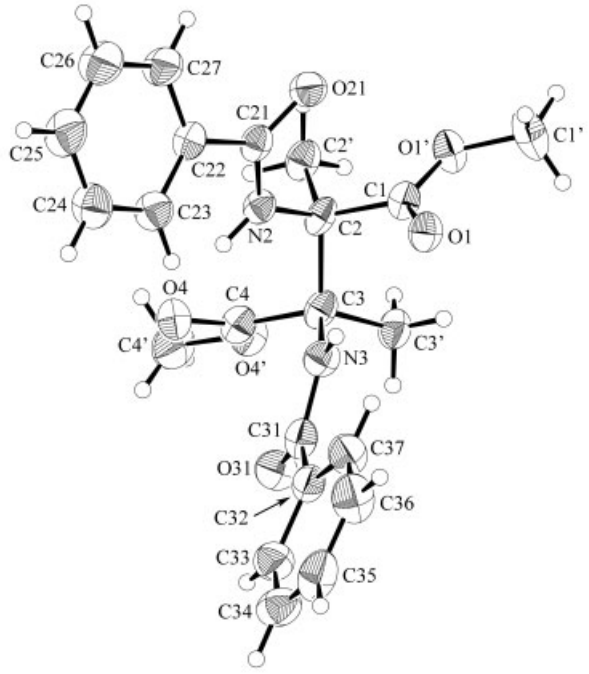
# Crystal structure of *meso*-dimethyl 2,3-dibenzamido-2,3-dimethyl-butanedioate, $C_{22}H_{24}N_2O_6$

D. R. Coghlan[I], C. J. Easton[II] and E. R. T. Tiekink*[,I,1]

[I] The University of Adelaide, Department of Chemistry, Australia 5005
[II] Australian National University, Research School of Chemistry, Canberra 0200, Australia

Received June 5, 2003, accepted and available on-line August 29, 2003; CCDC-No. 1267/1101




**Abstract**

$C_{22}H_{24}N_2O_6$, monoclinic, $P12_1/n1$ (No. 14), $a$ = 14.455(3) Å, $b$ = 10.239(2) Å, $c$ = 14.991(5) Å, $\beta$ = 101.01(2)°, $V$ = 2177.9 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.068, $wR_{ref}(F^2)$ = 0.244, $T$ = 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]. The isomers were separated [3] and analysed crystallographically to determine the structure of the particular isomer. Crystals of the meso isomer were obtained from the fractional crystallisation from an ethyl acetate/hexane solution (mp 451 K – 452.5 K).

**Discussion**

The molecule represents the *meso* isomer of $C_{22}H_{24}N_2O_6$ and the distance of the central C2—C3 bond is 1.603(8) Å, being equal to that reported for the *dl* isomer of 1.566(4) Å [4].

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless block, size 0.10 × 0.36 × 0.45 mm |
| Wavelength: | Cu $K_\alpha$ radiation (1.5418 Å) |
| $\mu$: | 7.67 cm$^{-1}$ |
| Diffractometer, scan mode: | Rigaku AFC6R, $\omega/2\theta$ |
| $2\theta_{max}$: | 120.2° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 5915, 2868 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2121 |
| $N(param)_{refined}$: | 274 |
| 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(2) | 4*e* | 0.6307 | 0.0110 | 0.2026 | 0.063 |
| H(3) | 4*e* | 0.5906 | 0.1982 | 0.2857 | 0.072 |
| H(1′1) | 4*e* | 0.9162 | 0.2608 | 0.4207 | 0.147 |
| H(1′2) | 4*e* | 0.9496 | 0.1813 | 0.5104 | 0.147 |
| H(1′3) | 4*e* | 0.8608 | 0.2718 | 0.5006 | 0.147 |
| H(2′1) | 4*e* | 0.7095 | –0.1843 | 0.3413 | 0.096 |
| H(2′2) | 4*e* | 0.7607 | –0.1151 | 0.4304 | 0.096 |
| H(2′3) | 4*e* | 0.8095 | –0.1225 | 0.3457 | 0.096 |
| H(3′1) | 4*e* | 0.6549 | 0.1370 | 0.4787 | 0.098 |
| H(3′2) | 4*e* | 0.6503 | –0.0136 | 0.4959 | 0.098 |
| H(3′3) | 4*e* | 0.5577 | 0.0699 | 0.4801 | 0.098 |
| H(4′1) | 4*e* | 0.4249 | –0.2643 | 0.3368 | 0.137 |
| H(4′2) | 4*e* | 0.4751 | –0.3273 | 0.4287 | 0.137 |
| H(4′3) | 4*e* | 0.5205 | –0.3389 | 0.3420 | 0.137 |
| H(23) | 4*e* | 0.5933 | 0.1006 | 0.0731 | 0.082 |
| H(24) | 4*e* | 0.5559 | 0.1001 | –0.0833 | 0.098 |
| H(25) | 4*e* | 0.6654 | 0.0421 | –0.1678 | 0.109 |
| H(26) | 4*e* | 0.8155 | –0.0137 | –0.0959 | 0.113 |
| H(27) | 4*e* | 0.8548 | –0.0148 | 0.0605 | 0.092 |
| H(33) | 4*e* | 0.2888 | 0.2300 | 0.2475 | 0.094 |
| H(34) | 4*e* | 0.2194 | 0.4135 | 0.1730 | 0.102 |
| H(35) | 4*e* | 0.3131 | 0.5765 | 0.1332 | 0.112 |
| H(36) | 4*e* | 0.4768 | 0.5604 | 0.1721 | 0.113 |
| H(37) | 4*e* | 0.5437 | 0.3794 | 0.2474 | 0.091 |

* Correspondence author (e-mail: chmtert@nus.edu.sg)
[1] Current address: The National University of Singapore, Department of Chemistry, Singapore 117543

**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* | 0.7452(3) | 0.2452(3) | 0.3383(2) | 0.076(3) | 0.045(2) | 0.062(2) | –0.005(2) | 0.011(2) | 0.001(2) |
| O(1') | 4*e* | 0.8358(3) | 0.1053(3) | 0.4312(2) | 0.073(3) | 0.059(2) | 0.060(2) | –0.010(2) | –0.006(2) | 0.004(2) |
| O(4') | 4*e* | 0.5362(3) | –0.1626(4) | 0.3991(2) | 0.084(3) | 0.056(2) | 0.070(2) | –0.013(2) | 0.022(2) | 0.003(2) |
| O(4) | 4*e* | 0.5221(3) | –0.1171(4) | 0.2512(2) | 0.071(3) | 0.069(2) | 0.062(2) | –0.009(2) | 0.005(2) | –0.007(2) |
| O(21) | 4*e* | 0.8370(3) | 0.0597(4) | 0.2229(2) | 0.063(3) | 0.073(2) | 0.055(2) | 0.009(2) | 0.011(2) | 0.001(2) |
| O(31) | 4*e* | 0.4151(3) | 0.0863(4) | 0.3354(3) | 0.074(3) | 0.069(3) | 0.087(3) | 0.001(2) | 0.015(2) | 0.008(2) |
| N(2) | 4*e* | 0.6875(3) | 0.0212(4) | 0.2318(2) | 0.060(3) | 0.059(3) | 0.039(2) | 0.003(2) | 0.011(2) | –0.000(2) |
| N(3) | 4*e* | 0.5580(4) | 0.1447(4) | 0.3114(3) | 0.067(4) | 0.051(2) | 0.062(3) | 0.006(2) | 0.011(2) | 0.004(2) |
| C(1) | 4*e* | 0.7639(4) | 0.1361(5) | 0.3653(3) | 0.071(4) | 0.045(3) | 0.045(3) | 0.002(3) | 0.008(2) | 0.002(2) |
| C(1') | 4*e* | 0.8957(5) | 0.2141(7) | 0.4690(5) | 0.093(5) | 0.086(5) | 0.099(5) | –0.028(4) | –0.022(4) | –0.003(4) |
| C(2') | 4*e* | 0.7503(4) | –0.1135(5) | 0.3653(4) | 0.088(4) | 0.045(3) | 0.057(3) | 0.007(3) | 0.010(3) | 0.001(2) |
| C(2) | 4*e* | 0.7045(4) | 0.0162(5) | 0.3306(3) | 0.083(4) | 0.043(3) | 0.039(2) | 0.006(2) | 0.016(2) | –0.001(2) |
| C(3') | 4*e* | 0.6182(4) | 0.0592(6) | 0.4635(3) | 0.078(4) | 0.068(3) | 0.050(3) | –0.008(3) | 0.016(3) | –0.009(3) |
| C(3) | 4*e* | 0.6050(4) | 0.0344(5) | 0.3615(3) | 0.080(4) | 0.047(3) | 0.045(3) | 0.004(3) | 0.016(2) | 0.001(2) |
| C(4') | 4*e* | 0.4848(5) | –0.2835(6) | 0.3745(5) | 0.104(6) | 0.055(4) | 0.123(6) | –0.021(3) | 0.042(4) | –0.006(4) |
| C(4) | 4*e* | 0.5462(4) | –0.0892(5) | 0.3298(3) | 0.074(4) | 0.056(3) | 0.050(3) | 0.006(3) | 0.011(3) | 0.004(2) |
| C(21) | 4*e* | 0.7553(5) | 0.0409(5) | 0.1841(3) | 0.072(4) | 0.042(3) | 0.048(3) | 0.008(3) | 0.005(3) | 0.000(2) |
| C(22) | 4*e* | 0.7281(4) | 0.0409(5) | 0.0837(3) | 0.073(4) | 0.049(3) | 0.047(3) | –0.002(3) | 0.014(3) | 0.001(2) |
| C(23) | 4*e* | 0.6385(5) | 0.0766(6) | 0.0395(4) | 0.076(5) | 0.074(4) | 0.055(3) | 0.005(3) | 0.013(3) | 0.004(3) |
| C(24) | 4*e* | 0.6163(5) | 0.0765(7) | –0.0542(4) | 0.093(5) | 0.096(5) | 0.052(3) | 0.002(4) | 0.003(3) | 0.007(3) |
| C(25) | 4*e* | 0.6813(6) | 0.0425(7) | –0.1047(4) | 0.113(6) | 0.114(6) | 0.046(3) | 0.006(5) | 0.015(4) | 0.000(3) |
| C(26) | 4*e* | 0.7706(6) | 0.0088(8) | –0.0617(4) | 0.101(6) | 0.133(6) | 0.055(4) | 0.005(5) | 0.031(4) | –0.005(4) |
| C(27) | 4*e* | 0.7941(5) | 0.0080(7) | 0.0320(4) | 0.078(5) | 0.100(5) | 0.054(3) | 0.008(4) | 0.018(3) | –0.004(3) |
| C(31) | 4*e* | 0.4638(5) | 0.1660(5) | 0.3038(4) | 0.078(5) | 0.051(3) | 0.056(3) | –0.002(3) | 0.007(3) | –0.005(2) |
| C(32) | 4*e* | 0.4249(4) | 0.2853(5) | 0.2552(3) | 0.070(5) | 0.055(3) | 0.058(3) | 0.002(3) | 0.007(3) | –0.007(2) |
| C(33) | 4*e* | 0.3265(5) | 0.2969(7) | 0.2323(4) | 0.073(5) | 0.087(5) | 0.072(4) | 0.009(3) | 0.009(3) | –0.002(3) |
| C(34) | 4*e* | 0.2847(5) | 0.4062(8) | 0.1874(4) | 0.087(5) | 0.099(5) | 0.070(4) | 0.027(4) | 0.014(3) | 0.004(4) |
| C(35) | 4*e* | 0.3406(7) | 0.5034(7) | 0.1643(4) | 0.137(7) | 0.066(4) | 0.068(4) | 0.022(5) | –0.003(4) | 0.000(3) |
| C(36) | 4*e* | 0.4389(6) | 0.4938(6) | 0.1872(5) | 0.111(7) | 0.064(4) | 0.098(5) | 0.002(4) | –0.006(4) | 0.010(4) |
| C(37) | 4*e* | 0.4784(5) | 0.3854(5) | 0.2321(4) | 0.077(5) | 0.055(3) | 0.089(4) | 0.003(3) | 0.000(3) | 0.003(3) |

*Acknowledgment.* The Australian Research Council is thanked for support.

## 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 *dl*-dimethyl 2,3-dibenzamido-2,3-dimethylbutanedioate, $C_{22}H_{24}N_2O_6$. Z. Kristallogr. NCS **218** (2003) 361-362.
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.