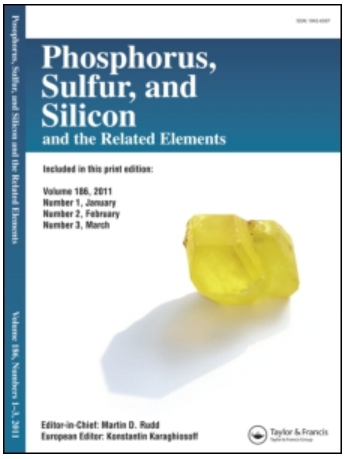

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The addition of trialkyl phosphites **2a,b** to 3-methylene-oxindoles, **8a,b** proceeded according to the 1:2 pattern to give **13a–d** as major products. Compounds **14a–d** (<10%) were also isolated and identified from the same reactions. Compounds **14** were exclusively obtained when controlled amounts of water were present in the reaction medium or when **8a,b** were reacted with the appropriate dialkyl phosphite (**15**). Analytical and spectroscopic (IR, ^{1}H, ^{31}P, ^{13}C NMR and MS) measurements are compatible with the proposed structures.

Key words: 3-Methylene-oxindoles, phosphorylation, carbon-alkylation.

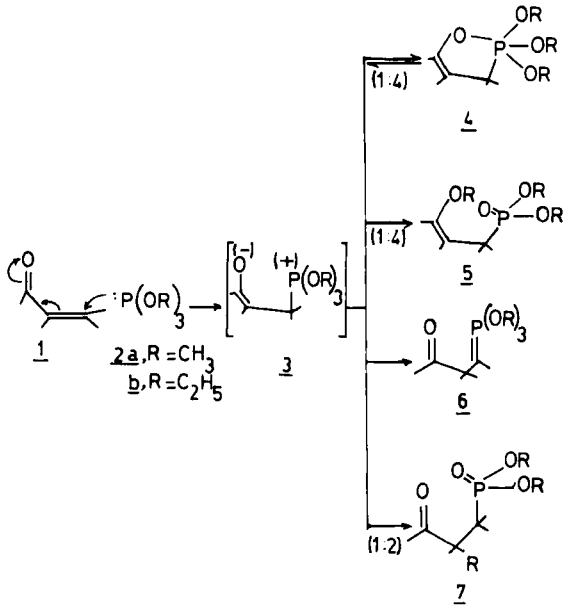
INTRODUCTION

The behaviour of α, β-unsaturated carbonyl compounds **1** toward attack by trialkyl phosphites (**2**) has attracted the interest of several groups of investigators. In principle, carbophilic attack on **1** by the nucleophilic reagents **2** occurs *via* 1:4 addition to give the dipolar form **3** (Scheme 1) which is either resonance-stabilized or suffers transformations depending upon the nature of reactants and/or the reaction conditions. Thus, betaine **3** cyclizes to (or exists in equilibrium with) a tetraoxyphosphorane structure (**4**)[2–6] or undergoes *O*-alkylation *via* alkyl group translocation to afford ether phosphonate adducts (**5**).[7–10] Stabilization of betaine **3** *via* proton transfer to give phosphite-methylenes (ylides, **6**) is also known.[11] In the present investigation, we report on a novel type of attack by **2** on systems **1** where *C*-alkylation at intermediate stage **3** occurs to give a new phosphonate structure (**7**) according to the 1:2 addition pattern.

RESULTS AND DISCUSSION

We have found that the reaction of trimethyl phosphite (TMP, **2a**) with 3-dicyanomethylene-oxindole (**8a**)† was completed (TLC) when the reactants

† Compounds **8a** and **8b** are also known as 2-(2-oxo-2,3-dihydro-1*H*-indolid-2-ene)malonitrile and ethyl 2-(2-oxo-2,3-dihydro-1*H*-indolid-2-ene)cyanoacetate, respectively.

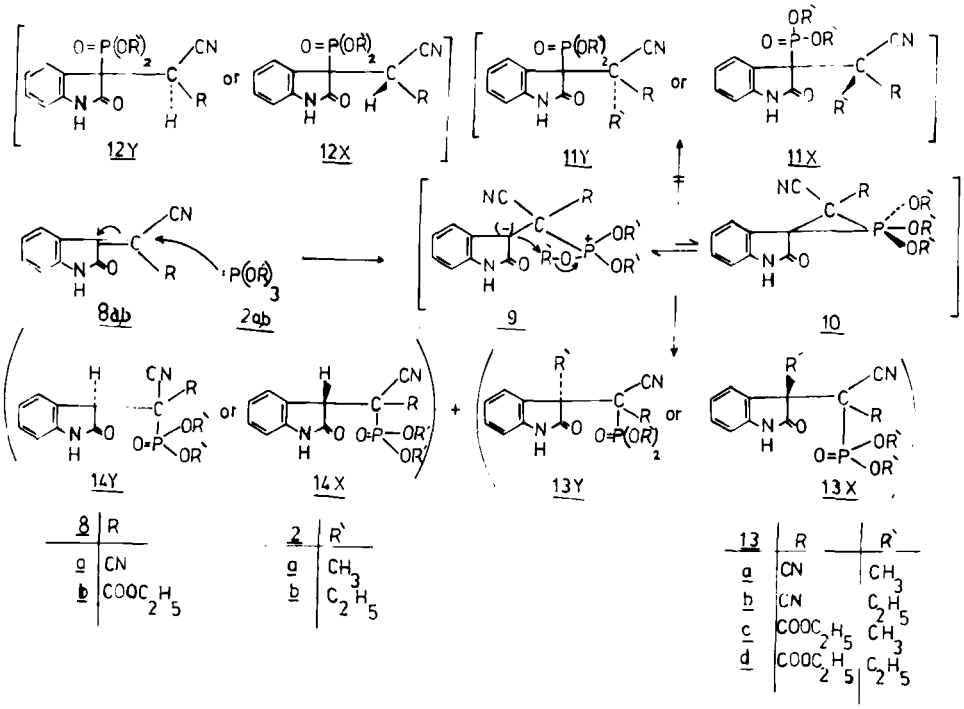


SCHEME 1

were boiled in dry toluene for 8 h. The crude reaction mixture indicated the presence of two products (TLC) which are phosphonate in nature[12] (^{31}P NMR, *vs.* 85% H_3PO_4: positive shifts around δ 29 ppm). Careful working up of the mixture by column chromatography yielded two colorless crystalline materials ("A" and "B") (Scheme 2). The first ("A", 90%, δ ^{31}P NMR: 29.6 ppm) was formulated as dimethyl[dicyano(2,3-dihydro-3-methyl-2-oxo-1*H*-indol-3-yl)methyl]-phosphonate (**13a**)† for the following reasons: a) The microanalytical data and molecular weight measurement (MS) for **13a** corresponded to $C_{14}H_{14}N_3O_4P$ (*m/z* 319, M^+, 6%). b) The strong amidic carbonyl band present in the spectrum of **8a** at 1720 cm^{-1} was also recorded in the spectrum of **13a** (1725 cm^{-1}). Presence of $>C{=}O$ group in **13a** was also attested by a signal at δ 169.9 ppm in its ^{13}C NMR spectrum. Meanwhile, presence of $-\overset{|}{\underset{|}{C}}-CH_3$ group in **13a** was strongly supported by a signal at δ 1.89 (3H, d, $^{1,4}J_{HP} = 4.5$ Hz) in its PMR spectrum[13] as well as by a signal at δ 19.3 ppm in its ^{13}C NMR spectrum.[13,14] The ^{13}C NMR spectrum also showed a signal at δ 35.2 ppm; a value which coincides with a chemical shift expected for the ring sp^3-carbon atom bearing a methyl group.[13,14]

The second product ("B", *ca.* 8%, ^{31}P NMR: δ 29.7 ppm) was formulated as dimethyl[dicyano(2,3-dihydro-2-oxo-1H-indol-3-yl)methyl] phosphonate (**14a**) for the following reasons: a) Its elemental analyses and molecular weight determination (MS) agreed with the molecular formula $C_{13}H_{12}N_3O_4P$ (*m/z* 305, M^+, 48%). b) Its IR spectrum showed strong absorption bands at 3160 (NH), 1720 (C=O),

† Nomenclutures are according to the current "Chemical Abstract" index names.



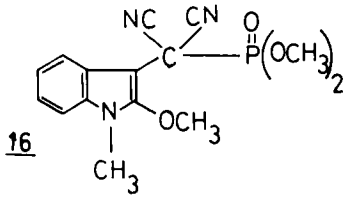
SCHEME 2

1250 (P=O) and 1035 cm^{-1} (P—O—CH_3).[15] c) The PMR spectrum of **14a** disclosed the presence of signals at δ 7.4 (4H, H ar., m), δ 11.43 (N*H*, s, exchangeable with D_2O), δ 3.73 (6H for the methoxyl groups attached to phosphorus, d, $^{1,3}J_{HP} = 12$ Hz). The signal due to the —C—C*H*$_3$ group present in the PMR spectrum of **13a** at δ 1.89 (*vide supra*) was absent in the spectrum of **14a**. Instead, a doublet of doublet appeared at δ 5.21 (1H, d.d., $^{1,3}J_{HP} = 8$ Hz) which is assignable to the ring methine proton. d) Moreover, compound **14a** was unequivocally obtained (in *ca.* 95% yield) when **8a** was allowed to react with dimethyl phosphite (**15a**) in absence of solvent at 100°C (Scheme 3).

8a,b + $(R'O)_2P(O)H \rightarrow$ **14a**, R = CN; R' = CH_3
15a, R' = CH_3 **b**, R = CN; R' = C_2H_5
b, R' = C_2H_5 **c**, R = CN; R' = C_3H_7-i
c, R' = C_3H_7-i **d**, R = $COOC_2H_5$; R' = CH_3
e, R = $COOC_2H_5$; R' = C_2H_5
f, R = $COOC_2H_5$; R' = C_3H_7-i

SCHEME 3

Treatment of **14a** with methyl iodide in acetone in the presence of anhydrous potassium carbonate did not afford **13a**. Instead, a colorless crystalline substance proved to have a structure like **16** was isolated and identified (*cf.* experimental).



16

Based upon these arguments, structures **13** and **14** seem to be more favourable than the other alternative structures **11** and **12** respectively for the reaction products of **8** with the appropriate alkyl phosphite.

Compounds **13b–d** and **14b–f** were similarly prepared by reacting **8a** or **8b** with the appropriate alkyl phosphite.

A mechanism that accounts for formation of compounds **13** from the reaction of **8a,b** with trialkyl phosphites **2a,b** is depicted in Scheme 2. This involves primary nucleophilic attack by the phosphite-phosphorus on the exocyclic methide-carbon in **8** to yield the dipolar species **9**; existing probably in equilibrium with the ring phosphorane form **10**. Structure **9** undergoes then intramolecular group translocation to yield **13**. Formation of compounds **14** from the reaction of trialkyl phosphites (**2a,b**) with **8a,b** may be attributed to partial hydrolysis of these phosphites to yield dialkyl phosphites (**15a,b**).[16] This is supported by the fact that compounds **14a,b,d,e** are formed almost quantitatively when controlled amounts of water are added to the reaction mixture of **8a** (or **8b**) with the appropriate trialkyl phosphite (experimental).

It should be noted that compounds **13** can exist in one of the diastereomeric forms **13X** or **13Y**. The same is also for compounds **14** (**14X** or **14Y**). However, the already available spectroscopic data can not decisively differentiate between the postulated structures. The specific spectroscopic facilities necessary to solve this problem are not available in the institute.

CONCLUSION

From the present study, it is safe to state that the exocyclic ethylenic part of the α,β-unsaturated carbonyl system in 3-methylene-oxindoles **8a,b** is the only vulnerable site of attack by trialkyl phosphites. This seems to be attributable, partly, to activation of the olefinic bond by the electronegative substituents (CN or $COOC_2H_5$) and, partly, to deactivation of the carbonyl function by its amidic nature.[17]

The present investigation thus offers a new pattern for the addition of trialkyl phosphites to α,β-unsaturated carbonyl systems. Moreover, a new area has been explored for utilizing trialkyl phosphites as alkylating agents,[18,19] and contributes to the conventional methods applied for carbon-alkylation.[20–24]

EXPERIMENTAL

All melting points are uncorrected. The IR spectra were recorded, in KBr, with Perkin-Elmer Infracord Spectrometer, 157 G. The PMR spectra were run on Varian Spectrometers at 60 MHz and/or 90 MHz, using TMS as an internal reference. ^{31}P and ^{13}C NMR spectra were recorded with a varian FT-80 Spectrometer. ^{31}P NMR spectra were recorded relative to external H_3PO_4 (85%). ^{13}C NMR spectra were recorded relative to internal TMS. The mass spectra were run at 70 eV on Kratos MS-50 equipment provided with a data system.

The appropriate precautions in handling moisture-sensitive compounds were observed. Solvents were dried by standard techniques, including high vacuum procedures.

Reagents: Trialkyl phosphites (**2a,b**) were purified by treatment with sodium ribbon followed by fractional distillation. Dialkyl phosphites were freshly distilled.

Action of Trialkyl Phosphites on 3-Methylene-oxindoles (**8**). *General Procedure:* A mixture of 3-methylene-oxindole (**8**)[25] (0.01 mol) and trialkyl phosphite (trimethyl- or triethyl-phosphite (4 ml) was refluxed in dry toluene for 8 h. After the reaction was completed (TLC), the volatile materials were evaporated, *in vacuo*. The residual substance was redissolved in methanol (100 ml) and evaporated to dryness in the presence of silica gel (5 g). The mixture was then added to a column previously charged with silica gel in cyclohexane. The column was developed with cyclohexane containing increasing amounts of ethyl-acetate.

Fraction (up to 5:5 v/v) eluted a colorless substance; recrystallized from the appropriate solvent (*cf.* Table I) to give colorless crystals shown to be compounds **14**.

Elution with pure ethyl acetate yielded a colorless material; recrystallized from the appropriate solvent (*cf.* Table I) to give a colorless crystalline product identified as **13**. Percentage yields, physical and analytical data for compounds **14** and **13** are given in Tables I, II and III.

Compounds **14** respond negatively to the $FeCl_3$ reaction.

TABLE I

Cpd.	%[a]	mp. °C	Mol. Form. (M. Wt.)	Anal (calcd./Found) C	H	N	P	M^+ *m/z*	IR(cm^{-1})* NH	C=O	P=O	P—O—C
13a	90	192[b]	$C_{14}H_{14}N_3O_4P$	52.66	4.42	13.16	9.70	319	3160	1725	1260	1030
			(319.27)	52.48	4.83	12.97	9.47					
b	90	147[c]	$C_{17}H_{20}N_3O_4P$	56.51	5.58	11.63	8.57	361	3200	1720	1240	1030
			(361.34)	56.42	5.37	11.59	8.49					
c	90	162[d]	$C_{16}H_{19}N_2O_6P$	52.46	5.22	7.64	8.45	366	3290	1735	1250	1030
			(368.32)	52.15	5.03	7.51	8.27			&1700		
d	90	137[b]	$C_{19}H_{25}N_2O_6P$	55.88	6.17	6.86	7.58	408	3200	1780	1260	1025
			(408.39)	55.76	6.08	6.69	7.47			&1720		
14a	8	222[e]	$C_{13}H_{12}N_3O_4P$	51.16	3.96	13.76	10.15	305	3160	1720	1250	1035
	(90)		(305.24)	50.47	3.88	13.65	10.30					
b	6	164[d]	$C_{15}H_{16}N_3O_4P$	54.05	4.84	12.60	9.29	333	3165	1720	1245	1010
	(90)		(333.29)	53.99	4.69	12.53	9.08					
c	—	150[e]	$C_{17}H_{20}N_3O_4P$	56.50	5.58	11.63	8.57	361	3175	1720	1235	1000
	(95)		(361.35)	56.52	5.56	11.58	8.39					
d	5	195[e]	$C_{15}H_{17}N_2O_6P$	51.14	4.86	7.95	8.79	352	3160	1750	1240	1050
	(95)		(352.28)	50.92	4.69	7.83	8.77			&1720		
e	5	138[d]	$C_{17}H_{21}N_2O_6P$	53.68	5.56	7.37	8.14	380	3170	1750	1240	1040
	(95)		(380.34)	53.36	5.48	7.18	8.93			&1720		
f	—	125[b]	$C_{19}H_{25}N_2O_6P$	55.87	6.17	6.86	7.58	408	3180	1750	1235	1000
	(95)		(408.41)	55.85	6.07	6.85	7.49			&1725		

[a] Yields are approximated. Values into parentheses are the % yields of **14a–f** originating from the reaction of **8a,b** with the appropriate dialkyl phosphite (**15**). [b] Solvent of crystallization is cyclohexane. [c] Solvent of crystallization is chloroform-n-pentane, (1:1 v/v). [d] Solvent of crystallization is benzene. [e] Solvent of crystallization is ethylacetate.

* The C≡N absorption bands are relatively weak or absent.

TABLE II

NMR spectral data[a]

Cpd.[b]	^{31}P	^{1}H					
13a	29.6	1.89 d		3.82 d		7.3	9.33
		$^{1,4}J = 4.5$		$^{1,3}J = 13.5$		m	s
		3H		6H		4H	1H
		C—CH_3		$POCH_3$		arom.	N*H*
b	29.3	1.35 t	1.95 q	4.25 q		7.27	9.61
		$^{1,4}J = 11.5$				m	s
		6H	2H	4H		4H	1H
		$POCH_2CH_3$	C—CH_2	$POCH_2$		arom.	N*H*
c	28.7	0.88 t	2.03 d	3.69 d		7.24	11.03
		$J_{HH} = 7$	$^{1,4}J = 3$	$^{1,3}J = 12$		m	s
		3H	3H	6H		4H	1H
		$OOCH_2CH_3$	C—CH_3	$POCH_3$		arom.	N*H*
d	29.4	1.36 m	2.4 q	4.18 m		7.25	11.5
						m	s
		12H	2H	8H		4H	1H
		$POCH_3$&$OOCH_3$	C—CH_2	$POCH_2$&CH_2		arom.	N*H*
		&C—CH_2CH_3		&$OOCH_2$			
14a	29.7			3.73 d	5.21 d	7.4	11.43
				$^{1,3}J = 12$	$^{1,3}J = 8$	m	s
				6H	1H	4H	1H
				$POCH_3$	C—*H*	arom.	N*H*
b	29.9	1.40 dt		4.25 q	4.81 d	7.26	9.25
		$^{1,4}J = 11.5$		$^{1,3}J = 11.5$	$^{1,3}J = 8$	m	s
		$_{6H}J_{HH} = 4$		4H	1H	4H	1H
		$POCH_2CH_3$		$POCH_2$	C—*H*	arom.	N*H*
d	30.3	0.85 t	3.76 q	4.22 d	4.93 d	7.2	10.88
		$J_{HH} = 6$		$^{1,3}J = 12$	$^{1,3}J = 6$	m	s
		3H	2H	6H	1H	4H	1H
		$OOCH_2CH_3$	$COOCH_2$	$POCH_3$	*C*—*H*	arom.	N*H*
e	29.2	0.83 t	1.13 t	3.86 m	4.86 d	7.14	10.95
		$J_{HH} = 6$	$^{1,4}J = 11.5$		$^{1,3}J = 6$	m	s
		3H	6H	6H	1H	4H	1H
		$COOCH_2CH_3$	$POCH_2CH_3$	$POCH_2$&$OOCH_2$	C—*H*	arom.	NH
f	29.9	0.93 t	1.24 d	3.76 q	4.52 sept.	4.71 d	7.69	11.05
		$J_{HH} = 7.5$	$J_{HH} = 6.5$	$J_{HH} = 7.5$	$^{1,3}J = 11.5$	$^{1,4}J = 6.5$	m	s
		3H	12H	2H	2H	1H	4H	1H
		$OOCH_2CH_3$	$CH(CH_3)_2$	$OOCH_2$	$POCH$	C—*H*	arom.	N*H*

[a] In δ scale ppm.;

[b] The solvent is $CDCl_3$/DMSO;

[c] The hydrogens of the CH_3 group are partially obscured.

Action of Dialkyl Phosphites on 3-Methylene-oxindoles (**8**). *General Procedure:* A mixture of 3-methylene-oxindole (**8**) (0.01 mol) and dialkyl phosphtie (dimethyl, diethyl or di-isopropyl phosphite) (4 ml) was heated in absence of solvent at 100°C for *ca.* 2 *h*. After removing the volatile materials, *in vacuo*, the residue was triturated with light petroleum and left to cool. The solid so formed was collected, and recrystallized from a suitable solvent to give compounds **14** almost in quantitative yields (90–95%). Physical and analytical data for compounds **14** are presented in Tables I, II and III.

Reaction of 3-Methylene-oxindoles 8a,b with Trialkyl Phosphites in the presence of Water. A mixture of **8a** (or **8b**) (0.01 mol), trimethyl phosphite (or triethyl phosphite) and water (1 ml) was boiled in toluene for 6 h. After the reaction has been completed (TLC), the volatile materials were evaporated, *in vacuo*. The residual substance was collected and recrystallized from the appropriate solvent (*cf.* Table I) to give compounds **14a,b,d,e** (yields: 60, 56, 63, 72%, respectively). Their identities were

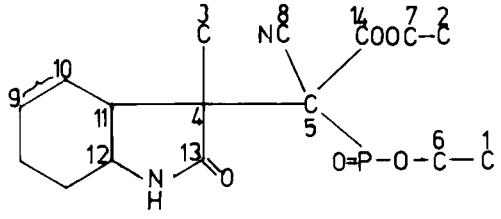
TABLE III

^{13}C NMR data[a,b]

Cpd.	Carbon #1	2	3	4	5	6	7	8	9	10	11	12	13	14
13a[c]			19.32	35.2	38.0	54.2		109.4	121.2	125.9	129.6	140.8	169.9	
14a[d]				26.8	37.9	55.3		110.3	121.2	125.3	130.6	142.6	169.7	
b[c]	13.5			27.2	38.2	63.1		109.7	123.2	125.6	130.7	142.0	169.8	
d[c]		14.3		26.2	39.4	55.0	62.2	109.6	121.3	125.8	129.7	143.2	169.2	171
e[c]	13.1	15.7		26.0	38.0	62.2[e]	64.2[e]	109.5	121.2	125.6	129.5	142.3	169.0	172

[a] See experimental for details of NMR experiments.

[b] The numbering system is as drawn:



[c] The solvent is DMSO.

[d] The solvent is $CDCl_3$.

[e] The resonance is obscured.

established by mp., mixed mps. and comparative IR spectral determinations with the corresponding reference samples.

Thermal Decomposition of the Adduct **14a**. The phosphonate adduct **14a** (1 g) was heated (bath temp., 230°C) for one hour under reduced pressure (5 mm/Hg) in a cold finger sublimator. The reaction vessel was left to cool and ethyl alcohol (5 ml) was added. The crystals which separated were recrystallized from ethanol to give the oxindole **8a** (identified by mp., mixed mps. and comparative IR spectra).[25] Dimethyl phosphite was detected in the receiver by the development of a violet color on addition of 3,5-dinitrobenzoic acid in the presence of alkali.[26]

Methylation of the Phosphonate **14a**. To a stirred solution of **14a** (1.5 g, 0.005 mol) in dry acetone (100 ml) was added 5 g of anhydrous K_2CO_3. Stirring was continued at room temperature for 1 h. Freshly distilled CH_3I (4.2 g, 0.03 mol) was then added and the mixture was gently heated under reflux for 10 h. The inorganic and volatile materials were removed to give **16** (1.7 g, 70%) in a semi-solid form which solidified after being triturated with cold pentane. This material was recrystallized from $CHCl_3$-light petroleum to yield **16** as white crystals mp. 78°C. Anal. Calcd. for $C_{15}H_{16}N_3O_4P$ (333.29). C, 54.05; H, 4.84; N, 12.60; P, 9.29. Found: C, 53.88; H, 4.69; N, 12.47; P, 9.18. MS: m/z 333. IR (KBr): Bands at 2260 (CN), 1245 (P=O) and 1040 cm^{-1} (P—O—CH_3). ^1H NMR ($CDCl_3$): Signals at δ 2.1 (s, NCH_3), δ 3.17 (dd, J_{HP} = 12 Hz, P—O—CH_3), δ 3.82 (s, OCH_3), δ 7.53 (m, arom.).

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