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REACTION OF DIMETHYL PHOSPHITE AND MONOMETHYL PHENYLPHOSPHONITE WITH 2-METHYL-3,4,5-TRIPHENYLCYCLOPENTADIENONE

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As a continuation of studying the reactions of cyclones with hydrophosphoryl compounds we studied the reaction of dimethyl phosphite (DMP) and monomethyl phenylphosphonite (MPP) with the tricyclone 2-methyl-3,4,5-triphenylcyclopentadienone. Little information exists in the literature on the nucleophilic addition reactions to this cyclone. It is known [1] that, in the presence of basic catalysts, such nucleophiles as alcohols add to the double bond with two phenyl substituents to give 4-alkoxy-substituted cyclopentenones. In a similar manner, alcohols attack the β-C atom (relative to the C=O group) in the tetracyclone (TC) [1] and 2,5-bis-(carbomethoxy)-3,4-diphenylcyclopentadienone (BCDC) [2, 3] to give the products of 1,4-addition to the C=C–C=O system. In contrast to alcohols, the hydrophosphoryl compounds DMP [4], MPP [5], and ethyl phenylphosphonite [6], when reacted with TC give, besides the 1,4-adducts, the products of 1,6-addition to the conjugated system C=C–C=C–C=O, and they can also add to the C=O group [4, 6]. The direction of the reactions depends on the reaction conditions, and also on the structure of the cyclone and hydrophosphoryl compound [3-7].

Starting with the unsymmetrical structure of the tricyclone, it could be expected that a larger number of regioisomers is formed in the reactions with DMP and MPP than in the case of the TC (Scheme 1), in which connection the formation of the α-hydroxyphosphonate (I) is improbable, since the reactions were run in the absence of catalysts [4].

Based on the DTA data, the reaction of DMP and MPP with the tricyclone proceeds at a lower temperature than with the TC (Table 1). (See Scheme 1, next page.)

In the absence of catalysts the tricyclone, in contrast to the TC and similar to the BCDC [7], fails to give the products of addition to the oxygen of the C=O group (II) with either MPP or DMP, and it also fails to form the γ-phosphonoketones (III) and (IV), the products of 1,4-addition to the conjugated system C=C–C=O. The studied reactions proceed regiospecifically to give the unconjugated β-phosphonoketones (V), while the isomeric

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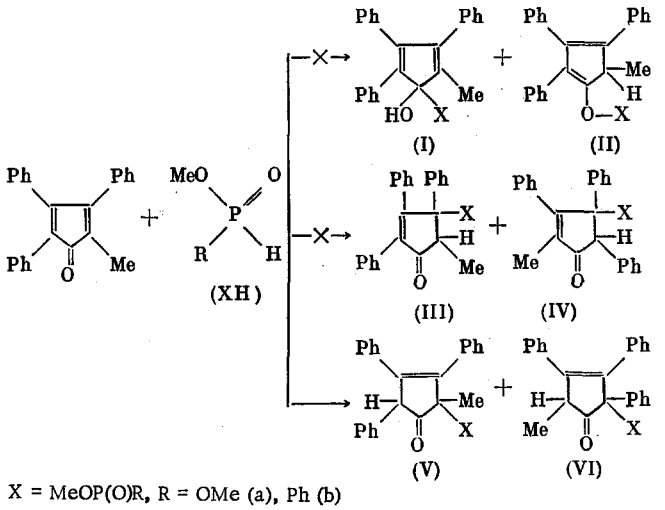
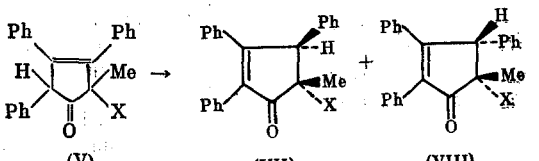
Scheme 1



X = MeOP(O)R, R = OMe (a), Ph (b)

ketones (VI) are not formed. Besides the unconjugated ketones (V), the conjugated β-phosphonoketones (VII) and (VIII), the prototropic isomerization products of (V), were also isolated from the reaction mixtures (Scheme 2).

Scheme 2



X = MeOP(O)R, R = OMe (a), Ph (b)

When the tricyclone is reacted with MPP, the amount of formed ketone (Vb) is substantially greater than that of ketone (Va) when reaction is with DMP. The structure of the products was established on the basis of the elemental analysis, and the IR and PMR spectral data (Tables 2 and 3).

The IR spectrum of the methyl ester of 2-methyl-3,4,5-triphenyl-3-cyclopenten-1-one-2-phenylphosphinic acid (Vb) has a ν C=O band at 1750 cm^{-1}, which indicates the unconjugated structure of this ketophosphinate, while the geminal arrangement of the CH_3 and $CH_3OP(O)Ph$ groups is confirmed by the fact that in the PMR spectrum the signal of the protons of the CH_3 group is split into a doublet, while the signal of the methine proton is a singlet (see Table 3).

The pure dimethyl ester of 2-methyl-3,4,5-triphenyl-3-cyclopenten-1-one-2-phosphonic acid (Va) could not be isolated due to the low yield of this product. Its formation is evidenced by the presence of the band ν C=O 1752 cm^{-1} in the IR spectrum of the reaction mixture. The formation of a smaller amount of (Va) than of (Vb) is explained by the higher temperature required for the reaction of the tricyclone with DMP than with MPP. Ketones (Va) and (Vb) are not stable under these conditions and isomerize to the conjugated ketones (VII) and (VIII). The conjugated structure of the latter is indicated by the ν C=O band in the 1690-1700 cm^{-1} region and the presence of a ν C=C band at 1630 cm^{-1} (see Table 2). The splitting of the signals of the CH_3 group and methine proton into doublets (see Table 3) definitely proves the geminal arrangement of the CH_3 and phosphono groups, since the PMR spectrum of the conjugated ketones (III) and (IV), the 1,4-addition products, would be different.

TABLE 1

Cyclone	Dimethyl phosphite		Monomethyl phenylphosphonite	
	start of exo effect, °C	maximum of exo effect, °C	start of exo effect, °C	maximum of exo effect, °C
Tetracyclone	152–160	188–192	83–86	116–120
Tricyclone	117–120	130–132	68–72	104–107

TABLE 2

Compound	Configuration	mp, °C	IR spectra (ν, cm^{-1})			
			POC	P=O	C=C	C=O
(Vb)	–	166–169	1030	1235	–	1750
(VIIa)	cis	–	1037, 1060	1252	1627	1705
(VIIIa)	trans	167–169	1040, 1066	1248	1630	1690
(VIIb)	cis	–	1038	1230	1632	1702
(VIIIb) *	trans	149–151	1045	1235	1630	1690
(VIIIb) *	trans	223–225	1034, 1038	1239	1632	1693

* Different diastereomers.

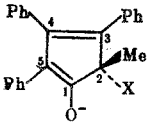
TABLE 3

Compound	Configuration	PMR spectra ($CHCl_3$, δ, ppm, J, Hz						^{31}P NMR spectra,* ppm
		CH	$^3J_{PH}$	$POCH_3$	$^3J_{PH}$	$PCCH_3$	$^3J_{PH}$	
(Vb)	–	4,86 s	–	3,36 d	11	1,30 d	17,5	–
(VIIa)	cis	5,09 d	15	3,82 i 3,79 d.d.	10,5	1,06 d	18	–37,6
(VIIIa)	trans	4,47 d	8	3,64 i 3,26 d.d.	11	1,79 d	15,5	–35,4
(VIIb)†	cis	5,34 d	14	3,72 d	10,5	0,82 d	18	–55,2
(VIIb)†	cis	5,03 d	15	3,77 d	10,5	1,15 d	15	–53,5
(VIIIb)†	trans	4,47 d	4,5	3,19 d	11	1,68 d	16	–53,5
(VIIIb)†	trans	4,51 d	5	3,17 d	11	1,82 d	14	–52

* Determined by the ^1H-$\{^{31}P\}$ double resonance method.
† Different diastereomers.

It should be mentioned that for the conjugated β-phosphonoketones (VII) and (VIII) the intensity and frequency of the νC=C bands are close to the ν C=C bands of the conjugated β-phosphonoketones, which were obtained by reacting DMP and MPP with TC [4, 5]. In the latter case the value ν C=C 1630-1640 cm^{-1} and the average intensity of this band served as a criterion of the β-position of the phosphono and C=O groups, since for the γ-phosphonoketones [4-6] the ν C=C band is located toward lower frequencies, is superimposed on the ν C=C band of the stretching vibrations of the aromatic ring, and has a weak intensity. As a result, this criterion is also operative for the conjugated β-phosphonoketones obtained from the tricyclone.

In contrast to the unconjugated β-phosphonoketones, obtained from the TC [4-6], the prototropic isomerization of (V) proceeds in a stereospecific manner to give ketones (VIII) with a trans arrangement of the methine proton and phosphono group, while the cis-isomers (VII) are formed to the extent of only 8-10%. This is explained by the fact that in the anion (IX), which is formed as an intermediate during the isomerization of (V), the approach of the proton toward the C^3 atom is less hindered from the side of the CH_3 group than from the side of the phosphono group. The formation of the trans-isomer is not as preferred, when instead of the CH_3 group the more bulky phenyl substituent is found in the molecule [4-6].



(IX)

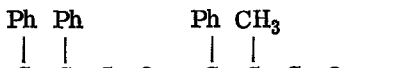
The separation of the reaction products of the tricyclone with DMP and MPP was effected by fractional crystallization and column chromatography. The trans-isomers (VIIIa) and (VIIIb) were isolated in the crystalline state, while the cis-isomers (VIIa) and (VIIb) were isolated as thick, noncrystallizing oils. The assignment of ketones (VII) and (VIII) to the cis- or trans-isomers was based on the value of the vicinal spin–spin coupling constant $^3J_{PH}$ [8, 9].

The correct assignment of the structure of ketones (VII) and (VIII) is also confirmed by the fact that in the cis-isomers (VII) the values of the δCH_3 chemical shifts are shifted upfield when compared with the trans-isomers (VIII). According to [10], this can be explained by the shielding effect of the phenyl group, found cis

to the CH_3. The trans-isomer (VIIIb) was isolated as two diastereomers, but the cis-isomer (VIIb) could not be separated into the diastereomers, although in one of the chromatography fractions the amount of one diastereomer greatly exceeded the amount of the other, which made it possible to correctly assign the signals of their protons in the PMR spectrum (see Table 3). It should be mentioned that when chromatographed on a Silufol plate the diastereomeric ketophosphinates (VIIb) and (VIIIb) have practically identical R_f values, and consequently their separation by chromatographing on a column presents great difficulties.

The reaction of DMP with the tricyclone was run previously [11] in refluxing DMP; (VIIIa) was isolated here, which, based on the $^3J_{PH}$ value (8 Hz), was erroneously assigned the structure of the cis-isomer (VIIa) (see Table 3).

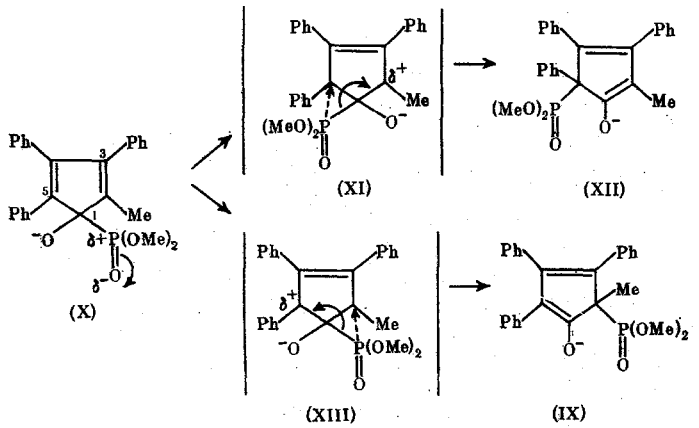
As a result, DMP and MPP, in contrast to alcohols [1], also form compounds with the tricyclone that can be regarded as being the products of 1,6-addition to the conjugated system of the cyclone. At the same time, from the standpoint of controlling the charges, nucleophilic attack on the α-C atom is improbable [7], since the most electrophilic center in the tricyclone molecule, the same as in other cyclones and α,β-unsaturated ketones, is the carbon atom of the C=O group. However, the addition products of nucleophiles, including phosphorus nucleophiles, to the C=O group are unstable, and under the influence of bases or at elevated temperature they decompose into the starting components, and consequently their formation cannot always be observed. In the case of an unsymmetrically substituted tricyclone it is also possible to form the 1,4-adducts at



either the C=C−C=O or C=C−C=O groupings, but neither DMP nor MPP forms the 1,4-addition products with the tricyclone.

The formation of the 1,6-adducts of hydrophosphoryl compounds with cyclones can be explained if it is assumed that attack by the nucleophiles is initially directed to the carbonyl C atom to give an anion, in which the phosphono group migrates to the adjacent carbon atom under the reaction conditions [7]. In anion (X), formed in the attack of DMP on the C atom of the C=O group of the tricyclone (Scheme 3), the phosphono group can migrate to both the C^5 and the C^2 atom. Under the conditions of controlling the charges the most probable would be migration of the phosphono group to the C^5 atom. However, for steric reasons, approach to the C^2 atom, which bears a less bulky substituent, is much easier, and consequently the formation of anion (IX) must be considered to be the most probable. In addition, when going to this anion not a single phenyl ring is withdrawn from the conjugation, which makes it more stabilized resonance wise than anion (XII).

Scheme 3



The reaction of DMP and MPP with the tricyclone gives, besides β-phosphonoketones (V), (VII), and (VIII), also the tricyclone reduction products, whose structure will be reported by us later.

EXPERIMENTAL

The PMR spectra were recorded on Varian T-60 and Varian HA-100D spectrometers, using $CHCl_3$ as the solvent and TMS as the internal standard. The IR spectra were taken on a UR-10 spectrophotometer. The thermographing was carried out in sealed Stepanov cups on an HTP-70 instrument equipped with a linear heating unit. The thermocouple was Chromel–Alumel, and the heating rate was 4 deg/min. The melting points were determined on a Kofler block.

Reaction of Tricyclone with Dimethyl Phosphite. a) A mixture of 2 g of the tricyclone and 1.3 g of DMP was placed in a bath heated to 150°C and kept there for 30 min. Treatment of the reaction mass with a mixture of ether and hexane gave white crystals of the trans-isomer of the dimethyl ester of 2-methyl-3,4,5-triphenyl-4-cyclopenten-1-one-2-phosphonic acid (VIIIa); yield 2.12 g (79%), mp 167-169°. Found: C 72.01; H 5.92; P 6.98%. $C_{26}H_{25}O_4P$. Calculated: C 72.21; H 5.82; P 7.16%. The residual reaction mass was chromatographed on a silica gel column to give 0.19 g (7%) of the cis-isomer (VIIa) as a thick oil and an additional 0.24 g (9%) of (VIIIa). For VIIa). Found: C 71.98; H 6.03; P 7.30%. $C_{26}H_{25}O_4P$. Calculated: C 72.21; H 5.82; P 7.16%. In addition, the tricyclone reduction products, namely the corresponding isomeric dihydro derivatives, were detected.

b) A mixture of 1 g of the tricyclone and 0.65 g of DMP was thermographed up to 130-132° (maximum temperature of exo effect). Treatment of the reaction mass with ether gave crystals of ketone (VIIIa). Based on the IR spectral data, the residual mass contained (VIIIa), the starting tricyclone, and a small amount of the unconjugated β-ketophosphonate (Va).

Reaction of Tricyclone with Methyl Phenylphosphonite. a) A mixture of 1 g of the tricyclone and 0.7 g of MPP was thermographed up to 104-107° (maximum temperature of exo effect). Treatment of the cold melt with ether gave 0.57 g (39% yield) of ketone (Vb) as white crystals with mp 166-169°. Found: C 77.50; H 5.91; P 6.37%. $C_{31}H_{27}O_3P$. Calculated: C 77.80; H 5.70; P 6.47%. Based on the IR and PMR spectral data the residual reaction mass contained a mixture of conjugated (VIIb) and (VIIIb) and unconjugated (Vb) ketones in a 1:1 ratio.

b) A mixture of 2 g of the tricyclone and 1.4 g of MPP was placed in a bath heated to 140° and kept there for 1 h. The obtained clear yellowish mass was dissolved in a little CH_2Cl_2. Then ether was added and the mixture on standing deposited a crystalline mixture of the diastereomers of the methyl ester of 2-methyl-3,4,5-triphenyl-4-cyclopenten-1-one-2-phenylphosphinic acid (VIIIb). Yield 1.92 g (65%). By repeated fractional crystallization from an ether–benzene–$CHCl_3$ mixture (VIIIb) was separated into the diastereomers with mp 149-151°. (Found: C 77.52; H 5.81; P 6.40%) and 223-225° (Found: C 77.95; H 5.85; P 6.51%). $C_{31}H_{27}O_3P$. Calculated: C 77.80; H 5.70; P 6.47%. The reaction mass remaining after separation of the crystalline portion was chromatographed on silica gel and here we isolated an additional 0.35 g (12%) of (VIIIb), and also 0.24 g (8%) of the cis-isomer (VIIb) as a thick oil. Some of the fractions also contained ketone (V) and the isomeric dihydro derivatives of the tricyclone.

Thermal Isomerization of β-Ketophosphinate (Vb). Employing thermographic control, 1 g of ketone (Vb) was heated up to 140°. Treatment of the reaction mass with ether gave 0.54 g of a crystalline mixture of the (VIIIb) diastereomers. The residual mass was chromatographed on silica gel to give an additional amount of crystalline (VIIIb), a small amount of the cis-isomer (VIIb) as a thick oil, and the reduction products of the tricyclone.

CONCLUSIONS

In the absence of catalysts, the reaction of dimethyl phosphite and monomethyl phenylphosphonite with 2-methyl-3,4,5-triphenylcyclopentadienone goes with the formation of unconjugated β-phosphonoketones, which under the reaction conditions undergo prototropic isomerization to the conjugated β-phosphonoketones. In contrast to alcohols, dimethyl phosphite and monomethyl phenylphosphonite when reacted with the tricyclone do not form the products of 1,4-addition to the conjugated C=C–C=O system of the cyclone.

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REACTION OF TRIALKYL PHOSPHITES WITH 1,1,1-TRIFLUOROACETONE

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The reactions of trialkyl phosphites with aliphatic aldehydes at 20°C [1], and also with chloral [2] and pentafluorobenzaldehyde [3] at low temperatures, give the corresponding 2,2,2-trialkoxy-1,4,2-dioxaphospholane derivatives, in which connection the 2,2,2-trialkoxy-3,5-bis(pentafluorophenyl)-1,4,2-dioxaphospholane when heated is converted to the 2,2,2-trialkoxy-4,5-bis(pentafluorophenyl)-1,3,2-dioxaphospholane [3]. According to [3], the formation of the 1,4,2-dioxaphospholanes is characteristic for aldehydes, while ketones, activated by electron-acceptor substituents, when reacted with trialkyl phosphites, give only the 1,3,2-dioxaphospholanes.

To obtain more detailed information on the mechanism of the reactions of activated carbonyl compounds with trialkyl phosphites we studied the reaction of the latter with 1,1,1-trifluoroacetone by the ^{31}P NMR method. In the ^{31}P NMR spectrum of a mixture of $(EtO)_3P$ and $MeCOCF_3$ (1:2) at 20° are observed the appearance, and then a decrease in the signal with δ 37 ppm of the corresponding 2,2,2-triethoxy-3,5-dimethyl-3,5-bis(trifluoromethyl)-1,4,2-dioxaphospholane. We recently isolated a similar dioxaphospholane in the pure state from the reaction of ethyl ethylene phosphite with $MeCOCF_3$ [4]. Later in the spectrum appears and increases the signal with δ 75 ppm of the end reaction product, namely triethoxy-α-trifluoromethyl-α-(1-methyl-2,2-difluorovinyloxy)ethoxyfluorophosphorane. For phosphoranes with a close structure, containing a P–F bond, δ 75 ppm [5]. A gradual decrease in the signal of the starting $(EtO)_3P$ (–138 ppm) is observed during reaction. The process is accompanied by the formation of a small amount of a phosphate with δ 3 ppm. The reaction is completed in 216 h (Fig. 1). The analogous reaction of $(MeO)_3P$ with $MeCOCF_3$ is completed in 168 h. Here, besides trimethoxy-α-trifluoromethyl-α-(1-methyl-2,2-difluorovinyloxy)ethoxyfluorophosphorane (δ 75 ppm) and the phosphate (δ 3 ppm), is formed a small amount of dimethyl methylphosphonate (δ –32 ppm) (Fig. 2a). It was found that the product with δ 3 ppm (Fig. 2b) could be removed by distillation of the reaction mixture. The PMR spectrum of the residual mixture has the signals (δ ppm, J, Hz): 1.35 (MeP, J_{HP} = 18) and 3.65 (MeOP, J_{HP} = 11), which coincide with those observed for the authentic $MePO(OMe)_2$.

The structure of trimethoxy-α-trifluoromethyl-α-(1-methyl-2,2-difluorovinyloxy)ethoxyfluorophosphorane was confirmed by the ^{19}F NMR spectrum (δ, ppm, J, Hz): 1.55 (CF_3, J_{FP} = 7), 5.48 (CF_2), –2.44 (FP, J_{FP} = 1115). For fluorophosphoranes of analogous structure, J_{FP} = 822 Hz [5]. The ratio of the integral intensity of the CF_3, $=CF_2$, and F signals is equal to 3:2:1. The IR spectrum has a very weak band at 1680 cm^{-1} (ν C=C), which has a medium intensity in the Raman spectrum.

The mass spectrum of the reaction products was obtained at 50 eV, m/e (relative intensity, % of total ionic current down to m/e 29, $\%\Sigma_{29}$): 333(0.23), 329(0.02), 319(0.04), 318(0.09), 317(1.3), 303(0.04), 302(0.32), 297(0.05), 295(0.14), 287(0.08), 282(0.091), 279(0.23), 272(0.14), 263(0.09), 262(0.18), 233(0.68), 223(0.23), 222(0.18), 221(0.13), 193(0.41), 185(0.319), 183(0.73), 171(0.52), 169(1.71), 153(0.64), 151(0.50), 149(0.32), 141(2.0), 140(0.41), 139(0.50), 127(2.7), 124(1.8), 121(0.82), 120(0.27), 110(0.32), 117(0.18), 113(5.9), 110(4.1), 109(5.9), 103(0.55), 97(0.39), 96(0.41), 95(2.7), 94(7.7), 93(4.1), 91(0.68), 90(0.41), 89(0.41), 85(0.23), 83(0.23), 82(0.32), 81(0.55), 80(0.55), 79(8.7), 78(0.23), 77(1.2), 76(0.23), 75(0.41), 69(1.7), 65(0.82), 64(0.32), 63(2.2), 59(0.91), 58(0.36), 57(0.36), 55(0.32), 53(0.18), 51(0.60), 50(0.18), 49(0.27), 48(0.36), 47(2.37), 46(0.23), 45(1.23), 44(2.0), 43(4.57), 42(0.41), 41(0.59), 40(2.3), 39(0.68), 33(0.55), 31(5.5), 30(1.7), 29(4.6). The peak with m/e 124 corresponds to the molecular ion $MePO(OMe)_2$, whose mass spectrum is known only at 70 eV [6]. We obtained the mass spectrum of this compound at 50 eV, m/e ($\%\Sigma_{29}$): 124(6.9), 109(9.6), 94(24.4), 93(9.9), 79(26), 63(4.7), 47(7.2), 45(1.1), 31(4.6), 29(5.4).

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