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$$\text{Ph}_3\text{P} + \text{ClCH}_2\overset{\text{O}}{\parallel}\text{CCH}_3 \longrightarrow [\text{Ph}_3\text{PCH}_2\overset{\text{O}}{\parallel}\text{CCH}_3]\text{Cl} \xrightarrow{\text{NaOH}} \text{Ph}_3\text{P}=\text{CH}\overset{\text{O}}{\parallel}\text{CCH}_3$$

**1**
**2**

spectra indicate that there is a CH at 70.68 ppm coupled to the  $^{31}\text{P}$  nucleus with  $J(\text{PC})$  99 Hz. An HMQCB (heteronuclear multiple-quantum coherence with bilinear rotation decoupling) experiment confirmed that the CH correlates with the proton resonance at 4.44 ppm and that there are two coincident methyl resonances.

$$\left[ \begin{array}{c} \text{Ph}_3\text{P} \\ | \\ \text{C} \\ / \quad \backslash \\ \text{H} \quad \text{C}=\text{O} \\ | \quad | \\ \text{H} \quad \text{CH}_3 \end{array} \right]^+ \rightleftharpoons \left[ \begin{array}{c} \text{Ph}_3\text{P} \\ | \\ \text{C} \\ / \quad \backslash \\ \text{H} \quad \text{C}=\text{OH} \\ | \quad | \\ \text{H} \quad \text{CH}_3 \end{array} \right]$$

Group	NMR <sup>a</sup>	Species		
		[Ph <sub>3</sub> PCH <sub>2</sub> C(O)CH <sub>3</sub> ]Cl <b>1</b>	[Ph <sub>3</sub> PCH=C(OH)CH <sub>3</sub> ]Cl	Ph <sub>3</sub> P=CHC(O)CH <sub>3</sub> <b>2</b>
CH <sub>2</sub>	<sup>1</sup> H	d 5.91, <i>J</i> (PH) 12	—	—
	<sup>13</sup> C	d 40.62, <i>J</i> 59	—	—
CH	<sup>1</sup> H	—	d 4.44 <i>J</i> (PH) 20	d 3.66, <i>J</i> (PH) 27
	<sup>13</sup> C	—	d 70.68, <i>J</i> (PC) 99	d 51.56 <i>J</i> (PC) 108
C=O/C—OH	<sup>1</sup> H	—	12.6	—
	<sup>13</sup> C	—	181.5 or 201.5	d 190.78 <i>J</i> (PC) 2
CH <sub>3</sub>	<sup>1</sup> H	—	2.32	2.06, <i>J</i> 2
	<sup>13</sup> C	32.11 <i>J</i> (PC) 7 & 23.63, <i>J</i> (PC) 13 <sup>b</sup>	—	d 28.54, <i>J</i> (PC) 15
<sup>31</sup> P shift/ppm <sup>c</sup>		21.87	15.37	14.52

<sup>a</sup>Recorded in CD<sub>2</sub>Cl<sub>2</sub>. The assignment is based on the relative integration from the <sup>1</sup>H spectrum. Shifts are in ppm relative to external H<sub>3</sub>PO<sub>4</sub>.

The spectroscopic information presented here will allow students to discuss these resonance structures with a little more authority.

The addition of D<sub>2</sub>O to the NMR sample did remove the signal attributed to the OH, but it also broadened and reduced the signals for the CH<sub>2</sub>/CH resonances. The treatment of the mixture of tautomers with aqueous NaOH gave a clean sample of the ylide, Ph<sub>3</sub>P=CHC(O)CH<sub>3</sub>. The reverse reaction, the addition of acid to the ylide, is reported to regenerate the phosphonium salt (2). By the published method, we regenerated the same tautomeric mixture of phosphonium ions.

Spectroscopic data are summarized in Table 1, but they do not contradict data previously described (1).

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### Note

1. Chicote, M. T.; Vicente, J. Personal communication (9 May, 2001). The NMR spectrum of the tautomeric mixture of **1** has been confirmed at 200 MHz. At 300 MHz, the methyl resonances separated into two singlets at 2.539 and 2.547 ppm. The latter resonance appeared as the larger of the two, but separate integration was not possible. The CH resonance was found at 4.60 ppm as a doublet of doublets (<sup>2</sup>J(PH) = 19 Hz, <sup>4</sup>J(HH) = 2 Hz). The CH<sub>2</sub> resonance was found at 6.14 ppm (d, <sup>2</sup>J(PH) = 11.4 Hz) and the OH at 12.93 ppm (d, J = 2 Hz). The relative integration between the resonance at 6.14 and 4.60 ppm was 9:2. No such tautomeric mixture has been found for [Ph<sub>3</sub>PCH<sub>2</sub>COR]Cl where R = OMe, OEt, Ph.

### Literature Cited

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