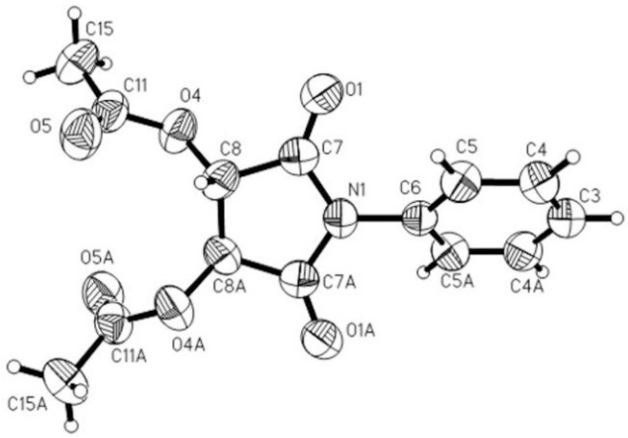
© by Oldenbourg Wissenschaftsverlag, München

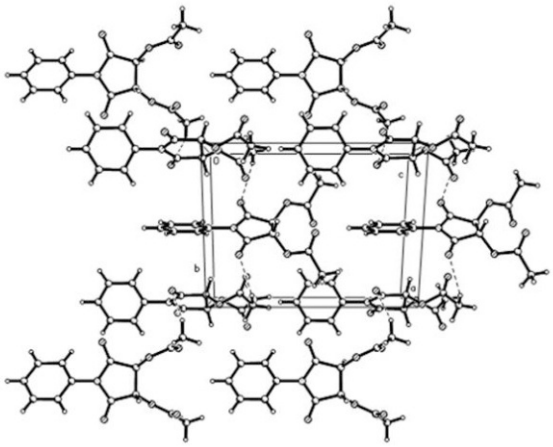
Crystal structure of 3,4-*bis*-(acetyloxy)-*N*-phenyl-succinimide, $C_{14}H_{13}NO_6$

Guo-Gang Tu, Chao Liu, Qiao-Li Lv, Bin-Hai Kuang, Shao-Hua Li*

Department of Pharmacy, Nanchang University Medical College, Nanchang 330006, Jiangxi Province, P. R. China

Received March 17, 2012, accepted June 11, 2012, available online August 09, 2012, CCDC no. 894537





Abstract

$C_{14}H_{13}NO_6$, hexagonal, $P6_2$ (no. 171), a = 10.085(1) Å, c = 11.677(2) Å, V = 1028.5 Å^3, Z = 3, $R_{gt}(F)$ = 0.0504, $wR_{ref}(F^2)$ = 0.1386, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.08×0.14×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.12 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, φ and ω
$2\theta_{max}$:	49.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4781, 633
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 408
$N(param)_{refined}$:	99
Programs:	SHELX [4]

Source of material

Aniline (0.93 g, 0.01 mol) was added dropwise to a solution of dihydro-3,4-*bis*-(acetyloxy)-2,5-furandione (2.16 g, 0.01 mol) in THF. After the mixture was stirred at room temperature for 30 min, *N*-hydroxysuccinimide (1.73 g, 15 mmol) was added to the mixture and then the solution of *N*,*N*'-dicyclohexylcarbodiimide (DCC) (2.06 g, 0.01 mol) in THF was added slowly to the mixture. The reaction mixture was stirred for 19 h at room temperature. The insoluble material was filtered off. The solvent was removed under reduced pressure to get a colourless oil. The target compound was washed with $NaHCO_3$ and crystallized from ethanol (2.36 g, yield 81%, m.p. 111-112°C). Colourless block-shaped single crystals of the title compound suitable for *X*-ray diffraction analysis precipitated after several days.

Experimental details

H atoms were positioned geometrically and refined with a riding model using SHELXL-97 default values (U_{iso}(H) = 1.2 U_{eq}(C) for CH groups and U_{iso}(H) = 1.5 U_{eq}(C) for CH_3). Refinement with all data (Friedel opposites not merged) led to an unsuitably large error of the Flack parameter. The final refinement was therefore performed with a data set with merged Friedel pairs, hence the calculated Flack parameter is meaningless.

Discussion

The title compound, $C_{14}H_{13}NO_6$, was synthesized [1]. The five-membered ring is non-planar and adopts a half-chair conformation. The dihedral angle between the mean plane of the five-membered ring and the phenyl ring is 42.5 (3)° the corresponding value between the mean plane of the five-membered ring and the acetoxy group is 73.7 (5)° (Fig., top). Bond lengths and angles are in normal ranges and comparable to those in related structures [2,3]. In the crystal structure, molecules are linked through intermolecular C–H···O hydrogen bonds forming a three-dimensional network (Fig., bottom).

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

Atom	Site	x	y	z	U_{iso}
H(3)	3*b*	½	½	–0.3119	0.079
H(4)	6*c*	0.3234	0.5364	–0.2165	0.074
H(5)	6*c*	0.3215	0.5376	–0.0164	0.065
H(8)	6*c*	0.3858	0.5417	0.3368	0.063
H(15A)	6*c*	0.2353	0.2173	0.6291	0.104
H(15B)	6*c*	0.2262	0.1297	0.5158	0.104
H(15C)	6*c*	0.0924	0.1593	0.5487	0.104

* Correspondence author (e-mail: tugg199@yahoo.com)

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}
C(3)	3*b*	½	½	–0.2323(8)	0.057(6)	0.072(7)	0.051(6)	0.020(5)	0.0	0.0
C(4)	6*c*	0.3943(8)	0.5219(7)	–0.1756(5)	0.057(4)	0.069(5)	0.055(4)	0.029(4)	–0.005(4)	0.003(4)
C(5)	6*c*	0.3926(8)	0.5225(7)	–0.0565(5)	0.054(4)	0.057(4)	0.055(4)	0.031(3)	0.003(3)	0.004(3)
C(6)	3*b*	½	½	0.0004(7)	0.051(5)	0.033(4)	0.045(5)	0.015(4)	0.0	0.0
C(7)	6*c*	0.3686(8)	0.4194(7)	0.1909(5)	0.051(4)	0.048(4)	0.055(4)	0.026(3)	0.000(3)	0.001(3)
C(8)	6*c*	0.4129(7)	0.4646(7)	0.3140(6)	0.058(4)	0.048(3)	0.052(4)	0.027(3)	0.009(3)	0.009(3)
C(11)	6*c*	0.2790(7)	0.3495(8)	0.4873(5)	0.057(4)	0.069(5)	0.050(4)	0.037(4)	0.008(3)	0.000(3)
C(15)	6*c*	0.2012(7)	0.2006(8)	0.5509(7)	0.075(4)	0.064(4)	0.070(4)	0.035(4)	0.024(4)	0.018(4)
N(1)	3*b*	½	½	0.1253(5)	0.059(5)	0.045(4)	0.041(5)	0.027(4)	0.0	0.0
O(1)	6*c*	0.2418(5)	0.3307(6)	0.1549(4)	0.058(3)	0.063(3)	0.062(3)	0.027(2)	0.006(2)	0.006(2)
O(4)	6*c*	0.3354(5)	0.3328(5)	0.3855(4)	0.078(3)	0.053(3)	0.052(3)	0.034(2)	0.015(2)	0.008(2)
O(5)	6*c*	0.2930(6)	0.4691(6)	0.5177(4)	0.090(4)	0.064(3)	0.061(3)	0.040(3)	0.011(3)	–0.006(2)

Acknowledgments. The work was supported by jiangxi province science foundation for youths (2010GQY0213) and jiangxi province science and technology planning project (2010BSA18500).

References

1. Marson, C. M.; Melling, R. C.: Enantioselective syntheses of *trans*-3,4-difluoropyrrolidines and investigation of their applications in catalytic asymmetric synthesis. J. Org. Chem. 70 (2005) 9771-9779.
2. Usman, A.; Razak, I. A.; Fun, H. K.; Chantrapromma, S.; Zhao, B. G.; Xu, J. H.:1,5-Dichloro-3,6-diphenyl-3-azabicyclo[3.2.0]hept-6-ene-2,4-dione. Acta Crystallogr. **E57** (2001). o990-o991.
3. Kwiatkowski, W.; Karolak-Wojciechowska, J.: Structure of 3-(*p*-chlorophenyl)-1-(3-pyridyl)-2,5-pyrrolidinedione. Acta Crystallogr. **C46** (1990) 913-915.
4. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.