Crystal structure of 3-(1′-nitropropyl)isobenzofuran-1(3*H*)-one, $C_{11}H_{11}NO_4$

R. Kakou-Yao[I], A. Saba[II], N. Ebby[I], M. Pierrot[III] and J. P. Aycard*,[IV]

[I] Université de Cocody, Laboratoire de Cristallographie et de Physique Moleculaire, UFR SSMT, 22 BP 582 Abidjan 22, Cote d'Ivoire
[II] Université de Ouagadougou, Laboratoire de Chimie Organique, Structure et Réactivité, 03 BP 7021 Ouagadougou 03, Burkina Faso
[III] LBS-UMR 6517, Centre Scientifique Saint-Jérôme, F-13397 Marseille, Cedex 20, France
[IV] Université de Provence, Laboratoire de Spectrometrie et Dynamique Moléculaire, Case 542 Avenue Escadrille Normandie Niemen, F-13397 Marseille Cedex 20, France

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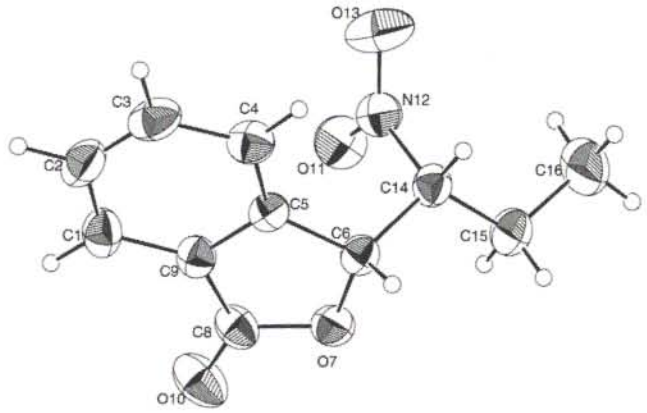

Abstract

$C_{11}H_{11}NO_4$, monoclinic, $P12_1/c1$ (No. 14), a = 8.861(1) Å, b = 11.794(1) Å, c = 10.891(1) Å, β = 103.682(1) Å, V = 1105.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.089, $wR(F)$ = 0.171, T = 298 K.

Source of material

The compound was synthetized according to the Hauser procedure [1]. The crystallization was done from CH_2Cl_2.

Discussion

The structure was determined to identify the diasteroisomer presence in the crystal. We observe the dl (*RS/SR*) ones. The atoms of the ring moiety are coplanar. The packing shows intramolecular (H6–O7 = 1.999(1) Å) and intermolecular hydrogen bonds (H6–O10 = 2.4268 Å, H3–O13 = 2.530(1) Å).

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.35 × 0.45 × 0.55 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.02 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	50.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2106, 2027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 1610
$N(param)_{refined}$:	145
Programs:	MULTAN [2], maXus [3], ORTEPII [4]

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

Atom	Site	x	y	z	U_{iso}
H(6)	4*e*	0.07243	0.24014	–0.02783	0.05
H(14)	4*e*	0.30583	0.31607	–0.00169	0.05
H(1)	4*e*	0.11297	–0.09673	0.25266	0.05
H(4)	4*e*	0.30961	0.07239	–0.07228	0.05
H(15A)	4*e*	0.13755	0.45420	0.03367	0.05
H(15B)	4*e*	0.19025	0.43040	0.17917	0.05
H(3)	4*e*	0.34885	–0.11904	–0.02120	0.05
H(2)	4*e*	0.25208	–0.20212	0.13777	0.05
H(16A)	4*e*	0.30687	0.58943	0.12911	0.05
H(16B)	4*e*	0.37857	0.51933	0.03451	0.05
H(16C)	4*e*	0.43127	0.49553	0.18001	0.05

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(5)	4*e*	0.1842(1)	0.10262(8)	0.06252(8)	0.0499(5)	0.0498(5)	0.0514(4)	–0.0003(3)	0.0040(3)	–0.0087(3)
C(6)	4*e*	0.1361(1)	0.22444(8)	0.05477(8)	0.0567(5)	0.0514(5)	0.0556(5)	0.0023(4)	0.0107(4)	–0.0014(3)
O(7)	4*e*	0.04958(8)	0.23718(6)	0.14981(8)	0.0631(5)	0.0537(4)	0.0938(5)	0.0003(3)	0.0287(4)	–0.0128(3)
N(12)	4*e*	0.3997(1)	0.27009(7)	0.18078(9)	0.0649(6)	0.0580(5)	0.0742(6)	–0.0033(4)	0.0118(4)	–0.0166(4)
C(14)	4*e*	0.2681(1)	0.31077(8)	0.07381(9)	0.0655(6)	0.0504(5)	0.0628(5)	0.0021(4)	0.0176(4)	–0.0006(4)
O(10)	4*e*	–0.0221(1)	0.13178(8)	0.29641(9)	0.1092(7)	0.0961(7)	0.0904(6)	–0.0310(5)	0.0554(5)	–0.0263(4)
C(1)	4*e*	0.1515(1)	–0.06133(9)	0.1868(1)	0.0774(7)	0.0556(6)	0.0731(6)	–0.0088(5)	0.0047(5)	0.0033(4)
C(9)	4*e*	0.1266(1)	0.05220(8)	0.15631(8)	0.0583(5)	0.0503(5)	0.0552(5)	–0.0052(4)	0.0075(4)	–0.0044(3)

* Correspondence author (e-mail: aycard@piimsdm3.univ-mrs.fr)

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	4*e*	0.0443(1)	0.13747(9)	0.21218(9)	0.0674(6)	0.0652(6)	0.0631(6)	–0.0143(5)	0.0234(4)	–0.0111(4)
C(4)	4*e*	0.2677(1)	0.0408(1)	–0.0062(1)	0.0684(6)	0.0684(7)	0.0711(6)	0.0012(5)	0.0218(5)	–0.0163(5)
O(11)	4*e*	0.3801(1)	0.26458(8)	0.28545(8)	0.0922(7)	0.1052(7)	0.0693(5)	–0.0032(5)	0.0109(4)	0.0025(4)
C(15)	4*e*	0.2222(1)	0.42980(9)	0.1009(1)	0.0842(8)	0.0489(6)	0.0990(8)	0.0063(5)	0.0303(6)	–0.0028(5)
O(13)	4*e*	0.5218(1)	0.2455(1)	0.1536(1)	0.0689(6)	0.1313(9)	0.1254(8)	0.0214(5)	0.0140(5)	–0.0393(6)
C(3)	4*e*	0.2905(1)	–0.0743(1)	0.0250(1)	0.0650(7)	0.0667(7)	0.115(1)	0.0089(5)	0.0136(6)	–0.0293(7)
C(2)	4*e*	0.2349(1)	–0.1230(1)	0.1190(1)	0.0685(7)	0.0520(6)	0.117(1)	0.0061(5)	–0.0058(7)	–0.0025(6)
C(16)	4*e*	0.3452(2)	0.5163(1)	0.1121(2)	0.129(1)	0.0551(8)	0.181(2)	–0.0095(8)	0.074(1)	–0.0169(8)

References

1. Hauser, F. M.; Baghdanon, V. M.:A new procedure forregiospecific syntheses of benzopyran-1-ones. J. Org. Chem. **53** (1988) 4676-4681.
2. Main, P.; Fiske, S. J.; Hull, S. E.; Lessinger, L.; Germain, G.; Declercq, J. P.; Woolfson, M. M.: MULTAN 11/82. A system of Computer Programs for the automatic Solution of Crystal Structures from X-ray Diffraction Data. Univs. of York, England and Louvain, Belgium 1982. Nonius. Kappa CCD Reference Manual. Nonius B.V., P.O. Box 811, 2600 Av, Delft, The Netherlands 1988.
3. Mackay, S.; Gilmore, C. J.; Edwards, C.; Tremayne, M.; Stewart, N.; Shankland, K.: University of Glasgow, Scotland, UK, Nonius BV, Delft, The Netherlands and MacScience Co. Ltd., Yokohama, Japan 1988.
4. Johnson, C. K.: ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.