Crystal structure of ethyl 3-(4-methoxyphenyl)-2-(triphenylphosphonioiminoyl)acrylate, $(C_6H_5)_3PNC(COOC_2H_5)(C_7H_5OCH_3)$

F.-L. Yang*,[I], G. Guo[II] and S. Bittner[III]

[I] Xuchang University, Department of Chemistry, Xuchang, 461000 P. R. China
[II] Nanyang Normal University, Department of Chemistry, Nanyang, 473061 P. R. China
[III] Ben-Gurion University of the Negev, Department of Chemistry, P.O. Box 653, Beer-Sheva 84105, Israel

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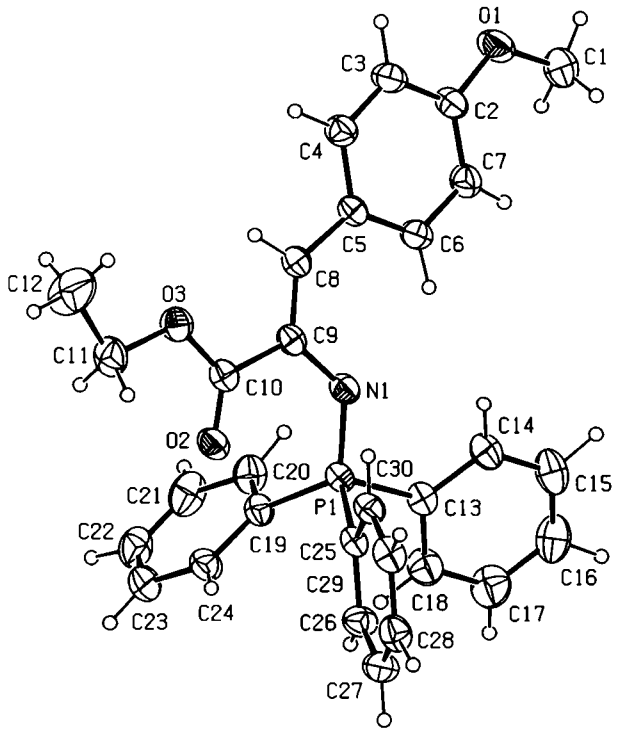

Abstract

$C_{30}H_{28}NO_3P$, monoclinic, $P12_1/c1$ (no. 14), $a = 10.169(4)$ Å, $b = 18.889(7)$ Å, $c = 14.336(5)$ Å, $\beta = 107.75(1)°$, $V = 2622.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.173$, $T = 301$ K.

Source of material

During our search for biological active imidazolinones [1,2], the title compound, a vinyliminophosphorane, was synthesized via the reaction of 2-azido-3-(4-methoxyphenyl)acrylic acid ethyl ester with triphenylphosphane at room temperature under inert and anhydrous conditions. The light yellow product was purified by crystallization from the mixtured solvents of petroleum ether and diethyl ether. Pure colorless single crystals, suitable for X-ray structure analysis, were obtained from dichloromethane (m.p. 122-124 °C).

Experimental details

All H atoms were located from Fourier difference maps and refined freely, except the H atoms attached to the C12 atom being slightly disordered. Those H atoms were added and treated as riding by applying of the rigid body approximation.

* Correspondence author (e-mail: yfling2000cn@yahoo.com.cn)

Discussion

The title compound possesses five distinct units: a 4-methoxyphenyl group, a triphenylphosphoranylimino group, an ester group, C8=C9 double bond and P1=N1 double bond. These units do not lie in the same molecular plane. Only the 4-methoxyphenyl group, the ester group and the N1 atom lie almost in the same plane with the torsion angles O2–C10–C9–C8, O3–C10–C9–N1, C8–C5–C6–C7 and C1–O1–C2–C3 being –177.6(2)°, –177.2(2)°, –177.9(2)° and 179.6(3)°, respectively. The large-scale conjugated system consists of 4-methoxy substituted phenyl ring, C10=O2 double bond and C8=C9 double bond. The four groups linked to the C8=C9 double bond show *Z* configuration. In the unit cell, the weak nonlinear intermolecular hydrogen interactions are formed between the C4–H4 of the 4-methoxy substituted phenyl ring and the C22 atom of one phenyl ring of the triphenylphosphoranyl group in a neighboring molecule, the C–H···C bond length and bond angle are 3.672 Å and 160.6°, respectively. Further weak nonlinear intermolecular hydrogen bonds are formed between the C5 atom of the 4-methoxyphenyl group and H18–C18 of one phenyl ring of the triphenylphosphoranyl group in another neighboring molecule, the C···H–C bond length and bond angle are 3.682 Å and 136.0°, respectively, being in the range known from literature data [2,3].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.47 × 0.52 × 0.67 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.36 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, ω/φ
$2\theta_{max}$:	56.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22335, 6538
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4182
$N(param)_{refined}$:	417
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL-plus [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	–0.251(3)	–0.028(2)	0.642(2)	0.09(1)
H(1B)	4*e*	–0.273(3)	0.049(2)	0.666(2)	0.10(1)
H(1C)	4*e*	–0.328(4)	–0.020(2)	0.721(3)	0.12(1)
H(3)	4*e*	0.082(2)	0.002(1)	0.904(2)	0.056(6)
H(4)	4*e*	0.291(2)	0.049(1)	0.900(2)	0.059(6)
H(6)	4*e*	0.123(2)	0.092(1)	0.610(2)	0.050(6)
H(7)	4*e*	–0.082(2)	0.057(1)	0.618(2)	0.060(6)
H(8)	4*e*	0.438(2)	0.099(1)	0.820(2)	0.049(6)

Table 2. Continued

Atom	Site	x	y	z	U_{iso}
H(11A)	4e	0.824(3)	0.152(1)	0.794(2)	0.076(8)
H(11B)	4e	0.781(3)	0.219(2)	0.836(2)	0.09(1)
H(12A)	4e	0.8089	0.1671	0.9849	0.188
H(12B)	4e	0.9418	0.1483	0.9562	0.188
H(12C)	4e	0.8236	0.0922	0.9419	0.188
H(14)	4e	0.116(3)	0.107(1)	0.453(2)	0.064(7)
H(15)	4e	−0.090(3)	0.100(2)	0.320(2)	0.10(1)
H(16)	4e	−0.128(3)	0.191(1)	0.192(2)	0.085(9)
H(17)	4e	0.040(3)	0.281(2)	0.212(2)	0.09(1)
H(18)	4e	0.237(3)	0.287(2)	0.342(2)	0.078(8)

Table 2. Continued

Atom	Site	x	y	z	U_{iso}
H(20)	4e	0.219(3)	0.297(2)	0.580(2)	0.080(8)
H(21)	4e	0.243(3)	0.416(2)	0.619(2)	0.089(9)
H(22)	4e	0.443(4)	0.478(2)	0.616(3)	0.13(1)
H(23)	4e	0.612(3)	0.417(2)	0.559(2)	0.091(9)
H(24)	4e	0.573(3)	0.300(1)	0.514(2)	0.062(7)
H(26)	4e	0.456(3)	0.238(2)	0.346(2)	0.073(8)
H(27)	4e	0.588(3)	0.183(1)	0.260(2)	0.070(7)
H(28)	4e	0.683(3)	0.071(1)	0.305(2)	0.068(7)
H(29)	4e	0.657(3)	0.017(2)	0.442(2)	0.080(8)
H(30)	4e	0.519(2)	0.072(1)	0.529(1)	0.035(5)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	4e	0.35723(5)	0.19484(3)	0.50917(4)	0.0403(3)	0.0377(3)	0.0533(3)	−0.0034(2)	0.0149(2)	0.0012(2)
O(1)	4e	−0.1414(2)	−0.0019(1)	0.7736(1)	0.0516(9)	0.095(1)	0.074(1)	−0.0188(8)	0.0169(8)	0.0190(9)
O(2)	4e	0.6079(2)	0.19231(8)	0.6606(1)	0.0495(8)	0.0609(9)	0.066(1)	−0.0131(7)	0.0193(7)	0.0032(7)
O(3)	4e	0.6344(2)	0.14545(9)	0.8088(1)	0.0409(8)	0.079(1)	0.0605(9)	−0.0064(7)	0.0088(7)	0.0042(8)
N(1)	4e	0.3322(2)	0.14665(9)	0.5920(1)	0.0433(9)	0.0490(9)	0.055(1)	−0.0068(7)	0.0151(8)	0.0031(7)
C(1)	4e	−0.2628(3)	−0.0010(2)	0.6924(3)	0.051(2)	0.096(2)	0.078(2)	−0.012(1)	0.012(1)	−0.002(2)
C(2)	4e	−0.0217(2)	0.0247(1)	0.7613(2)	0.051(1)	0.053(1)	0.064(1)	−0.0083(9)	0.019(1)	0.008(1)
C(3)	4e	0.0919(2)	0.0225(1)	0.8433(2)	0.055(1)	0.071(2)	0.059(1)	−0.008(1)	0.019(1)	0.016(1)
C(4)	4e	0.2166(2)	0.0478(1)	0.8387(2)	0.048(1)	0.060(1)	0.055(1)	−0.0032(9)	0.014(1)	0.007(1)
C(5)	4e	0.2335(2)	0.0757(1)	0.7525(2)	0.046(1)	0.038(1)	0.055(1)	−0.0018(8)	0.0174(9)	0.0004(8)
C(6)	4e	0.1164(2)	0.0769(1)	0.6705(2)	0.053(1)	0.064(1)	0.053(1)	−0.010(1)	0.015(1)	0.011(1)
C(7)	4e	−0.0103(2)	0.0512(1)	0.6748(2)	0.050(1)	0.066(1)	0.058(1)	−0.011(1)	0.006(1)	0.007(1)
C(8)	4e	0.3693(2)	0.1032(1)	0.7566(2)	0.045(1)	0.042(1)	0.054(1)	−0.0003(8)	0.0152(9)	0.0003(8)
C(9)	4e	0.4131(2)	0.1357(1)	0.6872(2)	0.041(1)	0.0372(9)	0.056(1)	−0.0004(7)	0.0170(9)	−0.0022(8)
C(10)	4e	0.5602(2)	0.1608(1)	0.7162(2)	0.042(1)	0.042(1)	0.057(1)	−0.0006(8)	0.0166(9)	−0.0034(8)
C(11)	4e	0.7771(2)	0.1687(2)	0.8394(2)	0.041(1)	0.088(2)	0.077(2)	−0.008(1)	0.011(1)	−0.009(2)
C(12)	4e	0.8437(3)	0.1417(3)	0.9393(3)	0.057(2)	0.223(5)	0.083(2)	−0.007(2)	0.001(2)	0.009(3)
C(13)	4e	0.1947(2)	0.1961(1)	0.4113(2)	0.043(1)	0.043(1)	0.059(1)	0.0024(8)	0.0153(9)	−0.0018(8)
C(14)	4e	0.0992(2)	0.1422(1)	0.4023(2)	0.046(1)	0.063(1)	0.076(2)	−0.006(1)	0.017(1)	0.001(1)
C(15)	4e	−0.0204(3)	0.1406(2)	0.3227(2)	0.046(1)	0.081(2)	0.101(2)	−0.008(1)	0.009(1)	−0.011(2)
C(16)	4e	−0.0443(3)	0.1919(2)	0.2524(2)	0.053(2)	0.090(2)	0.085(2)	0.012(1)	−0.006(1)	−0.011(2)
C(17)	4e	0.0496(3)	0.2453(2)	0.2597(2)	0.074(2)	0.081(2)	0.077(2)	0.011(2)	−0.001(2)	0.015(2)
C(19)	4e	0.3918(2)	0.2878(1)	0.5384(2)	0.052(1)	0.042(1)	0.054(1)	−0.0031(8)	0.0145(9)	−0.0022(8)
C(18)	4e	0.1678(3)	0.2481(1)	0.3386(2)	0.063(2)	0.060(1)	0.074(2)	0.000(1)	0.009(1)	0.012(1)
C(20)	4e	0.2938(3)	0.3240(1)	0.5698(2)	0.060(1)	0.059(1)	0.072(2)	0.003(1)	0.019(1)	−0.012(1)
C(21)	4e	0.3155(4)	0.3951(2)	0.5977(2)	0.091(2)	0.063(2)	0.074(2)	0.019(2)	0.016(2)	−0.017(1)
C(22)	4e	0.4319(4)	0.4290(1)	0.5939(2)	0.112(2)	0.043(1)	0.068(2)	−0.002(1)	0.011(2)	−0.007(1)
C(23)	4e	0.5286(4)	0.3939(1)	0.5623(2)	0.098(2)	0.050(1)	0.071(2)	−0.023(1)	0.023(2)	−0.004(1)
C(24)	4e	0.5094(3)	0.3235(1)	0.5350(2)	0.068(2)	0.046(1)	0.066(1)	−0.010(1)	0.026(1)	−0.002(1)
C(25)	4e	0.4764(2)	0.1603(1)	0.4478(1)	0.0393(9)	0.0377(9)	0.053(1)	−0.0059(7)	0.0124(8)	−0.0003(8)
C(26)	4e	0.4977(3)	0.1928(1)	0.3667(2)	0.070(1)	0.042(1)	0.062(1)	0.002(1)	0.027(1)	0.0048(9)
C(27)	4e	0.5754(3)	0.1601(1)	0.3149(2)	0.078(2)	0.056(1)	0.065(1)	−0.006(1)	0.035(1)	0.002(1)
C(28)	4e	0.6335(2)	0.0944(1)	0.3438(2)	0.053(1)	0.059(1)	0.077(2)	−0.006(1)	0.028(1)	−0.014(1)
C(29)	4e	0.6140(2)	0.0618(1)	0.4246(2)	0.049(1)	0.047(1)	0.073(2)	0.0052(9)	0.016(1)	0.001(1)
C(30)	4e	0.5349(2)	0.0943(1)	0.4761(2)	0.046(1)	0.043(1)	0.059(1)	−0.0020(8)	0.0145(9)	0.0061(9)

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