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Crystal structure of (4-ethoxynaphthalen-1-yl) (furan-2-yl)methanone, C₁₇H₁₄O₃

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Abstract

C₁₇H₁₄O₃, monoclinic, *C*2/*c* (no. 15), *a* = 17.373(4) Å, *b* = 7.8290(16) Å, *c* = 19.781(4) Å, β = 93.62(3)°, *V* = 2685.2(9) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0447, *wR*_{ref}(*F*²) = 0.1313, *T* = 293(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The 1-naphthol (2 mmol) and K₂CO₃ (14 mmol) were ground together into fine powder and stirred vigorously at 60°. Diethyl sulfate (3 mmol) was added directly to the mixture with stirring for 2 h. Then the solid was washed with water and filtrated. The crude products were extracted with diethyl ether, and the solvent was evaporated. The intermediate

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.52 × 0.35 × 0.27 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$\theta_{\max}$, completeness:	27.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	12701, 3074, 0.020
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2247
<i>N</i> (<i>param</i>) _{refined} :	182
Programs:	Rigaku [1], SHELX [2], Diamond [3]

1-ethoxynaphthalene was purified by column chromatography with petroleum ether and EtOAc (*v/v* = 10:1) as eluent. 1-Ethoxynaphthalene (10 mmol) and 2-furoyl chloride (12 mmol) were mixed in dry CH₂Cl₂ (50 mL) at 0 °C, and anhydrous AlCl₃ (22 mmol) was added with vigorous stirring. The mixture was refluxed until the reaction was completed (TLC monitored). The solvent was removed and the crude product was purified by column chromatography with petroleum ether-EtOAc (*v/v* = 15:1) as eluent.

Experimental details

The C–H atoms were constrained to an ideal geometry, with C–H = 0.97 Å. The *U*_{iso} values of the hydrogen atoms of methyl groups were set to 15*U*_{eq}(C_{methyl}) and all other hydrogen atoms were set to 12*U*_{eq}(C).

Comment

Diaryl ketones are important building blocks in the fields of medicinal, pharmaceutical and agricultural chemistry [4]. Their derivatives have been used as selective inhibitors of human immunodeficiency virus type 1 reverse transcriptase [5], antispasmodic agents [6], inhibitors of transforming growth factor-kinase [7]. Great efforts have been devoted to synthesis of the attractive target diaryl ketones skeleton due to its prevalent biological activity [8]. In our continuing effort to search for novel agrochemicals [9–11], herein we reported the synthesis and crystal structure of substituted diaryl ketone *via* alkylation and Friedel-Crafts acylation reactions.

The title compound contained one furan ring and one naphthyl moiety. The angle between these two moieties is

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.17320(6)	0.35899(15)	0.21681(6)	0.0389(3)
C2	0.09423(7)	0.31758(19)	0.21159(6)	0.0476(3)
H2	0.0667	0.3164	0.2504	0.057*
C3	0.05731(7)	0.2791(2)	0.15060(7)	0.0559(4)
H3	0.0050	0.2529	0.1479	0.067*
C4	0.09851(7)	0.27924(19)	0.09225(6)	0.0528(3)
H4	0.0734	0.2515	0.0508	0.063*
C5	0.17484(7)	0.31920(17)	0.09499(6)	0.0449(3)
H5	0.2010	0.3189	0.0554	0.054*
C6	0.21522(6)	0.36158(14)	0.15768(5)	0.0373(3)
C7	0.29635(7)	0.39760(15)	0.16425(6)	0.0403(3)
C8	0.33055(7)	0.42686(17)	0.22783(6)	0.0467(3)
H8	0.3833	0.4480	0.2321	0.056*
C9	0.28940(7)	0.42622(18)	0.28633(6)	0.0476(3)
H9	0.3146	0.4482	0.3283	0.057*
C10	0.21208(7)	0.39312(16)	0.28152(6)	0.0409(3)
C11	0.34308(7)	0.41589(16)	0.10439(6)	0.0442(3)
C12	0.42423(7)	0.36161(16)	0.11219(6)	0.0440(3)
C13	0.46460(8)	0.24736(18)	0.15150(7)	0.0551(3)
H13	0.4464	0.1848	0.1872	0.066*
C14	0.53961(8)	0.2412(2)	0.12809(8)	0.0608(4)
H14	0.5803	0.1735	0.1451	0.073*
C15	0.54066(8)	0.3516(2)	0.07672(7)	0.0576(4)
H15	0.5835	0.3738	0.0523	0.069*
C16	0.20218(8)	0.4033(2)	0.40191(6)	0.0541(3)
H16A	0.2282	0.5125	0.4074	0.065*
H16B	0.2399	0.3132	0.4102	0.065*
C17	0.14005(10)	0.3891(2)	0.45018(7)	0.0628(4)
H17A	0.1042	0.4814	0.4426	0.094*
H17B	0.1623	0.3944	0.4958	0.094*
H17C	0.1136	0.2822	0.4432	0.094*
O1	0.16642(5)	0.38841(13)	0.33440(4)	0.0508(3)
O2	0.31732(5)	0.47276(15)	0.04991(4)	0.0620(3)
O3	0.47091(5)	0.42707(13)	0.06503(4)	0.0519(3)

53.57(4)°. The bond lengths and bond angles were in the usual ranges. It was obviously that there was π - π conjunctive effect between furan ring, C11=O2 and naphthyl moiety which caused shorter bond length of C7—C11 [1.4843(18) Å] and C11—C12 [1.4712(17) Å] than the typical C—C bond length [1.54 Å]. The torsion angle of C10—O1—C16—C17 is 178.3(1)°. The presence of the van der Waals forces leads to the stability of the crystal structures.

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References

1. Rigaku/MS: CrystalClear. Rigaku/MS Inc., The Woodlands, TX, USA (2006).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany (2012).
4. Wang, D. H.; Cui, S. L.: Rh(III)-catalyzed aldehyde C—H bond functionalization of salicylaldehydes with arylboronic acids. *Tetrahedron* **71** (2015) 8511–8516.
5. Wyatt, P. G.; Bethell, R. C.; Cammack, N.; Charon, D.; Dodic, N.; Dumaitre, B.; Evans, D. N.; Green, D. V. S.; Hopewell, P. L.: Benzophenone derivatives: a novel series of potent and selective inhibitors of human immunodeficiency virus type 1 reverse transcriptase. *J. Med. Chem.* **38** (1995) 1657–1665.
6. Rubinstein, K.; Elming, N.; Fakstorp, J.; Hermansen, K.; Pedersen, J. G. A.; Natvig, J. T.: Antispasmodic ortho-substituted phenoxyalkylamines. *J. Med. Chem.* **9** (1966) 804–809.
7. Shimizu, T.; Kimura, K.; Sakai, T.; Kawakami, K.; Miyazaki, T.; Nakouji, M.; Ogawa, A.; Ohuchi, H.; Shimizu, K. J.: Synthesis and evaluation of [2-(4-quinolyloxy)phenyl]methanone derivatives: novel selective inhibitors of transforming growth factor- β kinase. *J. Med. Chem.* **51** (2008) 3326–3329.
8. Li, H. L.; Yang, M.; Qi, Y. X.; Xue, J. J.: Ligand-free Pd-catalyzed carbonylative cross-coupling reactions under atmospheric pressure of carbon monoxide: synthesis of aryl ketones and heteroaromatic ketones. *Eur. J. Org. Chem.* **14** (2011) 2662–2667.
9. Fu, Y.; Wang, M. X.; Zhang, D.; Hou, Y. W.; Gao, S.; Zhao, L. X.; Ye, F.: Design, synthesis, and herbicidal activity of pyrazole benzophenone derivatives. *RSC Adv.* **7** (2017) 46858–46865.
10. Fu, Y.; Sun, Y. N.; Yi, K. H.; Li, M. Q.; Cao, H. F.; Li, J. Z.; Ye, F.: Combination of virtual screening protocol by in silico toward the discovery of novel 4-hydroxyphenylpyruvate dioxygenase inhibitors. *Front. Chem.* **6** (2018) 14.
11. Ye, F.; Ma, P.; Zhang, Y. Y.; Li, P.; Yang, F.; Fu, Y.: Herbicidal activity and molecular docking study of novel ACCase inhibitors. *Front. Plant Sci.* **9** (2018) 1850.