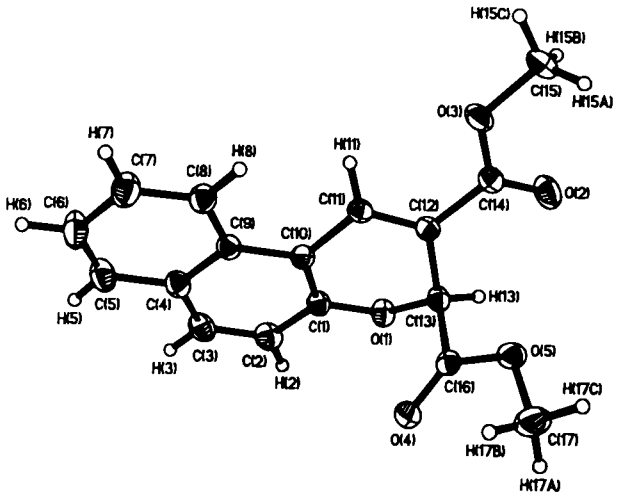
Crystal structure of dimethyl 3*H*-naphtho[2,1,*b*]pyran-2,3-dicarboxylate, $C_{17}H_{14}O_5$

A. Ramazani*, N. Noshiranzadeh, S. Kaffashy, A. Morsali, F. Jamali and F. Gouranlou

University of Zanjan, Department of Chemistry, P.O.Box 45195-313, Zanjan, Iran

Received March 4, 2002, accepted and available on-line May 8, 2002; CCDC-No. 1267/816



Abstract

$C_{17}H_{14}O_5$, monoclinic, $P12_1/n1$ (No. 14), $a = 7.069(2)$ Å, $b = 21.094(6)$ Å, $c = 9.721(3)$ Å, $\beta = 102.56(2)°$, $V = 1414.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.045$, $wR_{ref}(F^2) = 0.115$, $T = 163$ K.

Source of material

Dimethyl 3*H*-naphtho[2,1,*b*]pyran-2,3-dicarboxylate was synthesized in accordance with published procedure [1-3]. The solution of title compound (0.4 g) in acetonitrile-benzene (1:1) (25 ml) left in room temperature for five days. The light yellow crystals were filtered off, washed with cold acetonitrile-benzene (1:1) (7 ml) and dried in vacuum over P_4O_{10} (mp 407 K, yield 75%). Elemental analyses were consistent with the stiochiometry $C_{17}H_{14}O_5$ (Found: C, 68.35%; H, 4.69%. Calc.: C, 68.45%; H, 4.73%).

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer.

Discussion

Pyran derivatives are important heterocycles in bio-organic chemistry and are present in many natural products and pharmaceuticals [1-8]. There have been reported many studies on the synthesis of the heterocyclic pyran ring structure [1-8]. In the recent years there has been increasing interest in the determination of X-ray crystal structures of biologically active compounds [9-19].

In the crystal structure of title compound, the covalent bonds are found to be almost the same as in the previous reports [10,15,16].

Relevant bond lengths: d(O1—C1)= 1.3722(16) Å, d(O1—C13) = 1.4268 (16) Å, d(O2—C14) = 1.2029(17) Å, d(O3—C14) = 1.3353(17) Å, d(O3—C15) = 1.4452(17) Å, d(O4—C16) = 1.1974(16) Å, d(O5—C16) = 1.3352(16) Å, d(O5—C17) = 1.4474(19) Å, d(C10—C11) = 1.4491(18) Å, d(C11—C12) = 1.3386(19) Å, d(C12—C14) = 1.4713(18) Å, d(C12—C13) = 1.5079(18) Å, d(C13—C16) = 1.528(2) Å; angles at the O atoms: 115.64(10)° at O1, 115.50(11)° at O3 and 115.02(12)° at O5. The methyl groups at O3 and O5 are approximatly in the O3–C14–O2 ($\angle$C15–O3–C14–O2 = 0.5(2)°) and O5–C16–O4 ($\angle$C17–O5–C16–O4 = 2.16(19)°) planes, respectively, that agree well with a participation of O3 and O5 in the electron donation to the carbonyl groups. The torsion angle $\angle$C11–C12–C14–O2 = –167.72(14)°, shows clearly that the carbonyl group (C14–O2) and C=C double bond (C11–C12) share approximatly a common plane with *S-trans* conformation, probably is as a result of π-systems conjugations. π-π-ring stacking is observed within the crystal structure, probably is as a result of the presence of parallel aromatic naphthalene rings which stabilize the crystal packing.

Table 1. Data collection and handling.

Crystal:	light yellow prism, size 0.3 × 0.4 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.04 cm^{-1}
Diffractometer, scan mode:	Rebuild Syntex $P2_1$/Siemens P3, $\theta/2\theta$
$2\theta_{max}$:	58.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4062, 3785
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2878
$N(param)_{refined}$:	199
Programs:	SHELXTL-plus [20], SHELXL-97 [21]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.6924	0.1544	0.5251	0.033
H(3)	4*e*	0.7186	0.2535	0.6296	0.035
H(5)	4*e*	0.7196	0.3180	0.8355	0.043
H(6)	4*e*	0.7098	0.3317	1.0718	0.047
H(7)	4*e*	0.6820	0.2398	1.2125	0.043
H(8)	4*e*	0.6682	0.1394	1.1135	0.031
H(11)	4*e*	0.7519	0.0487	1.0257	0.032
H(13)	4*e*	0.6352	–0.0417	0.6531	0.020
H(15A)	4*e*	0.7132	–0.1540	1.1157	0.042
H(15B)	4*e*	0.9202	–0.1455	1.0962	0.043
H(15C)	4*e*	0.8699	–0.1146	1.2301	0.057
H(17A)	4*e*	0.0440	–0.0535	0.5714	0.044
H(17B)	4*e*	0.0373	–0.0166	0.7099	0.055
H(17C)	4*e*	0.0548	–0.0927	0.7147	0.051

* Correspondence author (e-mail: a-ramazani@mail.znu.ac.ir)

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}
O(1)	4*e*	0.6766(2)	0.04768(4)	0.6344(1)	0.0348(5)	0.0218(5)	0.0192(4)	–0.0027(4)	0.0114(4)	0.0010(4)
O(2)	4*e*	0.7276(2)	–0.11727(5)	0.8530(1)	0.0603(8)	0.0214(5)	0.0294(6)	0.0069(5)	0.0106(5)	–0.0006(4)
O(3)	4*e*	0.7680(2)	–0.06707(5)	1.0597(1)	0.0405(6)	0.0225(5)	0.0213(5)	0.0059(4)	0.0049(4)	0.0056(4)
O(4)	4*e*	0.2866(2)	0.04442(5)	0.6011(1)	0.0330(6)	0.0232(5)	0.0245(5)	0.0018(4)	0.0005(4)	0.0014(4)
O(5)	4*e*	0.2992(2)	–0.05160(5)	0.7036(1)	0.0256(5)	0.0289(5)	0.0254(5)	–0.0035(4)	0.0016(4)	0.0064(4)
C(1)	4*e*	0.6789(2)	0.10461(6)	0.7031(1)	0.0240(6)	0.0206(6)	0.0220(6)	–0.0007(5)	0.0070(5)	0.0017(5)
C(2)	4*e*	0.6955(2)	0.15880(7)	0.6231(2)	0.0358(8)	0.0290(7)	0.0229(7)	–0.0002(6)	0.0097(6)	0.0056(6)
C(3)	4*e*	0.7071(2)	0.21673(7)	0.6855(2)	0.0369(8)	0.0232(7)	0.0302(7)	–0.0003(6)	0.0096(6)	0.0093(6)
C(4)	4*e*	0.7010(2)	0.22347(7)	0.8299(2)	0.0256(7)	0.0208(6)	0.0297(7)	–0.0004(5)	0.0068(5)	0.0027(5)
C(5)	4*e*	0.7084(2)	0.28386(7)	0.8938(2)	0.0431(9)	0.0195(7)	0.0391(8)	–0.0005(6)	0.0111(7)	0.0040(6)
C(6)	4*e*	0.7019(3)	0.28989(7)	1.0322(2)	0.053(1)	0.0208(7)	0.0412(9)	–0.0017(7)	0.0129(8)	–0.0056(6)
C(7)	4*e*	0.6867(3)	0.23578(7)	1.1128(2)	0.0462(9)	0.0274(8)	0.0299(7)	–0.0017(7)	0.0105(7)	–0.0058(6)
C(8)	4*e*	0.6798(2)	0.17648(7)	1.0544(2)	0.0307(7)	0.0233(7)	0.0242(7)	–0.0009(6)	0.0071(6)	–0.0001(5)
C(9)	4*e*	0.6876(2)	0.16861(6)	0.9112(1)	0.0197(6)	0.0198(6)	0.0241(6)	–0.0004(5)	0.0046(5)	0.0012(5)
C(10)	4*e*	0.6805(2)	0.10769(6)	0.8457(1)	0.0199(6)	0.0200(6)	0.0208(6)	–0.0012(5)	0.0047(5)	0.0016(5)
C(11)	4*e*	0.7036(2)	0.04846(6)	0.9231(1)	0.0210(6)	0.0218(6)	0.0186(6)	0.0006(5)	0.0044(5)	0.0028(5)
C(12)	4*e*	0.6752(2)	–0.00675(6)	0.8538(1)	0.0212(6)	0.0211(6)	0.0183(6)	0.0006(5)	0.0059(5)	0.0017(5)
C(13)	4*e*	0.5974(2)	–0.00446(6)	0.6966(1)	0.0286(7)	0.0181(6)	0.0177(6)	–0.0002(5)	0.0076(5)	0.0007(5)
C(14)	4*e*	0.7248(2)	–0.06921(6)	0.9191(1)	0.0210(6)	0.0218(6)	0.0231(6)	0.0014(5)	0.0062(5)	0.0021(5)
C(15)	4*e*	0.8214(2)	–0.12696(7)	1.1297(2)	0.0309(8)	0.0269(7)	0.0309(7)	0.0065(6)	0.0059(6)	0.0114(6)
C(16)	4*e*	0.3765(2)	0.00049(6)	0.6603(1)	0.0296(7)	0.0213(6)	0.0131(5)	–0.0014(5)	0.0037(5)	–0.0023(5)
C(17)	4*e*	0.0895(2)	–0.05380(8)	0.6696(2)	0.0260(8)	0.0432(9)	0.0392(9)	–0.0071(7)	–0.0022(6)	0.0077(7)

Acknowledgment. Support of this investigation by Zanjan University Research Council (ZURC2233) is gratefully acknowledged.

References

1. Yavari, I.; Ramazani, A.: An efficient one-pot synthesis of dialkyl 3*H*-naphtho[2,1,*b*]pyran-2,3-dicarboxylates mediated by vinyltriphenylphosphonium salt. Synth. Commun. **27** (1997) 1385-1390.
2. Yavari, I.; Ramazani, A.: Vinyltriphenylphosphonium salt mediated preparation of dialkyl 2*H*-1-benzopyran-2,3-dicarboxylates. An efficient one-pot synthesis of 2*H*-chromene derivatives. J. Chem. Research (S) (1996) 382-383.
3. Ramazani, A.; Fattahi-Nujokamberi, G. R.: Synthesis of 2*H*-1-benzopyran derivatives with a strongly electron-withdrawing substituent at 6-position. Indian J. Chem. **41B** (2002) 572-574.
4. Yan, M.-C.; Jang, Y.-J.; Yao, C.-F.: An easy and efficient method for the synthesis of 2,2-dialkyl-3-nitrochromene. Tetrahedron Lett. **42** (2001) 2717-2721.
5. Kaye, P. T.; Nocanda, X. W.: Application of Baylis-Hillman methodology in a chemoselective synthesis of 3-acyl-2*H*-1-chromenes. J. Chem. Soc., Perkin Trans. 1 (2000) 1331-1332.
6. Subburaj, K.; Trivedi, G. K.: Microwave-assisted rate-enhanced method for the synthesis of 2,2-dimethyl-2*H*-chromenes. Bull. Chem. Soc. Jpn. **72** (1999) 259-263.
7. Matloubi-Moghaddam, F.; Sharifi, A.; Saidi, M. R.: Microwave promoted selective synthesis of naphthopyrans and naphthofurans: Application to the synthesis of natural lapachenole. J. Chem. Research (S) (1996) 338-339.
8. Majumdar, K. C.; Chatterjee, P.: Regioselective synthesis of pyrano[3,2-*f*] chromen-2(7*H*)-ones. J. Chem. Research (S) (1996) 462-463.
9. Parsons, S.; Smith, A. G.; Tasker, P. A.; White, D. J.: Acetone-4-methylthiosemicarbazone at 220 K. Acta Crystallogr. C**56** (2000) 237-238.
10. Lovejoy, D. B.; Richardson, D. R.; Bernhardt, P. V.: 2-Hydroxy-1-naphthaldehyde 2-methylthiosemicarbazone. Acta Crystallogr. C**56** (2000) 341-342.
11. Zhu, D.-R.; Xu, Y.; Zhang, Y.; Wang, T.-W.; You, X.-Z.: 4-Phenyl-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole. Acta Crystallogr. C**56** (2000) 895-896.
12. Jain, S. C.; Bharadvaja, A.; Kumar, R.; Agarwal, D.; Errington, W.: 3-[4-(3,4-Dimethylphenyl)-1,3-thiazol-2yl]-2-(2-hydroxyphenyl)-1,2,3,4-tetrahydroquinazolin-4-one. Acta Crystallogr. C**56** (2000) 592-593.
13. Janes, R. W.: 1-[5-[2-(Trifluoromethyl)phenyl]-1,3,4-thiadiazol-2-yl]-guanidinium chloride. Acta Crystallogr. C**56** (2000) 508-509.
14. Hu, N.-H.; Kiyota, Y.; Aoki, K.: Oxythiamine hexafluorophosphate-monohydrate, a thiamine antagonist with the same conformation as thiamine. Acta Crystallogr. C**56** (2000) 1284-1286.
15. Singh, U. P.; Thomas, M.; Seshadri, T. P.; Bhattacharya, S.: Ethyl 2-[*N*-(*tert*-butyloxycarbonyl)-L-alanylamino]-4-methyl-1,3-thiazole-5-carboxylate reveals a *trans* orientation of the preceding amide N-H with respect to the thiazole-ring sulfur. Acta Crystallogr. C**56** (2000) 1482-1483.
16. Boryczka, S.; Schreurs, A. M. M.; Kroon, J.; Steiner, T.: Bis(4-propargyloxy-3-quinolythio)-methane. Acta Crystallogr. C**56** (2000) 1234-1235.
17. Morsali, A.; Ramazani, A.; Jamali, F.; Gouranlou, F.: Crystal structure of diaqua-bis[N-(2-pyridyl)carbonylaniline]zinc(II) diperchlorate, $Zn(C_{12}H_{16}N_2O)_2(H_2O)_2(ClO_4)_2$. Z. Kristallogr. NCS **216** (2001) 605-606.
18. Morsali, A.; Ramazani A.; Jamali, F.; Gouranlou, F.: Crystal structure of pyridine-2-carbaldehyde thiosemicarbazonium perchlorate, $C_7H_9N_4S)(ClO_4)$. Z. Kristallogr. NCS **216** (2001) 607-608.
19. Ramazani A.; Morsali, A.; Jamali, F.; Gouranlou, F.; Jalilian, A. R.; Momeni-Movahhed, A.: Crystal structure of 4-[4-(dimethylamino)-phenyl]-5-(2,4-dimethyl-1,3-thiazol-5-yl)-2,4-dihydro-3*H*-1,2,4-triazol-3-thione, $C_{15}H_{17}N_5S_2$. Z. Kristallogr. NCS **217** (2002) 149-150.
20. Sheldrick G. M.: SHELXTL-plus v. 5.10, Structure Determination Software Suite, Bruker AXS, Madison, Wisconsin, USA 1998.
21. Sheldrick G. M.: SHELXTL-97. A Program for Refining Crystal Structures.Version 5.1o, Bruker AXS Inc., Madison, WI-53719, USA 1997.