCl + R_2NSO_2N = P(OAr)_3$$

These dialkylamines are colourless, crystalline, low melting and when melting non-decomposing compounds, with the exception of diethylamide which is liquid at room temperature. From the chemical point of view these dialhylamides are neutral and

Card 1/3

Dialkamides of Triaroxyphosphorosulphuric Acids and Aromatic Esters of the N,N-dialkylsulfamic - N' - Phosphoric Acids. 79-12-15/43

in boiling water very difficulty saponifiable products. With only few exceptions they can be saponified only by heating of alkalilyes on which occasion, however, saponification with good yields occurs only to the esters of the N,N-dialkyl-sulfamic - N- phosphoric acids (see pattern 2). They are insoluble in water, however, easily soluble in acetone, chloroform, dichlorethane, benzene and hot alcohol. The diaryl-esters of the N,N-phosphoric acids were produced from the dichloroanhydrides of the N,N-dialkylsulfamic-N'- phosphoric acids according to the formula:

$$R_2NSO_2NHPOCl_2 + 2\ ArONa \rightarrow 2\ NaCl + R_2NSO_2NHPO(OAr)_2$$

The diesters obtained in this way agreed with those which synthesized by saponification of the dialkylamides of the triaroxyphosphazoacids which indicates at the presumed structure. There are 3 references, 3 of which are Slavic.

Card 2/3

Dialkamides of Triaroxyphosphorosulphuric Acids and Aromatic Esters of the N,N-dialkylsulfamic - N' - Phosphoric Acids. 79-12-15/43

ASSOCIATION: Dnepropetrovsk Metallurgical Institute
(Dnepropetrovskiy metallurgicheskiy institut)

SUBMITTED: October 8, 1956

AVAILABLE: Library of Congress

1. Cyclic compounds - Synthesis 2. Cyclic compounds - Properties 3. Dialkamides of Triaroxyphosphoric-sulfuric acid 4. Aromatic esters of N,N-dialkylsulfamic-N'-phosphoric acids

Card 3/3

79-12-16/43

AUTHORS: Kirsanov, A. V., Derkach, G. I.

TITLE: Trichlorphosphazoaciles, Trichloroisophosphazoaciles and Their Derivatives (Trikhlorfosfazoatsily, trikhlorizofosfazoatsily i ikh proizvodnyye).

PERIODICAL: Zhurnal Obshchey Khimii 1957, Vol. 27, Nr 12, pp. 3248-3254 (USSR)

ABSTRACT: As it has been shown before trichlorophosphazoaciles of the RCON - PCl_3 type are obtained by the action of pentachloride on the amides of the carboxylic acids, which on the occasion of partial hydrolisis form dichloroanhydrides of the acil-amidophosphoric acids. Up to now only a trichloroisophosphazo-acile was known i. e. trichloroisophosphazotrichloracetyl and some corresponding triaroxyisophosphazoaciles. It is of interest whether also from other carboxylic acids trichloroiso-phosphazoaciles and triaroxyisophosphazoaciles occur or whether the trichloroisophosphazoaciles occur only in the case of trichloroacetic acid and its analogs. Trichlorophosphazo-aciles were produced for the diphenylchloracetic acid, tri-phenylacetic acid and p - nitrobenzoic acid and their thermal stability was investigated. It was demonstrated that

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79-12-16/43

Trichlorphosphazoaciles, Trichloroisophosphazoaciles and Their Derivatives.

trichloroisophosphazoaciles occur not only for trichloracetic acid and its analogs but also for diphenylchloracetic acid and p - nitrobenzoic acid. Trichloroisophosphazotriphenylacetyl could not be obtained, since dichloroanhydride of the triphenylacetylamidophosphoric acid when thermated has no durability. The authors obtained the di- and tri -α - naphtoxyderivatives from the corresponding synthesized trichlorophosphazo- and trichloroisophosphazo compounds. The synthesized Tri -α- naphtoxyisophosphazo - p - nitrobenzoyl is the first derivative of trichloroisophosphazoaciles obtained from crystals.
There are 6 references, 4 of which are Slavic.

ASSOCIATION: Dnepropetrovsk Metallurgical Institute (Dnepropetrovskiy metallurgicheskiy institut).

SUBMITTED: October 16, 1956

AVAILABLE: Library of Congress
1. Trichlorophosphazoaciles - Chemical analysis
2. Trichloroisophosphazoaciles - Chemical analysis

Card 2/2

KIRSANOV, A. V.

79-1-7/63

AUTHORS: Kirsanov, A. V., Molosnova, V. P.

TITLE: The Reaction of Phosphorus Pentachloride With Esters of Oxamic Acid (Reaktsiya pyatikhloristogo fosfora s efirami oksaminovoy kisloty) The Amides of Alkoxydichloroacetic Acids (Amidy alkoksidikhlorukusnykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 1, pp.30-35(USSR)

ABSTRACT: It was already earlier shown that under the influence of phosphorus pentachloride upon the ethyl ester of oxamic acid the amide of ethoxydichloroacetic acid and trichlorophospha zoethoxydichloroacetyl are consecutively obtained according to the following scheme:

$$C_2H_5OCOCONH_2 + PCl_5 \longrightarrow POCl_3 + C_2H_5OCCl_2CONH_2 \quad (1)$$

$$C_2H_5OCCl_2CONH_2 + PCl_5 \longrightarrow 2HCl + C_2H_5OCCl_2CON = PCl_3 \quad (2)$$

The synthesis of trichl[illegible]phosphazoethoxydichloroacetyl and its homologues was thoroughly investigated and it was shown that reaction (2) is generally valid for the synthesis of

Card 1/3

The Reaction of Phosphorus Pentachloride With Esters of Oxamic Acid . The Amides of Alkoxydichloroacetic Acids

trichlorophosphazoalkoxydichloroacetyls. It was of interest to find out whether reaction (1) might also be generally valid and whether it should also be considered a general method for the production of amides of alkoxydichloroacetic acids. It was found that reaction (1) takes place sufficiently fast at considerably lower temperatures than reaction (2) for the methyl-, ethyl-, butyl and isobutyl-ester of oxamic acid. This fact permitted to obtain the amides of the corresponding alkoxydichloroacetic acids with good yields, although reaction (2) in all cases takes place beside reaction (1). Quantitatively the amides are hard to be separated from the acetyls. The yield of amides is quite different according to the ester of oxamic acid used. Reaction (1) takes place most difficultly for the methylester, more easily for the ethyl- and quite easily for the butyl- and isobutyl ester of oxamic acid. The structure of the amides of alkoxydichloroacetic acids can quite incontestably be proved by their reaction with phenols (see formulae on page 33). There are 6 references, 5 of which are Slavic.

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79-1-7/63

The Reaction of Phosphorus Pentachloride With Esters of Oxamic Acid . The Amides of Alkoxydichloroacetic Acids

ASSOCIATION: Dnepropetrovsk Metallurgical Institute
(Dnepropetrovskiy metallurgicheskiy institut)

SUBMITTED: November 20, 1956

AVAILABLE: Library of Congress

Card 3/3 1. Chlorides 2. Oximidines 3. Chemistry

KIRSANOV, A.V.

AUTHORS: Kirsanov, A. V., Makitra, R. G. 79-1-8/63

TITLE: Diesters of Aromatic Acylamidophosphoric Acids (Diefiry aromaticheskikh atsilamidofosfornykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1950, Vol. 20, Nr 1, pp. 35-40 (USSR)

ABSTRACT: Like the diesters of arylsulfonamidophosphoric acids the diesters of acylamidophosphoric acids should also be representable by the influence of arylates or alcoholates of sodium upon dichloroanhydrides of acylamidophosphoric acids according to scheme (1), or by saponification of triaroxyphosphamacyls according to scheme (2)

$ArCONHPOCl_2 + 2ROR \rightarrow 2\ NaCl + ArCONHPO(OR)_2$ (1)

$ArCON{=}P(OR)_3 + H_2O \rightarrow ROH + ArCONHPO(OR)_2$ (2)

The dimethylester of benzoylamidophosphoric acid is obtained according to scheme (1) in the presence of much sodium methylate and methyl alcohol with a yield of 37,0%, which is not the case with the aromatic diesters (10 - 24,5%).

Card 1/3 According to scheme (2) the diarylesters are obtained with

79-1-8/43

Diesters of Aromatic Acylamidophosphoric Acids

a good yield, where one can start directly from trichloro-
phosphazoacyls. With dry sodium arylates in a benzene-,
ether- or dioxane-solution these acyls yield the
corresponding triaroxyphosphazoacyls according to the
following scheme: $ArCON = PCl_3 + 3NaOR \rightarrow 3NaCl + ArCON=P(OR)_3$

These acyls are much more easily saponified to the
diesters of triaroxyphosphazosulfonacyls, so that a
heating of 5 - 10 minutes with water is sufficient for
attaining the complete conversion of triaroxyphosphazoacyls
to the diarylesters of acylamidophosphoric acids. In the
synthesis of the diesters of p-chlorobenzoylamidophosphoric
acid p-chlorobenznitrile was in some cases liberated as by-
product. According to (2) the authors obtained the methyl-,
phenyl-, p-cresylic, p-chlorophenyl- and p-nitrophenyl-
-esters of benzoyl-, p-chlorobenzoyl- and p-nitrobenzoyl-
amidophosphoric acids as well as the di-α-naphtylester of
benzoylamidophosphoric acid.

ASSOCIATION: Laboratory for Insecticides of the Institute for Organic Chemistry AN Ukrainian SSR (Laboratoriya insektitsidov Instituta organicheskoy khimii Akademii nauk Ukrainskoy SSR)

Card 2/3

Diesters of Aromatic Acylamidophosphoric Acids 79-1-8/63

SUBMITTED: November 26, 1956

AVAILABLE: Library of Congress

Card 3/3 1. Chemistry 2. Arylates 3. Alcoholates

KIRSANOV, A. V.

AUTHORS: Kirsanov, A. V., Feshchenko, N. G.

TITLE: Trimethoxy- and Triaroxyphosphasosulfonnitrophenyls and Diesters of Nitrophenylsulfonamidophosphoric Acids (Trimetoksi- i triaroksifosfazosul'fonnitrofenily i diefiry nitrofenilsul'fonamidofosfornykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 2, pp. 339 - 343 (USSR)

ABSTRACT: Trimethoxy- and triaroxyphosphasosulfonnitrophenyls (I) and diesters of nitrophenylsulfonamidophosphoric acids (II) were synthesized for the purpose of investigating their insecticidic properties and as a starting point for the production of N-phosphoric acid derivatives of sulfanilamides. (I) was obtained by interaction of trichlorophosphasosulfonnitrophenyls (reference 1) with methylate and sodium arylates in a benzene solution (reference 2). Trimethoxy-, triphenoxy-, tri-p-chlorotriphenoxy-, tri-o- and tri-p-trinitrotriphenoxyphosphasosulfone-o-, m- and p-nitrophenyls (I) (table 1) were obtained in this manner. (I) represent colorless crystalline substances of a neutral character. They do not dissolve in water. Trimethoxyphosphasosulfonnitrophenyls (III) within one hour saponify on boiling with 96% alcohol to the corresponding diesters (XIV).

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79-2-13/54

Trimethoxy- and Triaroxyphosphasosulfonnitrophenyls and Diesters of Nitrophenylsulfonamidophosphoric Acids

Triphenoxyphosphasosulfonnitrophenyls (IV) do not change under the same conditions. Trimethoxy- and triphenoxyphosphasosulfonnitrophenyls melt at comparatively low temperatures (from 56 - 104°C). (I) do not saponify upon the influence of aqueous alkaline solutions, which is explained by their insolubility in water. In aqueous alkaline spirit solutions they easily saponify to (II). But (II) can more conveniently be produced from dichloroanhydrides of nitrophenylsulfonamidophosphoric acids (reference 1) by means of the influence of sodium arylates and - methylate in a dioxane solution. Thus dimethyl-, di-p-chlorodiphenyl-, di-o- and di-p-nitrodiphenyl ethers of o-, m- and p-nitrophenylsulfonamidophosphoric acids (II) (table 2) were produced. (II) can be eliminated in the form of salts, but it is more convenient in the form of free diesters. (II) represent comparatively high-melting (from 134 - 194°C), crystalline, colorless substances insoluble in water. They melt at far higher temperatures than the corresponding (I), with the exception of two p-nitrophenolethers (XI and XIII) which melt under the corresponding (I). (II) are monobasic acids which exactly titrate in the presence of phenolphthalein and yield well-crystallizing and water-soluble sodium salts. It is interesting that the sodium salts of the p-chlorophenylethers of nitrophenylsulfonamidophosphoric acids on withdrawal of the water solutions by the ether com-

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Trimethoxy- and Triaroxyphosphasosulfonnitrophenyls and Diesters of Nitrophenylsulfonamidophosphoric Acids

pletely go over into the ether layer. Sodium salts of other diesters are not withdrawn from the water solution by the ether. There are 2 tables, and 1 Slavic reference.

ASSOCIATION: Institute for Organic Chemistry AS Ukrainian SSR
(Institut organicheskoy khimii Akademii nauk USSR)

SUBMITTED: December 24, 1956

AVAILABLE: Library of Congress

KIRSANOV A V.

79-2-14/34

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

TITLE: Substituted Imido- and Monoarylsulfamides (Zameshchennyye imido- i monoarilsul'famidy)

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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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 Meybeck (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

Substituted Imido- and Monoarylsulfamides

and diarylsulfamides, however, is not large. This is easy to understand, as on the influence of amines upon sulfamide the imidosulfamide must also be produced beside the formation of substituted sulfamides. This imidosulfamide practically represents the only reaction product in the interaction of the sulfamide with tertiary amines (reference 2). Besides the already formed N-arylsulfamide on heating and under the influence of an amine excess may yield the corresponding 1,5-diarylimidosulfamide. At present it is impossible to find out whether the N-arylsulfamides are formed directly from the amines and the sulfamide according to the summary scheme (IV) or only as products of the hydrolysis of the 1,5-diarylsulfamide being in the reaction mixture, according to scheme (III). There are 1 table, and 7 references, 3 of which are Slavic.

ASSOCIATION: Institute for Metallurgy, Dnepropetrovsk
(Dnepropetrovskiy metallurgicheskiy institut)

SUBMITTED: January 17, 1957

AVAILABLE: Library of Congress

Card 3/3

KIRSANOV, A.V.

79-2-15/54

AUTHORS: Kirsanov, A. V., Molosnova, V. P.

TITLE: Trichlorophosphasoaroxydichloracetyls (Trikhlorfosfazoaroksidikhloratsetily)

PERIODICAL: Zhurnal Obshchey Khimii, 1959, Vol. 29, Nr 2, pp. 347 - 350 (USSR)

ABSTRACT: It was recently discovered that the aliphatic esters of oxamic acid react with pentachlorophosphorus and at first form amides of alkoxydichloroacetic acids (reference 1) according to scheme (I) and subsequently trichlorophosphasoalkoxydichloracetyls (reference 2) according to scheme (II)

$$ROCOCONH_2 + PCl_5 \longrightarrow POCl_3 + ROCCl_2CONH_2 \quad (I)$$

$$ROCCl_2CONH_2 + PCl_5 \longrightarrow 2HCl + ROCCl_2CON = PCl_3 \quad (II)$$

For aromatic esters of oxamic acid (reference 3) it was hitherto not possible to limit the reaction with scheme (I). Even in the presence of a large excess of oxamic-acid esters the reaction does not take place or it at once takes place according to scheme (III) with the formation of trichlorophosphasoaroxydichloracetyls:

$$ArOCOCONH_2 + 2PCl_5 \longrightarrow 2HCl + POCl_3 + ArOCCl_2CON = PCl_3 \quad (III)$$

A possible explanation of this phenomenon is given in reference 1. Under favorable conditions the reaction takes place according to

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Trichlorophosphasoaroxydichloracetyls

scheme (III) quantitatively or almost quantitatively with the formation of colorless, crystalline trichlorophosphasoaroxydichloracetyls (IV). The "raw products" as a rule melt at temperatures only a few degrees lower than the recrystallized ones and are usually colorless. Only in the case of naphthoxy derivatives (XI and XII) the raw products are colored and melt at considerably lower temperatures than the pure substances. According to its physical and chemical properties (IV) is similar to trichlorophosphasoalkoxydichloracetyl (reference 2). Under the influence of water vapors or, still better, of dehydrated formic acid (IV) yield dichloranhydrides of aroxydichloracetylamidophosphoric acids (V) with a good yield according to the scheme:

$ArOCCl_2CON = PCl_3 + HCOOH \longrightarrow CO + HCl + ArOCCl_2CONHPOCl_2$. But on heating (IV), in contrast to trichlorophosphasoalkoxyacetyls, decompose considerably more difficult. On that occasion they do not split off any chlorobenzene and considerably more difficult and slowly (only at 160°C) phosphorus chloroxide. (V) represent colorless, crystalline substances. They melt at considerably higher temperatures than the corresponding trichlorophosphasoaroxydichloracetyls (IV) and in the usual organic solvents they are more difficult to solve than (IV). At room temperatures (V) slowly react with

Trichlorophosphasoaroxydichloracetyls

water. Therefore thy can be stored without any special precautionary measures. On heating with water (V) rapidly hydrolylates and energetically reacts with substances whose molecules contain active hydrogen atoms. All solvents and initial substances were most exactly dehydrated. All tests were performed in a manner that the reaction mixtures and reaction products as far as possible did not get in touch with atmospheric moisture. There are 3 references, all of which are Slavic.

ASSOCIATION: Institute for Metallurgy, Dnepropetrovsk
(Dnepropetrovskiy metallurgicheskiy institut)

SUBMITTED: January 17, 1957

AVAILABLE: Library of Congress

79-28-4-43/60

AUTHORS: Kirsanov, A. V., Feshchenko, N. G.

TITLE: Ester of Aminophenylsulfonamido-Phosphoric Acids (Efiry aminofenilsul'fonamidofosfornykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol.28, Nr 4, pp.1049-1052(USSR)

ABSTRACT: Dimethyl- and diphenyl ester of o-, m-, and p-aminophenylsulfonamido-phosphoric acids (formula I) were produced according to the following reaction scheme:

$$NO_2C_6H_4SO_2NH_2 \xrightarrow{PCl_5} NO_2C_6H_4SO_2N{=}PCl_3 \xrightarrow{HCOOH} NO_2C_6H_4SO_2NHPOCl_2 \longrightarrow$$

$$\longrightarrow NO_2C_6H_4SO_2NHPO(OR)_2 \xrightarrow{6\,H} NH_2C_6H_4SO_2NHPO(OR)_2$$

(2NaOR)

The reduction of diesters of the nitrophenylsulfonamido-phosphoric acids was performed with hydrogen at presence of a palladium catalyst in alcoholic solution at room temperature and at a pressure of ca. 100 torr. The yields were quantitatively. The diester produced by this way form colorless crystals,

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Ester of Aminophenylsulfonamido-Phosphoric Acids

which have only weakly basic, but strong acid properties. They solve readily in aqueous soda solution and can be titrated as monobasic acids. Their aqueous solutions react acid with Congo red. The dimethyl ester of the aminophenylsulfonamido phosphoric acids are readily soluble in hot water and can be diazotized on the usual conditions (as aniline). The corresponding diphenyl ester are in hot water difficultly to solve, in alcohol more readily soluble. They can be diazotized on the same conditions as aromatic amines with electronegative substituents (e.g. nitroaniline).

The amides of the o-, m- and p-aminobenzenesulfonic acids, to the derivatives of which belong the compounds of the formula I, have little different melting points (153°, 142°, 163°). The lowest melting point has the m-isomeric, the highest the p-isomeric (Ref 3). At the corresponding isomeric compounds of the formula I on the contrary the melting points are far from each other. The highest melting point here has the m-isomeric, the lowest the o-isomeric. The ortho amino-compounds of the formula I are at room temperature very readily soluble in acetone, the para-isomeric only with difficulty and in case of heating. The meta-isomerics are practically

insoluble in acetone. For the strong change of the melting points as well as for the great differences of the solubility in acetone the following explanation is probable: In the case of the o- and p-isomerics intra- and intermolecular hydrogen bindings form, the formation of which in case of the m-isomerics is complicated or impossible. The influence of the hydrogen bridges upon the melting points of the amides of the aminobenzenesulfonic acids is because of absence of the polarizing influence of the phosphoric acid rest essentially lower; therefore the melting points of the isomerics are close together. For the characteristic of the compounds with the formula I their benzoyl derivatives were produced according to the method by Schotten-Baumann. These compounds form colorless crystals, which have strong acid and no basic properties. Their aqueous solutions react acid with Congo red. The melting points partly are higher and partly are lower than the melting points of the corresponding compounds of the formula I.

 In an experimental part the synthesis and the properties

Ester of Aminophenylsulfonamido-Phosphoric Acids

of the compounds of the formula I and of their benzoyl derivatives are described exactly. There are 2 tables and 4 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR (Institute for Organic Chemistry, AS Ukrainian SSR)

SUBMITTED: February 14, 1957

Card 4/4

AUTHORS: Kirsanov, A. V., Yegorova, N. L. 79-28-4-44/60

TITLE: Triaroxyphosphorazosulfonalalkyls and Aromatic Esters of Alkylsulfonamidophosphoric Acids (Triaroksifosfazosul'fonalkily i aromaticheskiye efiry alkilsul'fonamidofosfornykh kislot)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 4, pp. 1052-1055 (USSR)

ABSTRACT: Triaroxyphosphorazosulfonalkyls were producet by reaction of sodium arylates with trichlorophosphorazosulfonalkyls (ref 1) in benzene solution:

$$RSO_2N{=}PCl_3 + 3\ ArONa \longrightarrow \underset{(I)}{RSO_2N = P(OAr)_3} + 3\ NaCl$$

Sodium phenolate and sodium-p-chlorphenolate react very with trichlorophosphorazosulfonalkyls. With dry sodium phenolate without solvent, the reaction very violently takes place and the reaction mixture carbonizes. Sodium-p-nitrophenolate reacts less violently; the reaction takes place on heating in the water bath in the course of 2-3 hours. Yields, melting points

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and analytical data of the compounds of formula I obtained in this way are exactly mentioned. The compounds are colorless crystallized substances (with the one exception of the liquid derivative Ar = C_6H_5, R = n-C_4H_9) which are easily soluble in acetone, dioxane, a little more difficultly in benzene, ether and alcohol. They dissolve in boiling tetrachloride and petroleum ether as well, whilst in hot water very difficultly, in cold water they are insoluble. According to their physical and chemical properties the compounds of the formula I are very similar to triaroxyphosphorazosulfonaryls (ref 2), however, they differ by an higher solubility in boiling water and polar solvents. The compounds of formula I are neutral substances which are relatively constant against water and atmospheric moisture. An exception is represented by the derivative with Ar = p-$NO_2C_6H_4$, R = C_2H_5 (II), which is very easily hydrolyzable. Already in its solutions in 96 % ethanol a complete saponification takes place, where p-dinitrophenyl

ester of ethylsulfonamidophosphoric acid (III) and nitrophenol are formed:

$$C_2H_5SO_2N=P(OC_6H_4NO_2\text{-}p)_3 + H_2O \longrightarrow C_2H_5SO_2NHPO(OC_6H_4NO_2\text{-}p)_2 + p\text{-}NO_2C_6H_4OH.$$

All the other compounds of formula I are not modified on heating with alcohol, however, under the action of caustic alkalies in alcoholic-aqueous solution they are easily saponified. In this case salts of the corresponding diaryl ester of alkylsulfonamidophosphoric acids (IV) form, the yields, melting points and analytical data of which are mentioned. They easily dissolve in acetone and hot alcohol, very difficultly in cold - more easily in boiling water. In most of the unpolar solvents they are difficultly soluble. In chemical respects they are strong monobasic acids. With respect to hydrolysis in an acid, as well as in an alkaline

medium they are essentially more constant than the compounds of formula I. There structure not only unequivocally results from the formation by saponification of the compounds of formula I, but was also proved by the synthesis of diacetic chlorides of alkylsulfonamidophosphoric acids with sodium arylates:

$$RSO_2NHPOCl_2 + 2ArONa \longrightarrow RSO_2NHPO(OAr)_2 + 2NaCl$$

Syntheses and analytical data of the mentioned compounds are exactly described in an experimental part.
There are 2 tables and 3 references, 3 of which are Soviet

ASSOCIATION: Dnepropetrovskiy metallurgicheskiy institut (Dnepropetrovsk Metallurgical Institute)

SUBMITTED: February 4, 1957

AUTHORS: Kirsanov, A. V., Derkach, G. I., Makitra, R. G. 79-28-5-21/69

TITLE: Triaroxyphosphazoacyl (Triaroksifosfazoatsily)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 5, pp. 1227-1232 (USSR)

ABSTRACT: The similarity of triaroxyphosphazoacyls (I) and triaroxyphosphazosulfone-compounds (II) shows up in a number of common chemical properties so that their reactions of formations are in common:

$$RSO_2N = PCl_3 + 3NaOR' \longrightarrow 3NaCl + RSO_2N = P(OR')_3$$

$$RCON = PCl_3 + 3NaOR' \longrightarrow 3NaCl + RCON = P(OR')_3$$

Between them, however, also specific chemical differences with regard to heating and hydrolysis. The compounds (I) split off rather easily (depending on the radical) from the corresponding triesters of phosphoric acid and produce nitriles according to the scheme $RCON = P(OR')_3 \rightarrow OP(OR')_3$ +RCN (III). The compounds (II) are very much stable against

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heating so that until now there has been no case of splitting according to scheme (III). They saponify easily with alkali liquors under the formation of salts of the diesters of the corresponding alkyl- or aryl--sulfonamidophosphoric acids, but they do not saponify with water in neutral solutions. Therefore the synthesis and the separation of the products (II) do not meet with any difficulties because of the easy saponifiability. All compounds (I) saponify on boiling practically quantitatively to the diesters of the acylamidophosphoric acids for which reason the synthesis, separation and purification of the triaroxyphosphazoacyls takes place so difficulty; for the same reason in the experiments care must be taken that they do not come into contact with atmospheric humidity. This difference can apparently be explained by the fact that in the saponification of the compounds (I) in alkali solutions the carbon- and oxygen atoms of the carboxyl-group take part in the hydrolysis and increase the positive charge of the phosphorus atom according to the given scheme 1. In the saponification of the compounds (II) mainly only the nitrogen- and phospho-

rus atoms take part in the hydrolysis (see scheme 2). There are 2 tables and 7 references, 6 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii AN Ukrainskoy SSR (Institute for Organic Chemistry, AS Ukrainian SSR)

SUBMITTED: February 22, 1957

AUTHOR: [illegible], [illegible] SOV/79-28-6-[illegible]/63

TITLE: Anilides of Alkylsulfon[illegible]idophosphoric Acids (Anilidy alkilsul'fon[illegible]idofosfornykh kislot)

PERIODICAL: Zhurnal obshchey Khimii, 1958, Vol. 28, Nr 6, pp. 1587-1593 (USSR)

ABSTRACT: On the action of aniline on trichlorophosphazosulfonalkyls the formation of anilidodichlorophosphazosulfonalkyls of the type $RSO_2N{=}PCl_2(NHC_6H_5)$ (I), of dianilidochlorophosphazosulfonalkyls of the type $RSO_2N{=}PCl(NHC_6H_5)_2$ and of trianilidophosphazosulfonalkyls of the type $RSO_2N{=}P(NHC_6H_5)_3$ can be expected. The products (I) could not be obtained (Ref 2). The compounds (II) are obtained in sufficiently good yields in the conversion of trichlorophosphazosulfonalkyls with aniline in carbon tetrachloride. They are crystalline substances of neutral character and hydrolize easily to dianilides of the alkylsulfonamidophosphoric acids (III) on heating their solutions in 96% alcohol or in boiling water according to

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the scheme $RSO_2H + PCl(NHC_6H_5)_2 + H_2O \rightarrow HCl +$ + $RSO_2NHPO(NHC_6H_5)_2$. The salt formation of (II) by action of alkali liquors and ammonia takes place without difficulty. On an acidification of these easily soluble salts the free (III) compounds separate so that the reaction mixture for the production of (III) can be directly separated from the alkali liquor. The compounds (III) are colorless bodies of bitter taste and cannot be hydrolized with alkali solutions; this can, however, be achieved by a heating with dilute mineral acids under the formation of amides of the alkylsulfo acids and anilides of the phosphoric acids. They are mono- -basic, rather strong acids. Trianilidophosphazosulfonalkydes (IV) are obtained in good yields on a longer heating of the trichlorophosphazosulfonalkyls with excess aniline in benzene solution. The properties of the products (II-IV) are mentioned in the experimental part. There are 3 references, 3 of which are Soviet.

ASSOCIATION: Dnepropetrovskiy metallurgicheskiy institut
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Anilides of Alkylsulfonamidophosphoric Acids SOV/79-28-6-34/65

SUBMITTED: February 21, 1957

1. Anilines--Chemical reactions

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AUTHORS: Kirsanov, A. V., Levchenko, Ye. S. SOV/79-28-6-35/63

TITLE: Fluoranhydride Salts of the Arylsulfonamidophosphoric Acids (Soli ftorangidridov arilsul'fonamidofosfornykh kislot)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 6, pp. 1589-1594 (USSR)

ABSTRACT: In continuation of their own previous papers on trichlorophosphazosulfonaryls and on products of their hydrolysis, the dichloranhydrides of the arylsulfonamidophosphoric acids (Refs 1, 2) the authors were interested in synthesizing and investigating the fluorine containing analogues of the trichlorophosphazosulfonaryls and of the dichloroanhydrides of the arylsulfonamidophosphoric acids. The direct substitution of the chlorine in these compounds by fluorine, as for instance , with potassium fluoride, did not succeed. As is known chlorine is easily replaced by fluorine in the chloroanhydrides of various acids when potassium fluoride in aqueous solution is allowed to act on them. In the conversion of the trichlorophosphazosulfonaryls with a saturated solution of potassium fluoride heat is produced, on which occasion besides the substitution of chlorine by fluorine a partial

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Fluoranhydride Salts of the Arylsulfonamidophosphoric Acids

hydrolysis takes part and potassium salts of the difluoranhydrides of the arylsulfonamidophosphoric acids are formed according to the summary scheme 1. The same products are obtained in the conversion of the dichloroanhydrides of the same phosphoric acids with potassium fluoride according to scheme 2. As was to be expected the potassium salts of the dichloroanhydrides of the arylsulfonamidophosphoric acids, the potassium salts of the difluoranhydrides of the arylsulfonamidophosphoric acids and the dipotassium salts of the monofluoranhydrides of the same acids are formed depending on the reaction conditions. The structure of the potassium salts of the difluoranhydrides of the same acids is proved by their conversion to the esters of these acids by means of sodium methylate. It was shown that the potassium salts of the difluoranhydrides of the arylsulfonamidophosphoric acids have a still greater resistance to hydrolysis than the potassium salts of the dichloroanhydrides of the same acids. The reasons for this phenomenon are discussed in detail. There are 3 tables and 10 references, which are Soviet.

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Fluoranhydride Salts of the Arylsulfonamidophosphoric Acids

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR (Institute of Organic Chemistry, AS Ukr SSR)

SUBMITTED: February 21, 1957

1. Phosphoric acid--Hydrolysis

Card 3/3

AUTHORS: Kirsanov, A. V., Nekrasova, Z. D. SOV / 79-28-6-36/63

TITLE: The Diphenylamide of Trichlorophosphazocarbonic Acid and Its Derivatives (Difenilamid trikhlorfosfazougol'noy kisloty i yego proizvodnyye)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 6, pp. 1595-1601 (USSR)

ABSTRACT: Only one method for the synthesis of the N-phosphoric acid derivatives of urea (carbamidephosphoric acids) has been published until now, that is to say, the binding of the primary and secondary amines to the chlorine anhydride or to the isocyanatephosphates (Ref 1). Besides, a bis-trichlorophosphazocarbonyl (Ref 2) was obtained on the action of phosphorpentachloride on urea; this product being a derivative of the N,N'-carbamide biphosphoric acid. In order to develop a common method of synthesis for the N,N-double substituted carbamide-N'-phosphoric acids and their derivatives, and at the same time to extend the possibilities for using the phosphorpentachloride reaction with acid amines, the reaction of phosphorpentachloride with N,N-diphenylurea was carried out. Phosphorpentachloride reacts on N,N-di-

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