Crystal structure of *dl*-dimethyl 2,3-dibenzamido-2,3-dimethyl-butanedioate, $C_{22}H_{24}N_2O_6$

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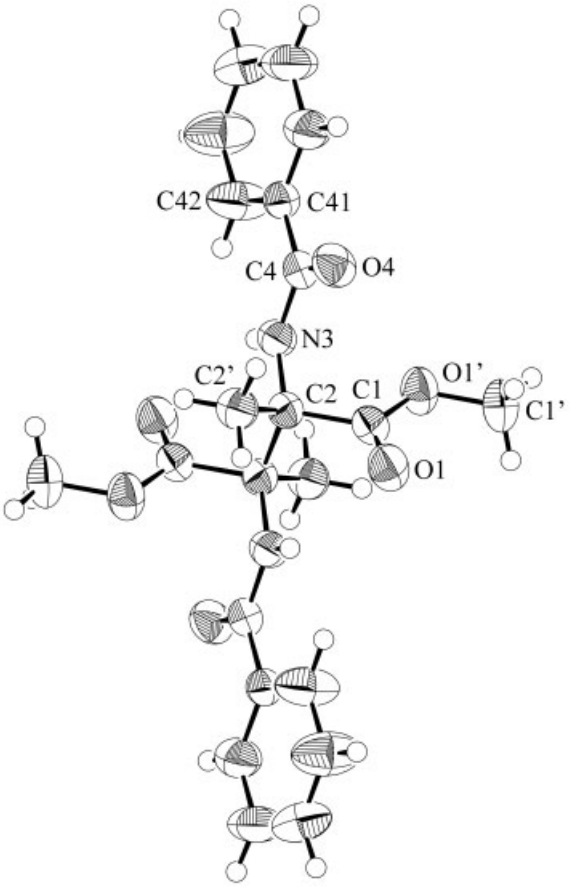

Abstract

$C_{22}H_{24}N_2O_6$, monoclinic, $P12_1/c1$ (No. 14), a = 10.982(2) Å, b = 9.440(1) Å, c = 10.375(3) Å, β = 111.06(1)°, V = 1003.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.074, $wR_{ref}(F^2)$ = 0.222, T = 293 K.

Source of material

The title compound, as a mixture of isomers, was prepared from the reaction between di-*tert*-butyl peroxide and *N*-benzoylalanine methyl ester in accord with literature methods [1,2]. These were separated [3] and the analysed crystallographically to determine the structure of the particular isomer. Colourless crystals of the *dl*-isomer were obtained from the crystallisation of an ethyl acetate/hexane solution of the compound (mp 421.5 K – 424 K).

Discussion

The molecule is centrosymmetric about the C2—C2^i bond with d(C2—C2^i) = 1.566(4) A; i: –x, 1–y, 1–z. Significant elongation of the central bond was sought as a result of strain and stability of the product radicals that would form through bond scission but, this was not found in this structure nor in that of the *meso* isomer [4].

Table 1. Data collection and handling.

Crystal:	colourless cube, size 0.57 × 0.57 × 0.57 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
$2\theta_{max}$:	55.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2526, 2393
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1767
$N(param)_{refined}$:	137
Programs:	teXsan [5], SHELXS-86 [6], SHELXL-97 [7], DIFABS [8], ORTEPII [9]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	–0.1759	0.4258	0.5084	0.057
H(11a)	4*e*	0.1410	0.0466	0.6419	0.098
H(11b)	4*e*	0.2445	0.1586	0.6369	0.098
H(11c)	4*e*	0.1644	0.0736	0.5035	0.098
H(21a)	4*e*	–0.1189	0.5763	0.2794	0.078
H(21b)	4*e*	–0.0826	0.4304	0.2305	0.078
H(21c)	4*e*	0.0258	0.5449	0.2945	0.078
H(42)	4*e*	–0.3490	0.3905	0.5160	0.100
H(43)	4*e*	–0.5514	0.3328	0.5141	0.115
H(44)	4*e*	–0.6612	0.1467	0.3888	0.094
H(45)	4*e*	–0.5648	0.0094	0.2729	0.097
H(46)	4*e*	–0.3674	0.0723	0.2623	0.079

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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*	0.0624(2)	0.2253(2)	0.5494(2)	0.063(1)	0.0515(9)	0.066(1)	0.0113(7)	0.0327(8)	0.0161(8)
O(1)	4*e*	0.1662(2)	0.3380(2)	0.4330(2)	0.064(1)	0.053(1)	0.083(1)	0.0066(8)	0.0434(9)	0.0065(8)
O(4)	4*e*	–0.1687(2)	0.2149(2)	0.2965(2)	0.065(1)	0.0506(9)	0.075(1)	–0.0003(7)	0.0348(9)	–0.0114(8)
N(3)	4*e*	–0.1453(2)	0.3874(2)	0.4512(2)	0.0456(9)	0.0426(9)	0.056(1)	–0.0025(7)	0.0207(8)	–0.0040(8)
C(1)	4*e*	0.0774(2)	0.3282(2)	0.4723(2)	0.051(1)	0.039(1)	0.049(1)	–0.0002(8)	0.0210(9)	–0.0010(9)
C(1')	4*e*	0.1613(3)	0.1170(3)	0.5860(3)	0.076(2)	0.054(1)	0.067(2)	0.020(1)	0.026(1)	0.015(1)
C(2)	4*e*	–0.0266(2)	0.4426(2)	0.4422(2)	0.0428(9)	0.040(1)	0.043(1)	0.0003(8)	0.0152(8)	0.0030(8)
C(4)	4*e*	–0.2102(2)	0.2783(2)	0.3740(2)	0.047(1)	0.037(1)	0.052(1)	0.0020(8)	0.0155(9)	0.0023(9)
C(2')	4*e*	–0.0531(2)	0.5043(2)	0.2982(2)	0.059(1)	0.054(1)	0.039(1)	–0.003(1)	0.0138(9)	0.0063(9)
C(41)	4*e*	–0.3367(2)	0.2410(2)	0.3868(2)	0.046(1)	0.042(1)	0.047(1)	–0.0009(8)	0.0123(9)	0.0046(9)
C(42)	4*e*	–0.3933(3)	0.3144(4)	0.4628(4)	0.071(2)	0.094(2)	0.099(2)	–0.035(2)	0.048(2)	–0.048(2)
C(43)	4*e*	–0.5140(4)	0.2792(5)	0.4629(4)	0.076(2)	0.127(3)	0.102(3)	–0.035(2)	0.054(2)	–0.044(2)
C(44)	4*e*	–0.5781(3)	0.1689(4)	0.3903(3)	0.054(1)	0.109(2)	0.069(2)	–0.023(2)	0.019(1)	–0.005(2)
C(45)	4*e*	–0.5222(3)	0.0898(4)	0.3194(4)	0.061(2)	0.080(2)	0.092(2)	–0.028(1)	0.016(1)	–0.016(2)
C(46)	4*e*	–0.4034(3)	0.1261(3)	0.3148(3)	0.059(1)	0.059(1)	0.076(2)	–0.009(1)	0.020(1)	–0.014(1)

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References

1. Burgess, V. A.; Easton, C. J.; Hay, M. P.; Steel, P. J.: Dimerization of glycine derivatives. Aust. J. Chem. **41** (1988) 701-710.
2. Burgess, V. A.; Easton, C. J.; Hay, M. P.: Selective reaction of glycine residues in hydrogen atom transfer from amino acid derivatives. J. Am. Chem. Soc. **111** (1989) 1047-1052.
3. Coghlan D. R.: Ph.D. Thesis, The University of Adelaide, 1994.
4. Coghlan, D. R.; Easton, C. J.; Tiekink, E. R. T.: Crystal structure of *meso*-dimethyl 2,3-dibenzamido-2,3-dimethylbutanedioate, $C_{22}H_{24}N_2O_6$. Z. Kristallogr. NCS **218** (2003) 359-360.
5. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
6. Sheldrick, G. M.: SHELXS-86. Program for the solution of crystal structure. University of Göttingen, Germany 1986.
7. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
8. Walker, N.; Stuart, D.: An empirical method for corrrecting diffractometer data for absorption effects. Acta Crystallogr. A**39** (1983) 158-166.
9. Johnson, C. K.: ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, 1976.