A Novel Diastereoselective Synthesis of β-Phosphonato Unsaturated Thioimidates

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Abstract: A novel, convenient, and efficient approach to the synthesis of α,β-unsaturated thioimidates with a phosphonate functional group in β-position has been reported based on a multicomponent reaction. The generated zwitterions from the reaction of trialkyl phosphites and dialkyl acetylenedicarboxylates are trapped with benzenecarbonyl isothiocyanate to produce the title compounds in good yield.

Key words: thioimidate, trialkyl phosphite, dialkyl acetylenedicarboxylate, benzenecarbonyl isothiocyanate, multicomponent reaction

Thioimidates,[1] especially unsaturated thioimidates[2] are important building blocks in organic synthesis for the synthesis of heterocyclic compounds. Stereoselective synthesis of γ-lactams by iodine-induced lactamization of γ,δ-unsaturated thioimidates has been reported by Takahata and co-workers.[3] Coupling reaction of a cyclic thioimidate with oxazolones is a key step in the synthesis of carzinophilin, which is an antitumor and natural antibiotic.[4] Rearrangement of thiomethylimidate cyclopropane to a pyrrolothiomethylimidate intermediate was reported in the synthesis of dihydropyrroles.[5] Synthesis of amidrazones using an engineered papain nitrile hydratase and cysteine protease-like structures by trapping thioimidate also shows their importance.[6] Macrocyclic ligands containing the thioimidate group also exhibited good affinity towards metal ions, the complexes of which are known to act as a catalyst for RNA cleavage.[7] Recently, glycosyl thioimidates are being used in glycol conjugates and oligosaccharide synthesis.[8]

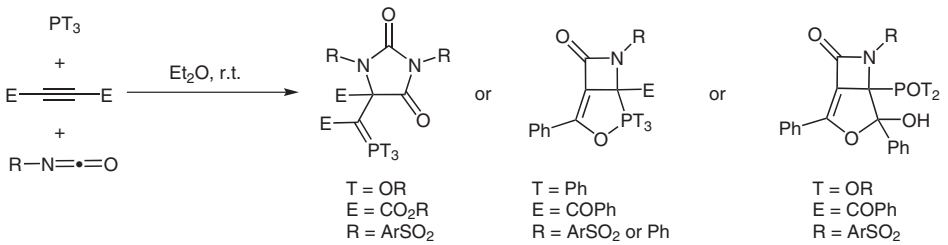
The most important method for their preparation includes the reaction of imidoyl halides[9] or nitriles[6a,10] with S-nucleophiles and conversion of thioamides to thioimidates via an alkylating agent.[7,11] Palladium-catalyzed coupling reaction between bromobenzene, *tert*-butyl isocyanide and anionic S-nucleophiles is also reported for their synthesis.[12] A more recent method is based on the use of imidoylbenzotriazoles as synthetic equivalents of the very hydrolysis sensitive imidoyl chlorides.[13] Synthesis of α,β-unsaturated thioimidates from imines and alkynyl sulfides in the presence of $Sc(OTf)_3$ was reported by Kobayashi and co-workers.[14]

In 1962, Johnson and Tebby established the reaction of P(III) with dimethyl acetylenedicarboxylate (DMAD). This early finding forms the basis for a large class of new backbones thus accessible.[15]

Some of our previous findings included the trapping of 1:1 intermediate of phosphorus nucleophiles and acetylenes (TPP/DMAD) with various electrophiles such as isocyanates (Scheme 1).[16a,b,d]

Along the same line, we became interested in the application of benzenecarbonyl isothiocyanate in these types of reactions for the synthesis of title compounds. Our new synthetic method, which has not been previously reported to our knowledge, is illustrated in Table 1. Reaction between trialkyl phosphites **1**, dialkyl acetylenedicarboxylates **2**, and benzenecarbonyl isothiocyanate proceeds in toluene at room temperature to produce β-phosphonato unsaturated thioimidates **3** in 70–75% yields (Table 1).

The data obtained from elemental analyses, IR, ^1H, ^{13}C and ^{31}P NMR, and mass spectra confirmed the molecular structure of products. The mass spectrum of **3a** displayed molecular ion peak at *m*/*z* 429. The most important absorption band in IR spectrum is due to the P=O stretching frequency of phosphonate group, which appeared at 1249 cm^{-1}. Absorption bands at 1731 and 1690 cm^{-1} are due to



Scheme 1 Three-component reaction between phosphorus nucleophiles, acetylenes, and isocyanates studied earlier

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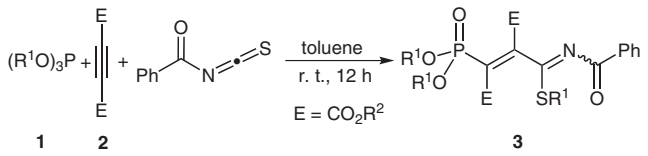
Table 1 β-Phosphonato Unsaturated Thioimidates **3** Prepared

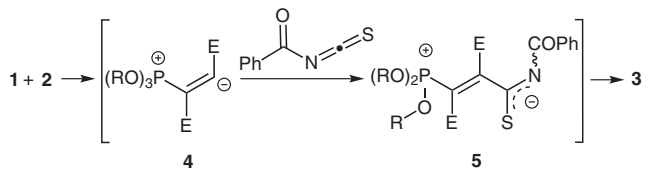


Product	R^1	R^2	Yield (%)
3a	Me	Me	75
3b	Me	Et	72
3c	Et	Me	75
3d	Et	Et	70

the two C=O of esters and one carbonyl group. In the ^{1}H NMR spectrum of **3a**, characteristic singlet signal at δ = 2.67 showed that S-alkylation had taken place, and this signal is due to the thiomethoxy group while the hydrogen chemical shift of NR group appears at about 4–5 ppm.[17] The spectrum exhibited also two sharp singlets recognized as arising from methoxy groups (δ = 3.76 and 3.85). A doublet at 3.62 ppm ($^3J_{P,H}$ = 11.6 Hz) is due to the $PO(OCH_3)_2$ group. The phenyl moiety gave rise to characteristic signals in the aromatic region of the spectrum. The ^{1}H decoupled ^{13}C NMR spectrum of **3a** showed 19 distinct resonances compatible with the proposed structure that 12 peaks of them are due to the six carbons, which split with the P atom. The signal at δ = 15.20, which is due to the thiomethoxy group also confirmed S versus N-alkylation.

The presence of ^{31}P in **3a**–**d** helps in the assignment of the signals by long range coupling with ^{1}H and ^{13}C nuclei. Vicinal ^{31}P-^{13}C coupling constant through a π-bond is a useful tool for assigning the *Z*/*E*-structure. In general, $^3J_{C,P}$-*trans* coupling constant is much larger than $^3J_{C,P}$-*cis*.[18] The comparison of observed $^3J_{C,P}$ for the C=N group with the desired values indicates the product geometries. In the ^{13}C NMR spectrum of **3a**, the $^3J_{C,P}$ coupling constant of 24.1 Hz (δ = 161.93, C=N) showed that there is *E* relationship between $PO(OMe)_2$ and C=N.[18a,19] Finally, it is found that, there is only one of the two probable diastereoisomers (*E* or *Z*), in the products. Thus, the reaction is diastereoselective.

Although we have not established the mechanism of this reaction in an experimental manner, a possible explanation is proposed in Scheme 2. Based on the established chemistry of phosphorus nucleophiles,[16] it is reasonable to assume that the zwitterion **4** is readily formed from the reaction of a trialkyl phosphite **1** and a dialkyl acetylenedicarboxylate **2**. Zwitterion **4** is trapped by benzenecarbonyl isothiocyanate to generate intermediate **5**. In the presence of another molecule of intermediate **5** and probably because of polarizability and nucleophilicity of sulfur atom, S-alkylation takes place to form the products **3** in 70–75% yield.



Scheme 2 Plausible mechanism for the formation of phosphonato unsaturated thioimidates **3**

Overall, we have succeeded in reporting a novel synthetic method for β-phosphonato unsaturated thioimidates via benzenecarbonyl isothiocyanates in a multicomponent reaction. More importantly, the reaction is stereoselective and only one of the two isomers is obtained. In comparison with other reported synthetic methods, the reaction is performed in neutral conditions and substrates are mixed with no bases or catalysts. The simplicity of the present procedure makes it an interesting alternative to the complex multistep approaches.

Trialky phosphites, dialkyl acetylenedicarboxylates, and benzenecarbonyl isothiocyanate, were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Elemental analyses for C, H and N were performed using a Heraeus CHN–O–Rapid analyzer. Mass spectra were recorded on a Finnigan Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded ($CDCl_3$ solution) on a Bruker DRX-500 Avance spectrometer at 500.1 and 125.7 MHz, respectively. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Chromatography columns were prepared from Merck silica gel (230–240 mesh).

Dimethyl 2-[(Benzoylimino)(methylsulfanyl)methyl]-3-(dimethoxyphosphoryl)but-2-enedioate (3a); Typical Procedure

To a magnetically stirred mixture of DMAD (0.14 g, 1 mmol) and benzenecarbonyl isothiocyanate (0.16 g, 1 mmol) in anhyd toluene (3 mL) in a 5 mL flat-bottomed flask was added trimethyl phosphite (0.12 g, 1 mmol) and allowed to stir for 12 h. The solvent was removed under reduced pressure and product was separated by column chromatography over silica gel (Merck 230–240 mesh) using a hexane–EtOAc (7:1) mixture as eluent; yield: 0.32 g (75%); yellow oil.

IR (KBr): 1728 (C=O of ester), 1654 (PhC=O), 1611 (C=C), 1565 and 1433 (Ar), 1249 (P=O), 1188 and 1155 (C–O of ester), 1050 and 1022 cm^{-1} (P–OMe).

^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 2.67 (s, 3 H, SCH_3), 3.62 [d, $^3J_{P,H}$ = 11.6 Hz, 6 H, $PO(OCH_3)_2$], 3.76 (s, 3 H, OCH_3), 3.85 (s, 3 H, OCH_3), 7.44 (t, $^3J_{H,H}$ = 7.4 Hz, 2 H, 2 CH of Ar), 7.54 (t, $^3J_{H,H}$ = 7.7 Hz, 1 H, CH of Ar), 8.16 (d, $^3J_{H,H}$ = 7.9 Hz, 2 H, 2 CH of Ar).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 15.20 (SCH_3), 53.24 (OCH_3), 53.43 (OCH_3), 53.61 [d, $^2J_{P,C}$ = 5.2 Hz, $PO(OCH_3)_2$], 128.28 (2 CH of Ar), 130.41 (2 CH of Ar), 133.16 (CH of Ar), 133.98 (C_{ipso} of Ar), 134.71 (d, $^1J_{P,C}$ = 169.82 Hz, PO*C*=C), 144.03 (d, $^2J_{P,C}$ = 6.4 Hz, POC=*C*), 161.93 (d, $^3J_{P,C}$ = 24.1 Hz, *C*=NCO), 164.88 (d, $^2J_{P,C}$ = 9.4 Hz, *C*O_2Me), 171.88 (d, $^3J_{P,C}$ = 7.0 Hz, *C*O_2Me), 175.88 (Ph*C*=O).

^{31}P NMR (202.4 MHz, $CDCl_3$): δ = 9.49.

MS: *m*/*z* (%) = 429 (5, [M^+]), 382 (15), 370 (10), 320 (10), 105 (100), 77 (30), 51 (10).

Anal. Calcd for $C_{17}H_{20}NO_8PS$ (429.38): C, 47.55; H, 4.69; N, 3.26. Found: C, 47.58; H, 4.71; N, 3.28.

Diethyl 2-[(Benzoylimino)(methylsulfanyl)methyl]-3-(dimethoxyphosphoryl)but-2-enedioate (3b)
Yield: 0.33 g (72%); yellow oil.

IR (KBr): 1724 (C=O of ester), 1660 (PhC=O), 1611 (C=C), 1568 and 1443 (Ar), 1241 (P=O), 1176 and 1151 (C–O of ester), 1089 and 1021 cm^{-1} (P–OMe).

^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.17 (t, $^3J_{H,H}$ = 7.4 Hz, 3 H,OCH_2CH_3), 1.32 (t, $^3J_{H,H}$ = 7.3 Hz, 3 H,OCH_2CH_3), 2.64 (s, 3 H, SCH_3), 3.65 [d, $^3J_{P,H}$ = 11.5 Hz, 6 H, $PO(OCH_3)_2$], 4.21 (q, $^3J_{H,H}$ = 7.0 Hz, 2 H, OCH_2CH_3), 4.31 (q, $^3J_{H,H}$ = 7.1 Hz, 2 H, OCH_2CH_3), 7.43 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, 2 CH of Ar), 7.53 (t, $^3J_{H,H}$ = 7.1 Hz, 1 H, CH of Ar), 8.15 (d, $^3J_{H,H}$ = 7.9 Hz, 2 H, 2 CH of Ar).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 13.67 (OCH_2CH_3), 13.81 (OCH_2CH_3), 15.21 (SCH_3), 53.63 [d, $^2J_{P,C}$ = 5.3 Hz, $PO(OCH_3)_2$], 62.38 (OCH_2CH_3), 62.81 (OCH_2CH_3), 128.35 (2 CH of Ar), 130.36 (2 CH of Ar), 133.10 (CH of Ar), 134.05 (C_{ipso} of Ar), 134.69 (d, $^1J_{P,C}$ = 169.0 Hz, PO*C*=C), 144.14 (d, $^2J_{P,C}$ = 6.3 Hz, POC=*C*), 161.40 (d, $^3J_{P,C}$ = 24.2 Hz, *C*=NCO), 164.31 (d, $^2J_{P,C}$ = 9.6 Hz, CO_2Et), 171.50 (d, $^3J_{P,C}$ = 7.1 Hz, CO_2Et), 175.87 (Ph*C*=O).

^{31}P NMR (202.4 MHz, $CDCl_3$): δ = 8.61.

MS: *m*/*z* (%) = 457 (7, [M^+]), 410 (21), 384 (19), 339 (27), 279 (58), 105 (100), 77 (42), 51 (12).

Anal. Calcd for $C_{19}H_{24}NO_8PS$ (457.43): C, 49.89; H, 5.29; N, 3.06. Found: C, 49.92; H, 5.27; N, 3.08.

Dimethyl 2-[(Benzoylimino)(ethylsulfanyl)methyl]-3-(diethoxyphosphoryl)but-2-enedioate (3c)
Yield: 0.35 g (75%); yellow oil.

IR (KBr): 1729 (C=O of ester), 1657 (PhC=O), 1612 (C=C), 1565 and 1430 (Ar), 1250 (P=O), 1178 and 1155 (C–O of ester), 1041 and 1013 cm^{-1} (P–OEt).

^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.17 [t, $^3J_{H,H}$ = 7.0 Hz, 6 H, $PO(OCH_2CH_3)_2$], 1.47 (t, $^3J_{H,H}$ = 7.4 Hz, 3 H, SCH_2CH_3), 3.29 (q, $^3J_{H,H}$ = 7.4 Hz, 2 H, SCH_2CH_3), 3.72 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 3.95–4.02 [m, 4 H, $PO(CH_2CH_3)_2$], 7.44 (t, $^3J_{H,H}$ = 7.7 Hz, 2 H, 2 CH of Ar), 7.52 (t, $^3J_{H,H}$ = 7.4 Hz, 1 H, CH of Ar), 8.17 (d, $^3J_{H,H}$ = 7.1 Hz, 2 H, 2 CH of Ar).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 13.29 (SCH_2CH_3), 15.90 [d, $^3J_{P,C}$ = 6.6 Hz, $PO(OCH_2CH_3)_2$], 26.66 (SCH_2CH_3), 53.06 (OCH_3), 53.35 (OCH_3), 63.53 [d, $^2J_{P,C}$ = 4.9 Hz, $PO(OCH_2CH_3)_2$], 128.25 (2 CH of Ar), 130.50 (2 CH of Ar), 133.07 (CH of Ar), 134.16 (C_{ipso} of Ar), 135.37 (d, $^1J_{P,C}$ = 167.5 Hz, PO*C*=C), 143.52 (d, $^2J_{P,C}$ = 6.3 Hz, POC=*C*), 162.19 (d, $^3J_{P,C}$ = 24.1 Hz, *C*=NCO), 165.08 (d, $^2J_{P,C}$ = 9.2 Hz, CO_2Me), 171.89 (d, $^3J_{P,C}$ = 7.1 Hz, CO_2Me), 175.98 (Ph*C*=O).

^{31}P NMR (202.4 MHz, $CDCl_3$): δ = 5.34.

MS: *m*/*z* (%) = 442 (12), 410 (35), 334 (23), 306 (17), 262 (33), 105 (100), 77 (65), 51 (15).

Anal. Calcd for $C_{20}H_{26}NO_8PS$ (471.46): C, 50.95; H, 5.56; N, 2.97. Found: C, 50.99; H, 5.57; N, 2.98.

Diethyl 2-[(Benzoylimino)(ethylsulfanyl)methyl]-3-(diethoxyphosphoryl)but-2-enedioate (3d)
Yield: 0.35 g (70%); yellow oil.

IR (KBr): 1725 (C=O of ester), 1655 (PhC=O), 1616 (C=C), 1565 and 1459 (Ar), 1244 (P=O), 1170 and 1161 (C–O of ester), 1090 and 1014 cm^{-1} (P–OEt).

^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.18 (t, $^3J_{H,H}$ = 7.0 Hz, 3 H, OCH_2CH_3), 1.22 (t, $^3J_{H,H}$ = 7.1 Hz, 3 H, OCH_2CH_3), 1.32 [t, $^3J_{H,H}$ = 7.1 Hz, 3 H, $PO(OCH_2CH_3)_2$], 1.46 (t, $^3J_{H,H}$ = 7.3 Hz, 3 H, SCH_2CH_3), 3.28 (q, $^3J_{H,H}$ = 7.3 Hz, 2 H, SCH_2CH_3), 3.98 (q, $^3J_{H,H}$ = 7.2 Hz, 2 H, OCH_2CH_3), 4.16–4.25 [m, 4 H, $PO(CH_2CH_3)_2$], 4.31 (q, $^3J_{H,H}$ = 7.2 Hz, 2 H, OCH_2CH_3), 7.44 (t, $^3J_{H,H}$ = 7.3 Hz, 2 H, 2 CH of Ar), 7.53 (t, $^3J_{H,H}$ = 7.4 Hz, 1 H, CH of Ar), 8.17 (d, $^3J_{H,H}$ = 7.0 Hz, 2 H, 2 CH of Ar).

^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 13.40 (SCH_2CH_3), 13.69 (OCH_2CH_3), 13.85 (OCH_2CH_3), 15.99 [d, $^3J_{P,C}$ = 6.5 Hz, $PO(OCH_2CH_3)_2$], 26.58 (SCH_2CH_3), 62.19 (OCH_2CH_3), 62.65 (OCH_2CH_3), 63.42 [d, $^2J_{P,C}$ = 4.7 Hz, $PO(OCH_2CH_3)_2$], 128.22 (2 CH of Ar), 130.45 (2 CH of Ar), 132.98 (CH of Ar), 134.23 (C_{ipso} of Ar), 135.28 (d, $^1J_{P,C}$ = 167.1 Hz, PO*C*=C), 144.30 (d, $^2J_{P,C}$ = 6.2 Hz, POC=*C*), 162.31 (d, $^3J_{P,C}$ = 24.0 Hz, *C*=NCO), 164.71 (d, $^2J_{P,C}$ = 9.5 Hz, CO_2Et), 171.62 (d, $^3J_{P,C}$ = 7.2 Hz, CO_2Et), 175.91 (Ph*C*=O).

^{31}P NMR (202.4 MHz, $CDCl_3$): δ = 5.67.

MS: *m*/*z* (%) = 448 (8), 410 (21), 384 (31), 334 (41), 277 (17), 192 (45), 105 (100), 77 (79), 43 (55).

Anal. Calcd for $C_{22}H_{30}NO_8PS$ (499.51): C, 52.90; H, 6.05; N, 2.80. Found: C, 52.95; H, 6.08; N, 2.79.

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