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# Kinetics and Mechanism of the Pudovik Reaction in the Azomethine Series: II.[1] Hydrolysis and Catalysis in the Reaction of Dialkyl Phosphites with Benzalaniline

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**Abstract**—A $^{31}$P NMR study show that diisopropyl phosphite and benzalaniline do not react with each other as individual components. Adduct formation is made possible by partial hydrolysis of the dialkyl phosphite.

Proceeding with research into the mechanism of reaction of dialkyl phosphites with azomethines [1] we found that the reaction have an induction period (3–5 min) when fast prepared solutions of freshly purified reagents are used, while with time the apparent rate constant steadily increases. Assuming that this fact is explained by absorption of atmospheric moisture, we showed that addition of water eliminates the induction period and prevents increase in the reaction constant (kinetic details of this study will be described in the following communication).

In the present work we held, like Vorkunov and Levin [2], the opinion that dialkyl phosphites are more likely bases than proton donors, and this predetermines their low activity in noncatalyzed proton-transfer reactions. Presumably, the effect of water is primarily associated the fact that it hydrolyzes dialkyl phosphites, thus inducing an acid-catalyzed reaction pathway.

Preliminary study of the reaction of benzalaniline (**I**) with diisopropyl phosphite (**II**) by differential thermal analysis (DTA) gave convincing evidence to show that it is much facilitated by addition of water and possible products of hydrolysis of the dialkyl phosphite, specifically, phosphorous acid and its salt with azomethine **I** (salt **III**). As seen from Table 1, the above additives much reduce the temperature of onset ($T_o$) and maximum ($T_{max}$) of exothermic effects compared with the control reaction. The $^{31}$P NMR spectra of the reaction mixtures in 2-propanol after thermography contained, along with signals of aminophosphonate **IV** and unreacted phosphpite **II**, other signals assignable to adducts of azomethine **I** with hydrolysis products of phosphite **II**: isopropyl phosphite {PhCH[$PO_2$(OPr-*i*)]$^-$$N^+H_2$Ph (**V**), $\delta_P$ ~19 ppm}, salt **III**, and analogous salt of monoester **V** (salt **VI**,

**Table 1.** Compositions of mixtures of azomethine **I** and phosphite **II** and their thermoanalytical data

| Components, mmol | | | Thermoanalytical data | | |
|---|---|---|---|---|---|
| **I** | **II** | additives | $T_o$, °C | $T_m$, °C | compound: relative content (%), $\delta_P$, ppm, ($J_{PH}$, Hz) |
| 1.4 | 1.4 | – | 120 | 143 | **IV**: 88, 21.9 (23.3);<br>**II**: 5, 5.1 (692.3);<br>**V**: 5, 19.4 (23.3);<br>**VI**: 2, 2.9 (643.3) |
| 1.4 | 1.4 | $H_2O$ (0.6) | 108 | 126 | **IV**: 74, 21.9 (23.8);<br>**V**: 8, 19.3 (26.7);<br>**III**: 15, 2.8 (653.5)<br>**VI**: 3, 2.6 (644.7); |
| 1.4 | 1.4 | Salt **III** (0.02) | 92 | 115 | **IV**: 90, 21.8 (22.6);<br>**II**: 3, 5.1 (696.7);<br>**V**: 5, 18.9 (ill-defined);<br>**III**: 2, 2.6 (641.8) |
| 1.5 | 1.4 | $H_3PO_3$ (0.1) | 79 | 110 | **IV**: 72,[a] 21.8 (24.4);<br>**V**: 15, 18.9 (29.3)<br>**III**, **VI**: 13, 2.6 (649.1);<br>2.8 (646.5) |

[a] The actual yield of aminophosphonate **IV** is higher, since this product was not initially dissolved completely ($\delta_P$ 21.9 ppm, d, $^2J_{PH}$ 22.3 Hz).

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[1] For communication I, see [1].

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**Table 2.** $^{31}P$ NMR spectra of dialkyl phosphite solutions

| Run no. | Solvent | Concentration of $(RO)_2PHO$, M | R | Concentration of $H_2O$, M | Handling time, days | $\delta_R$, ppm ($^1J_{PH}$, Hz); content, % | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | $(RO)_2PHO$ | (RO)(OH)PHO | $H_3PO_3$ |
| 1 | Dioxane | 1.46 | Me | – | 2 | 11.6 (694.5) | – | – |
| 2 | | 1.46 | Me | – | 15 | 11.8 (696.8); 82 | 9.1 (685.4); 18 | – |
| 3 | | 1.42 | Me | 1.53 | 2 | 12.3 (702.7); 50 | 8.6 (686.3); 49.5 | 5.4 (677.8); 0.5 |
| 4 | | 1.42 | Me | 1.53 | 15 | 12.5 (713.1); 5 | 9.4 (697.7); 60 | 6.2 (686.9); 35 |
| 5 | | 2.20 | *i*-Pr | 2.20 | 5 | 5.5 (690.6); 91.5 | 4.5 (672.0); 8.5 | – |
| 6 | | 2.20 | *i*-Pr | 2.20 | 24 | 6.6 (695.5); 40 | 7.1 (683.8); 60 | – |
| 7 | DMSO | 1.46 | Me | – | 1 | 13.6 (700.0) | – | – |
| 8 | | 1.46 | Me | – | 15 | 13.2 (698.8); 13 | 7.6 (665.3); 75 | 2.8 (649.1); 12 |
| 9 | | 1.41 | Me | 1.61 | 1 | 13.7 (702.2) | – | – |
| 10 | | 1.41 | Me | 1.61 | 8 | 13.2 (700.5); 56.6 | 7.5 (669.6); 42.1 | 2.4 (646.3); 1.3 |
| 11 | | 1.41 | Me | 1.61 | 15 | 13.1 (725.1); 2 | 7.6 (665.0); 78 | 2.8 (650.0); 19 |
| 12 | Acetonitrile | 1.47 | Me | – | 2 | 12.5 (694.3) | – | – |
| 13 | | 1.47 | Me | – | 22 | 12.4 (697.8) | 8.1 (671.2); traces | – |
| 14 | | 1.41 | Me | 1.62 | 2 | 12.9 (699.5); 92.6 | 8.4 (677.0); 7.4 | – |
| 15 | 2-Propanol | 1.46 | Me | – | 1 | 11.9 (704.1) | – | – |
| 16 | | 1.46 | Me | – | 8 | 11.1 (704.4); 96.6 | 6.1 (670.4); 1.0 | 7.9 (694.5); 2.4[a] |
| 17 | | 1.46 | Me | – | 15 | 11.9 (704.2); 93.6 | 6.9 (671.3); 2.7 | 8.7 (699.4); 3.7[a] |
| 18 | | 1.5 | *i*-Pr | – | 1 | 5.2 (693.8) | – | – |
| 19 | | 1.5 | *i*-Pr | – | 14 | 5.3 (693.6) | – | – |
| 20 | | 0.57 | *i*-Pr | – | 1 | 5.2 (694.1); 81.5 | 3.3 (667.1); 18.5 | – |
| 21 | | 0.60 | *i*-Pr | 1.37 | 1 | 5.4 (694.8); 81 | 3.5 (667.9); 19 | – |

[a] (MeO)(*i*-PrO)PHO.

$\delta_P$ ~2.6–2.9 ppm). The resulting data prompted us to consider in more detail the possibility of hydrolysis of dialkyl phosphites under conditions similar to those we applied in our kinetic experiments and to assess the effect of hydrolysis products on the reaction in hand.

The hydrolytic instability of dialkyl phosphites is generally recognized, and quantitative data are available on their hydrolysis in neutral, acidic, and basic media [3]. At the same time, since the reactions are accomplished in organic solvents, we considered it of interest to trace, on a qualitative level, the effects of the medium and the time of handling of dialkyl phosphites in solutions or in the pure state under conditions not excluding moisture absorption or in the presence of specially added water on reactions of these compounds with azomethines. $^{31}P$ NMR spectroscopy was chosen as measurement technique, and solutions of dimethyl phosphite **VII** and phosphite **II** in dioxane, DMSO, acetonitrile, and 2-propanol, as objects for study. We followed changes in the spectra of solutions on their handling in under usual conditions without and with water additives. Dialkyl phosphites were specially purified before use (vide infra).

The results of spectral measurements are presented in Table 2. The spectrum of phosphite **VII** in dioxane (run no. 1) (hereinafter, the run numbers correspond to those in Tables 2 and 3) is a doublet of septets ($\delta_P$ 11.6 ppm, $^1J_{PH}$ 694.5 Hz). After two-week handled at room temperature in an NMR ampule, the spectrum (run no. 2) contains, along with the signal of phosphite **VII**, a doublet of quartets ($\delta_P$ 9.1 ppm, $^1J_{PH}$ 685.4 Hz) from methyl phosphite. With the same solution handling with special water additives (run nos. 3 and 4), quite a different pattern is observed. Two days after preparation of the solution (run no. 3), phosphite **VII** has hydrolyzed by half; along with the major hydrolysis product, methyl phosphite (49.5%), the mixture contained some phosphoric acid (0.5%) appearing as a doublet ($\delta_P$ 5.4 ppm, $^1J_{PH}$ 677.8 Hz). At room temperature, hydrolysis is almost complete within 2 weeks (run no. 4). Analogous experiments with solutions of phosphite **II** showed that it is more resistant to hydrolysis compared with phosphite **VII** (this, however, is well known; see, for example, [3]); isopropyl phosphite **VIII**, too, is hydrolytically stable: We detected no phosphorous acid formation the whole time of observation (run no. 6).

**Table 3.** Reaction of azomethine **I** with phosphite **II** in 2-propanol

| Run no. | Component concentrations, M | | | Handling time, days | Compound: $\delta_R$, ppm ($J_{PH}$, Hz); relative content, % |
|---|---|---|---|---|---|
| | **I** | **II** | others | | |
| 22 | 0.03 | 0.07 | – | 15 | **II**: 5.1 (693.9) |
| 23 | 0.50 | 0.50 | – | 8[a] | **II**: 5.0 (693.9); 99.4<br>**IV**: 21.9; 0.6 |
| 24 | 0.50 | 0.50 | $H_2O$, 0.003 | 8[a] | **II**: 5.0 (695.7); 99.2<br>**IV**: 21.8 (22.9); 0.8 |
| 25 | 0.49 | 0.49 | $H_2O$, 0.58 | 8[a] | **II**: 5.0 (696.0); 98.8<br>**IV**: 21.7 (22.9); 1.2 |
| 26 | 0.48 | 0.23 | $H_3PO_3$, 0.16 | 21 | **IV**: 21.9 (24.9); 56<br>**III**: 2.8 (650.3); 44 |
| 27 | 0.55 | 0.55 | $H_3PO_3$, 0.27 | 5[a] | **II**: 5.0 (689.3); 4.5<br>**IV**: 21.7 (25.2); 36<br>**III**: 3.0 (655.7); 59.5 |
| 28 | 0.54 | 0.54 | $H_3PO_3$, 0.72<br>$H_2O$, 0.27 | 5[a] | **II**: 5.1 (695.3); 18<br>**IV**: 21.7 (23.0); 32<br>**III**: 2.9 (652.9); 50 |
| 29 | 0.38 | 0.38 | **III** | 9[b] | **II**: 5.1 (694.6); 61<br>**IV**: 21.9 (25.7); 25<br>**III**: 2.5 (639.9); 14 |
| 30 | – | 0.12 | **III** | 9[b] | **II**: 5.1 (694.7)<br>**III**: 2.5 (646.4) |
| 31 | 0.79 | 0.7 | – | 2[a] | **II**: 5.1 (691.4); 99.5<br>**IV**: 21.9, 0.5 |
| 32 | 0.79 | 0.7 | **VIII** 1 drop | 2[a] | **II**: 5.1 (690.8); 67<br>**IV**: 21.9 (25.0); 27<br>**III**: 2.5 (647.8); 6 |
| 33 | 1.30 | 1.30 | – | Heating | **II**: 5.1 (691.6); 77<br>**IV**: 21.9 (23.5) 23 |
| 34 | 1.25 | 1.25 | **IX**, ~0.2 | 5 h | **II**: 5.1 (697.9); 95<br>**IV**: 20.8, 5 |
| 35 | 1.23 | 1.23 | **X**, ~0.085 | at 50°C | **II**: 5.1 (692.1); 91.5<br>**IV**: 21.8 (23.5); 8.5 |
| 36 | 1.14 | 1.14 | **X**, ~0.5 | | **II**: 5.0 (680.5); 97<br>**IV**: 21.7, 3 |

[a] With heating for 5 h at 50°C. [b] With heating for 16 h at 50°C.

It should be noted that the spectra of dialkyl phosphite solutions with water additives (in all the solvents) display well-defined multiplet signals of all the phosphorus-containing compounds present, whereas in the spectra of specially prepared mixtures of phosphite **VII** and phosphorous acid we observe one to three broad signals, depending of the component ratio.

On replacement of dioxane by DMSO, the spectral dynamics is generally preserved. The spectrum of the solution (run no. 7) without water additives contains no other signals than that of phosphite **VII**, and after two-week handling of this solution in an NMR ampule, the hydrolysis degree attains 90% (run no. 8). An almost identical result was obtained in experiments with water additives (run nos. 9–11). The similarity in the hydrolysis patterns with and without water additives (run nos. 8 and 11) probably derives from a high moisture-absorbing capacity of DMSO.

Unlike DMSO, in acetonitrile we observed no hydrolysis of phosphite **VII** via absorption of air moisture (run nos. 12 and 13) within three weeks. With special water additives, hydrolysis does occur (run no. 14) but at a lower rate than in DMSO.

In 2-propanol, appreciable hydrolysis of phosphite **VII** with air moisture is observed within one week (run nos. 15–17). Interestingly, the second product of transformation of phosphite **VII** (apart from methyl phosphite) was found to be isopropyl methyl phosphite formed ether by reesterification of phosphite **VII** with 2-propanol or esterification of methyl phosphite formed by hydrolysis. At the same time, with phosphite **II** under similar conditions we observed no spectral changes (run nos. 18 and 19). However, in the sample of phosphite **II** handled without solvent (run no. 20), much monoester **VIII** was found. It is noteworthy that addition of water to this sample does not increase the amount of the hydrolysis product (run no. 21). Thus, phosphite **II** proves to be resistant to hydrolysis in 2-propanol, whereas on handling without solvent it gives an appreciable amount of monoester **VIII**. The spectra of phosphites **II** and **VII** handled in sealed vessels or not specially purified after preparation, revealed monoalkyl phosphites in amounts depending on handling conditions.

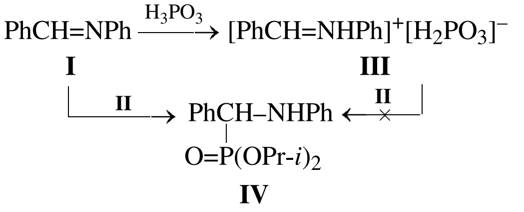
In the available publications concerning the kinetics of reactions of dialkyl phosphites with electrophylic reagents nothing has been said as to the authors take account for the possible presence in dialkyl phosphites of acidic admixtures. Purification of dialkyl phosphites most commonly involved distillation to published constants; however, this purity criterion is insufficient for kinetic studied. We earlier mentioned [4] that even multiple distillation of phosphite **VII** provides no purification from acidic admixtures (at that time we considered that it was simply hydrogen chloride). This is readily seen in the UV spectrum of 1-cyclohexylimino-2-butene. The spectrum of the azomethine contains an $n,\pi^*$-transition band ($\lambda_{max}$ 219 nm); addition of phosphite **VII** produces either shifting or attenuation of this band and appearance of a new band ($\lambda_{max}$ 243 nm), depending of the content of acidic components; such spectral changes are characteristic of protonation of azomethines [5].

To free dialkyl phosphites of acidic admixtures proves possible only via fractionation over metallic sodium followed by rectification under a dry inert gas to remove possible disproportionation products. The fact that acidic admixtures are impossible to remove by simply repeatedly fractionating dialkyl phosphites is probably explained by the ability of the latter to form stable adducts with strong proton donors [6, 7].

Evidence for the catalytic effect of hydrolysis products of dialkyl phosphites in the reaction with azomethines was obtained from spectral studies of model reactions between benzalaniline (**I**) and diisopropyl phosphite (**II**).

In the spectra of a mixture of freshly purified phosphite **II** and azomethine **I** in 2-propanol kept at room temperature we detected no adduct, aminophosphonate **IV**, within two weeks (run no. 22); upon heating of the same mixture for 5 h at 50°C, only traces of the adduct were found (run no. 23). Contrary to expectations, the effect of water proved very weak (run nos. 24 and 25). Addition to the starting mixture of phosphorous acid leads to aminophosphonate **IV** formation already at room temperature (run no. 26). Changing the component ratio in the mixture (run no. 27) and addition of water (run no. 28) results in decreased consumption of phosphite **II**, which implies competitive formation of aminophosphonate **IV** and salt **III**. In the latter case (run no. 28), salt **III** forms immediately as the solution is prepared, which is assignable to ionization of phosphorous acid with the water added.

The fact that salt **III** forms within some minutes after mixing phosphorous acid and azomethine **I** (analogous 1 : 1 benzalanilinium salt was earlier obtained by the reaction with phosphous acid independent of the reagent ratio [8]), whereas aminophosphonate **IV** forms rather slowly, puts the question whether salt **III** catalyzes this reaction. Moreover, salt **III** may mediate formation of amine **IV**.



$$\begin{tikzcd} \underset{\mathbf{I}}{PhCH{=}NPh} \arrow[r, "H_3PO_3"] \arrow[d, "\mathbf{II}"] & \underset{\mathbf{III}}{[PhCH{=}NHPh]^+[H_2PO_3]^-} \arrow[dl, "\mathbf{II}"', "\times"] \\ \underset{\mathbf{IV}}{\underset{O=P(OPr\text{-}i)_2}{PhCH}\text{–}NHPh} & \end{tikzcd}$$

Heating the reaction mixture in 2-propanol in the presence of salt **III** (run no. 29) leads to compound **IV**; at the same time, salt **III** fails to react with phosphite **II** under the same conditions (run no. 30). Thus, salt **III** catalyzes rather than mediate aminophosphonate **IV** formation.

Since the initial hydrolysis product of dialkyl phosphite is monoalkyl phosphite, whereas for phosphorous acid to form requires longer time [the more so as with phosphite **II** in 2-propanol and dioxane we did not detect the second stage of hydrolysis at all (Table 1), it might be suggested that monoalkyl phosphite, too, catalyzes the reaction studied.

We failed to find in the literature any procedure which might provide individual monoisopropyl ester **VIII** or monomethyl phospite.

An attempt was made to isolate the former from a dioxane solution of equimolar mixture of phosphite **II** and water, that contained, according to $^{31}P$ NMR data, 70% of ester **VIII** and 30% of phosphite **II**. However, distillation gave a fraction [bp 75–76°C (0.5 mm), $n_D^{20}$ 1.4135] containing, apart from ester **VIII** (64%), phosphite **II** (30%) and phosphorous acid (6%). Heating solutions of **I** or **II** in 2-propanol without (run no. 31) and with (run no. 32) addition of the obtained fraction revealed catalytic action of the additive, but the purity of the experiment was disturbed by the presence of phosphoric acid in the additive. The catalytic action of ester **VIII** is also confirmed by the fact that the reaction of azomethine **I** with phosphite **II** handled for several days after purification and containing some ester **VIII** formed by hydrolysis, too, gives aminophosphonate **IV** (run no. 33, cf. run nos. 22 and 23).

Since acidic admixtures catalyze the reaction, we proposed that basic additives would produce the opposite effect. Actually, addition of triethylamine (**IX**) or diethylaniline (**X**) to phosphite **II** handled as described above much decreased the yield of aminophosphonate **IV** (run nos. 33–36).

Thus, individual phosphite **II** in the absence of catalysts does not form adducts with azomethine **I**. Presumably, individual dialkyl phosphites are indifferent toward compounds with C=N and, not unlikely, C=O bonds. For instance, we could effect addition of phosphite **VII** to benzalacetone by the carbonyl group only under conditions of acid catalysis [9]. However, Arbuzov *et al.* could obtain the adduct in the absence of catalysts [10], by handling the reaction mixture for a long time. This result can be explained by partial hydrolysis of phosphite **VII** as a result of its unavoidable contact with air moisture.

Obviously, the synthetic result of reactions of azomethines and, probably, carbonyl compounds with dialkyl phosphites depends on the purity and the time of handling of the latter, since, because of contact with air moisture and subsequent hydrolysis we most probably dial here with a catalytic process.

## EXPERIMENTAL

The IR spectra were obtained on a Specord M-80 instrument for suspensions in mineral oil. The $^{31}$P NMR spectra were recorded on a Varian Unity-300 instrument (121 MHz), external reference 85%-naya $H_3PO_4$.

The thermograms were obtained on a home-made installation with electronic recording, heating rate 5 deg/min, chromel–alumel thermocouples, reference mineral oil. The reaction mixtures (0.5 g) were placed in Stepanov vessels and, after the exothermic effect had been passed, analyzed by $^{31}$P NMR and IR spectroscopy.

The solvents were purified by known procedures [11, 12].

Commercial benzalaniline was twice recrystallized from 2-propanol and dried in a vacuum. Commercial dimethyl phosphite and diisopropyl phosphite obtained by a known procedure were twice distilled in a vacuum and then twice distilled over metallic sodium under argon.

**Benzalanilinium phosphite (III).** A solution of 1.6 g of phosphorous acid in 15 ml of 2-propanol was added to a solution of 3.3 g of azomethine **I** in 20 ml of 2-propanol. Some minutes after, crystals formed and were filtered off (0.6 g). The mother liquor was reduced in part to obtain an additional 1.8 g of the reaction product (total yield 49%), mp 154–180°C. IR spectrum, ν, cm$^{-1}$: 1095, 1150 (POO$^-$), 1633 (C=N), 2000–3000 br (P–OH, P–H, NH$^+$). $^{31}$P NMR spectrum (2-propanol), $\delta_P$, ppm: 1.6 d ($^1J_{PH}$ 664.2 Hz).

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