"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR

UDC 547.241

PUDOVIK, A. N., and SUDAKOVA, T. M., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Addition of Oxides and Sulfides of Secondary Phosphines to Acrylic and Methacrylic Acid Derivatives"

Leningrad, Zhurnal Obshchey Khimii, Sep 71, Vol 41, No 9, pp 1962-1964

Abstract: It is shown that the reactivity of oxides of secondary phosphines in addition reactions to acrylonitriles is considerably higher than that of appropriate phosphites and phosphinates. The reactivity of thione derivatives is higher than that of phosphoryl compounds. The study involved addition of oxides and sulfides of secondary phosphines containing butyl and phenyl radicals as well as a series of partial esters of phosphoric acids with the same radicals to acrylonitriles. The reactivity was evaluated from the degree of completeness of the addition over specific time intervals. The reactions were conducted in solutions of butyl alcohol and dioxane (10:1); potassium tertiary butoxide was used as the catalyst. Data on the reactivities of secondary phosphines versus dialkylphosphinates and dialkyl phosphates cited in tables in the original article imply that the inductive effect of the radicals at the phosphorus is the factor most responsible for the reactivities of these compounds in addition reactions.

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USSR

UDC 547.26'118

PUDOVIK, A. N., GUR'YANOVA, I. V., and KAKURINA, V. P.

"Reaction of Diethylphosphorus Acid Monoisocyanate With Ethyl Pyruvate"

Leningrad, Zhurnal Obshchey Khimii, Sep 71, Vol 41, No 9, pp 1976-1978

Abstract: In furthering research on reactions of trivalent phosphoric acid derivatives with carbonyl compounds activated by electron acceptor substituents, a study was made of the reaction of diethylphosphorus acid monoisocyanate with ethyl pyruvate. The reaction product is 2-ethoxy-2-oxo-2-ethyl-5-methyl-5-carboethoxy-2-phosphaoxazolin-4-one. The new compound is a very viscous liquid which can be distilled under vacuum without being decomposed and can be dissolved in organic solvents. On prolonged storage, the product gradually thickens and turns into a glassy insoluble mass. The initial reaction yields optimum results at low temperatures and a 1:1 ratio of reagents in methylene chloride solution.

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USSR

UDC 547.26'118

TARASOVA, R. I., KISLITSYNA, R. M., and PUDOVIK, A. N.

"Reaction of the Isocyanate of Diethylphosphorus Acid With Aldehydes"

Leningrad, Zhurnal Obshchey Khimii, Sep 71, Vol 41, No 9, pp 1972-1976

Abstract: The reaction of diethylphosphorus acid isocyanate with benzaldehyde, p-chloro- and p-bromobenzaldehyde yields crystalline cyclic diethoxyalkylphosphazo carbonates and polymeric 1:1 adducts; the latter are formed on heating the cyclic diethoxybromobenzylphosphazo carbonate to its melting point. Analysis of the reaction products reveals two ethoxy groups. The IR spectra of the reaction products of diethylphosphorus acid isocyanate with p-bromobenzaldehyde show absorption at 1350 cm^{-5} for P=N and absorption at 1710-1720 cm^{-1} for the group C=O. Reactions with anisaldehydes and other aldehydes yielded oily products consisting of two fractions. According to analytical data, both fractions could be the addition products of diethylphosphorus acids isocyanates and aldehydes in a 1:1 ratio. Both have identical IR spectra, which greatly differ from those of crystalline 1:1 adducts. The IR spectra of the oils show absorption at 1260-1265 (P=O), at 1735-1746 (C=O) and 3200-3400 cm^{-1} (NH).

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USSR

UDC 547.37:547.26'118

PUDOVIK, A. N., ZIMIN, M. G., and KURGUZOVA, A. M., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Addition of α-Hydroxyalkylphosphonate Esters to Unsaturated Compounds"

Leningrad, Zhurnal Obshchey Khimii, Sep 71, Vol 41, No 9, pp 1964-1967

Abstract: The study of addition of α-hydroxyalkylphosphonates to unsaturated compounds indicates that α-hydroxyalkylphosphonates have a tendency to add to acrylonitriles, methacrylate and methyl vinyl ketone in the presence of a saturated solution of sodium ethoxide to form α-(dialkoxyphosphono)alkyl β-cyano(aceto, carbomethoxy)alkyl ethers. The addition products were identified from the tabular data obtained by elementary analysis, molecular refraction and IR and NMR spectra. A comparison of the yields of the addition products shows diethyl α-hydroxymethylphosphonate to be most reactive, diethyl α-hydroxyethylphosphonate somewhat less reactive and diethyl α-hydroxyisopropylphosphonate to be least reactive in the addition reactions. There appears to exist a specific regularity of an increasing tendency to addition reactions (primary > secondary > tertiary) for aliphatic alcohols.

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UDC 541.127.3:542.938:547.26'118

BEL'SKIY, V. Ye., BEZZUBOVA, N. N., YELISEYENKOV, V. N., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, USSR Academy of Sciences

"Kinetics of the Hydrolysis of Mixed Esters of Methylphosphonic Acid Which Contain P-O-C and P-S-C Bonds"

Leningrad, Zhurnal Obshchey Khimii, Vol XL, No 12, Dec 70, pp 2557-2560

Abstract: As a continuation of studies begun in 1969, the authors examined the reactions between water and the OH^- ion, and a number of the derivatives containing P-O-C and P-S-C bonds.

It was concluded from kinetic data that the reaction between water and the mixed esters having the P-O-Alk and P-S-Alk groups includes breaking of the C-O bond. Further, the effect of $p_\pi - d_\pi$ interaction between phosphorus and thiol sulfur on reactivity is insignificant. The linear relationship between the rate constants of hydrolysis for the mixed thiolphosphonates and fluorides of phosphorus-containing acids of similar structure was shown.

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USSR

UDC 542.91:661.718.1

VASYANINA, M. A., KHAYRULLIN, V. K., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Substituted 2,5-Dioxo-1,2-oxaphospholanes With Mercaptans"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 8, Aug 71, pp 1722-1726

Abstract: The reactions of substituted 2,5-dioxo-1,2-oxaphospholanes with mercaptans give a mixture consisting of two isomers, viz. ethyl- or phenyl-(β-carbalkylthioalkyl)phosphinic acids and thioalkyl esters of ethyl- or phenyl-β-carboxyalkylphosphinic acids. The reactions to a significant extent proceed contrary to the principle of strong and mild acids and bases with attack on the carbonyl group by the mercaptan, probably because of steric hindrances.

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USSR

UDC 139.143.43:661.718.1

ISHMAYEVA, E. A., CHERKASOV, R. A., OVCHINNIKOV, V. V., and PUDOVIK, A. N.,
Kazan' State University imeni V. I. Ul'yanov-Lenin

"Dipole Moments of Cyclic Thio- and Dithiophosphonates"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 6, Jun 71, pp 1317-1318

Abstract: The authors determined the dipole moments of cyclic thio- and dithiophosphonates. It is suggested that there is conformational ring mobility.

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USSR

UDC 542.91:661.718.1

KHAYRULLIN, V. K., DMITRIYEVA, G. V., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Substituted Bicyclic Phospholanes"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 6, Jun 71, pp 1249-1254

Abstract: The interaction of 2-ethyl-2,5-dioxo-1,2-oxa-3-phospholene and 3-methyl-2-ethyl-2,5-dioxo-1,2-oxa-3-phospholene with 2,3-dimethyl-1,3-butadiene gives the corresponding diene synthesis reaction adducts. Alcoholysis of 5,6-dimethyl-1-ethyl-1,3-dioxo-2-oxa-1-phosphabicyclo-[3,0,4]-no-5,6-nene goes counter to R. G. PEARSON's rule with the attack of alcohol on the carbon of the carbonyl group of the ring and the formation of 4-ethylhydroxyphosphonyl-5-carbethoxy-1,2-dimethylcyclohexene. This can be explained by the presence of steric factors which hinder the attack on the phosphorus atom by alcohol.

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UDC 542.91:661.718.1

KHAYRULLIN, V. K., DMITRIYEVA, G. V., and FUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Chloromethyldichlorophosphine with Propiolic Acid"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 6, Jun 71, pp 1254-1259

Abstract: The article describes results of a study of the reaction of chloromethyldichlorophosphine with propiolic acid. The reaction gives chloromethyl-β-chloroformyl vinylphosphinic acid chloride, which reacts with alcohol in the presence of triethylamine to give ethyl ester of chloromethyl-β-carbethoxy vinylphosphinic acid. When the latter is treated with PCl_5, of the two ethoxy groups only the ethoxy group at the phosphorus is replaced by a chlorine atom. This can be seen by comparing spectrograms of the initial ester and the resultant chloromethyl-β-carbethoxy vinylphosphinic acid chloride. Heating of chloromethyl-β-chloroformyl vinylphosphinic acid with acetic anhydride gives acetyl chloride and 2-chloromethyl-2,5-dioxo-1,2-oxa-3-phospholene, which readily enters into diene synthesis reaction with 2,3-dimethyl-1,3-butadiene to give 5,6-dimethyl-1-chloromethyl-1,3-dioxo-2-oxa-1-phosphabicyclo-[3,0,4]-no-5,6-nene. In the alcoholysis of 2-chloromethyl-2,5-dioxo-1,2-oxa-3-phospholene

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KHAYRULLIN, V. K., et al., Izvestiya Akademii Nauk SSR, Seriya Khimicheskaya, No 6, Jun 71, pp 1254-1259

the attack by alcohol is directed against the phosphorus atom, while in the alcoholysis of 5,6-dimethyl-1-chloromethyl-1,3-dioxo-2-oxa-1-phosphabicyclo-[3,0,4]-no-5,6-nene the attack by alcohol, contrary to the ideas developed by R. G. PEARSON, is directed against the carbon atom of the carbonyl group to give 4-chloromethylhydroxyphosphinyl-5-carbethoxy-1,2-dimethylcyclohexene, probably as a result of steric factors.

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UDC 542.944 + 546.14 + 661.718.1

PASHINKIN, A. P., GAZIZOV, T. Kh., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences, USSR

"Ethyl o-Benzoylenephosphite Reaction With Bromine"

Moscow, Izvestiya Akademii Nauk USSR, Seriya Khimicheskaya, No 2, Feb 71, pp 437-439

Abstract: The reaction of ethyl o-benzoylenephosphite with bromine follows the Arbuzov reaction, forming ethyl o-bromoformylphenylphosphonic acid bromide which then decomposes to ethyl bromide and o-benzoylenephosphonic acid bromide. Bromine was added slowly to ethyl o-benzoylenephosphite, the temperature was then brought up to 100°, ethyl bromide was evaporated, the residue treated with an equimolar mixture of ethanol and triethylamine in benzene. The mixture was refluxed in benzene for 2 hrs, filtered and ethyl o-benzoylenephosphate isolated by distillation. When the same reaction was carried out at temperatures below -10°C, the product was diethyl o-carbetnoxyphenylphosphate, b.p. 124-125°/0.006 mm, d_4^{20} 1.1893, n_D^{20} 1.4843.

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USSR

UDC 541.127.3:542.938:547.26'118

BEL'SKIY, V. Ye., BEZZUBOVA, N. N., AKAMSIN, V. D., YELISEYENKOV, V. N., RIZPOLOZHENSKIY, N. I., and PUDOVIK, A. N., Corresponding Member of the USSR Academy of Sciences

"Reactivity of Phosphonic Acid Esters and Their Thio-Analogs in Alkaline Hydrolysis"

Moscow, Doklady Akademii Nauk SSSR, Vol 197, No 1, March-April 1971, pp 85-87

Abstract: Nucleophilic substitution on the phosphorous atom decreases on replacement of the phosphoryl oxygen by sulfur. This was established as a result of studying the kinetics of hydrolysis of various esters and acid chlorides of phosphoryl oxygen. However, it still remained unclear whether the reaction rate changed to an equal extent on replacement of the P=O by P=S for a series of compounds with close structure. This paper contains a study of the kinetics of alkaline hydrolysis of some esters and thio esters of substituted phosphonic and thiophosphonic acids. The data obtained permits the conclusion to be drawn that the change in reactivity on converting from phosphoryl to thiophosphoryl compounds essentially depends on the nature of the substitutions on the phosphorus, and a decrease in the reaction rate constant does not

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BEL'SKIY, V. Ye., et al, Doklady Akademii Nauk SSSR, Vol 197, No 1, March-April 1971, pp 85-87

always occur. The mechanism of this effect is discussed, the experimental procedure is presented, and tables of values are given for the activation energy E and the pre-exponential factor A in the equation $K = A \exp(-E/RT)$ for the reaction with OH^--ion, and the hydrolysis rate constants of KOH.

A definite tendency toward an increase in absolute magnitude of the pre-exponential factor is noted on the replacement of oxygen atoms by sulfur in phosphonates. If it is considered that nucleophilic substitution on the phosphorus requires the formation of a pd-bond in the activated complex, the increase in the pre-exponential factor indicates high availability of the d-orbitals of phosphorus in the thio-derivatives for nucleophilic attack.

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USSR

UDC 547.26'118

PUDOVIK, A. N., CHERKASOV, R. A., and SHERGINA, I. V., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Reactions of the Ammonium Salts of Dithiophosphonates With Dialkoxyphosphanemethylchloromethyl Esters"

Leningrad, Zhurnal Obshchey Khimii, Vol 41 (103), No 3, Mar 71, pp 708-709

Abstract: The reaction of dialkoxyphosphanemethylchloromethyl esters with ammonium salts of 0,0-dialkyldithiophosphoric acids and polyalkyleneglycoldithiophosphates was studied. The reaction occurred easily at 60-70° in alcohol solution yielding α'-diethoxyphosphane-α-diethoxydithiophosphoryldimethyl ester, b.p. 160-160°/1 mm, d_4^{20} 1.2026, n_D^{20} 1.4898; α-diethoxyphosphane-α'-diisopropoxydithiophosphoryldimethyl ester, b.p. 153-54°/0.6 mm, d_4^{20} 1.1934, n_D^{20} 1.4915; polydiethyleneglycol-S-methyloxymethyldiethoxyphosphoryldithiophosphate, and its isopropyl permutations — all yellow resins.

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USSR

UDC 547.241

PUDOVIK, A. N., GUR'YANOVA, I. V., ROMANOV, G. V., and LAPIN, A. A., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Reaction of Diphenylphosphine With Methyl Ester of Pyruvic Acid"

Leningrad, Zhurnal Obshchey Khimii, Vol 41 (103), No 3, Mar 71, pp 709-710

Abstract: Diphenylphosphine was added dropwise to an excess of methyl pyruvate, the mixture heated to 45-50°, and then cooled. After several days crystals appeared and were recrystallized from hexane to give (α-hydroxy-α-carbomethoxy)-ethyldiphenylphosphine, m.p. 126-128°. In the temperature range 129-148 the production isomerised:

$$(C_6H_5)_2\overset{O}{\overset{\|}{P}}-\overset{OH}{\overset{|}{\underset{CH_3}{\underset{|}{C}}}}-COOCH_3 \xrightarrow{t^\circ} (C_6H_5)_2-\overset{O}{\overset{\|}{P}}-O-\overset{CH_3}{\overset{|}{CH}}-COOCH_3$$

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USSR

UDC 547.512+547.341+547.772.2

PUDOVIK, A. N., GAREEV, R. D., RAYEVSKAYA, O.E., Kazan State University imeni V. I. Ul'yanov Lenin

"Synthesis and Properties of 3-Dialkoxyphosphono-5,5-diphenyl-Δ^1-and Δ^2-pyrazolines"

Leningrad, Zhurnal Obshchei Khimii, Vol 40, No 6, Jun 70, pp 1189-1195

Abstract: The reactions of diphenyldiazomethane (I) with diethyl and dimethyl vinylphosphonates were studied. Whereas in the reaction with diethyl vinylphosphonate the Δ^2-pyrazoline derivative, namely 3-diethoxy-phosphono-5,5-diphenyl-Δ^2-pyrazoline, is obtained, the Δ^1-analog is obtained when dimethyl vinylphosphonate reacts with I. The characteristic P-O-CH_3 IR bands at 1030, 1060, and 1190 cm^{-1} are observed together with the characteristic bands of the P=O group, the C-H bonds of the benzene rings, and the N=N bond. The Δ^1-pyrazolines are easily isomerized into the thermodynamically more stable Δ^2-pyrazolines. Strong bases and acids catalyze this transformation. Thus, the reaction of dimethyl vinylphosphonate with I in acetonitrile at room temperature yields within 1 hr the Δ^2-pyrazoline derivative in 70.1% yield. It could be shown in a special experiment that the Δ^1-pyrazoline is completely transformed into the Δ^2-pyrazoline within 10 hours at room temperature.

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PUDOVIK, A. N., et al, Zhurnal Obshchei Khimii, Vol 40, No 6, Jun 70, pp 1189-1195

if triethylamine is present. The Δ^1-pyrazoline, when heated for half-an-hour at 75°, was completely decomposed to yield quantitatively N_2 and 1-dimethoxyphosphono-2,2-diphenylcyclopropane (II) (92.5%), whereas the Δ^2-pyrazoline derivative remains unchanged under these conditions and is transformed into II only after heating to 160-170°. This indicates that the reaction of diphenyldiazomethane with vinylphosphonates yields first Δ^1-pyrazolines which in a second stage can be isomerized to the corresponding Δ^2-pyrazolines or which can be decomposed to N_2 and the corresponding cyclopropanes. The presence of the cyclopropane ring could be shown by NMR spectra. IR spectral studies showed that the Δ^2-pyrazolines form associations as a result of intermolecular hydrogen bonds.

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UDC 547.341'139.81+547.391

PUDOVIK, A. N., BATYYEVA, E. S., SHAGIDULLIN, R. R., RAYEVSKIY, O. A., ~~PUDOVIK, M.~~ A., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Amides of Diphenylphosphinous Acid with α, β-Unsaturated Acids"

Leningrad, Zhurnal Obshchei Khimii, Vol 40, No 6, Jun 70, pp 1195-1202

Abstract: The mechanism of the reaction of diphenylphosphinous acid amides with α, β-unsaturated acids was investigated. Protonation of the nitrogen atom of the amide leads to the formation of an amine and subsequently the amide of the unsaturated acid and siphenylphosphinous acid. Association of the latter leads to the final product, namely β-carbamoyl-alkyl-or alkenyl-diphenylphosphine oxide, depending on whether an α, β-unsaturated acid of the ethylene or acetylene series was used. The proposed mechanism was confirmed by IR spectrometry.

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UDC 547.391.1+546.183.325

GAZIZOV, T. Kh., PUDOVIK, A. N. Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov Academy of Sciences USSR

"Reaction of Dialkyl Acylphosphites with Acrylic Acid"

Leningrad, Zhurnal Obshchei Khimii, Vol 40, No 6, Jun 70, pp 1202-1205

Abstract: The reaction mechanism of mixed anhydrides of dialkyl phosphites and carboxylic acids with α,β-unsaturated carboxylic acids was studied. Initial attack of the proton from the α,β-unsaturated acid on the P atom was found to be the determining step. Diethyl phosphite can react only in this way with the mixed anhydride of acrylic and acetic acid. In the presence of a small amount of triethyl phosphite, the reaction leads to the formation of the mixed anhydride of β-(diethylphosphono) propionic acid and acetic acid which subsequently disproportionates to β-(diethylphosphono)propionic acid anhydride and acetic anhydride. $(C_2H_5O)_2POCOCH_2=CH_2$ reacted with acetic acid according to the same mechanism to form diethyl phosphite and the anhydride of β-(diethylphosphino)propionic acid.

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UDC: 547.26'118

PUDOVIK, A. N., GUR'YANOVA, I. V., KAKURINA, V. P., GOL'DFARB, E. M., Kazan' State Institute imeni V. I. Ul'yanov-Lenin

"On the Reaction of Dialkylchlorophosphites With Pyroracemic Acid Esters"

Leningrad, Zhurnal Obshchey Khimii, Vol 40 (102), No 11, Nov 70, pp 2374-2376

Abstract: The authors study reactions of methyl and ethyl pyroracemates with diethyl- and dibutylchlorophosphates. Various proportions of the reagents were reacted at temperatures ranging from 0 to 100°C. The reaction is most complete when the reagents are taken in an equimolecular ratio and reacted with moderate heating or allowed to stand at room temperature for a considerable length of time. Colorless viscous liquids are formed with quantitative release of alkyl chlorides. Elementary analysis and the molecular weight of the resultant products indicate that two molecules of pyroracemate and two molecules of dialkylchlorophosphite participate in the synthesis. Two molecules of alkyl chloride are given off during the reaction. On the basis of these considerations, it is concluded that the reactions produce cyclic phosphonates -- 3,6-dimethyl-3,6-dicarbalkoxy-1,4-dialkoxy-2,5-dioxa-1,4-diphosphorinanes. Formation of intermediate products was investigated by studying the P^{31} NMR spectra.

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UDC 547.26'118

GAZIZOV, T. KH., PASHINKIN, A. P., PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Diethylacetylphosphite Reaction With Amines"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 9, Sep 70, p 2130

Abstract: Reaction of diethylacetylphosphite (I) with diethylamine (II) carried out in petroleum ether at -5° gave diethylammonium acetate and diethylphosphorous acid diethylamide when the reagent ratio was 1:3. A 1:1 ratio of (I) to (II) gave a mixture of diethylphosphorous acid (III) and diethylamide of acetic acid. Reaction of (I) with dibutylamine is also dependent on the ratio of reagents. When aniline was reacted with (I), acetanilide and (III) were formed, but when the reaction was carried out in the presence of triethylamine, the anilide of (III) was obtained. It is proposed that the reaction between dialkylacylphosphites and amines is a reversible reaction.

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USSR

UDC 547.26'118:541.124

PUDOVIK, A. N., CHERKASOV, R. A., KUTYREV, G. A., SAMITOV, YU. YU., MUSINA, A. A., GOL'DFARB, E. I., Kazan' State University imeni V. I. Ul'yanov-Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Reactivity of Phosphorus Dithioacids in Reactions With Acrylonitrile"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 9, Sep 70, pp 1982-1988

Abstract: This paper is concerned with the effect of substituents A and B in phosphorus dithioacids of the type ABP(S)SH on reaction rates with 1,3-conjugated reagents such as acrylonitrile. It was shown by means of kinetic measurements and differential-thermal analysis that the reactivity of phosphorus dithioacids with acrylonitrile increases in the order dithiophosphinates, dithiophosphonates, and dithiophosphates. A linear correlation lg k to the total values of substituents $\sum\sigma_\phi$ was determined. Through the use of NMR (P31) spectroscopy, it was found that reactivity of

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PUDOVIK, A. N., et al, Zhurnal Obshchey Khimii, Vol 40, No 9, Sep 70, pp 1982-1988

dithioacids increases with increase in the total number of electrons in the d orbitals of the phosphorus atom. It was also determined that the dominant role of the effect of the substituent conjugation at the dithioacid phosphorus atom in the transitional state is determined by the reactivity of dithioacids of the phosphate and phosphonate structures.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR

UDC: 547.26+547.233+546.185.325

PASHINKIN, A. P., GAZIZOV, T. Kh., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan', Academy of Sciences USSR

"Rupture of the Phosphorus-Carbon Bond in Alpha-Ketophosphonates"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 28-30

Abstract: Reactions of esters alpha-ketophosphonic acid (I), where R is methyl, isopropyl, ter. butyl, or phenyl, with nucleophilic agents, such as alcohols and amines, proceed under relatively mild conditions and result in rupture of the P-C bond. Nucleophilic substitution is thought to be the mechanism of the bond rupture. The composition of the reaction products is greatly affected by the ratio of the reactants. Thus, heating a 1:10 mixture of (I) with ethanol without a catalyst, gave diethylphosphorous acid (yield 92%), and ethyl acetate. The same mixture but in the presence of 3-4 drops triethylamine gave at room temperature an 85% yield of ethanol and ethyl acetate. Adding 2-3 drops of triethylamine to a 1:1 mixture of (I) and ethanol gave diethylphosphorous acid (yield 31.9%) and diethyl (alpha-diethylphosphon-ethyl) phosphate (II) (yield 55.5%). Apparently, II was formed by phosphonate-phosphate isomerization under the influence of the basic catalyst. Formation

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PASHINKIN, A. P., et al, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 28-30

of (II) may be minimized by the presence of a large excess of the nucleophilic agent. The reactions with primary aliphatic amines proceed similarly but much easier, and the exothermic effect is significantly higher than with ethanol. Dialkylphosphorous acid and an amide of the corresponding carboxylic acid are formed. Formation of (II) was also observed in these reactions. The reactions with aniline are more complex. Dialkylphosphorous acid and anilides of carboxylic acids were obtained in low yields only.

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USSR

UDC: 547.26+547.233+546.185.325

PASHINKIN, A. P., GAZIZOV, T. Kh., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan', Academy of Sciences USSR

"Rupture of the Phosphorus-Carbon Bond in Alpha-Ketophosphonate Esters"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 28-30

Abstract: Reactions of alpha-ketophosphonate esters (I), where R at P is methyl, isopropyl, tert-butyl, or phenyl with nucleophilic agents, such as alcohols and amines, proceed under relatively mild conditions and result in rupture of the P-C bond. Nucleophilic substitution is thought to be the mechanism of the bond rupture. The composition of the reaction products is greatly affected by the ratio of the reactants. Adding 2-3 drops of triethylamine to a 1:1 mixture of (I) and ethanol gave diethylphosphorous acid (yield 31.9%) and diethyl alpha-diethylphosphonethyl phosphate (II) (yield 55.5%). Apparently, II was formed by phosphonate-phosphate isomerization under the influence of the basic catalyst. Formation of (II) may be minimized by the presence of a large excess of the nucleophilic agent. The reactions with primary aliphatic amines proceed similarly but much easier, and the exothermic effect is significantly higher than with ethanol. Dialkylphosphorous acid and an amide of the corresponding carboxylic acid are formed.

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PASHINKIN, A. P., et al, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 28-30

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USSR

UDC: 547.391+547.398.1'118

PUDOVIK, A. N., PUDOVIK, M. A., and TERENT'YEVA, S. A.

"Reaction of Amides of Dialkylphosphorous and Alkylarylphosphinous Acids with alpha, beta-Unsaturated Carboxylic Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 33-36

Abstract: The mixture of acrylic or methacrylic acid and N,N-diethylethylphenylphosphonous amide kept below 90° for one day gave 35 or 38% yields, respectively, of beta-(N,N-diethylcarbamyl)ethyl(isopropyl)-ethylphenylphosphine oxides, liquids identified by their physical constants and IR spectra. Diethylamides of P,P-dialkyl phosphonylpropionic(isobutyric) acids (I) were the main products of the reaction of N,N-diethylamidodialkylphosphite with acrylic or methacrylic acids, respectively. The reaction products contained also a maximum 5% admixture of ethyl P,P-dialkylamidophosphonylpropionate (isobutyrate) (II) which were detected by IR spectra and gas-liquid chromatography. Pure I, where alkyl = ethyl, was obtained in 68% yield by heating at 150° triethyl phosphite with N,N-diethyl-beta-bromopropionamide. Pure II, where alkyl=ethyl, was prepared in 71% yield from N,N-diethylamidodiethylphosphite and ethyl beta-bromopropionate. Physical constants and IR spectrum of pure II differed from those of the amide I, prepared by the reaction of N,N-diethylamidodiethylphosphite with acrylic acid.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR

UDC 542.91 + 661.718.1

PUDOVIK, A. N., KHAYRULLIN, V. K., and KONDRAT'YEVA, R. M., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Synthesis of 5-Methyl-2-ethyl-4-chloro-2-keto-1,2-oxa-4-phospholene"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 11, Nov 70, pp 2548-2553

Abstract: The article reports the counter synthesis of 5-methyl-2-ethyl-4-chloro-2-keto-1,2-oxa-4-phospholene (III). Chlorination of 5-methyl-2-ethyl-2-keto-1,2-oxa-4-phospholene (I) gives 5-methyl-2-ethyl-4,5-dichloro-2-keto-1,2-oxaphospholane (II), which is readily dehydrochlorinated with triethylamine in benzene to give III. Subsequent conversion of III gives the ethyl ester of ethyl-(β-chloro-γ-keto-butyl)phosphinic acid (IV) and 5-methyl-2-ethyl-4,4,5-trichloro-2-keto-1,2-oxaphospholane (V). V is readily dehydrochlorinated on treatment with 2 M triethylamine in benzene to give triethylamine hydro-

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USSR

PUDOVIK, A. N., et al., Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 11, Nov 70, pp 2548-2553

chloride and 2-ethyl-5-methylene-4-chloro-2-keto-1,2-oxa-3-phospholene (VI).

The reaction of ethyl- or phenyldichlorophosphine with methyl isopropenyl ketone in the presence of acetic annydride gives 4,5-dimethyl-2-ethyl-(VII) and 4,5-dimethyl-2-phenyl-2-keto-1,2-oxa-4-phospholenes (VIII). VII and VIII are readily chlorinated at room temperature to give oxaphospholanes, which react with 2 M triethylamine in an inert solvent to give 4-methyl-2-ethyl-(IX) and 4-methyl-2-phenyl-5-methylene-2-keto-1,2-oxa-3-phospholene (X). IX and X readily react with water or alcohols to give phosphorylated α,β-unsaturated ketones.

The IR spectra for the various products are described.

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USSR

UDC 547.26'118

MURATOVA, A. A., YARKOVA, E. G., PLEKHOV, V. P., MUSINA, A. A., RUDOVIK, A. N., Kazan' State University imeni V. I. Ul'yanov-Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Study of the Complexes of Tin Halides With Incomplete Esters of Ethylphosphonous and Dialkylphosphinous Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 9, Sep 70, pp 1978-1982

Abstract: Complexes of tin tetrachloride, tetrabromide, and tetraiodide with methyl, ethyl, n-propyl, isopropyl, and n-butyl esters of ethylphosphonous acid and of tin tetrachloride with di-n-butyl, di-n-hexyl and di-n-octylphosphonous acids were synthesized and studied by IR spectroscopy. Comparison of the IR spectra of phosphonous and phosphinous acids and their complexes with tin tetrahalides in the range of 400-4000 cm^{-1} shows that the type of changes observed is analogous to spectral changes of the dialkylphosphorous acids and their complexes. Consequently, all of them participate in these complexes in their pentavalent state. It was

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MURATOVA, A. A., et al, Zhurnal Obshchey Khimii, Vol 40, No 9, Sep 70, pp 1978-1982

shown that the coordination bond is through the oxygen of the phosphoryl group. The P-H bond was found to be sensitive both to the changes in the structure of the addendum and towards the ability of tin halides to act as electron acceptors.

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USSR

UDC 546.183.325 + 547.446.26'118

PASHINKIN, A. P., GAZIZOV, T. KH., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Avademy of Sciences USSR

"Some Reactions of Mixed Anhydrides of Carboxylic and Dialkylphosphorous Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 7, Jul 70, pp 1481-1485

Abstract: The authors studied the interaction of chloral with mixed anhydrides of diethylphosphorous acid and formic, isobutyric, pivalic and acrylic acids, as well as the mixed anhydride of diisopropylphosphorous and acetic acids. It was found that the reaction of chloral with the mixed anhydride of diethylphosphorous acid and formic acid follows an Arbuzov reaction scheme to give the ester of formic acid and diethoxyphosphonotrichloromethylcarbinol. The reactions with the other mixed anhydrides proceed analogously. The mixed anhydride of diethylphosphorous acid and benzoic acid or its p-substituted derivatives reacts with chloral in two directions, viz. according to the Ar-

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PASHINKIN, A. P., et al., Zhurnal Obshchey Khimii, Vol 40, No 7, Jul 70, pp 1481-1485

buzov scheme and the Perkov scheme. If a methyl group possessing a positive inductive effect is introduced into the p-position, there is an increase in the yield of the Perkov scheme product and a decrease in the Arbuzov scheme product. The introduction of a nitro group possessing a negative inductive effect directs the reaction completely towards the formation of a phosphonate.

The reactions of the mixed anhydrides of dialkylphosphorous and carboxylic acids with iodine, bromine and acetyl halides were studied. It was found that the reactions of diisopropyl acetylphosphite and diethyl benzoylphosphite with bromine at a low temperature proceed according to the Arbuzov scheme to give carboxylic and dialkylphosphoric acid halides, which subsequently interact to give an alkyl halide.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR UDC 547.26'118

PUDOVIK, A. N., PUDOVIK, M. A., SHULYNDINA, O. S., and NAGAYEVA, KH. KH., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"2-Substituted N-Phenyl(benzyl)-1,3,2-oxaazaphospholanes"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 7, Jul 70, pp 1477-1480

Abstract: The interaction of phosphorus trichloride with β-phenyl-(benzyl)aminoethanol gives 2-chloro-N-phenyl(benzyl)-1,3,2-oxaazaphospholane. These acid chlorides readily react with alcohols, secondary amines to form corresponding amides and esters. The same products are obtained by a transesterification reaction -- transamidation of some trivalent phosphorus acid derivatives. Thus, heating of hexaethyltriaminophosphine with β-phenylaminoethanol in a benzene solution gives 2-diethylamino-N-phenyl-1,3,2-oxaazaphospholane. The latter on heating with alcohol readily becomes 2-ethoxy-N-phenyl-1,3,2-oxaazaphospholane.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR UDC 547.26'118

FUDOVIK, A. N., GUR'YANOVA, I. V., and RAKHMATULLINA, L. Kh., Kazan' State University imeni Ul'yanov-Lenin

"Reactions of Dialkyl Anilidophosphites with Esters of Pyruvic and Glyoxylic Acid"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 7, Jul 70, pp 1485-1489

Abstract: The authors studied reactions of the methyl, ethyl, butyl esters of pyruvic acid and the butyl ester of glyoxylic acid with diethyl and dibutyl anilidophosphites and diethyl-p-anisidino- and diethyl-p-toluidinophosphites at -5° and +100° with an equimolecular reagent ratio or with an excess of the carbonyl compound. It was found that, regardless of the reaction temperature or the reagent ratio, the reactions give O, O-dialkyl-O- α-carbalkoxyalkyl-N-phenylimidophosphates.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR UDC 547.398 + 661.718.1

PUDOVIK, A. N., TERENT'YEVA, S. A., PUDOVIK, M. A., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Complete Phosphites With Amides of α, β-Unsaturated Carboxylic Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 8, Aug 70, pp 1707-1711

Abstract: The reaction of tertiary phosphorites with unsubstituted amides of α, β-unsaturated carboxylic acids occurs with participation of the amide group, yielding dialkyl esters of N-alkylcarbamoylalkylphosphonates and carbamoylalkylphosphonates. A mixture of 8.5gm methacrylamide and 16.6gm triethyl phosphite was refluxed for 3 hrs at 140°, yielding 2,3 diethyl-N-ethylcarbamoylisopropylphosphonate, b.p. 143-145/0.05 mm, d_4^{20} 1.0954, n_D^{20} 1.4600. When 14.2gm acrylamide was reacted with 33.2gm triethylphosphite for 2 hrs at 130°, followed by another hour at 150°, a complex mixture of products was obtained, which beside some unreacted material and

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PUDOVIK, A. N., et al, Zhurnal Obshchey Khimii, Vol 40, No 8, Aug 70, pp 1707-1711

alcohol contained a mixture of diethyl-β- cyanoethylphosphonate and diethyl-β-carbethoxyethylphosphonate, diethyl-N-ethylcarbamoylethylphosphonate, b.p. 135-136°/0.02 mm, d_4^{20} 1.1205, n_D^{20} 1.4590 and solid diethylcarbamoylethylphosphonate, m.p. 76.5-78°. Reacting a mixture of acrylamide, diethylphosphite and triethylphosphite yielded diethyl-β-cyanoethylphosphonate, d_4^{20} 1.1081, n_D^{20} 1.4381 and diethylcarbamoylethylphosphonate m.p. 74-75°.

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USSR

PUDOVIK, A. N., ISHMAYEVA, E. A., SHARGINA, I. V., Kazan University

"A Method for Preparing 2,6-bis-(dialkoxyphosphono)-dioxanes-1,4"

USSR Author's Certificate No 253804, class 12o, 26/01 (C 07 f), filed 24 Jan 68, published 3 Mar 70 (from RZh-Khimiya, No 21 (II), 10 Nov 70, Abstract No 21 N599 P by G. V. Kuznetsova)

Translation: These substances, potentially useful as fungicides, are obtained by reaction of disodium derivatives of bis-(dialkoxyphosphono)-dimethyl ester with dihaloiddimethyl esters; 3.34 g of Na is gradually added to a solution of 23.1 g of $(EtO)_2$-$P(O)CH_2OCH_4$ $(P(O)(OEt)_2$ in 100 ml of absolute ether. After all the Na reacts, the small amount of excess $(ClCH_2)_2O$ is added to the mixture a drop at a time. The precipitated NaCl is filtered out and 2,6-bis-(diethoxyphosphono)-dioxane-2,4 (boiling point 154°-7°/1, d_4^{20} 1.1632, $n^{20}D$ 1.4360) is formed from the filtrate.

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USSR UDC: 547.183.325+547.387

GAZIZOV, T. Kh., PASHINKIN, A. P., and PUDOVIK, A. N.

"Thermal Isomerization of a Mixed Anhydride of Diethylphosphorous and Acrylic Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 1, Jan 70, pp 31-32

Abstract: An investigation of the product of thermal isomerization of the anhydride confirmed the formation of 1,3-di(diethoxyphosphonyl)propenyl acrylate (I) according to the proposed two-step reaction scheme. Presumably, (diethoxy)phosphonylmethylketene (II) formed in the first step of isomerization subsequently reacts with the starting anhydride to give I. Heating the final product of thermal isomerization of the title anhydride with excen absolute ethanol and two drops triethylamine gave 48% diethylphosphorous acid and 61.3% ethyl beta-diethylphosphonylpropionate. The result was explained as alcoholysis of the ester function in I followed by breaking of the P-C bond in the presence of the ethoxide anion. Passing ketene through the title anhydride at 20° yielded about 60% alpha-diethoxyphosphonylvinyl acrylate, identified by its physical constants and IR spectrum. The above reactions were seen as confirmation of the proposed thermal isomerization scheme of the mixed anhydride.

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1/2 015 UNCLASSIFIED PROCESSING DATE--27NOV70

TITLE--REACTION OF DIETHYL PHOSPHITE WITH ACETOACETIC ESTER -U-

AUTHOR-(03)-PUDOVIK, A.N., ZIMIN, M.G., SOBANOV, A.A.

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(4), 936-7

DATE PUBLISHED-------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--ALKYL PHOSPHITE, ACETOACETATE, CHEMICAL DECOMPOSITION, CARBONYL COMPOUND, HYDROXYL RADICAL, ORGANIC PHOSPHORUS COMPOUND

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--3006/1518 STEP NO--UR/0079/70/040/004/0936/0937

CIRC ACCESSION NO--AP0135179

UNCLASSIFIED

APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 015 UNCLASSIFIED PROCESSING DATE--27NOV70

CIRC ACCESSION NO--AP0135179

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. REACTION OF ACCH SUB2 CO SUB2 ET WITH (ETO) SUB2 PHO IN THE PRESENCE OF ETONA FAILED TO YIELD IDENTIFIABLE PRODUCTS, BUT THE SAME REACTION RUN AT ROOM TEMP. WITH ET SUB2 NH AS THE BASE GAVE 95PERCENT (ETO) SUB2 P(O)CME(OH)CH SUB2 CO SUB2 ET (I), M. 46DEGREES, WHICH WITH ACCL AR AC SUB2 O AND A CATALYTIC AMT. H SUB2 SO SUB4 GAVE 96PERCENT CORRESPONDING ACETATE, B SUB10 169-70DEGREES, N PRIME20 SUBD 1.4425, D PRIME10 1.1447. AN ATTEMPT TO VERIFY THE EARLIER REPORT OF DEHYDRATION OF (RO) SUB2 P(O)CH(R)OH (US 2,579,810) SHOWED THAT LOSS OF WATER FROM THEM UNDER THE REPORTED CONDITIONS DID NOT OCCUR, BUT THAT THE ESTERS SMOOTHLY DECOMPD. INTO THE CARBONYL COMPD. AND (RO) SUB2 PHO; DEHYDRATION ALSO FAILED FOR (RO) SUB2 P(O)NHCH(OH)CCLS UB3, BUT HYDROXYPHOSPHONATES IN WHICH A CH SUB2 GROUP, ACTIVATED BY AN ELECTRON ACCEPTOR GROUP, IS PRESENT DID UNDERGO AT LEAST PARTIAL DEHYDRATION. THUS (ETO) SUB2 P(O)CME:CHCO SUB2 ET, 10PERCENT, B SUB15 156-70DEGREES, 1.4500, 1.0936, WAS FORMED FROM I BY HEATING WITH A TRACE OF PIPERIDINE IN C SUB6 H SUB6. THE SAME PRODUCT FORMED IN 63PERCENT YIELD FROM I ACETATE ON HEATING WITH DRY NA SUB2 CO SUB3.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 013 UNCLASSIFIED PROCESSING DATE--30OCT70

TITLE--ALPHA,OXOPHOSPHONATES IN REACTIONS WITH DIPHENYLDIAZOMETHANE -U-

AUTHOR-(03)-PUDOVIK, A.N., GAREYEV, R.D., STABROVSKAYA, L.A.

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(3), 698

DATE PUBLISHED----------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--HETEROCYCLIC OXYGEN COMPOUND, BENZENE DERIVATIVE, POLYNUCLEAR HYDROCARBON, ORGANIC PHOSPHORUS COMPOUND, AZO COMPOUND, ORGANIC SYNTHESIS

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--2000/0895 STEP NO--UR/0079/70/040/003/0698/0698

CIRC ACCESSION NO--AP0124558

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APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

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2/2 013 UNCLASSIFIED PROCESSING DATE--30OCT70

CIRC ACCESSION NO--AP0124558

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. REACTION OF (MEO) SUB2 P(O)AC AND PH SUB2 CH SUB2 OCCURRED RAPIDLY AT 80DEGREES TO GIVE 63PERCENT I AND N SUB2; I, M. 87-8DEGREES. SIMILAR REACTION WITH (MEO) SUB2 P(O)BZ GAVE 83PERCENT II, M. 129-30DEGREES.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 012 UNCLASSIFIED PROCESSING DATE--30OCT70

TITLE--DIETHOXY PROPYNYLPHOSPHONATE IN 1,3,DIPOLAR ADDITION REACTIONS -U-

AUTHOR-(02)-PUDOVIK, A.N., KHUSAINOVA, N.G.

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(3), 697

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--HETEROCYCLIC NITROGEN COMPOUND, ORGANIC PHOSPHORUS COMPOUND, ETHYL ETHER, KETONE

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--2000/0872 STEP NO--UR/0079/70/040/003/0697/0697

CIRC ACCESSION NO--AP0124535

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APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 012 UNCLASSIFIED PROCESSING DATE--30OCT70

CIRC ACCESSION NO--AP0124535

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. HEATING N,PHENYLSYDNONE WITH (ETO)SUB2 P (O)C:CME 10 HR AT 150-60DEGREES GAVE 53PERCENT I, B SUB0.03 158-61DEGREES, D PRIME20 1.1692, N SUBD PRIME20 1.5335, PROBABLY FORMED BY ELIMINATION OF CO SUB2 FROM INTERMEDIATE II. (FORMULA SHOWN ON MICROFICHE). FACILITY: KAZAN. GOS. UNIV., KAZAN, USSR.

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APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 016 UNCLASSIFIED PROCESSING DATE--02OCT70

TITLE--REACTION OF AMIDOPHOSPHITES WITH AMMONIUM SALTS OF ACIDS -U-

AUTHOR-(04)-YELISEYENKOV, V.N., PUDOVIK, A.N., FATTAKHOV, S.G., SERKINA, N.A.

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(2) 498

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--AMINE DERIVATIVE, AMMONIUM SALT, HYDROGEN CHLORIDE, ORGANIC PHOSPHORUS COMPOUND, TRIETHYLAMINE

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1992/1570 STEP NO--UR/0079/70/040/002/0498/0498

CIRC ACCESSION NO--AP0112564

UNCLASSIFIED

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2/2 016 UNCLASSIFIED PROCESSING DATE--02OCT70

CIRC ACCESSION NO--AP0112564

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. MIXING 12.4 G P(NET SUB2) SUB3 (I) AND 6.9 G ET SUB3 N.HCL AT 80-90DEGREES IN VACUO (WATER PUMP) GAVE 53PERCENT (ET SUB2 N) SUB2 PCL, B SUB0.02 60DEGREES, N PRIME20 SUBD 1.4900; SIMILARLY 17.35 G I AND 7.7 G ET SUB2 NH.HCL GAVE ET SUB2 NH AND 61PERCENT (ET SUB2 N) SUB2 PCL. AT 40DEGREES, 12.3 G I AND 10.6 G ETNEP(S)CH.ET SUB2 NH GAVE 100PERCENT ET SUB2 NH AND 61PERCENT (ETSUB2 N) SUB2 PCP(S)MEET, B SUB0.007 82-3DEGREES, D PRIME20 1.0368, N PRIME20 SUBD 1.4890. SIMILARLY, 8.5 G ACOH.ET SUB3 N AND 13.1 G (ET SUB2 N)P-(OBU) SUB2 GAVE A MIXT. CONTG. 6.7 G (BEO) SUB2 PHO, 0.9 G (BUO) SUB2 POAC, AND 3.8 G ACNET SUB2. THUS, REACTION OF P(III) AMIDES WITH AMINE SALTS IS REVERSIBLE, AND REMOVAL OF THE AMINE AS FORMED MAY BE USED TO DISPLACE THE EQUIL. IN THE DIRECTION OF FORMATION OF PRODUCTS SHOWN ABOVE BY REMOVAL OF R SUB2 NH.

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1/2 013 UNCLASSIFIED PROCESSING DATE--09OCT70

TITLE--REACTIONS OF DIBUTYL AND DIPHENYLPHOSPHINOUS ACIDS WITH SATURATED AND UNSATURATED CARBOXYLIC ACID CHLORIDES -U-

AUTHOR-(02)-PUDOVIK, A.N., SUDAKOVA, T.M.

D

COUNTRY OF INFO--USSR

SOURCE--DOKL. AKAD. NAUK SSSR 1970, 190(5), 1121-3

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--ORGANIC PHOSPHORUS COMPOUND, ACID CHLORIDE, CHEMICAL REACTION, CARBOXYLIC ACID

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1992/2026 STEP NO--UR/0020/70/190/005/1121/1123

CIRC ACCESSION NO--AT0112981

UNCLASSIFIED

APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

UNCLASSIFIED PROCESSING DATE--09OCT70

2/2 013

CIRC ACCESSION NO--AT0112981

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. THE EASE OF ADDN. OF THE FOLLOWING COMPDS. TO UNSATD. ELECTROPHILIC REAGENTS DECREASES IN THE ORDER: R SUB2 PHO, PH SUB2 PHO, RP(OR)(O)H, AND (RO) SUB2 PHO. THUS, ACCL AND BU SUB2 PHO REACT WITH MUCH HEAT EVOLUTION TO YIELD 24.1PERCENT BU SUB2 P(O)AC (I), B SUB2 62-3.5DEGREES, D PRIME20 0.9319, N PRIME20 SUBD 1.4755, AND 27PERCENT BU SUB2 P(O)CHMEOP(O)BU SUB2 (II), M. 59-60DEGREES. THE LATTER FORMS FROM ADDN. OF BU SUB2 PHO TO I, FORMING PRESUMABLY BU SUB2 P(O)CME(OH)P(O)BU SUB2, WHICH ISOMERIZES UNDER THE REACTION CONDITIONS INTO II. THIS WAS PROVEN BY RUNNING THIS REACTION WITH ISOLATED I AND BU SUB2 PHO. PH SUB2 PHO AND ACCL REACTED WITH LESS HEAT EVOLUTION TO GIVE 21PERCENT PH SUB2 P(O)AC, B SUB2 125-7DEGREES, 1.2083, 1.6319, AND 26PERCENT PH SUB2 P(O)CHMEOP(O)PH SUB2, M. 130-1DEGREES. BU SUB2 PHO AND CH SUB2:CHCLCL REACTED VERY VIGOROUSLY AND GAVE 23PERCENT BU SUB2 P(O)CH SUB2 CH SUB2 C(O)BU SUB2, B SUB2 152-4DEGREES, AND 21PERCENT BU SUB2 P(O)CH(CH:CH SUB2)OP(O)BU SUB2, B SUB1 95-6DEGREES, 1.0354, 1.4665, FORMED PROBABLY FROM AN INTERMEDIATE SUCH AS BU SUB2 P(O)COCH:CH SUB2. BU SUB2 PHO AND CH SUB2:CHCOCL WERE ALLOWED TO REACT UNDER MILD CONDITIONS (6DEGREES IN C SUB6 H SUB6) TO YIELD ONLY THE ADDN. PRODUCT BU SUB2 P(O)CH SUB2 CH SUB2 COCL, WHICH WAS TOO UNSTABLE TO BE ISOLATED, BUT TREATED WITH ETOH GAVE ITS ET ESTER, 42PERCENT, B SUB1 158-60DEGREES, 1.0117, 1.4678. METHACRYLOYL CHLORIDE REACTED SIMILARLY (NO DETAILS). FACILITY: KAZAN. GOS. UNIV. IM. UL'YANOVA-LENINA, KAZAN, USSR.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 021 UNCLASSIFIED PROCESSING DATE--02OCT70

TITLE--SYNTHESIS AND SOME PROPERTIES OF POLY(ALKYLENE GLYCOL S,ALKYL DITHIOPHOSPHATES) -U-

AUTHOR-(03)-PUDOVIK, A.N., CHERKASOV, R.A., SHERGINA, I.V.

COUNTRY OF INFO--USSR

SOURCE--VYSOKOMOL. SOEDIN., SER. A 1970, 12(2), 343-7

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--CHEMICAL SYNTHESIS, POLYMER, GLYCOL, PHOSPHORUS SULFIDE, EXOTHERMIC REACTION, THIOL, ISOMERIZATION, AMMONIUM SALT, COMPLEX COMPOUND, AMINE

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1989/0255 STEP NO--UR/0459/70/012/002/0343/0347

CIRC ACCESSION NO--AP0106911

UNCLASSIFIED

APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

UNCLASSIFIED PROCESSING DATE--02OCT70

2/2 021

CIRC ACCESSION NO--AP0106911

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. THE REACTION OF H(OROP(S)(SH)) SUBN OET (POLY(ALKYLENE GLYCOL DITHIOPHOSPHATES)) (I) WITH 30PERCENT P(OR PRIME) SUB3 (WHERE R PRIME EQUALS ME, ET, ISO,PR, BU, ISO,BU, AND C SUB5 H SUB11) WAS EXOTHERMIC AND GAVE THE H(OROP(S)(SR PRIME)) SUBN OET (II) CONTG. 10.0-16.3PERCENT P. AN ABSORPTION BAND AT 670 CM PRIME NEGATIVE1 (WHICH MAY BE ASSIGNED TO VIBRATIONS OF P:S GROUPS) SUGGESTED THAT A THIONE THIOL ISOMERIZATION MAY HAVE OCCURRED IN THE OLIGOMERIC UNIT DURING TRANSESTERIFICATION. THE REACTION OF I AND II WITH NH SUB3, RNH SUB2, R SUB2 NH, AND R SUB3 N (WHERE R IS ALKYL) WAS ALSO STUDIED. I DISSOLVED IN DIOXANE GAVE WITH NH SUB3 (IN THE COLD) AN AMMONIUM SALT WHICH COULD BE TREATED WITH R PRIME BR TO GIVE II. RNH SUB2, R SUB2 NH, AND R SUB3 N REACTED WITH II TO GIVE COMPLEXES.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 015 UNCLASSIFIED PROCESSING DATE--23OCT70

TITLE--REACTIONS OF ORGANOPHOSPHORUS COMPOUNDS CONTAINING ACTIVE METHYLENE GROUPS WITH METHYL BETA,CHLOROVINYL KETONE AND ALPHA,HALO ETHERS -U-

AUTHOR-(03)-PUDOYIK, A.N., NIKITINA, V.I., KURGUZOVA, A.M.

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(2), 291-4

DATE PUBLISHED-------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--ORGANIC PHOSPHORUS COMPOUND, METHYLENE, CHLORINATED ORGANIC COMPOUND, KETONE, HALOGENATED ORGANIC COMPOUND, ETHER, TRIETHYLAMINE, AMMONIUM SALT, CYANIDE

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1995/1434 STEP NO--UR/0079/70/004/002/0291/0294

CIRC ACCESSION NO--AP0116881

UNCLASSIFIED

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 015 UNCLASSIFIED PROCESSING DATE--23OCT70

CIRC ACCESSION NO--AP0116881

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. TREATING THE K OR NA DERIV. OF APPROPRIATE ACTIVE METHYLENE COMPDS. WITH CLCH:CHAC OVER 1-1.5 HR WITH ICE COOLING GAVE, AFTER HEATING TO COMPLETE THE REACTION, THE FOLLOWING ACCH:CHCRR PRIME R DOUBLE PRIME (R, R PRIME AND R DOUBLE PRIME SHOWN): (FORMULA SHOWN ON MICROFICHE). TREATING THE P COMPD. CONTG. AN ACTIVE METHYLENE GROUP, WITH ET SUB3 N AND EQUIMOLAR AMT. CLCH:CHAC GAVE OVERNIGHT A SOLID MASS WHICH WAS HEATED 8-10 HR IN MEPH TO YIELD THE PRODUCTS OF KETOVINYLATION BY MEANS OF TRIALKYL,ACYLVINYLAMMONIUM SALTS. FOR ALKYLATION WITH HALOETHERS THE K DERIV. OF THE ACTIVE METHYLENE COMPD. WAS TREATED WITH HALOETHER AND HELD 1-2 HR AT ROOM TEMP. FOLLOWING WERE REPORTED: (FORMULA SHOWN ON MICROFICHE).

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR UDC 547.558.1

PUDOVIK, A. N., and YASTREBOVA, G. YE., Kazan' State University imeni V. I. Ulyanov Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Organophosphorus Compounds with Active Methylene Group"

Moscow, Uspekhi Khimii, Vol 39, No 7, Jul 70, pp 1190-1219

Abstract: A review is presented of the present state of the chemistry of organophosphorus compounds with active methylene groups such as esters and nitriles of phosphonacetic acids, phosphonacetone, and other compounds in which the methylene group is directly bound with a phosphoryl and any electronegative group. Compounds of this type are analogs of acetoacetic, malonic, cyanacetic and phenylacetic esters, the importance of which for theoretic and synthetic organic chemistry is well known. In the first part of the review, methods are described for synthesis of phosphorus-containing compounds with active methylene group, and in the second part their chemical properties, associated with the mobility of hydrogen atoms of the methylene group, are discussed. These include alkylation, acylation, addition, condensation, PO-olefination and other reactions such as

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USSR

PUDOVIK, A. N., et al., Moscow, Uspekhi Khimii, Vol 39, No 7, Jul 70, pp 1190-1219

bromination, chlorination and azo coupling.

In the discussion of synthesis of organophosphorus compounds with an active methylene group, details were presented on the following: derivatives of phosphorylated acetic acid and its nitrile; phosphorylated ketones; phosphorylated alkanes; phosphorylated alkenes; derivatives of substituted methylphosphonic acid containing aryl, carbocyclic, and heterocyclic radicals; esters of halogenemethyl-, aminomethyl-, oxymethyl- and alkozymethylphosphonic acids.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 010 UNCLASSIFIED PROCESSING DATE--30OCT70

TITLE--ADDITION OF ALDEHYDES TO ALPHA,OXOPHOSPHONIC ACID ESTERS -U-

AUTHOR-(03)-PUDOVIK, A.N., NIKITINA, V.I., YEVDOKIMOVA, V.V.

P

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(2), 294-8

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--CHEMICAL SYNTHESIS, PHOSPHATE ESTER, ALDEHYDE, BENZENE DERIVATIVE, CHLORINATED ORGANIC COMPOUND

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1992/1750 STEP NO--UR/0079/70/040/002/0294/0298

CIRC ACCESSION NO--AP0112736

UNCLASSIFIED

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 010 UNCLASSIFIED PROCESSING DATE--30OCT70

CIRC ACCESSION NO--AP0112736

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. HEATING (RO) SUB2-P(O) AC OR (RO) SUB2 P(O)BZ WITH SLIGHT EXCESS ALDEHYDE AND A TRACE OF PIPERIDINE IN MEPH OR C SUB6 H SUB6 20-5 HR GAVE THE FOLLOWING ADDUCTS: SHOWN ON MICROFICHE.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 025 UNCLASSIFIED PROCESSING DATE--23OCT70

TITLE--REACTIONS OF ETHYL AND ARYLALKOXYCHLOROPHOSPHINES WITH PROPIOLIC ACID -U-

AUTHOR-(03)-VASVANINA, M.A., KHAYRULLIN, V.K., PUDOVIK, A.N.

COUNTRY OF INFO--USSR

SOURCE--IZV. AKAD. NAUK SSSR, SER. KHIM. 1970, (2), 452-5

DATE PUBLISHED-------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--HETEROCYCLIC BASE COMPOUND, ORGANIC PHOSPHORUS COMPOUND, BENZENE DERIVATIVE, MERCAPTAN, IR SPECTRUM

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1997/0846 STEP NO--UR/0062/70/000/002/0452/0455

CIRC ACCESSION NO--AP0119750

UNCLASSIFIED

APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2"

"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 025 UNCLASSIFIED PROCESSING DATE--23OCT7C

CIRC ACCESSION NO--AP0119750

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. TO 0.05 MOLE RP(OR PRIME1)CL IN C SUB6 H SUB6 UNDER CO SUB2 WAS ADDED 0.05 MOLE PROPIOLIC ACID SMALLER THAN 30DEGREES, THE MIXT. KEPT 2 HR AT ROOM TEMP., AND 0.05 MOLE ABS. ETOH ADDED OVER 2 HR TO GIVE RP(O)(OR PRIME1)CH:CHCO SUB2 ET (R AND R PRIME1 SHOWN): ET, CCL SUB3 CME SUB3, 33PERCENT B SUB0.04 142-3DEGREES, D PRIME20 1.3039, N PRIME20 SUBD 1.4920; ET, 1,TRICHLOROMETHYLCYCLOPENTYL, 40PERCENT, B SUB0.04 176-8DEGREES, 1.3108, 1.5050; PH, CCL SUB3 CME SUB2, 29PERCENT, B SUB0.04 190-2DEGREES, 1.3249, 1.5400; AND P,MEC SUB6 H SUB4, CCL SUB3 CME SUB2, 29PERCENT, B SUB0.04 192-4DEGREES, 1.3010, 1.5360. REACTION OF RP(OCME SUB2 CCL SUB3)CL WITH PROPIOLIC ACID IN C SUB6 H SUB6 2 HR AT ROOM TEMP., FOLLOWED BY I MOLE BUSH AND KEEPING THE WHOLE 3 HR GAVE THE FOLLOWING RP(O)(OCME SUB2 CCL SUB3)CH:CHCOSBU; ET, 30PERCENT, B SUB0.04 177-9DEGREES, 1.2695, 1.5160; AND PH, 23PERCENT, B SUB0.04 208-10DEGREES, 1.2931, 1.550. TO 16 G PHPCL(OCME SUB2 CCL SUB3) IN C SUB6 H SUB6 UNDER CO SUB2 WAS ADDED 3.5 G PROPIOLIC ACID TO YIELD AFTER 2 HR AT ROOM TEMP. 30PERCENT I, B SUB0.04 153-4DEGREES, 1.3454, 1.5700: IR SPECTRAL CURVES (3) ARE SHOWN. FACILITY: INST. ORG. FIZ. KHIM. IM. ARVUZOVA, KAZAN, USSR.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 010 UNCLASSIFIED PROCESSING DATE--23OCT70

TITLE--SYNTHESIS OF PHENYL,BETA,CHLOROFORMYLVINYL PHOSPHINIC ACID CHLORIDE -U-

AUTHOR-(03)-KHAYRULLIN, V.K., DMITRIEVA, G.V., PUDOVIK, A.N.

COUNTRY OF INFO--USSR

SOURCE--IZV. AKAD. NAUK SSSR, SER. KHIM. 1970, (2), 468-72

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--CHEMICAL SYNTHESIS, CHLORINATED ORGANIC COMPOUND, BENZENE DERIVATIVE, PHOSPHINIC ACID, HETEROCYCLIC BASE COMPOUND, PHOSPHORUS SULFIDE

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1997/0851 STEP NO--UR/0062/70/000/002/0468/0472

CIRC ACCESSION NO--AP0119755

UNCLASSIFIED

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 010 UNCLASSIFIED PROCESSING DATE--23OCT70

CIRC ACCESSION NO--AP0119755

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. ADDING 7 G PROPIOLIC ACID TO 17.8 G PH PCL SUB2 IN C SUB6 H SUB6 (EXOTHERM) GAVE ON THE FOLLOWING DAY 50PERCENT PHP(O)CLCH:CHCOCL, B SUB0.04 140DEGREES, D PRIME20 1.3884, N PRIME20 SUBD 1.5795. THIS (10.5 G) AND 4.5 G AC SUB2 O IN C SUB6 H SUB6 HEATED 1 HR AT 60DEGREES GAVE BZCL AND 80PERCENT I, B SUB0.04 165DEGREES, 1.3464, 1.5720. THIS (1 G) AND 0.2 ML H SUB2 O IN 20 MIN AT 60DEGREES GAVE 94PERCENT PHP(O)(OH)CH:CHCO SUB2 H, M. 76-8DEGREES. SIMILARLY, ETOH GAVE 75PERCENT PHP(O)(OH)CH:CHCO SUB2 ET (IA), M. 138-40DEGREES. TO 18.3 G PHP(O)(OH)CH:CHCO SUB2 ET WAS ADDED 14.2 G PCL SUB5 AND CCL SUB4 AND THE MIXT. HEATED 1 HR AT 60DEGREES TO GIVE 91.3PERCENT PHP(O)CLCH:CHCO SUB2 ET (II), B SUB0.04 152DEGREES, 1.2713, 1.5455. TO 2 G II IN C SUB6 H SUB6 HEPTANE WAS ADDED 0.5 ML H SUB2 O AND 1 G ET SUB3 N TO YIELD 54PERCENT IA. II IN HEPTANE,C SUB6 H SUB6 TREATED WITH BUSH AND ET SUB3 N GAVE 83PERCENT PHP(O)(SBU)CH:CHCO SUB2 ET, B SUB0.04 180-2DEGREES, 1.1339, 1.5510. IR CURVES (6) WERE SHOWN.

FACILITY: INST.ORG. FIZ. KHIM. IM. ARBUZOVA, KAZAN, USSR.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 026 UNCLASSIFIED PROCESSING DATE--18SEP70

TITLE--REACTION OF 1,3,2, OXAAZAPHOSPHOLANES WITH ACETIC ANHYDRIDE -U-

AUTHOR-(03)-PUDOVIK, A.N., PUDOVIK, M.A., SHULYNDINA, O.S.

P

COUNTRY OF INFO--USSR

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(2), 501-2

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--ACETIC ANHYDRIDE, EXOTHERMIC REACTION, HETEROCYCLIC NITROGEN COMPOUND, ORGANIC PHOSPHORUS COMPOUND, HETEROCYCLIC OXYGEN COMPOUND, IR SPECTRUM, NMR SPECTRUM

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1985/1406 STEP NO--UR/0079/70/040/002/0501/0502

CIRC ACCESSION NO--AP0101496

UNCLASSIFIED

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 026 UNCLASSIFIED PROCESSING DATE--18SEP70

CIRC ACCESSION NO--AP0101496

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. ADDING AC SUB2 O TO I IN C SUB6 H SUB6 (EXOTHERM) (70 TO 80DEGREES MAX.) GAVE AFTER 12 HR 90PERCENT AC NMECH SUB2 CHMEOP(OET)(OAC), B SUB0 TIME SUB001 115 TO 18DEGREES, N PRIME20 SUBD 1.4592, D PRIME20 1.1177. THE PRODUCT ADDED S AND REACTED VIGOROUSLY WITH CCL SUB3 CHO. SIMILAR REACTION OF II WITH AC SUB2 O REQUIRED REFLUXING 1 HR IN C SUB6 H SUB6, TO GIVE ACNPHCH SUB2 CH SUB2 OP (OET)(OAC), WHICH ON ATTEMPTED DISTN. UNDERWENT DISPROPORTIONATION AND GAVE 47PERCENT ACNPHCH SUB2 CH SUB2 OAC, B SUB0 TIMES SUB05 116 TO 17DEGREES, 1.5158, 1.1205. IR AND NMR SPECTRAL DATA WERE GIVEN.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

1/2 025 UNCLASSIFIED PROCESSING DATE--18SEP70

TITLE--CONDENSATION AND ADDITION TO UNSATURATED COMPOUNDS OF TETRAETHOXYDIPHOSPHONOMETHANE -U-

AUTHOR-(03)-PUDOVIK, A.N., YASTREBOVA, G.YE., PUDOVIK, O.A.

COUNTRY OF INFO--USSR

P

SOURCE--ZH. OBSHCH. KHIM. 1970, 40(2), 499

DATE PUBLISHED--------70

SUBJECT AREAS--CHEMISTRY

TOPIC TAGS--CONDENSATION REACTION, ORGANIC PHOSPHORUS COMPOUND, ETHYL ETHER, COMPLEX COMPOUND, ACRYLONITRILE, ACRYLATE, IR SPECTRUM

CONTROL MARKING--NO RESTRICTIONS

DOCUMENT CLASS--UNCLASSIFIED

PROXY REEL/FRAME--1985/1405 STEP NO--UR/0079/70/040/002/0499/0499

CIRC ACCESSION NO--AP0101495

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

2/2 025 UNCLASSIFIED PROCESSING DATE--18SEP70

CIRC ACCESSION NO--AP0101495

ABSTRACT/EXTRACT--(U) GP-0- ABSTRACT. CH SUB2(P(O)(OET SUB2) SUB2 (I) HEATED 8 HR IN C SUB6 H SUB6 IN THE PRESENCE OF SATD. ETONA,ETOH WITH ELECTROPHILIC REAGENTS GAVE THE CORRESPONDING ADDUCTS. WITH CH SUB2:CHCN THE PRODUCT WAS 54PERCENT (NCCH SUB2 CH SUB2) SUB2 C(P(O)(OET) SUB2) SUB2, M. 78-9DEGREES; ME ACRYLATE GAVE ((ETO) SUB2 P(O)) SUB2:CHP(O)(OET) SUB2 GAVE 30PERCENT ((ETO) SUB2 P(O)) SUB2 CHCH SUB2 CH SUB2 P(O)(OET) SUB2, B SUB0.08 183-5DEGREES, 1.1805, 1.4540, WHILE BZH REQUIRED HEATING 22 HR IN XYLENE IN THE PRESENCE OF PIPERIDINE WITH CONTINUOUS REMOVAL OF H SUB2 O AND GAVE 10PERCENT ((ETO) SUB2 P(O)) SUB2 C:CHPH, B SUB0.5 186-8DEGREES, 1.1626, 1.4980. IR SPECTRAL DATA WERE GIVEN.

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"APPROVED FOR RELEASE: 09/17/2001 CIA-RDP86-00513R002202610006-2

USSR

UDC 542.91 + 661.718.1

KHAYRULLIN, V. K., DMITRIYEVA, G. V., and RUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Synthesis of Phenyl-β-chloroformylvinylphosphinic Acid Chloride"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 2, Feb 70, pp 468-472

Abstract: Phenyldichlorophosphine reacts with propiolic acid to give phenyl-β-chloroformylvinylphosphinic acid chloride. Moderate heating of the latter with acetic anhydride in a benzene solution gives 2-phenyl-2,5-dioxo-1,2-oxaphospholene. Hydrolysis of the latter gives phenyl-β-carboxyvinylphosphinic acid, alcoholysis phenyl-β-carbethoxyvinylphosphinic acid. Treatment of the ethyl ester of phenyl-β-carbethoxyvinylphosphinic acid with phosphorus pentachloride gives phenyl-β-carbethoxyvinylphosphinic acid chloride.

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Organophosphorus Compounds

USSR UDC 542.91 + 661.718.1

VASYANINA, M. A., KHAYRULLIN, V. K., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reactions of Ethyl- and Arylalkoxychlorophosphines With Propiolic Acid"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 2, Feb 70, pp 452-455

Abstract: Ethyl- and arylalkoxychlorophosphines react with propiolic acid to give esters of ethyl- or aryl-β-chloroformylvinylphosphinic acids (A). On distillation they eliminate alkyl chloride and convert to 2-ethyl- or 2-aryl-2,5-dioxo-1,2-oxa-3-phospholenes. Treatment of the undistilled products with alcohol gives esters of ethyl- or aryl-β-carbethoxyvinylphosphinic acids. The interaction of type A adducts with butyl mercaptan gives tert.-(1,1,1-trichloro)butyl esters of ethyl- and phenyl-β-carbobutylthiovinylphosphinic acids.

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UDC 547.26'118

PUDOVIK, A. N., ZIMIN, M. G., and SOBANOV, A. A.

"Interaction of Diethyl Phosphite With Acetoacetic Ester"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 4, Apr 70, pp 936-937

Abstract: The authors were unable to isolate pure addition products with a satisfactory yield as a result of the reaction of acetoacetic ester with diethylphosphorous acid in the presence of sodium ethylate. However, when the reaction was staged at room temperature in the presence of diethylamine, an addition product was obtained, viz. diethyl ester of α-hydroxy-α-methyl-β-carboethoxyethylphosphonic acid (I). Heating of I with acetyl chloride or with acetic anhydride in the presence of a catalytic amount of sulfuric acid gives its acetate. I undergoes partial dehydration on heating with a catalytic amount of piperidine in a benzene medium to give diethyl ester of α-methyl-β-carboethoxyvinylphosphonic acid.

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UDC 547.241 + 547.298.1

PUDOVIK, A. N., KHAYRULLIN, V. K., and DMITRIYEVA, G. V., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Chlorophosphines With Acrylamides and Methacrylamides in the Presence of Acetic Acid"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1034-1040

Abstract: The authors studied reactions of alkyldichlorophosphines, phenyldichlorophosphine and diethylchlorophosphine with substituted and unsubstituted amides of acrylic and methacrylic acids in the presence of acetic acid. It was found that alkyl- or aryldichlorophosphines react with dialkylamides of acrylic acid to form alkyl- or aryl(β-dialkylcarbamoylethyl)phosphinic acid chlorides. Diethylchlorophosphine reacts with the diethylamide of acrylic acid to give diethyl(β-diethylcarbamoylethyl)phosphine oxide. Dichlorophosphines react with amides of monosubstituted amides of α, β-unsaturated car-

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PUDOVIK, A. N., et al., Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1034-1040

boxylic acids in the presence of acetic acid to form substituted 2,5-dioxo-1,2-azaphospholanes. The likeliest mechanism for reactions of chlorophosphines with amides of α,β-unsaturated acids in the presence of acetic acid is one whereby the reaction begins with a nucleophilic attack of the β-carbon atom of the amide by the three-coordinate phosphorus atom.

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UDC 547.341

PUDOVIK, A. N., KHUSAINOVA, N. G., and TIMOSHINA, T. V., Kazan' State University imeni V. I. Ulyanov Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Dialkoxyphosphonoalkylalkynyl, -aminoalkynyl Ethers and Dialkoxyphosphonoisopropylalkynylamines"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1040-1042

Abstract: Continuing their studies on the synthesis of organophosphorus compounds, the authors obtained α-dialkoxyphosphonoalkylpropargyl ethers as a result of the reaction of propargyl bromide with the sodium derivative of dialkyl esters of α-hydroxyalkylphosphonic acid. The interaction of dialkoxyphosphonomethylenepropargyl ethers with secondary amines and paraform in the presence of copper acetate gives 4-dialkylamino-2-butynyl ethers. The reaction of propargyl bromide with dialkyl esters of α-aminoisopropylphosphonic acid in the presence of triethylamine gives dialkoxyphosphonoisopropylpropargylamines. The biological activity of the resultant compounds was stud-

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PUDOVIK, A. N., et al., Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1040-1042

ied. Thus, according to data obtained at the Chair of Pharmacology of Kazan' State Medical Institute by V. G. DUNAYEV and N. G. ABDRAKHMANOVA, 1-diethoxyphosphonomethylenehydroxy-4-diethylamino-2-butyne possesses low toxicity for warm-blooded animals, does not suppress cholinesterase activity or induce depression of the stem region of the central nervous system.

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USSR UDC 541.49 + 547.241

TOROPOVA, V. F., CHERKASOV, R. A., SAVEL'YEVA, N. I., and PUDOVIK, A. N., Kazan' State University imeni V. I. Ul'yanov-Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Effect of Substituents in Molecule of Phosphorus Dithioacids on Stability of Their Complex Compounds With Mercury Ions"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1043-1046

Abstract: The authors studied the effect of substituents at the phosphorus atom on the stability of complex mercury compounds with a series of phosphorus dithioacids of the type $(RO)_2PSSH$, $(RO)R'PSSH$ and R_2PSSH in water-ethanol solutions. The complexing was studied by the potentiometric method with a mercury indicator electrode. The stability constants (β) of the complex compounds were determined, and the redox potentials of some systems were measured. It is shown that log β_2 can be correlated with the sum of substituent constants at the phosphorus atom. In the reaction series under consideration (n=16) $\rho = 2.60$, $r = 0.905$.

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PUDOVIK, A. N., and GAREYEV, R. D., Kazan' State University imeni V. I. Ul'yanov-Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Reactions of Diphenyldiazomethane With Isopropenylphosphonic Acid Derivatives"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1025-1030

Abstract: For purposes of studying thermal reactions of diphenyldiazomethane, as well as reactions of diphenylmethylene with isopropenylphosphonic acid derivatives, the authors studied the behavior of diphenyldiazomethane towards dimethoxy-, diethoxy- and diphenoxyisopropenyl phosphonates under thermal reactions staged at 75°. Phosphorus-containing cyclopropane derivatives were obtained. Thermal reactions with isopropenyl phosphonates proceed according to a "pyrazoline" mechanism rather than a "diazonium" mechanism. Isopropenylphosphonic acid dichloride reacts with diphenyldiazomethane to give 1-dichlorophosphono-1-methyl-2,2-diphenylcyclopropane. A study of cata-

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PUDOVIK, A. N., and GAREYEV, R. D., Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1025-1030

lytic reactions of isopropenyl phosphonates with diphenyldiazomethane at 75° in the presence of anhydrous copper sulfate showed that in the case of diethoxyisopropenyl phosphonate only a small quantity of cyclopropane derivative is formed. The reaction is accompanied by the formation of benzophenonazine and benzophenone. The diphenylmethylene resulting from the catalytic decomposition of diphenyldiazomethane possesses nucleophilic properties.

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PUDOVIK, A. N., VASYANINA, M. A., and KHAYRULLIN, V. K., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Esters of Aryl(β-carbamoylalkyl)phosphinic Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1030-1034

Abstract: Reactions of ethyl- and arylalkoxychlorophosphines with acrylamides and methacrylamides in the presence of acetic acid give ethyl- and aryl(β-carbamoylalkyl)phosphinic acid esters. The article considers the direction of the reactions.

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Organometallic Compounds

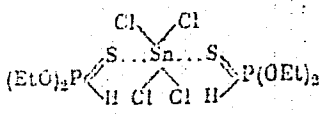
USSR UDC 547.26'118

MURATOVA, A. A., PLEKHOV, V. P., and PUDOVIK, A. N., Kazan' State University imeni V. I. Ulyanov Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Interaction of Diethyl Thiophosphite With Mercuric Chloride and Stannic Chloride"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 5, May 70, pp 1168-1169

Abstract: The reaction of mercuric chloride with diethyl thiophosphite, in which phosphorus is in the tetracoordinate state, gives diethyl chlorophosphite with a trivalent phosphorus atom. The interaction of diethyl thiophosphite with stannic chloride at room temperature gives a complex mixture of products with the structure



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KHAYRULLIN, V. K., DMITRIYEVA, G. V., and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan, Academy of Sciences USSR

"Reaction of Ethyldichlorophosphine With Acrylic Acid Esters"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, Vol 4, Apr 70, pp 871-876

Abstract: The reaction of ethyldichlorophosphine with acrylic acid esters was studied in the presence of a proton donor to determine the reaction center of the conjugated system C:C-C:O in reactions with substituted dichlorophosphines. On the basis of their experimental data the authors propose the following mechanism for the reaction. The tricoordinated phosphorus atom of ethyldichlorophosphine carrying the unshared electron pair attacks the beta-carbon atom of acrylic ester. The bipolar ion formed adds a proton from the acetic acid by its negative end, while its anion adds to phosphorus. The intermediate complex formed is stabilized by splitting off an acyl chloride molecule. As a result of such a reaction the chloroanhydride of ethyl-(β-carbalkoxyethyl)-phosphinic acid and

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KHAYRULLIN, V. K., et al, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, Vol 4, Apr 70, pp 871-876

acetyl chloride are formed. When the reaction is carried out in the presence of acetic acid, the main products are the chloroanhydrides of ethyl-(β-carbalkoxyethyl)-phosphinic acid.

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PUDOVIK, A. N., PUDOVIK, M. A., and SHULYNDINA, O. S., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR, Kazan'

"Reaction of 1,3,2-Oxaazaphospholanes With Acetic Anhydride"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, pp 501-502

Abstract: It is shown that ring substituted and unsubstituted N-alkyl-1,3,2-oxaazaphospholanes react under mild conditions with acetic anhydride resulting in ring cleavage on the nitrogen-phosphorus bond and the formation of corresponding dialkyl acetylphosphites in high yield.

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ACC. NR. AP0049135 Abstracting Service: CHEMICAL ABST. 5 Ref. Code: UR 0039

100829g Reaction of amides of dialkyl phosphorous and alkylarylphosphinous acids with α,β-unsaturated carboxylic acids. Pudovik, A. N.; Pudovik, M. A.; Terent'eva, S. A. (USSR). *Zh. Obshch. Khim.* 1970, 40(1), 33-6 (Russ). Mixing $CH_2{:}CHCO_2H$ with $Et_2NPEtPh$ at below 90° and holding 1 day gave 35% $EtPhP(O)CH_2CH_2CONEt_2$, $b_{0.07}$ 174-7°, d^{20} 1.0937, n_D^{20} 1.5357. Similarly was prepd. 38% $EtPhP(O)CH_2CHMeCONEt_2$, $b_{0.03}$ 160-1°, —, 1.5317. Treating 19.3 g $(EtO)_2PNEt_2$ with 7.2 g $CH_2{:}CHCO_2H$, maintaining the temp. below 80° gave $(EtO)_2P(O)CH_2CH_2CONEt_2$ contg. some 5% mixed $EtOP(O)(NEt_2)CH_2CH_2CO_2Et$, the mixed product, b_{11} 191°, 1.0706, 1.4567; the pure diethylamide, b_{10} 188-90°, 1.0720, 1.4571, was prepd. in 68% yield from $(EtO)_3P$ and $Et_2NCOCH_2CH_2Br$ at 150° in 2 hr $(PrO)_2PNEt_2$ (22.1 g) treated with 7.2 g $CH_2{:}CHCO_2H$ below 80° gave 29% $(PrO)_2P(O)CH_2CH_2CONEt_2$, b_{10} 187-9°, 1.0372, 1.4555. Similarly was prepd. 25% $(PrO)_2P(O)CH_2CHMeCONEt_2$, b_3 186-7.5°, 1.0231; 1.4545. Also reported: $EtOP(O)(NEt_2)CH_2CH_2CO_2Et$, $b_{0.08}$ 105-7°, 1.0538, 1.4472; $(EtO)_2P(O)CH_2CHMeCONEt_2$, $b_{0.03}$ 120-1°, 1.0503, 1.4560. Ir spectra are shown. G. M. Kosolapoff

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Acc. Nr: AP0049134 Abstracting Service: CHEMICAL ABST. 5-70 Ref. Code: UR 0079

100826d Thermal isomerization of a mixed anhydride of diethyl phosphorous and acrylic acids. Gazizov, T. Kh.; Pashinkin, A. P.; Pudovik, A. N. (USSR). *Zh. Obshch. Khim.* 1970, 40(1), 31-2 (Russ). $(EtO)_2POC(O)CH:CH_2$ (I) formed from thermal isomerization of the mixed anhydride of $(EtO)_2POH$ and $CH_2:CHCO_2H$ heated with excess abs. EtOH in the presence of a drop of Et_3N 2 hr gave 48% $(EtO)_2PHO$ and 61.3% $(EtO)_2P(O)CH_2CH_2CO_2Et$, $b_{1.5}$ 100-10°, d^{20} 1.1016, n_D^{20} 1.4310. Passing $CH_2:CO$ into $(EtO)_2P(O)CCH:CH_2$) 50 min at room temp. resulted in an exothermic reaction that yielded 59.9% $(EtO)_2P(O)C(:CH_2)O_2CCH:CH_2$, $b_{0.04}$ 84-5°, 1.1089, 1.4510.

G. M. Kosolapoff

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PUDOVIK, A. N., Corresponding Member of the Academy of Science USSR, and SUDAKOVA, T. M., Kazan State University imeni V. I. Ul'yanov-Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Reactions of Dibutyl- and Diphenylphosphinic Acids With Acid Chlorides of Saturated and Unsaturated Carboxylic Acids"

Moscow, Doklady AN SSSR, Vol 190, No 5, Feb 70, pp 1121-1123

Abstract: Dibutyl phosphinic acid reacts with acrylic and metacrylic acid chloride under mild conditions basically by an addition reaction and at higher temperatures by an addition and a replacement reaction. Examples follow. The reaction of dibutylphosphinic acid with acrylic acid chloride is accompanied by a strong exothermic effect yielding β-(dibutylphosphonium)-propionyldibutylphosphine oxide, b.p. 152-154°/2 mm and α-(dibutylphosphonium)-propenyl ester of dibutylphosphinic acid, b.p. 95-96°/1 mm, d_4^{20} 1.0354, n_D^{20} 1.14665. The same reaction carried out in benzene solution at +6°C yields dibutylphosphonylpropionic acid chloride which decomposes on distillation and therefore is converted to the ethyl ester, b.p. 158-160°/1 mm, d_4^{20} 1.0117, n_D^{20} 1.4678. An analogous reaction occurs between dibutyl-

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PUDOVIK, A. N. and SUDAKOVA, T. M., Doklady AN SSSR, Vol 190, No 5, Feb 70, pp 1121-1123

phosphinic acid and acetyl chloride, the mixture being heated by the exothermic reaction to 60 C, and it yields the dibutylacetylphosphine oxide, b.p. 62-63.5°/2 mm, d_4^{20} 0.9319, n_D^{20} 1.4755, and α-(dibutylphosphonic)-ethyl ester of dibutylphosphinic acid, m. p. 59-60°. The reaction of acetyl chloride with diphenylphosphinic acid is less energetic, yielding acetyldiphenylphosphine oxide, b.p. 125-127°/2 mm, d_4^{20} 1.2083, n_D^{20} 1.6319, and ~(diphenylphosphonic)-ethyl ester of the diphenylphosphinic acid, m.p. 130-131°.

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USSR UDC 547.341'139.81 + 547.391

PUDOVIK, A. N., BATYYEVA, E. S., and NESTERENKO, V. D.

"Reaction of Phenylsulfamide of Diphenylphosphinous Acid With Benzaldehyde"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, pp 502-503

Abstract: Phenylsulfamide of diphenylphosphinous acid reacts energetically with benzaldehyde in the absence of a catalyst to give a crystalline product of composition 1 : 1, representing phenylsulfamide of diphenyl(α-hydroxybenzyl)phosphine.

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USSR UDC: 547.26'118

~~PUDOVIK,~~ A. N., YASTREBOVA, G. YE., and PUDOVIK, O. A., Kazan' State University imeni V. I. Ulyanov Lenin, Kazan, Ministry of Higher and Secondary Specialized Education RSFSR

"Reactions of Tetraethoxydiphosphonomethane Condensation and Addition to Unsaturated Compounds"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, p 499

Abstract: It is shown that tetraethyl ester of methylenediphosphonic acid in the presence of a saturated alcoholic solution of sodium ethylate adds to the unsaturated electrophilic compounds nitriloacrylic acid, methyl acrylate and diethyl vinylphosphonate. The addition of tetraethoxydiphosphonomethane to nitriloacrylic acid gives bis(2-cyanoethyldiethoxyphosphono)methane. In the case of methyl acrylate and diethyl vinylphosphonate 1-substituted bis(3,3-diethoxyphosphono)-propanes are obtained. It was found that the condensation reaction of tetraethoxydiphosphonomethane with benzaldehyde proceeds under considerably more severe conditions than analogous condensation reactions of other organophosphorus compounds containing an active methylene group.

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PUDOVIK, A. N., NIKITINA, V. I., and KURGUZOVA, A. M.

"Reactions of Organophosphorus Compounds Containing Active Methylene Groups With Methyl-β-chlorovinyl Ketone and α-Halogen Ethers"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, pp 291-294

Abstract: The authors studied ketovinylation reactions of a series of organophosphorus compounds with an active methylene group (diethylphosphonoacetic ester, diethyl phosphonoacetone, diethyl esters of cyanoacetic and benzylphosphonic acids, as well as their monoalkyl derivatives). Ketovinylation of the sodium and potassium derivatives of phosphonoacetic ester and nitrilophosphonoacetic acid results in the formation of complex mixtures of products, of which mono- and diketovinylation products were obtained in low yields. Reactions with monoalkyl derivatives of the above compounds, as well as with monoalkyl derivatives of dimethyl and diethyl phosphonoacetone proceed more smoothly. Monoketovinylation products were obtained in satisfactory yields in a number of reactions. In addition to ketovinylation, all the studied reactions displayed self-condensation of methyl-

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PUDOVIK, A. N., et al., Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, pp 291-294

β-chlorovinyl ketone to give triacetylbenzene.

The interaction of (β-acylvinyl)ammonium salts with monoalkylated dimethyl phosphonoacetone and diethyl ester of benzylphosphonic acid was studied. High yields of the ketovinylation products were obtained in this case, and no formation of self-condensation product was observed. No ketovinylation product could be obtained in the case of diethyl ester of benzylphosphonic acid.

Reactions of α-chloromethyl propyl and α-chlorodimethyl ethers with potassium derivatives of phosphonoacetic ester, phosphonoacetone and cyanophosphonomethane were studied. Monoalkylation products were obtained in most cases, as well as dialkylation products in a number of cases. According to IR spectral data the alkylation products of phosphonoacetone are derivatives of its enol form.

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USSR UDC: 547.26'118

YELISEYENOKOV, V. N., PUDOVIK, A. N., FATTAKHOV, S. G., and SERKINA, N. A., Kazan, Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

"Reaction of Amidophosphites With Ammonium Salts of Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, p 498

Abstract: The authors report finding that reactions of amidophosphites with amine hydrochlorides and salts of carboxylic and phosphorus- containing acids are reversible and proceed according to the equation

$$>P-NR_2 + B \cdot HX \rightleftarrows >P-X + B + HNR_2$$

B = amine; X = Hlg, Ac, P(O)C and P(S)O; R = Alk.

The authors regard the results as important for eludicating the mechanism involved in the phosphorylation of amines and alcohols with amidophosphites, and the question will be considered in a special article.

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USSR UDC 547.26'118

PUDOVIK, A. N., NIKITINA, V. I., and YEVDOKIMOVA, V. V.

"Addition Reactions of Aldehydes to α-Ketophosphonic Acid Esters"

Leningrad, Zhurnal Obshchey Khimii, Vol 40, No 2, Feb 70, pp 294-298

Abstract: Continuing their study of addition and condensation reactions of organophosphorus compounds with an active methylene group, the authors studied reactions of aceto- and benzoylphosphonic acids with aldehydes. It was found that reactions of chloral with dimethyl and diethyl esters of acetophosphonic acid and diethyl ester of benzoylphosphonic acid in benzene at 80° in the presence of piperidine proceed on the carbonyl groups to give dialkyl esters of α-hydroxy-α-acetoxy- or α-hydroxy-α-benzoylhydroxyethylphosphonic acids. At a higher temperature (110°) in toluene there is a phosphonate-phosphate rearrangement to give phosphates. The reactions of dimethyl and diethyl esters of acetophosphonic acid with benzaldehyde and butyl ester of glyoxylic acid, as well as of diethyl ester of benzoylphosphonic acid with benzaldehyde in toluene in the presence of piperidine give the corresponding phosphates.

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TOROPOVA, V. F., CHERNASOV, R. A., SAVEL'YEVA, N. I., SLYUSAR', N. V., PUDOVIK, A. N.

"Investigation of Complex Compounds of Dithio Acids of Phosphorus with Bivalent Nickel and Cobalt Ions, and Application of the Hammett Equation with σ_p Constants to the Complex-Forming Reactions"

Leningrad, Zhurnal Obshchey Khimii, Vol 42(104), No 7, Jul 72, pp 1485-1489

Abstract: Complex compounds of dithio acids of phosphorus with bivalent nickel and cobalt ions were studied. The composition and stability constants of the complexes were determined in 90% ethanol-water solutions at an ionic strength of 0.3 and a temperature of 25°C. It was shown that the stability constants log β of the complexes conform to the Hammett equation with σ_p constants -- specific constants of the substituents associated with the phsophorus aton in the dithio acid molecule. Correlation parameters are compared for the reaction series of complex compounds of dithio acids of phosphorus with ions of various metals.

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PUDOVIK, A. N., DMITRIYEVA, G. V., ANOSHINA, N. P., ZYABLIKOVA, T. A., and KHAYRULLIN, V. K., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences USSR

UDC 542.91:547.1'118

"Reaction of Chlorophosphines with β-Chloroacrylic Acid"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 5, May 72, pp 1159-1164

Abstract: The article describes the reaction of methyl-, ethyl-, and phenyldichlorophosphine with β-chloroacrylic acids, as well as results of thermographic studies of some reactions of this type. The reaction of methyl-, ethyl- and phenyldichlorophosphine with cis-β-chloroacrylic acid gives alkyl- or aryl-(β-chloroformylvinyl)phosphinic acid chlorides, while the reaction with trans-β-chloroacrylic acid gives trans-β-chloroacrylic acid chloride.

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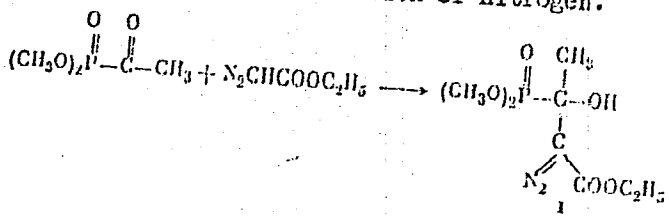
UDC: 547.26'118

PUDOVIK, A. N., REMIZOV, A. B., STABROVSKAYA, L. A., SHTIL'MAN, S. Ye., LUSHCHITS, I. G., GAREYEV, R. D., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Adduct of the 'Aldol' Type of Ethyl Diazoacetate With Dimethyl Acetophosphonate"

Leningrad, Zhurnal Obshchey Khimii, Vol 42(104), No 6, Jun 72, p 1421

Abstract: The authors investigated the reaction of dimethyl acetophosphonate with ethyl diazoacetate at room temperature. It was found that an "aldol" addition product is formed without elimination of nitrogen.



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UDC: 547.26'118

REMIZOV, A. B., GAREYEV, R. D., PUDOVIK, A. N., Kazan' State University imeni V. I. Ul'yanov-Lenin

"Rotational Isomerism of Dialkyl Esters of Aceto- and Benzoylphosphonic Acids"

Leningrad, Zhurnal Obshchey Khimii, Vol 42(104), No 6, Jun 72, pp 1238-1240

Abstract: The authors studied the infrared spectra of dimethyl and diethyl phosphonate (I and II) and dimethyl benzoylphosphonate (III). The spectra were taken on the UR-20 spectrophotometer. All three compounds were studied in the liquid state, and compound (I) was studied in the gaseous state as well. The effect of temperature was investigated. An analysis of the experimental material showed dynamic equilibrium of two isomers in compounds (I) and (II) due to rotation about the P-C bond. The rotational isomerism is observed in the liquid state both with and without a solvent.

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USSR UDC: 538.27+541.67+547.341+547.772

SAMITOV, Yu. Yu., GAREYEV, R. D., STABROVSKAYA, L. A., PUDOVIK, A. N.,
Kazan' State University imeni V. I. Ul'yanov-Lenin

"Stereochemistry of Organophosphorus Compounds. II. NMR Spectra, Conformations of 3- and 5-Phosphorylated Δ^1-and Δ^2-Pyrazolines and Angular Correlation $^3J_{PCCH}$"

Leningrad, Zhurnal Obshchey Khimii, Vol 42(104) No 6, Jun 72, pp 1227-1235

Abstract: 3-Methyl-3-dialkoxyphosphinyl-5,5-dimethyl- Δ^1- and 3-phenyl-5-methyl-5-dimethoxyphosphinyl- Δ^2-pyrazolines were synthesized. The paramagnetic resonance spectra of 3- and 5-phosphorylated Δ^1- and Δ^2-pyrazolines were studied, and their preferred conformations were determined. The angular correlation was empirically established for the vicinal constant of spin-spin interaction type $^3J_{PCCH} = f(\phi)$ for the case where the carbon atoms in the P-C-C-H fragment have sp3 hybridization, and where there is no steric hindrance to rotation of the dimethoxyphosphinyl group about the P-C bond. It was established by ultraviolet and infrared spectroscopy that the tetrahedral phosphorus atom falls behind the phenyl group with respect to ability to enter into conjugation.

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UDC: 547.26'118

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PUDOVIK, A. N., BATYYEVA, E. S., AL'FONSOV, V. A., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Academy of Sciences of the USSR

"Concerning the Reaction of Dialkyl Phosphorous Acid Anilides With Methylcarbonic Acid Chloride"

Leningrad, Zhurnal Obshchey Khimii, Vol 42(104), No 6, Jun 72, pp 1235-1238

Abstract: The authors studied the reaction of secondary amides of dialkyl phosphorous acids with methylcarbonic acid chloride. Reactions of anilide, p-toluidide and p-anisidide of diethyl, dipropyl and dibutyl phosphorous acid with methylcarbonic acid chloride were accompanied by an exothermal effect with the formation of alkyl halide. The reaction of diethyl phosphorous acid anilide with methylcarbonic acid chloride yielded diethyl chlorophosphite (42%), methylcarbonic acid anilide (55%) and ethyl O-ethyl anilidocarbomethoxyphosphonate (20%). The results indicate that the reaction goes in two directions simultaneously: with initial attack of the carbon in the carbonyl group both by a phosphorus atom and by a nitrogen atom:

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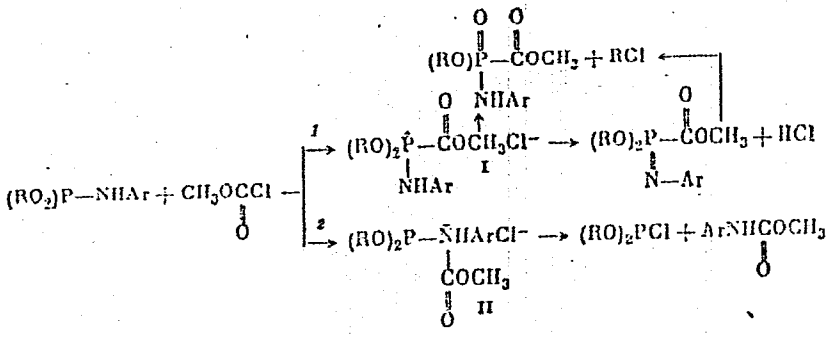
PUDOVIK, A. N., Zhurnal Obshchey Khimii, Vol 42(104), No 6, Jun 72, pp 1235-1238



The reaction through the phosphorus atom yields phosphonic derivatives, while that through the nitrogen atom yields dialkyl phosphorous acid chlorides and methylcarbonic acid anilides.

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UDC 539.143.43+661.718.1

ISHMAYEVA, E. A., ZIMIN, M. G., GALEYEVA, R. M., and PUDOVIK, A. N., Kazan State University imeni V. I. Ul'Yanob-Lenin, Kazan

"The Dipole Moments of Organophosphorus Compounds. II. Benzoylphosphonates, Benzoylphosphinates, and Benzoylphosphine Oxides"

Moscow, Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 3, Mar 71, pp 538-543

Abstract: The diethyl esters of benzoylphosphonic acids $X\text{-}C_6H_4COP(O)(OEt)_2$ (I) with X = p-Cl, M-Cl, M-Br, P-NO_2 (bp 150° at 2 mm, 146° at 2 mm, 164° at 2 mm; and bp 145° at 1 mm, m.p. 51.5°, respectively) and diethyl(diphenyl) benzoylphosphine oxides $PhCOP(O)R_2$ (II) with R = Et, Ph (bp 155-6° at 6 mm and 167-8° at 1.5 mm, respectively) were prepared. Compounds I were obtained by the reaction of $X\text{-}C_6H_4COCl$ with triethyl phosphite and compounds II by reacting diethyl- or diphenylphosphinous acid $HP(O)R_2$ with PhCOCl. The dipole moments of compounds I and II were determined experimentally and the spatial structure of these compounds was elucidated from the data obtained, that of ethyl esters of ethyl- and phenylbenzoylphosphinic acids from

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ISHMAYEVA, E. A., et al., Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No 3, Mar 71, pp 538-543

available experimental data. Differences between the experimentally determined and calculated values of the dipole moments of I indicated an interaction between X and the $-P(O)(OEt)_2$ group.

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