Crystal structure of (4-phenyl-1-*p*-tolylbut-1-en-3-ynyl)-4-methylbenzoate, C₂₅H₂₀O₂

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Abstract

C₂₅H₂₀O₂, monoclinic, *P*12₁/*c*1 (No. 14), *a* = 10.709(1) Å, *b* = 10.663(1) Å, *c* = 17.478(2) Å, β = 90.37(1)°, *V* = 1995.8 Å³, *Z* = 4, *R*(*F*) = 0.052, *wR*_{ref}(*F*²) = 0.152, *T* = 293 K.

Source of material

The title compound is obtained as an unexpected product in the reaction of 3-phenyl-1-propyne on 4-methylbenzoyl chloride, in triethylamine in presence of Cu(I). In these conditions described in [1] as a general method of alkyne acylation, the formation of 1-(4-methyl benzoyl)-3-phenyl-1-propyne is normally expected. Yellow crystals were grown from heptane solution.

Discussion

A molecule of the title compound (1) is illustrated in the figure (thermal ellipsoids of 50% probability). The crystal structure of 1 is characterised by an alternation of different type of bonds in a complex π system, located in the sequence C6–C8–C9–C10–C11–C12. The central π system is almost planar as indicated by the angle C6–C8–C9–C12, which has a value of –179.9(4)°. In analogous compounds, this value becomes respectively –179.7(3)°, –174.9(4)° and –173.5(3)° when the oxygen atom O18 is replaced by an iodide atom (2) [2], a tellure atom (3) [3] or a *sp*² carbon atom (4) [4]. For these compounds, the bond length values (in Å) of the sequence C6–C8, C8–C9, C9–C10, C10–C11 and C11–C12 are not much different: 1: 1.483(6), 1.333(6), 1.425(7), 1.194(7), 1.426(7); 2: 1.466(7), 1.317(7), 1.426(7), 1.211(8), 1.432(8); 3: 1.481(4), 1.322(3), 1.406(4), 1.213(3), 1.413(4); 4: 1.498(5), 1.342(4), 1.413(4), 1.189(5), 1.444(5), which indicates a very weak sensibility of this bond sequence to the neighbourhood atoms.

Table 1. Data collection and handling.

Crystal:	yellow square block, size 0.32 × 0.36 × 0.45 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.73 cm ^{−1}
Diffraction, scan mode:	Nonius CAD4, ω/2θ
2θ _{max} :	49.96°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	2801, 2801
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1366
<i>N</i> (<i>param</i>) _{refined} :	246
Programs:	SHELXS-86 [5], SHELXL-97 [6], ORTEP-II [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(18)	4e	0.4976	0.2203	0.5273	0.104
H(19)	4e	0.5339	0.1873	0.6120	0.104
H(20)	4e	0.4714	0.3166	0.5930	0.104
H(15)	4e	−0.2615	−0.7142	0.5616	0.079
H(16)	4e	−0.1416	−0.7318	0.5118	0.079
H(17)	4e	−0.2579	−0.6612	0.4780	0.079
H(1)	4e	−0.1310	−0.2570	0.5106	0.063
H(2)	4e	−0.0020	−0.4380	0.6966	0.058
H(3)	4e	0.0934	0.2035	0.6935	0.061
H(4)	4e	0.2887	0.2897	0.6744	0.065
H(5)	4e	0.1679	−0.0559	0.5318	0.062
H(6)	4e	−0.2120	−0.4490	0.4629	0.067
H(7)	4e	−0.1070	0.1401	0.6784	0.057
H(8)	4e	−0.0850	−0.6240	0.6532	0.071
H(9)	4e	0.3646	0.0344	0.5122	0.080
H(10)	4e	−0.5360	−0.0140	0.7241	0.050
H(11)	4e	−0.7100	−0.1240	0.7254	0.050
H(12)	4e	−0.7080	−0.3330	0.6932	0.050
H(13)	4e	−0.5230	−0.4010	0.6328	0.050
H(14)	4e	−0.3600	−0.2950	0.6427	0.050

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	0.1940(5)	0.0153(4)	0.5604(3)	0.080(4)	0.044(3)	0.063(3)	-0.003(3)	-0.009(3)	-0.004(2)
C(2)	4e	0.3085(5)	0.0690(5)	0.5491(3)	0.074(4)	0.068(3)	0.077(3)	0.005(3)	0.000(3)	0.005(3)
C(3)	4e	0.3471(5)	0.1722(5)	0.5915(3)	0.073(3)	0.075(4)	0.067(3)	-0.018(3)	-0.009(3)	0.007(3)
C(4)	4e	0.2663(5)	0.2170(5)	0.6447(3)	0.089(4)	0.065(3)	0.059(3)	-0.026(3)	-0.014(3)	-0.002(3)
C(5)	4e	0.1501(5)	0.1663(4)	0.6560(3)	0.081(3)	0.053(3)	0.054(3)	-0.011(3)	-0.005(2)	-0.009(2)
C(6)	4e	0.1127(4)	0.0624(4)	0.6138(2)	0.071(3)	0.042(2)	0.044(2)	-0.002(2)	-0.010(2)	0.003(2)
C(7)	4e	0.4740(5)	0.2292(6)	0.5799(4)	0.095(4)	0.115(5)	0.123(5)	-0.023(4)	-0.010(4)	0.008(4)
C(8)	4e	-0.0111(5)	0.0066(4)	0.6251(2)	0.080(3)	0.037(2)	0.047(2)	-0.006(2)	-0.014(2)	0.002(2)
C(9)	4e	-0.1104(5)	0.0564(4)	0.6583(3)	0.077(3)	0.045(3)	0.059(3)	0.001(3)	-0.009(3)	-0.001(2)
C(10)	4e	-0.2258(5)	-0.0093(5)	0.6666(3)	0.075(4)	0.062(3)	0.069(3)	0.005(3)	-0.010(3)	0.002(3)
C(11)	4e	-0.3198(5)	-0.0673(5)	0.6721(3)	0.073(4)	0.083(4)	0.069(3)	-0.002(3)	-0.015(3)	0.007(3)
C(12)	4e	-0.4313(5)	-0.1387(6)	0.6768(3)	0.061(4)	0.101(5)	0.066(3)	-0.011(3)	-0.009(3)	0.016(3)
C(13)	4e	-0.5388(7)	-0.0909(6)	0.7048(4)	0.075(4)	0.128(5)	0.097(5)	-0.006(4)	-0.006(4)	0.023(4)
C(14)	4e	-0.6446(7)	-0.160(1)	0.7099(5)	0.068(5)	0.206(9)	0.111(6)	0.010(6)	-0.008(4)	0.051(7)
C(15)	4e	-0.6455(9)	-0.279(1)	0.6849(6)	0.096(6)	0.182(9)	0.129(7)	-0.055(8)	-0.025(5)	0.062(7)
C(16)	4e	-0.5423(9)	-0.3285(7)	0.6569(5)	0.124(6)	0.118(6)	0.134(6)	-0.024(6)	-0.020(5)	-0.008(5)
C(17)	4e	-0.4339(6)	-0.2600(8)	0.6508(4)	0.078(4)	0.120(6)	0.127(6)	-0.015(4)	-0.019(4)	0.000(5)
O(18)	4e	-0.0270(3)	-0.1136(2)	0.5930(2)	0.088(2)	0.040(2)	0.049(2)	-0.009(2)	-0.014(2)	0.002(1)
C(19)	4e	-0.0129(4)	-0.2122(4)	0.6412(3)	0.060(3)	0.048(3)	0.051(3)	-0.004(2)	-0.005(2)	0.003(2)
O(20)	4e	0.0321(3)	-0.2008(3)	0.7039(2)	0.089(2)	0.055(2)	0.057(2)	-0.005(2)	-0.023(2)	0.003(2)
C(21)	4e	-0.0606(4)	-0.3297(4)	0.6081(2)	0.055(3)	0.046(3)	0.044(2)	-0.002(2)	-0.003(2)	-0.002(2)
C(22)	4e	-0.1228(4)	-0.3342(4)	0.5387(2)	0.076(3)	0.048(3)	0.059(3)	-0.008(2)	-0.015(2)	0.007(2)
C(23)	4e	-0.1706(4)	-0.4462(4)	0.5122(3)	0.079(3)	0.058(3)	0.060(3)	-0.014(3)	-0.025(2)	-0.006(2)
C(24)	4e	-0.1588(4)	-0.5546(4)	0.5532(3)	0.064(3)	0.044(3)	0.073(3)	-0.011(2)	0.004(2)	-0.002(2)
C(25)	4e	-0.0958(5)	-0.5493(4)	0.6218(3)	0.106(4)	0.046(3)	0.065(3)	0.000(3)	-0.012(3)	0.007(2)
C(26)	4e	-0.0472(5)	-0.4394(4)	0.6490(2)	0.092(4)	0.047(3)	0.054(3)	0.001(3)	-0.017(3)	0.001(2)
C(27)	4e	-0.2095(5)	-0.6764(5)	0.5235(3)	0.094(4)	0.067(3)	0.095(4)	-0.023(3)	-0.001(3)	-0.006(3)

References

1. Chowdhury, C.; Kundu, N. G.: Copper (I) catalysed acylation of terminal alkynes. *Tetrahedron Lett.* **37** (1996) 7323-7324.
2. Barluenga, J.; Llorente, I.; Alvarez-Garcia, J. L.; Gonzalez, J. M.; Campos, P. J.; Diaz, M. R.; Garcia-Granda, S.: Reaction of (*tert*-Butyldimethylsilyl)alkynes with IP₂BF₄: Selective Synthesis of Novel Head-to-Head Dimers. *J. Am. Chem. Soc.* **119** (1997) 6933-6934.
3. Zukerman-Schpector, J.; Caracelli, I.; Dabdoub, M. J.; Dabdoub, V. B.: Structure of (*Z*)-1-*P*-Methoxyphenyltelluro-1,4-Diphenyl But-1-en-3-Yne. *J. Chem. Cryst.* **26** (1996) 389-392.
4. Borbulevych, O. Ya.; Golding, I. R.; Kharitonkin, A. B.; Shishkin, O. V.; Gololobov, Yu. G.: The *N*-phenylamide of 2-cyano-5-phenylpent-2-en-4-ynoic acid. *Acta Crystallogr. C55* (1999) 1019-1020.
5. Sheldrick, G. M.: SHELXS-86. Program for the Determination of Crystal Structure, University of Göttingen, Germany 1985.
6. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structure, University of Göttingen, Germany 1997.
7. Johnson, C. K.: ORTEP-II. Report ORNL- 3974, Oak Ridge National Laboratory, Tennessee, USA 1976.