

**Table 1.** Phosphonic Acid Diesters **2**, **3**, and **4** Prepared from Phosphonic Dichlorides **1**, Benzyl Alcohol, and a Second Alcohol

Di-chloride	Mixture of Alcohols	Products	Yield <sup>a</sup> (%)	Molecular Formula <sup>b</sup>	MS <sup>c</sup> <i>m/z</i>
<b>1a</b>	PhCH <sub>2</sub> OH + <i>n</i> -C <sub>14</sub> H <sub>29</sub> OH	<b>2a</b>	19	C <sub>20</sub> H <sub>39</sub> O <sub>3</sub> P (338.3)	339
		<b>3a</b>	20	C <sub>34</sub> H <sub>63</sub> O <sub>3</sub> P (550.8)	551
		<b>4a</b>	42	C <sub>27</sub> H <sub>41</sub> O <sub>3</sub> P (444.6)	444
<b>1a</b>	PhCH <sub>2</sub> OH + CH <sub>3</sub> (OCH <sub>2</sub> CH <sub>2</sub> ) <sub>2</sub> OH	<b>2a</b>	23	see above	
		<b>3c</b>	18	C <sub>16</sub> H <sub>27</sub> O <sub>7</sub> P (362.35)	363
		<b>4c</b>	57	C <sub>18</sub> H <sub>23</sub> O <sub>5</sub> P (350.3)	351
<b>1b</b>	PhCH <sub>2</sub> OH + <i>n</i> -C <sub>14</sub> H <sub>29</sub> OH	<b>2b</b>	27	C <sub>16</sub> H <sub>18</sub> ClO <sub>3</sub> P (324.7)	325
		<b>3b</b>	19	C <sub>30</sub> H <sub>62</sub> ClO <sub>3</sub> P (537.2)	537
		<b>4b</b>	40	C <sub>23</sub> H <sub>40</sub> ClO <sub>3</sub> P (431.0)	431
<b>1b</b>	PhCH <sub>2</sub> OH + CH <sub>3</sub> (OCH <sub>2</sub> CH <sub>2</sub> ) <sub>2</sub> OH	<b>2b</b>	13	see above	
		<b>3d</b>	20	C <sub>12</sub> H <sub>26</sub> ClO <sub>7</sub> P (348.75)	349
		<b>4d</b>	48	C <sub>14</sub> H <sub>22</sub> ClO <sub>5</sub> P (336.75)	337

<sup>a</sup> Yield of isolated pure product. All products **2**, **3**, and **4** were obtained as oils.

<sup>b</sup> Satisfactory microanalyses obtained: C  $\pm 0.39$ , H  $\pm 0.35$ . Exception: **3d**, C  $-0.48$ .

<sup>c</sup> FAB technique on a JEOL DX 300 spectrometer.

**Table 2.** Phosphonic Acid Monoesters **6** Prepared

Product	Yield <sup>a</sup> (%)	mp (°C)	Molecular Formula <sup>b</sup>	MS <sup>c</sup> <i>m/z</i>
<b>6a</b>	98	oil	C <sub>20</sub> H <sub>35</sub> O <sub>3</sub> P (354.45)	355
<b>6b</b>	98	33	C <sub>16</sub> H <sub>34</sub> ClO <sub>3</sub> P (340.9)	341
<b>6c</b>	98	oil	C <sub>11</sub> H <sub>17</sub> O <sub>5</sub> P (260.2)	260
<b>6d</b>	98	oil	C <sub>7</sub> H <sub>16</sub> ClO <sub>5</sub> P (246.6)	247

<sup>a</sup> Yield of isolated pure product.

<sup>b</sup> Satisfactory microanalyses obtained (except for 2 values): C  $+0.50$ , H  $\pm 0.33$ ; unsatisfactory: **6c**, C  $+0.50$ ; **6d**, C  $-0.52$ .

<sup>c</sup> FAB technique on a JEOL DX 300 spectrometer.

**Dibenzyl 2-Chloroethylphosphonate (2b), Ditetradecyl 2-Chloroethylphosphonate (3b), and Benzyl Tetradecyl 2-Chloroethylphosphonate (4b); Typical Procedure:**

Under N<sub>2</sub> at 20 °C, 2-chloroethylphosphonic dichloride<sup>+</sup> (**1b**; 5.4 g, 29 mmol) is added to a stirred mixture of anhydrous benzyl alcohol (8.1 g, 29 mmol), tetradecanol (6.2 g, 29 mmol), and pyridine (4.6 g, 58 mmol) in anhydrous benzene (20 mL), and stirring is continued at 20 °C for 24 h. Pyridine hydrochloride is filtered off and the filtrate is evaporated. The pure components of the residue are isolated by preparative HPLC (silica gel, hexane/acetone, 7:3).

**Tetradecyl 2-Chloroethylphosphonate (6b); Typical Procedure:**

A mixture of diester **4b** (1.02 g, 2.39 mmol) and 10% Pd on activated coal (78 mg) in AcOH (100 mL) is hydrogenated at 20 °C and atmospheric pressure. After 1 h, the theoretical amount of H<sub>2</sub> (53.5 mL) has been absorbed. The coal is filtered off. Evaporation of the filtrate gives **6b**; yield: 0.81 g (~100%); mp 33 °C.

**Table 3.** Spectral Data of Phosphonic Acid Diesters **2**, **3**, and **4** and Monoesters **6**

Compound	IR (CCl <sub>4</sub> ) <sup>a</sup> $\nu_{P=O}$ (cm <sup>-1</sup> )	<sup>1</sup> H-NMR (CDCl <sub>3</sub> ) <sup>b</sup> $\delta$ , <i>J</i> (Hz)	<sup>31</sup> P-NMR (CDCl <sub>3</sub> /85% H <sub>3</sub> PO <sub>4 ext</sub> ) <sup>c</sup> $\delta$
<b>2a</b>	1255	5.1 (d, 4H, <i>J</i> = 8); 7.2–8.2 (m, 15H)	19.5
<b>3a</b>	1250	0.7–2.5 (m, 54H); 4.1 (q, 4H, <i>J</i> = 6.5); 7.3–8.1 (m, 5H)	18.7
<b>4a</b>	1253	0.7–2.2 (m, 27H); 4.1 (q, 2H, <i>J</i> = 6.5); 5.1 (de, 2H, <i>J</i> = 8); 7.3–8.1 (m, 10H)	19.0
<b>3c</b>	1250	3.4 (s, 6H); 3.6 (m, 12H); 4.2 (m, 4H); 7.3–8.2 (m, 5H)	18.8
<b>4c</b>	1255	3.4 (s, 3H); 3.6 (m, 6H); 4.2 (m, 2H); 5.1 (d, 2H, <i>J</i> = 8); 7.2–8.2 (m, 10H)	19.2
<b>2b</b>	1245	2.2 (m, 2H); 3.6 (dt, 2H, <i>J</i> = 9, 7); 5.0 (d, 2H, <i>J</i> = 8); 7.4 (s, 10H)	26.6
<b>3b</b>	1243	0.7–2.5 (m, 56H); 3.5–4.3 (m, 6H)	25.4
<b>4b</b>	1246	0.7–1.8 (m, 27H); 2.3 (m, 2H); 3.4–4.2 (m, 4H); 5.1 (d, 2H, <i>J</i> = 9); 7.3 (s, 5H)	26.0
<b>3d</b>	1243	2.4 (m, 2H); 3.4 (s, 6H); 3.3–4.5 (m, 18H)	26.5
<b>4d</b>	1245	2.3 (m, 2H); 3.4 (s, 3H); 3.6 (m, 6H); 4.1 (m, 2H) 5.1 (s, 5H)	26.9

<sup>a</sup> Recorded on a Perkin-Elmer 377 Infrared spectrophotometer.

<sup>b</sup> Recorded on a Varian EM 360 spectrometer.

<sup>c</sup> Recorded on a Bruker WP 80 spectrometer.

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### Transylidations with Phenyliodonium Bis(aryl/alkylsulfonyl) Methylides

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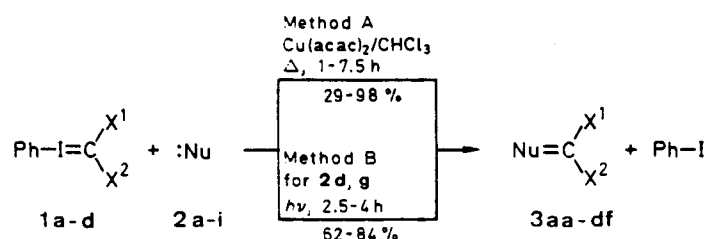
The title compounds have been used to prepare a stable triethoxyphosphonium ylide and also several new ylides of pyridine, triphenylphosphine, triphenylarsine and some sulfur compounds.

Aryliodonium ylides are readily accessible and versatile reagents with interesting synthetic applications.<sup>1,2,3</sup> One of their most characteristic reactions is transylation, i.e. formation of new ylides of other elements upon treatment with the appropriate nucleophile, where X<sup>1</sup> and X<sup>2</sup> are good electron acceptors and Nu is a non-charged nucleophile in which the nucleophilic character is centered at a N.P.As or S atom. The nucleophilic attack at the carbanionic carbon of the iodonium ylide is

apparent and is catalyzed by copper derivatives.<sup>4</sup> The reaction most likely involves the initial complexing of the reactants with the Cu atom, followed by transylidation.

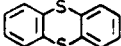


In a preliminary note from this laboratory, it has been reported that phenyliodonium bis(phenylsulfonyl)methylide (**1a**) reacts with pyridine, triphenylphosphine and some sulfur nucleophiles to afford new pyridinium, triphenylphosphonium and sulfonium ylides, respectively.<sup>5</sup> We have extended the range of nucleophiles that react with **1a** and also tested some other phenyliodonium ylides **1b-d** containing the bis(sulfonylmethylene) functionality; all of them give the transylidation reaction and afford new ylides as shown in Table 1. The reactions can be performed either photolytically or thermally in the presence of cupric acetylacetonate ( $\text{Cu}(\text{acac})_2$ ). There is not any clear trend for optimum conditions, and in most of the examples cited both sets of conditions have been applied; the best yields are reported. The use of a nitrogen atmosphere is beneficial in the thermal reactions, but not necessary in the photochemical reactions. With the exception of pyridine, which served as solvent, thermal reactions were carried out in dry chloroform with a ratio of iodonium ylide/nucleophile equal to 1:2; in the photochemical reactions the nucleophile was also the solvent.



I	X <sup>1</sup>	X <sup>2</sup>
a	PhSO <sub>2</sub>	PhSO <sub>2</sub>
b	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>2</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>2</sub>
c	CH <sub>3</sub> SO <sub>2</sub>	CH <sub>3</sub> SO <sub>2</sub>
d	-SO <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> SO <sub>2</sub> -	

2	:Nu	2	:Nu
a	pyridine	e	S(Ph)Me
b	PPh <sub>3</sub>	f	S=C(NMe <sub>2</sub> ) <sub>2</sub>
c	P(OEt) <sub>3</sub>	g	S(O)Me <sub>2</sub>
d	SMe <sub>2</sub>	h	AsPh <sub>3</sub>
		i	

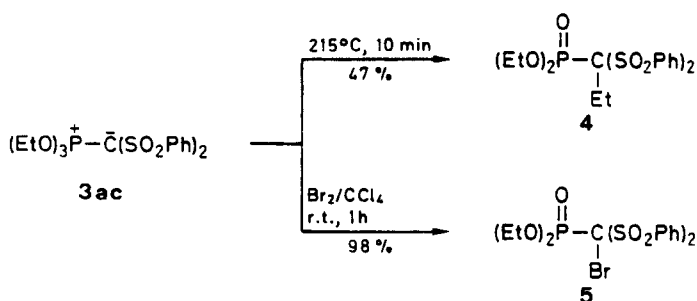
Ylides **3ab**, **3ad**, **3af**, and **3ah** have been prepared previously using several approaches.<sup>6-9</sup> but in most cases yields were low or the starting materials not readily available or both. Special interest presents ylide **3ac**, because it is one of the few known stable<sup>10-12</sup> trialkoxyphosphonium ylides, most of which are only reaction intermediates. Its stability is associated with the dispersal of the negative charge from carbanionic carbon, which becomes very weakly nucleophilic, whereas it is also sterically hindered by the sulfonyl groups. Isomerization of **3ac** to the phosphonic ester **4** was effected under vigorous conditions, by heating it in the absence of solvent up to 215°C.

Table 1. Ylides **3** Prepared

Prod- uct	Condi- tions	Yield <sup>a</sup> (%)	mp (°C) (solvent)	Molecular Formula <sup>b</sup> or Lit. mp (°C)
3aa	Δ, 4 h	75	270-273 (CH <sub>2</sub> Cl <sub>2</sub> )	C <sub>18</sub> H <sub>15</sub> NO <sub>4</sub> S <sub>2</sub> (373.4)
3ab	Δ, 3 h	92	260-264	267-269 <sup>6</sup>
3ac	Δ, 7.5 h	87	131-133 (EtOH)	C <sub>19</sub> H <sub>25</sub> O <sub>4</sub> PS <sub>2</sub> (460.5)
3ad	hν, 3.5 h	84	209-211	209-211 <sup>7</sup>
3ae	Δ, 1 h	29	191-194	C <sub>20</sub> H <sub>18</sub> O <sub>4</sub> S <sub>3</sub> (418.5)
3af	Δ, 7 h	78	172-174	170-173 <sup>9</sup>
3ag	hν, 2.5 h	62	176-178	178-179 <sup>7</sup>
3ah	Δ, 4 h	72	245-247	244-246 <sup>8</sup>
3ai	Δ, 2.5 h	45	212-214 (EtOH/benzene)	C <sub>25</sub> H <sub>18</sub> O <sub>4</sub> S <sub>4</sub> (510.7)
3ba	Δ, 3 h	62	245-247 (EtOAc/hexane)	C <sub>20</sub> H <sub>19</sub> NO <sub>4</sub> S <sub>2</sub> (401.5)
3bb	Δ, 4.5 h	48	279-280 (PhCl)	C <sub>33</sub> H <sub>29</sub> O <sub>4</sub> PS <sub>2</sub> (584.6)
3bd	hν, 3.5 h	76	233-235 (EtOAc/hexane)	C <sub>17</sub> H <sub>20</sub> O <sub>4</sub> S <sub>3</sub> (384.5)
3bf	Δ, 4.5 h	92	183-185 (CHCl <sub>3</sub> /hexane)	C <sub>20</sub> H <sub>26</sub> N <sub>2</sub> O <sub>4</sub> S <sub>3</sub> (454.6)
3bh	Δ, 4.5 h	59	253-255 (PhCl)	C <sub>33</sub> H <sub>29</sub> AsO <sub>4</sub> S <sub>2</sub> (628.6)
3cb	Δ, 2 h	81	312-315 (PhCl)	C <sub>21</sub> H <sub>21</sub> O <sub>4</sub> PS <sub>2</sub> (432.5)
3cd	hν, 4 h	79	197-200 (EtOAc/hexane)	C <sub>5</sub> H <sub>12</sub> O <sub>4</sub> S <sub>3</sub> (232.3)
3cf	Δ, 2 h	90	151-152 (EtOAc/hexane)	C <sub>8</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub> (303.4)
3df	Δ, 4 h	98	190-192 (EtOAc/hexane)	C <sub>9</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub> (314.5)

<sup>a</sup> Yields of pure isolated products.

<sup>b</sup> Satisfactory microanalyses obtained: C ± 0.38, H ± 0.25, N ± 0.22; exceptions: **3bd** (C + 0.50) and **3aa** (C - 0.45).



In the presence of a good electrophile **3ac** reacts readily, e.g. a solution of bromine in carbon tetrachloride was instantly decolorized to afford the bromophosphonate **5**.

The structures of new ylides were fully supported by spectral data. With the exception of triphenylarsonium ylides **3ah** and **3bh**, all others had a molecular ion of small intensity in their mass spectra. Some selected IR and <sup>13</sup>C-NMR data have been collected in Table 2. It is of interest to note that sulfonium ylides are characterized by the lowest IR absorptions for the sulfonyl stretching modes and the highest chemical shifts for the carbanionic carbon.

In conclusion, ylides of various kinds containing the bis(aryl/alkylsulfonyl)methylene functionality can be readily obtained from the corresponding phenyliodonium ylides. Even the weakly nucleophilic thianthrene **2i** is capable of taking part in this reaction.

Table 2. Selected Spectral Data of 1a and Some Ylides 3

Compound	IR (Nujol) (cm <sup>-1</sup> )		<sup>13</sup> C-NMR (DMSO- <i>d</i> <sub>6</sub> /TMS), $\delta$	
	$\nu_{\text{asSO}_2}$ <sup>a</sup>	$\nu_{\text{SO}_2}$ <sup>a</sup>	carbanionic C	C <sub>ipso</sub> <sup>b</sup>
1a	1295	1125	119.6	147.1
3aa	1290	1120		
3ab	1305	1130	68.3 ( <i>J</i> = 108 Hz)	146.3
3ac <sup>c</sup>	1320	1140	73.9 ( <i>J</i> = 226 Hz)	146.3
3ad	1300	1130	91.2	146.2
3ae	1330	1160		
3af	1285	1130	66.6	146.5
3ag	1305	1130		
3ah	1300	1130		

<sup>a</sup> The stronger absorptions of each mode are mentioned.

<sup>b</sup> It refers to phenyl C attached to the SO<sub>2</sub> group.

<sup>c</sup> The full <sup>13</sup>C-NMR spectrum of 3ac is:  $\delta$  = 15.5 (CH<sub>3</sub>, *J*<sub>POCC</sub> = 7.2 Hz); 67.3 (CH<sub>2</sub>, *J*<sub>POC</sub> = 7.4 Hz); 73.9 (C, *J*<sub>PC</sub> = 226 Hz); 146.3 (C-1); 128.2 (C-2, C-6); 126.7 (C-3, C-5); 131.4 (C-4). The <sup>1</sup>H-NMR spectrum of 3ac (CDCl<sub>3</sub>/TMS) is:  $\delta$  = 1.32 (t, 9H, *J* = 7 Hz); 4.25 (m, 6H); 7.25–7.62 (m, 6H); 7.83–8.13 (m, 4H).

IR spectra were recorded with a Perkin-Elmer Model 297 spectrophotometer. <sup>1</sup>H-NMR spectra with a Varian A-60A instrument, <sup>13</sup>C-NMR spectra with a Varian CFT-20 instrument and mass spectra with a Hitachi-Perkin Elmer RMU-61 single focusing mass spectrometer at 70 eV.

The iodonium ylides 1a–d have been prepared from (diacetoxyiodo)benzene and the corresponding disulfone as described previously.<sup>13</sup>

#### Ylides from Thermal Reactions: General Procedure:

Ylides 1a–d (1 mmol) and the nucleophile 2a–i (2 mmol) in dry CHCl<sub>3</sub> (30 mL) are refluxed in the presence of Cu(acac)<sub>2</sub> (1 mg, 0.00004 mmol) under a nitrogen atmosphere for a time indicated in Table 1 [for pyridine ylides 3aa and 3ba and for thioanisole ylide 3ae the iodonium ylides are refluxed in pyridine (25 mL) or thioanisole (6 mL), respectively, without solvent]. After removal of the solvent, the residue is triturated with ether, which is discarded, and the crude ylide is recrystallized (Table 1).

#### Ylides from Photochemical Reactions: General Procedure:

The iodonium ylide 1 in Me<sub>2</sub>S or DMSO (30 mL) is irradiated in a Pyrex tube with a Philips 400 W low-pressure mercury lamp for the time shown in Table 1. A work-up similar as above gives the new ylides. An exception for 3ag is that irradiation is performed under a nitrogen atmosphere with the following work-up: brine (150 mL) is added to the reaction mixture followed by extraction with CH<sub>2</sub>Cl<sub>2</sub> (3 × 50 mL); the combined extract is washed with water, dried (MgSO<sub>4</sub>), and freed from the solvent by evaporation. The residue is purified by column chromatography (silica gel, EtOAc/hexane).

#### Diethyl [Bis(phenylsulfonylethyl)phosphonate] (5):

A quantity of 3ac (88 mg, 0.00019 mmol) in CCl<sub>4</sub> (5 mL) is treated with a 0.01 M Br<sub>2</sub> solution in CCl<sub>4</sub> until the color of Br<sub>2</sub> persisted. After 1 h at room temperature volatiles are evaporated, and the residue (96 mg, 98% yield) is recrystallized from chloroform/hexane to give pure 5, mp 153–155°C.

C<sub>17</sub>H<sub>20</sub>BrO<sub>7</sub>PS<sub>2</sub> calc. C 39.93 H 3.91  
(511.35) found 40.05 3.78

IR (Nujol):  $\nu$  = 1340, 1140, 1020 cm<sup>-1</sup>.

<sup>1</sup>H-NMR (CDCl<sub>3</sub>, TMS):  $\delta$  = 1.27 (t, 6H, *J* = 7 Hz); 4.30 (m, 4H); 7.48–7.77 (m, 6H); 8.10–8.33 (m, 4H).

MS: *m/z* = 512.510 (M<sup>+</sup>, 0.3%).

#### Isomerization of 3ac:

A quantity of 3ac (324 mg, 0.0007 mmol) is heated under N<sub>2</sub> in an oil bath at 215°C for 10 min. The residue is chromatographed (silica gel, EtOAc/hexane) to afford a compound assigned the structure diethyl 1,1-bis(phenylsulfonylethyl)propylphosphonate (4) on the basis of spectral evidence: yield: 152 mg (47%); mp 335–337°C (from CHCl<sub>3</sub>/hexane).

IR (Nujol): 1295, 1160, 1030 cm<sup>-1</sup>.

<sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>/TMS):  $\delta$  = 0.88–1.23 (m, 9H), 3.50–4.08 (m, 6H), 7.33–7.67 (m, 6H), 7.83–8.17 (m, 4H).

MS: *m/z* = 460 (M<sup>+</sup>, 1%).

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#### A New Oxidative Cleavage of 2-Nitrocycloalkanones by Hydrogen Peroxide: An Important, Efficient Method for Dicarboxylic Acid or Ketoacid Synthesis

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2-Nitrocycloalkanones are smoothly converted into dicarboxylic acids or ketoacids, depending on whether the nitro group is secondary or tertiary, by treatment with aqueous 30% hydrogen peroxide and potassium carbonate in methyl alcohol solution for 8–10 h at room temperature.

The synthetic utility of 2-nitrocycloalkanones as well as the preparation of these synthetic intermediates has been reviewed.<sup>1</sup> The C–C bond between the carbonyl group and the nitro substituted atom of cyclic 2-nitroketones can be cleaved by nucleophilic agents under mild reaction conditions. This retro Claisen condensation is useful for synthesizing open-chain,  $\alpha,\omega$ -disubstituted compounds, which are difficult to prepare by other methods.