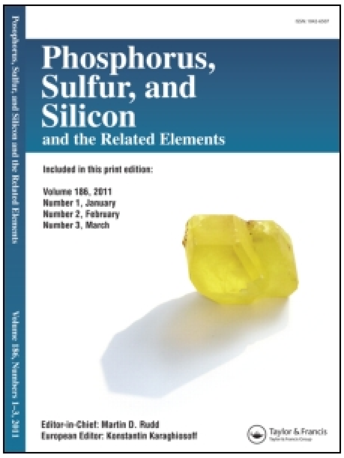

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Synthesis and Study of Dynamic ^{1}H NMR of Carbon-Carbon Double Bond Rotation Energy Barrier in Stable Phenothiazine Phosphorus Ylides

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To cite this Article Maghsoodlou, Malek Taher , Khorassani, Sayyed Mostafa Habibi , Niroumand, Uranous , Charati, Faramarz Rostami and Khosrosharodi, Maryam(2007) 'Synthesis and Study of Dynamic ^{1}H NMR of Carbon-Carbon Double Bond Rotation Energy Barrier in Stable Phenothiazine Phosphorus Ylides', Phosphorus, Sulfur, and Silicon and the Related Elements, 182: 3, 647 — 655

To link to this Article: DOI: 10.1080/10426500601047149

URL: http://dx.doi.org/10.1080/10426500601047149

Phosphorus, Sulfur, and Silicon, 182:647–655, 2007
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ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500601047149

Synthesis and Study of Dynamic ^{1}H NMR of Carbon–Carbon Double Bond Rotation Energy Barrier in Stable Phenothiazine Phosphorus Ylides

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The 1:1 intermediates generated by the addition of triphenylphosphine to dialkyl acetylenedicarboxylates were trapped by an NH-acidic compound such as phenothiazine to give dialkyl-2-(phenothiazino)-3-(triphenylphosphoranylidene)-butanedioates in high yields. The ylide moiety of these compounds was strongly conjugated with the adjacent carbonyl group, and rotation around the partial double bond in the (E) and (Z) geometrical isomers was slow on the NMR time scale. The dynamic effects in the ylide moiety were investigated by ^{1}H NMR. They are attributed to a restricted rotation around the carbon–carbon double bond.

Keywords Rotational energy; phenothiazine; triphenylphosphine; phosphorus ylides; dynamic NMR

INTRODUCTION

Phenothiazines have proven to be a pharmaceutically important class of heterocycles. Due to their pharmacological efficacy, they are applied as sedatives, tranquilizers, antiepilectica, antipyretica, and antitumor agents, as well as bactericides and pesticides.[1,2] Interestingly, phenothiazines are able to cleave DNA upon photochemical induction.[3] It was recognized quite early that the low oxidation potential of this class of

Received August 2, 2005; accepted August 23, 2006.

The authors gratefully acknowledge financial support from the Research Council of The University of Sistan and Balouchestan, Zahedan, Iran.

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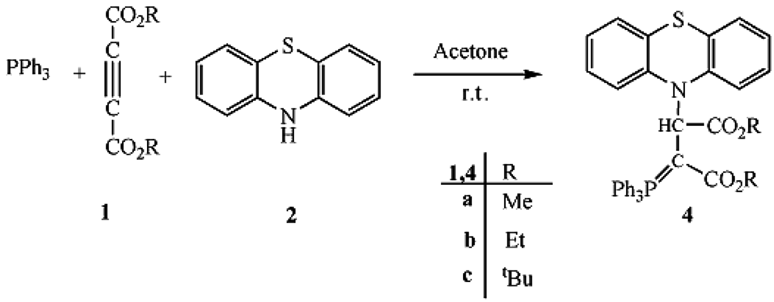
tricyclic nitrogen-sulfur heterocycles and their propensity to form stable radical cations play a key role in their physiological activities.[4]

With respect to the importance of heterocycles, the development of simple synthetic routes for widely used organic materials, and especially for organophosphorus compounds, from readily available reagents is one of the major tasks in organic synthesis. Organophosphorous compounds are of particular interest as synthetic targets due to their importance in industrial, biological, and chemical syntheses.[5]

Many strategies have appeared describing novel syntheses of organophosphorus compounds, including our previous report.[13] Here we report an efficient synthetic route to sterically congested nitrogen-containing phosphorus ylides **4** using triphenylphosphine, dialkyl acetylenedicarboxylates **1**, and phenothiazine **2** (Scheme 1).

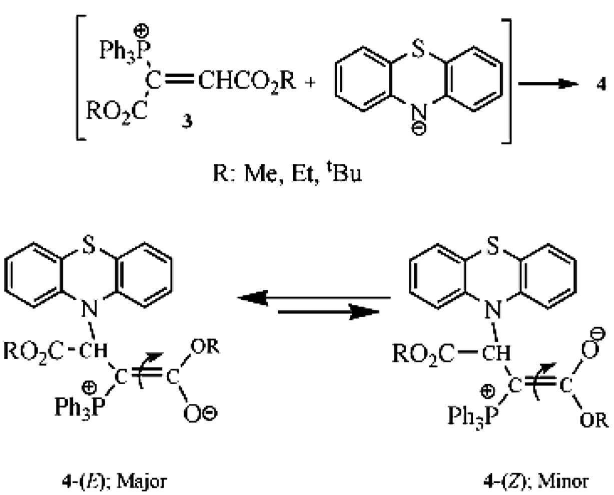


SCHEME 1

RESULTS AND DISCUSSION

On the basis of well-established chemistry of trivalent phosphorus nucleophiles,[13–17] it is reasonable to assume that the first step in the formation of phosphorus ylides **4** is performed by the nucleophilic reaction of triphenylphosphine with dialkyl acetylenedicarboxylates **1**. Subsequent protonation of the highly reactive 1:1 adduct by phenothiazine **2** leads to the vinyl triphenylphosphonium salts **3**. The nucleophilic attack of the phenothiazine anion on the vinyl triphenylphosphonium cation finally yields phosphoranes **4** as mixtures of the *E* and *Z* isomers (Scheme 2).

All of the compounds **4a–c** are stable solid powders whose structures are supported by ^{1}H, ^{13}C, and ^{31}P NMR as well as the IR spectral data. The mass spectra of these compounds display the molecular ion peak at $m/z = 405$, 457, and 489 (M^{+}-phenothiazine) for **4a**, **4b**, and **4c**,



SCHEME 2

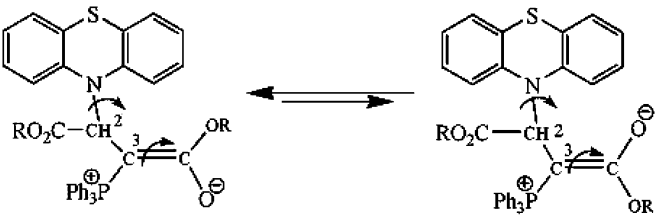
respectively. ^{1}H NMR data of compounds **4a** and **4b** show the presence of the *Z* and *E* rotamers, while phosphorane **4c** is found only as one rotamer. The ^{1}H NMR spectrum of **4a** displays sharp signals at 2.99 and 3.93 ppm for the major rotamer and 3.65 and 3.79 ppm for the minor rotamer arising from the methyl protons of the methoxy groups. The signal at $\delta = 3.65$ for the methyl group of the minor rotamer appears at a higher field because of the anisotropic effect of phenyl groups of the triphenylphosphine moiety.[18] The ^{1}H NMR spectrum of **4a** also displays two doublets at $\delta = 4.22$ (${}^3J_{\mathrm{PH}} = 14.6$ Hz) and $\delta = 4.25$ (${}^3J_{\mathrm{PH}} = 15.9$ Hz) for the methine protons of the major and minor rotamer, respectively (Table I).

The ^{1}H NMR spectrum of **4b** is similar to that of **4a** except for the ethyl group, which exhibits characteristic signals with appropriate chemical shifts. The ^{1}H NMR spectrum of **4c** shows a different pattern from those of **4a** and **4b**. It shows one doublet and two sharp singlets for the methine and the methyl protons, repectively. This indicates the presence of only one rotamer. Evidence for the presence of two rotamers in the case of **4a** and **4b** and of only one rotamer in the case of **4c** indicated by ^{1}H NMR data is further confirmed by ^{13}C and ^{31}P NMR spectra. In each ^{31}P NMR spectrum of **4a** and **4b**, two signals were observed at $\delta = 25.3$ and 24.7 and $\delta = 25.1$ and 24.8 ppm, respectively, which were attributed to the *E* and *Z* rotamers (Table I and Scheme 3). These shifts are similar to those observed for other phosphorus ylides.[19,20] The first-order rate constant ($k = \pi\Delta\upsilon/\sqrt{2}$) in the interconversion

TABLE I Selected ^{1}H, ^{13}C, and ^{31}P NMR Chemical Shifts (ppm) and Coupling Constants (Hz) for the Major (M) and Minor (m) Rotamers of Compounds 4a–c

			^{1}H NMR data			^{13}C NMR Data		
Entry	Yield (%)	Percent of Rotamers	δ, H-2 ($^{3}J_{PH}$)	δ, OR	δ, CO_2R	C-2 ($^{2}J_{PC}$)	C-3 ($^{1}J_{PC}$)	^{31}P NMR δ
4a	95	52 (M)	4.2 (14.6)	2.9	3.9	65.3 (14.6)	42.5 (123.7)	25.3
		48 (m)	4.3 (15.9)	3.7	3.9	64.8 (14.9)	43.8 (125.7)	24.7
4b	90	56 (M)	4.1 (18.5)	3.4[a]	4.3[a]	65.5 (15.2)	42.2 (123.3)	25.1
		48 (m)	4.2 (14.9)	3.8[a]	4.4[a]	64.8 (13.6)	44.5 (124.4)	24.8
4c	86	88	4.1 (16.5)	0.9	1.7	66.3 (14.7)	41.6 (123.7)	24.7

[a] The methylene group of the OR moiety.



4-(E); Major

4-(*Z*); Minor

SCHEME 3

between the two rotamers was studied by variable temperature ^{1}H NMR spectroscopy for compound **4a** in $CDCl_3$ as a solvent.

The ^{1}H NMR spectrum of **4a** in $CDCl_3$ solution at an ambient temperature displayed two doublets for the CH proton at $\delta = 4.22$ and 4.28 ppm. Increasing the temperature to 65°C resulted in coalescence. The same was observed in the variable temperature ^{1}H NMR spectrum of **4b**. From the colescence temperature of the proton resonances of the CH group and using the equation for the rate constant,[18] we calculated the first-order rate constant (k) for the bond rotation in **4a** to 66.6 s^{-1} at 25°C (Table II).

Application of absolute-rate theory equation with a transmission coefficient of 1 gave a free-energy of activation ($\Delta G^{\neq}$) for **4a** of 62.5 ± 2 kJ mol^{-1}, for which all known sources of errors were estimated and included (Table II).[6,21]

The experimental data available are not suitable for obtaining meaningful values of $\Delta H^{\neq}$ and $\Delta S^{\neq}$, although the errors in $\Delta G^{\neq}$should not be large.[8] In summary, dynamic NMR effects were observed in the ^{1}H NMR spectra of compounds **4,** which are attributed to a restricted rotation around the carbon–carbon double bond.

TABLE II Selected ^{1}H Chemical Shifts (ppm) and Activation Parameters (kJ mol^{-1}) for 4a,b in $CDCl_3$

Compound	Temp. (°C)	Resonance (CH of Ylide)	$\Delta\upsilon$	K (S^{-1})	T_C(K)	$\Delta G^{\#}$(KJ mol^{-1})
4a	25	4.22, 4.28	30.0	66.6	298	62.5
	65	4.22	—	—	—	—
4b	25	4.11, 4.24	65	144.4	298	60.6
	60	4.17	—	—	—	—

In conclusion, we have prepared novel phenothiazine-containing phosphorus ylides via a one-pot reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of a NH-acid such as phenothiazine. The present method has the advantage that the reaction is performed under neutral conditions. The substances can be mixed without any activation or modification (without using support or a catalyst). The phosphoranes **4a–c** may be considered potentially useful synthetic intermediates.

EXPERIMENTAL

Melting points and IR spectra were measured on an Electrotermal 9100 apparatus and a Shimadzu IR 470 spectrophotometer, respectively. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded with a BRUKER DRX-500 AVANCE instrument in $CDCl_3$ as a solvent at 500.1, 125.8, and 202.4 MHz, respectively. Mass spectra were obtained with a Finigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. Dialkyl acetylenedicarboxylates **1**, triphenylphosphine, and phenothiazine **2** were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General Procedure for Preparation of Ylides (4)

To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and phenothiazine (0.199 g, 1 mmol) in 5 mL of acetone was added dropwise a mixture of the dialkyl acetylenedicarboxylate (1 mmol) in 1 mL of acetone at −5°C over 10 min. The reaction mixture was then allowed to warm up to r.t. and stirred for 4 h. The solid product was filtered and washed with (3 × 10 mL) cold diethyl ether to give ylides **4a–c**. Characteristic analytical and spectroscopic data of dialkyl-2-(phenothiazine-*N*-yl)-3-(triphenylphosphoranilidene)butandioates **4a–c** follow.

Dimethyl-2-(phenothiazino)-3-(triphenylphosphoranylidene)-butanedioate (4a)

Yellow solid, yield 95%, m.p. 186–188°C IR (KBr), $\upsilon_{\max}$ (cm^{-1}): 1730 (C=O), 1600 (C=C), MS (*m/z*, %): 405 (M$^+$-phenothiazine, 100), 262.2 (Ph_3P, 60), 199.2 (phenothiazine, 65), 183 ($(CH_3)_3CO_2CCHCO_2H$,60), 167.2 (phenothiazine-S, 35), 77.1 (Ph, 18); major isomer **4a**-(*E*) (52%), ^{1}H NMR ($CDCl_3$): δ 2.9 (s, 3H, OCH_3) and 3.9 (s, 3H, OCH_3), 4.2 (d, $^3J_{PH}$ = 14.6 Hz, 1H, P—C—CH), 6.6–7.9 (m, 23H, arom-H). ^{13}C NMR

(CDCl$_3$): δ 42.5 (d, $^1J_{PC}$ = 123.7 Hz, P=C), 49.0 (s, O*C*H$_3$), 52.4 (s, OCH$_3$), 65.3 (d, $^2J_{PH}$ = 14.6 Hz, P—C—CH), 115.3 (s, C—S), 121.8 (s, CH) 122.3 (s, CH), 122.6 (s, CH), 125.7 (s, CH), 126.1 (d, $^1J_{PC}$ = 92.8 Hz, C$_{ipso}$), 128.4 (d, $^3J_{PC}$ = 12.3 Hz, C$_{meta}$), 131.7 (d, $^4J_{PC}$ = 2.8 Hz, C$_{para}$), 133.5 (d, $^2J_{PC}$ = 9.7 Hz, C$_{ortho}$), 146.6 (s, C—N), 169.2 (d, $^2J_{PC}$ = 12.8 Hz, C=O), 174.2 (d, $^3J_{PC}$ = 17.2 Hz, C=O). ^{31}P NMR (CDCl$_3$): δ 25.3; minor isomer **4a**-(*Z*) (48%), ^{1}H NMR (CDCl$_3$): δ 3.7 (s, 3H, OCH$_3$), 3.9 (s, 3H, OCH$_3$), 4.3 (d, $^3J_{PH}$ = 15.9 Hz, 1H, P-C-CH), 6.6–8.0 (m, 23H, arom-H). ^{13}C NMR (CDCl$_3$): δ 43.8 (d, $^1J_{PC}$ = 125.7 Hz, P=C), 50.3 (s, O*C*H$_3$), 52.2 (s, OCH$_3$), 64.8 (d, $^2J_{PC}$ = 14.9 Hz, P—C—*C*H), 114.8 (s, C—S), 121.6 (s, CH), 122.5 (s, CH), 122.7 (s, CH), 125.4 (s, CH), 126.2 (d, $^1J_{PC}$ = 92.8 Hz, C$_{ipso}$), 128.5 (d, $^3J_{PC}$ = 15.6 Hz, C$_{meta}$), 131.9 (d, $^4J_{PC}$ = 2.8 Hz, C$_{para}$), 133.6 (d, $^2J_{PC}$ = 9.8 Hz, C$_{ortho}$), 146.5 (s, C—N), 169.1 (d, $^2J_{PC}$ = 12.4 Hz, C=O), 174.1 (d, $^3J_{PC}$ = 17.2 Hz, C=O). ^{31}P NMR (CDCl$_3$): δ 24.7.

Diethyl-2-(phenothiazino)-3-(triphenylphosphoranylidene)-butanedioate (4b)

Yellow solid, yield 85%, m.p. 180–182°C, IR (KBr), υ_{max}, (cm^{-1}): 1720 (C=O), 1630 (C=C), MS (*m*/*z*, %): 457 (M-2CO$_2$Et, 8), 359.3 (M-phenothiazine-CH$_2$CH$_{3,}$ 18), 262.2 (PPh$_3$, 90), 199.2 (phenothiazine, 90), 183.1 (t-EtO$_2$CCCHCO$_2$H, 95); major isomer **4b**-(*E*) (56%), ^{1}H NMR (CDCl$_3$): δ 0.4 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH$_3$), 1.3 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH$_3$), 3.4 (m, 2H, CH$_2$), 4.1 (d, 1H, $^3J_{PH}$ = 18.5 Hz, P—C—CH), 4.3 (m, 2H, CH$_2$), 6.5–8.0 (m, 23H, arom-H). ^{13}C NMR (CDCl$_3$): δ 13.9 (s, CH$_3$), 14.4 (s, CH$_3$), 42.2 (d,$^1J_{PC}$ = 123.3 Hz, P=C), 57.5 (s, CH$_2$), 61.4 (s, CH$_2$), 65.5 (d, $^2J_{PC}$ = 15.2 Hz, P—C—CH), 117.9 (s, C—S), 121.8 (s, CH) 122.3 (s, CH), 122.6 (s, CH), 125.7 (s, CH), 127.1 (d, $^1J_{PC}$ = 85.0 Hz, C$_{ipso}$), 128.3 (d, $^3J_{PC}$ = 12.2 Hz, C$_{meta}$), 131.6 (d, $^4J_{PC}$ = 2.8 Hz, C$_{para}$), 133.6 (d, $^2J_{PC}$ = 9.7 Hz, C$_{ortho}$), 146.7 (s, C—N), 168.6 (d, $^2J_{PC}$ = 12.8 Hz, C=O), 173.67 (d, $^3J_{PC}$ = 14.6 Hz, C=O). ^{31}P NMR (CDCl$_3$): δ 25.1; minor isomer **4b**-(*Z*) (44%), ^{1}H NMR (CDCl$_3$): δ 0.5 (t, $^3J_{HH}$ = 6.9 Hz, 3H, CH$_3$), 1.4 (t, $^3J_{HH}$ = 6.9 Hz, 3H, CH$_3$), 3.8 (m, 2H, CH$_2$), 4.2 (d, $^3J_{PH}$ = 14.9 Hz, 1H P—C—C*H*), 4.4 (m, 2H, CH$_2$), 6.5–8.0 (m, 23H, arom-H). ^{13}C NMR (CDCl$_3$): δ 14.0 (s, CH$_3$), 14.1 (s, CH$_3$), 44.5 (d, $^1J_{PC}$ = 124.4 Hz, P=C), 58.6 (s, CH$_2$), 61.2 (s, CH$_2$) 64.8 (d, $^2J_{PC}$ = 13.6 Hz, P—C—CH), 117.9 (s, C—S), 121.6 (s, CH), 122.1 (s, CH), 122.5 (s, CH), 125.7 (s, CH), 126.3 (d, $^1J_{PC}$ = 74.8 Hz, C$_{ipso}$), 128.5 (d, ^{3}J$_{PC}$ = 12.2 Hz, C$_{meta}$), 131.5 (d, $^4J_{PC}$ = 2.5 Hz, C$_{para}$), 133.7 (d, $^2J_{PC}$ = 8.7 Hz, C$_{ortho}$), 146.8 (C—N), 168.6 (d, $^2J_{PC}$ = 12.6 Hz, C=O), 173.7 (d, $^3J_{PC}$ = 14.6 Hz, C=O). ^{31}P NMR (CDCl$_3$): δ 24.8.

Di-*tert*-butyl-2-(phenothiazino)-3-(triphenylphosphoranylidene)-butanedioate (4c)

Yellow crystals, yield 90%, m.p. 198–199°C, IR (KBr), υ_{max}, (cm^{-1}): 1720 (C=O), 1620 (C=C), MS (*m/z*, %): 488.9 (M-phenothiazine-H, 18), 433 (M-phenothiazine-*t*Bu, 18), 377.3 (M-phenothiazine-CO$_2$-*t*Bu, 100), 262.1 (Ph$_3$P, 38), 199.4 (phenothiazine, 90), 183.1 (*t*BuO$_2$CCCHCO$_2$H, 35). ^{1}H NMR (CDCl$_3$): δ 0.9 (s, 18H, CH$_3$), 1.7 (s, 9H, CH$_3$), 4.1 (d, $^3J_{PH}$ = 16.5 Hz, 1H, P—C—CH), 6.0–8.0 (m, 23H, arom-H). ^{13}C NMR (CDCl$_3$): δ 28.3 (s, CH$_3$), 28.4 (s, CH$_3$), 41.6 (d, $^1J_{PC}$ = 123.7 Hz, P=C), 66.3 (d, $^2J_{PC}$ = 14.7 Hz, P—C—CH), 76.8 (s, CMe$_3$), 80.2 (s, CMe$_3$), 115.2 (s, C—S), 121.1 (s, CH), 122.2 (s, CH), 122.6 (s, CH), 125.8 (s, CH), 127.0 (d, $^3J_{PC}$ = 94.0 Hz, C$_{ipso}$), 128.1 (d, $^3J_{PC}$ = 12.2 Hz, C$_{meta}$), 131.4 (s, C$_{para}$), 133.6 (d, $^2J_{PC}$ = 10.6 Hz, C$_{ortho}$), 146.9 (s, C—N), 147.0 (s, C—N), 167.7 (d, $^2J_{PC}$ = 12.2 Hz, C=O), 172.1 (d, $^3J_{PC}$ = 17.6 Hz, C=O). ^{31}P NMR (CDCl$_3$): δ 24.6.

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