SCOPE AND LIMITATION OF THE REACTIONS OF PHENANTHRENE-9,10-QUINONE MONOXIME WITH PHOSPHORUS YLIDES.

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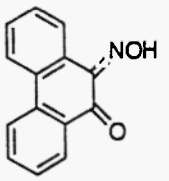
Abstract : The reactions of the entitled compound **1** with some phosphorus ylides of different types **2a-d** and **3** were studied under different experimental conditions with the aim of evaluating the synthetic potenual of this approach for the synthesis of heterocyclic systems. However, the formation of phenanthrene[9,10-*b*]-fused compounds with four, five and six membered heterocyclic rings was accomplished. Structures of the new compounds were established on the basis of elemental and spectroscopic analyses.

Introduction

Phosphorus ylides were shown to be versatile synthons for introducing nucleophilic substituents into aromatic molecules (1-3), and for preparing new fused heterocycles (4, 5). However, a limited amount of work have been done (6a-8) to evaluate the synthetic potential of this reaction pathway toward ∝-carbonyl monoxime compounds. It is therefore of obvious interest to continue our studies of using Wittig reagents for the synthesis of heterocyclic compounds (9,10), applying this chemistry to phenanthrene-9,10-quinone monoxime **1**, reporting in some detail, its behaviour toward some ylides such as methoxycarbonyl-, ethoxycarbonyl-, acetyl-, and formyl- **2a-d**, as well as diphenyl-methylenetriphenylphosphorane **3**, respectively, .



1

$(C_6H_5)_3\overset{+}{P}-\overset{-}{C}HCOR$

2

a, R = OCH_3
b, R = OC_2H_5
c, R = CH_3
d, R = H

$(C_6H_5)_3\overset{+}{P}-\overset{-}{C}(C_6H_5)_2$

3

Earlier work on o-quinone monoximes led to conflicting conclusions reporting that they exist in a quinonoid-benzenoid equilibrium and undergo reactions characteristic of both forms (11).

Results and Discussion

I. Reaction of **1** *and* methoxycarbonylmethylenetriphenylphosphorane **2a**.

Monoxime **1** on treatment with **2a** (2 equiv.) in refluxing tetrahydrofuran (THF), the reaction did not complete, even after 3 days. The product mixture was then subjected to column chromatography to give some edducts and a red solid (68%). For this product the structure of methyl [2-oxo-2*H*-dibenzo[*f,h*]quinolene-4yl]carboxylate **6a** (Scheme 1, route A) was proposed and it is the only adduct occurred regardless of the ratio of the reactants employed. The structure of compound **6a** is elucidated by its analytical and spectral properties (Tables 1 and 2).

Even though there are existing (4) the oxygen analogs of **6** which obtained from similar reactions of the parent o-quinone, we presume a different mechanism for the formation of **6**. In this context, Wittig mono-olefination of **1** by **2a** affords the intermediate **4a** followed by the attack of nitrogen on **2a** to generate the ylide **5** accompanied by elemination of a molecule of the appropriate alcohol. Intramolecular transformation of **5** to **5A** and further dehydration of the latter can account for the formation of **6** *via* its reaction with advintitious water and elemination of TPPO. However, attacking both the carbonyl- and the

hydroxylimino-group into the same molecule is in accordance with the mechanism, previously reported by Nicolaide etal (6).

Repetition of the reaction between the substrate **1** and two equiv. of ylide **2a** in refluxing chloroform containing triethylamine for ~70 h gave, besides compound **6a** (52%) a yellow substance (8%) could be isolated and identified as methyl ([*14H*]diphenanthro[9,10-*b*:9`,10`-*e*]pyran-14yl) carboxylate **9a** (Scheme 1, route C).

Furthermore, the reaction of monoxime **1** with the ylide **2a** in toluene (containing Et_3N) represents another, even more interesting variation of the same reaction in THF or in $CHCl_3$ (Scheme 1, routes A and C). As in case with the above two reactions, the primary condensation product is the quinolene-derivative **6a** which obtained as a minor product (12%) under these experimental conditions. Additionally, 10-(carbomethoxymethylimino)-9-hydroxyphenanthrene **11a** (38%), ketazine **13** (22%) and the parent quinone **14** were obtained. Formation of pyran-derivative **9a** was not hitherto been observed.The identity of compound **11a** is inferred from its correct analytical and spectroscopic data. However, an alternative tautomeric structures of type **4a** or **15**, previously reported in similar instances (6b,2), respectively, can be dismissed.

TABLE 1: Analytical Data, Physical Properties and IR spectra for the products **6**, **9**, **11**, **13**, **20-22**, **25**, **27** and **30**.

Compound.	Yield[a] in %	mp °C	Mol. Form. (M. wt.)	Anal. Found (Calcd.) % C	H	N	M^+ m/z%	IR, ν cm^{-1} NH/ OH/NOH	C=O ester	C=O (ring)	Others
6a	68[b]	164-66	$C_{19}H_{13}NO_3$	74.99	4.11	4.27	303	3370	1733	1681	
	52[c]		(303.321)	(75.24)	(4.32)	(4.62)	46				
	12[d]										
6b	59[b]	139-41	$C_{20}H_{15}NO_3$	75.37	4.42	4.15	317	3380	1739	1678	
			(317.348)	(75.69)	(4.76)	(4.41)	48				
9a	8[c]	136-38	$C_{31}H_{20}O_3$	84.19	4.4		440		1730		1225
			(440.501)	(84.53)	(4.58)		14				(-C-O-)
9b	32[c]	125-28	$C_{32}H_{22}O_3$	84.66	4.56		454		1720	1662	1278
			(454.528)	(84.56)	(4.88)		10				(-C-O-)
11a	38[d]	169-71	$C_{17}H_{13}NO_3$	72.78	4.43	4.76	279	3435	1725		1620, 1500
			(279.299)	(73.11)	(4.69)	(5.02)	18	(chelated)			(C=C/N=C)
11b	15[c]	140-42	$C_{18}H_{15}NO_3$	73.34	5.03	4.55	293	3415	1718		1620, 1500
	42[d]		(293.326)	(73.71)	(5.15)	(4.78)	23	(chelated)			(C=C, N=C)
13	22[c]	322-24	$C_{28}H_{16}N_2O_2$	81.74	3.72	6.46	412			1690	1622, 1570
			(412.452)	(81.54)	(3.91)	(6.79)	13				(C=C/C=N)
20[e]	12[c]	195-97	$C_{34}H_{24}NO_2P$	79.73	4.28	2.63	509	3415		1700	1575, 980
			(509.554)	(80.14)	(4.75)	(2.75)	8				(C=P, Ar-P)
21	25	194-96	$C_{20}H_{17}NO_2$	78.84	5.47	4.36	303	3308	1735		1625, 1615
			(303.364)	(79.19)	(5.65)	(4.62)	28		(acetyl)		(C=CH)
22	45[f]	165-68	$C_{20}H_{15}NO$	83.87	5.16	4.75	285		1730		
	72[g]		(285.348)	(84.18)	(5.3)	(4.9)	20		(acetyl)		
25	53	108-11	$C_{16}H_{11}NO_2$	76.85	4.33	5.47	249	3378,			
			(249.272)	(77.09)	(4.45)	(5.62)	52	3305			
27	28	139-42	$C_{27}H_{19}NO$	86.57	5.01	3.49	373	3370			1225
			(373.457)	(86.84)	(5.13)	(3.75)	32				(vinyl ether)
30	36	267-69	$C_{40}H_{28}$	94.22	5.51		508				
			(508.664)	(94.45)	(5.55)		12				

a) Yields are approximated. b) Yield, was obtained from the reaction I.a. c) Yield was obtained from the reaction I.b. d) Yield was obtained from the reaction I.c. e) Compound **20** has elemental analysis for P= 5.82 (6.08). f) Yield was obtained from the reaction II.b. Yield was obtained from the reaction II.C.

On the other hand, the structure of ketazine **13** was attested by the following evidences: a) Compound **13** is insoluble in dilute alkali and gives no color with alcoholic ferric chloride solution. b) Correct elemental analysis and molecular weight measurement (MS). Actually, the mass spectrum of the dimeric product displayed the molecular ion peak at m/z = 412 [M^+, $C_{28}H_{16}N_2O_2$,13%] which supports structure **13**. Loss of $2H^\bullet$ radicals from M^+, which is frequently observed in the behaviour of condensed aromatics under electron impact (12), yields the radical cation at m/z 410 (base peak) which loses then N_2 molecule to give the ion peak m/z 382 [$(C_{28}H_{14}O_2)^+$, 22%].

TABLE 2 : ^{1}H NMR (δ, ppm) data[a,b] for the products **6**, **9**, **11**, **13**, **20**-**22**, **25**, **27** and **30**.

Compound	C-CH_3	OCH_3/OCH_2	C-H/ N=CH/ C=CH	-NH/OH/ N-OH
6a*		3.85 (s, 3H)		8.75 (br, 1H)
6b	1.38 (t, 3H)	4.34 (q, 2H)		8.55 (br, 1H)
	J_{HH} = 6 Hz	J_{HH} = 6 Hz		
9a		3.82 (s, 3H)	4.41 (s, 1H)	
9b	1.39 (t, 3H)	4.35 (q, 2H)	4.58 (s, 1H)	
	J_{HH} = 6 Hz	J_{HH} = 6 Hz		
11a*		4.13 (s, 3H)	6.58 (s, 1H)	9.54 (s, 1H)
11b	1.62 (t, 3H)	4.61 (q, 2H)	6.62 (s, 1H)	9.63 (s, 1H)
	J_{HH} = 7 Hz	J_{HH} = 7 Hz		
13			7.65-8.32 (m, 16H, Ar-H)	
20[c]*			7.25-8.46 (m, 23H, Ar-H)	
21	2.43 and 2.55		6.27 and 6.43	8.75 (br., 1H)
	(2s, 6H)		(2H, =CH, 2s)	
22	2.43 and 2.55			
	(2s, 6H)			
25*				3.33 (d, 1H)
				J = 7.5 Hz
27				8.55 (br., 1H)
30*			7.23-8.35 (m, 28H, Ar-H)	

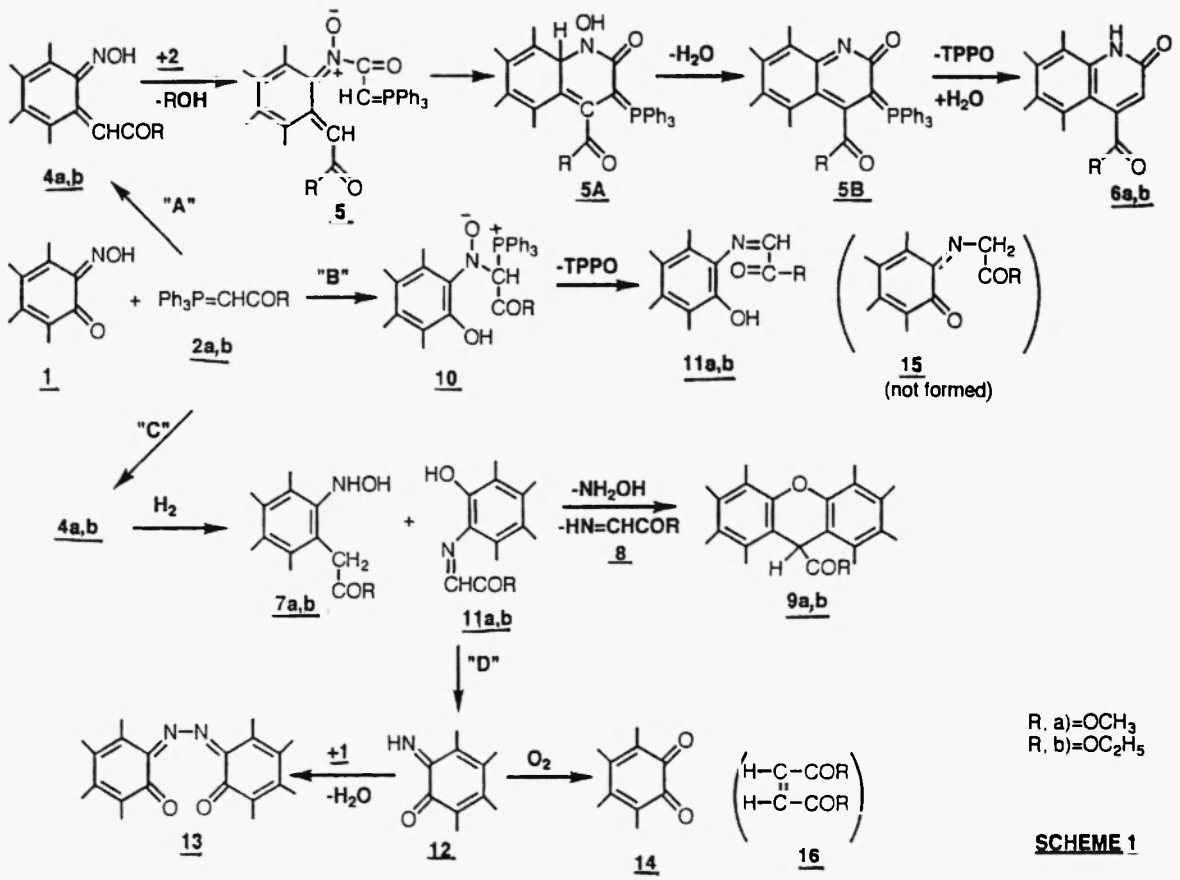
a) See experimental for details for NMR experiments.

b) Quinoline, pyridine, pyrrole and aromatic hydrogen protons lie in δ 6.6-8.55 ppm region.

c) ^{31}P NMR spectrum of compound **20** showed a singlet at δp = 24.4 ppm.

* ^{13}C NMR measurements for compounds designated with asterisk have been performed: **6a**, δc : 51.4 (OCH_3), 167.9 (C=O, ester); 172.5 ppm (C=O, amide). **11a**, δc: 50.8 (OCH_3), 147.4 (N=CH), 153.1 (C-OH), 160.5 (C=O). **20**, δc : 151.5 (d, Jcp = 124.9 Hz, C=P), 170.6 ppm (d, J= 8 Hz (C=O). **22**, δc: 15.41 (CH_3), 22.35 (C(O)CH_3), 193.8 ppm (C=O); **30**, δc: 28.73 ppm (C-Ph_2).

A mechanism for the formation of **11a** and **13** is based on direct attack by the nitrogen lone pair electrons of the anionic centre in the oxime **1** on the carbanion centre in the Wittig reagent **2a** by an addition elemination mechanism (13, 14), this would give the ylidic intermediate **10a** (Scheme 1, pathway B). Interestingly, the stability of **10a** could be attained through intramolecular Wittig reaction to give compound **11a** or suffers slowly decomposition, intrigued by the base (TEA), to give (path D), the appropriate symmetrical unsubstituted ethylene **16** (not detected) and ∝-imino ketone **12**. The further oxidation (15) of **12** affords phenanthrene-9,10-quinone **14**, meanwhile its condensation with **1** yields the ketazine **13** through the loss of water. The latter step (path D) and formation of **13** and **14** would probably be accelerated by the relatively long heating. Moreover, the conversion of **1** to **12** by the influence of phosphorus ylides is in accordance with the



concept of "Hard and Soft Bases" discussed by Pearson (16) and Hudson (17). Phosphorus ylides which acting as a Lewis Base (18) forms relatively unstable intermediate **10** catalyzing its decomposition and formation of **13**, **14** and **16** *via* **12**.

Furthermore, the formation of nitrogen-to-nitrogen bond by the action of nucleophilic phosphorus reagents on ∝-keto monoximes or o-quinone monoximes should be linked with the production of acenaphthenequinoneketazine formed by the reaction of the parent ∝-imino ketone with phosphite esters (19) and formation of the azo-derivative from the reaction of 1,2-naphthoquinone monoxime with phosphorus ylides (8).

A plausible mechanistic sequence for formation of **9** is proposed in Scheme 1 "C". Hydrogenation of **4a** to **7a**, followed by further condensation of the latter with **11** accompanied by imino-ester **8** elemination can account for the formation of **9**.

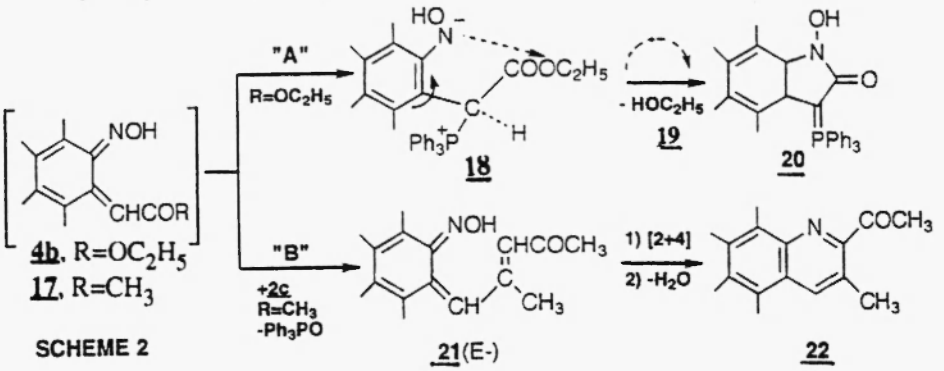
II. *Reaction of* **1** *and* ethoxycarbonylmethylenetriphenylphosphorane **2b**.

In a systematic study, the reaction of monoxime **1** with **2b**, has been investigated in THF, $CHCl_3$ and toluene. A tetrahydrofuran solution of **1** and the ylide **2b** (2 equiv.) was heated under reflux for 3 days and the product mixture was then resolved by column chromatography to give red solid (59%) assigned structure **6b** on the basis of comparable spectroscopic data arguments with **6a**. Compound **6b** appeared likewise, the sole product accompanied by some edducts.

On the other hand, the reaction was completed (as in I) when it was carried out in refluxing chloroform containing TEA for 70 h affording, irrespectively, four crystalline substances, pyran-derivative **9b** (32%), imino-phenanthrol **11b** (15%), ketazine **13** (22%) and the parent quinone **14** (6%). Compound **6b** has not

hitherto been observed (<5%). The structures of the isolable products **9b**, **11b** and **13** are consistent with spectroscopic data available (*cf.* Tables 1 and 2) and on the basis of comparable arguments with their analogues **9a**, **11a** and ketazine **13**, previously identified.

When the same reaction of **1** and **2b**, using the same amounts, was performed in toluene and in the presence of TEA, as expected, and likwise with **2a** in toluene, the major product (42%) was the 1:1 adduct **11b**. Ketazine **13** was also isolated in a moderate yield (~20%) along with a yellow substance assigned structure **20** (12%) (Scheme 2"**A**"). Neither compound **6b** nor **9b** was obtained from this reaction. Assignment of 3-triphenylphosphoranylidenephenanthro-1-hydroxypyrrole-2-(*3H*)-one **20** was based on the analytical and spectroscopic data, e.g., its IR spectrum revealed absorption bands at 1575 (C=P), 1410 and 980 cm^{-1} (P-C, aryl). Despite the IR spectrum of **20** disclosed a broad OH stretching frequency at 3415, the OH resonance was not detected in its PMR spectrum, a phenomenon that had previously been noted in the case of 1-hydroxyoxyindoles (7, 20). The mass spectrum showed the molecular ion m/z 509 (8%). Fragmentation took by the loss of $HO^{\bullet}$ from the molecular ion $(M\text{-}HO^{\bullet})^{+}$, this pattern of fragmentation being in accord with that occurring in substituted 1-hydroxyindoles (12, 20).



SCHEME 2

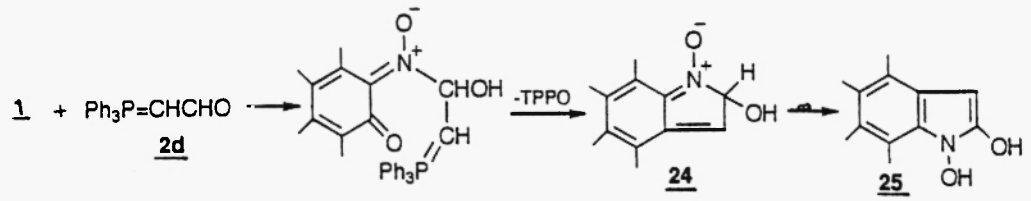
Obviously, a mechanism for the formation of **20** can be explained as occurring in Scheme 2 (pathway **A**) through Michael addition of triphenylphosphine, generated *in situe* to the quinone-methanide intermediate **4b** to give the betaine **18** which, in consequence of extrusion of ethyl alcohol molecule, yields the ylide **20**. The aformentioned conversion (**4** ⟶ **20**) meets a well documented analogy in the literature (1, 2, 4b).

III. *Reaction of* **1** *and* **2c**.

Acetylmethylenetriphenylphosphorane **2c**, in contrast to **2a** or **2b**, does not react to any appreciable extent with the monoxime **1** in refluxing THF, and the starting materials were recovered practically unchanged even after 3 days. In $CHCl_3$ containing TEA, the reaction afforded two green products **21** (25%) and **22** (45%) Scheme 2, pathway **B**). Changing the reaction medium, using toluene, only compound **22** was obtained as a major product (>70%) along with the ketazine **13** (10%). Obviously, compounds **21** and **22** are produced *via* the corresponding primary mono-olefinated intermediate **17**, which by further Wittig reaction of the acetonyl-carbonyl (2, 6) of **17** with a second ylide species **2c** affords compound **21**. However, the latter step can be followed by [4+2] intramolecular cyclization to give the pyridine-derivative **22** through the elemination of a molecule of water. Synthesis of compounds **21** and **22** as two stable products from the same reaction is not surprising. A reasonable rationalization for the observed results can most probably be attributed to the spatial arrangement of the substituents in the initially formed **21** (*E* and *Z* conformers), thereby, we presume that the isolated product **21** is absolutely *E* configuration, while pyridine derivative **22** derived from the *Z* isomer.

IV. *Reaction of* **1** *and* formylmethylenetriphenylphosphorane **2d**.

Next, the scope of the present study was also extended to **2d**, which is the least reactive of the four phosphorus ylides **2a-d**. The reaction of **1** with excess of **2d** (chloride salt) in ethanol containing TEA gave the ketazine **13** (12%) and the unexpected 1,2-dihydroxypyrrole derivative **25** (53%).



SCHEME 3 **23**

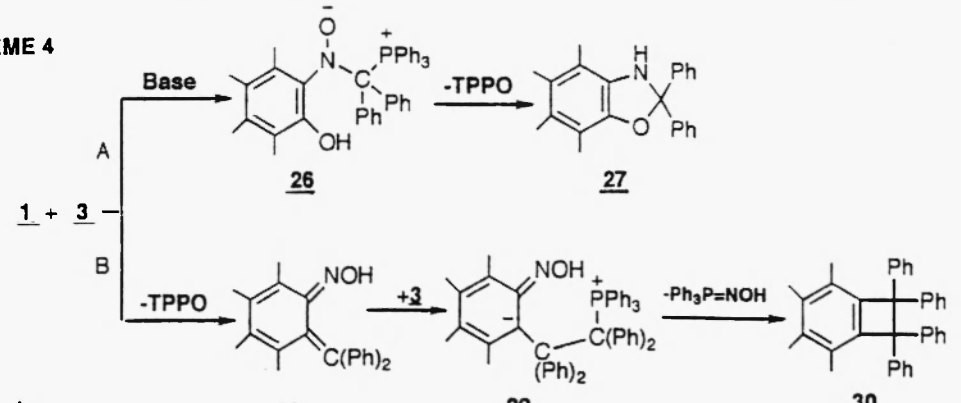
A mechanism proposed for the formation of compound **25** from the reaction of **1** and **2d** is depicted in Scheme 3. The attack of nitrogen on the aldehydic-carbonyl in **2d** results in its reduction with formation of the ylide **23**. Attack of ylide **23** on the aryl-carbonyl in normal internal Wittig reaction would lead to the amine oxide **24**, which is the tautomeric form of **25**. This behaviour is parallel to the reaction course of phenanthrenequinone imine with acetaldehyde, previously reported (15a).

V. Reaction of **1** *and* diphenylmethylenetriphenylphosphorane **3**.

In the same way, the monoxime **1** was allowed to react with **3** (bromide salt, 2 equiv.) in refluxing ethanol containing triethylamine to give 2,2-diphenyl-2,3-dihydrophenanthro[9,10-*b*]-1,3-oxazole **27** (28%) and 1,1,2,2-tetraphenylphenanthro[9,10-*b*]cyclobutene **30** (36%).

Mechanistically, formation of compounds **27** and **30** may be considered to proceed *via* the two intermediates **26** and **28** initially formed, concurrently. Oxazole **27**, probably results from intramolecular cyclization of the crowded intermediate **26** accompanied by TPPO elemination. On the other hand, the cyclobutene **30** apparently arose (Scheme 4) by further Michael addition of **3** to **28** followed by a Wittig-type reaction of the second ylide group with the hydroxyimino-group and elemination of hydroxyiminotriphenylphosphorane species (Ph_3P=NOH species). An analogous Wittig-type-reaction has been reported to proceed between phosphorus ylides and Schiff bases with elemination of Ph_3 P=NOH (2,6, 21).

SCHEME 4



Conclusion

Significantly, the five reactions reported here are indicative of the broad reaction spectrum of which the phosphorus ylides are cabable. However, the foregoing observations, unambiguously show that compound **1** reacts with ylides **2** and **3** both in quinone oxime structure **1a** (*cf* **4**) and in the benzenoid nitroso form **1b** (*cf.*

10) at least under the prevailing experimental conditions. In this respect, the behaviour of **1** is in marked disparity with the behaviour of ∝-nitroso-ß-naphthol toward the same reagents whereby it reacts absolutely in benzenoid nitroso form (8).

Even though, the structural products indicated two positions in **1**, are susceptible to nucleophilic attack, and hence a competition between two options available to the stabilized ylides in the reactions with **1**, i.e. olefination or conjugate addition, it is obvious that the aryl carbonyl is a preferable site of attack by the nucleophilic reagents **2** and **3**. On the other hand, the findings also support the assumption that the basic medium stimulate the course of the reaction at the imino-group.

Experimental

Melting points are uncorrected. IR spectra were obtained with a Phillips Infracord Spectrometer Model PU 9712 in KBr. ^{1}H- and ^{13}C-NMR spectra were recorded in $CDCl_3$ or [D_6] DMSO as solvents on a Jeol-270 MHz Spectrometer. The ^{31}P-NMR spectrum was taken with a Varian CFT-20 (*vs.* external 85% H_3PO_4).

I. Reaction of Phenantherene-9,10-quinone Monoxime **1** *with Phosphorus Ylides* **2a,b**.*General Procedure:*

a) In Tetrahydrofuran (THF). A suspension of oxime **1** (22) (2.2 g, 10 mmol) and **2a** or **2b** (23) (21 mmol) in dry THF (50 ml) was refluxed for 3 days. After evaporation of the solvent, the remainder was subjected to column chromatography on silica gel. Elution with hexane-chloroform afforded methyl 2-oxo-*2H*-dibenzo *[f,h]*quinolene-4-carboxylate **6a** or ethyl 2-oxo-*2H*-dibenzo*[f,h]*quinolene-4-carboxylate **6b**, respectively. Percentage yields, physical and spectral data of compounds **6a,b** are listed in Tables **1** and **2.**

Unchanged oxime **1** (*ca*, 20%), unchanged ylide **2a** or **2b** (ca. 22%) and TPPO (*ca* 63%), were also isolated and identified by mp., mixed mps. and comparative IR and MS spectra.

The reaction between equimolar amounts of compounds **1** and **2a** or **2b** under the conditions described above, afforded again compounds **6a** (31%) or **6b** (27%) along with **1** (38%) and TPPO (29%).

I.b- In Chloroform. The same reaction was repeated in boiling chloroform (TEA). After usual workup we obtained: a) red crystals of **6a** or yellow crystals (chloroform-pentane) of **11b**, respectively; b) orange solid (0.24 g, 6%), (ethyl alcohol) of **14** (mp., mixed mps. and comparative IR and MS spectra) (24); c) red substance (acetonitrile) of **9a** or **9b**, respectively; d) compound **13** as yellow crystals (benzene) (see tables 1, 2).

Compounds **6b** and **11a** were not isolated from this reaction. Compound **13** was obtained, only with ylide **2b**.

I.c- In Toluene. The same reaction was repeated in boiling toluene (TEA). After usual workup we obtained: a) yellow crystals (chloroform-pentane) of **11a** or **11b**, respectively; b) compound **6a**. The parallel compound **6b** was not obtained from this reaction; c) pale yellow crystals (dichloromethane) of **20** (from **2b**); d) **13** (*ca.* 20%). Data for compounds **6a, 11a.b** and **20** are tabulated in Tables **1** and **2.**

II. Reaction of oxime **1** *with* **2c**.*a- In THF.* No reaction was observed when equimolar amounts of **1** and acetylmethylenetriphenylphosphorane **2c** (25) were refluxed in THF even after 3 days. Compounds **1** and **2c** were recovered practically unchanged in ~90% yield.

II.b- In Chloroform. To a solution of **2c** (6.5 g, 21 mmol) in 30 ml chloroform was added a solution of oxime **1** (2.2 g, 10 mmol) in 30 ml of the same solvent (TEA) and the reaction mixture was refluxed for 56 h. After usual workup we obtained: a) **22** as green crystals (acetone); b) green needles (CH_3Cl-hexane) of 10-hydroxyimino-9-phenanthrylidene derivative **21** (see Tables **1** and **2**).

II.c- In Toluene. When the same reaction (**11b**) was repeated in boiling toluene (TEA) for 32 h, only **22** as a major product (see Table **1**) was obtained along with ketazine **13** (~10%).

III. Reaction of Oxime **1** *with* **2d**. A mixture of oxime **1** (1.1 g, 5 mmol) and **2d** (chloride salt) (26) (1.7 g,7.1 mmol) in ethyl alcohol (30 ml) containing TEA (2.5 ml) was refluxed for 3 days. After usual workup we obtained: a) **13** (*Ca.* 12%); b) **25** as brown crystals (ethanol-ether).

IV. Reaction of oxime **1** *with* **3**. A suspension of **1** (1.1 g, 5 mmol) and **3** (bromide salt) (27) (2.78 g, 11 mmol) in ethyl alcohol (30 ml) containing TEA (2.5 ml) was refluxed for 3 days. After usual workup we obtained: a) yellow crystals (acetonitrile) of **27**; b) orange crystals (diethyl ether) of **30** (see Tables **1** and **2**).

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