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Diethyl 2,3-Diacetamidosuccinate

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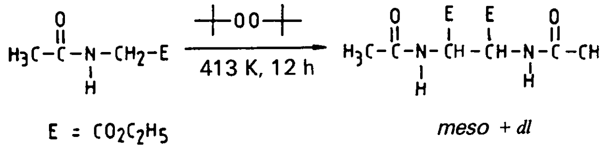
Abstract. $C_{12}H_{20}N_2O_6$, $M_r = 288.30$, monoclinic, $P2_1/n$, $a = 10{\cdot}815\,(3)$, $b = 9{\cdot}398\,(2)$, $c = 7{\cdot}687\,(3)$ Å, $\beta = 106{\cdot}01\,(2)°$, $V = 751{\cdot}0\,(4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}11$ cm^{-1}, $F(000) = 308$, $T = 291$ K, $R = 0{\cdot}061$ for 958 observed reflections. The compound has the *meso* configuration in its centrosymmetric conformation. All bond lengths, including the central C—C distance of 1·530 (5) Å, agree with standard values [Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19]. An intermolecular hydrogen bond involving the amide group is observed: N(7)—H···O(9) = 2·913 (5) Å [O(9) at $1{\cdot}5 - x$, $y - 0{\cdot}5$, $0{\cdot}5 - z$].

Experimental. The title compound is known (Obata & Niimura, 1977), but we have developed another synthetic route involving selective dehydrodimerization of the corresponding monomer, ethyl *N*-acetylglycine. A 10 *M* excess of the glycinate is heated with 1 *M* di-*tert*-butyl peroxide in a sealed tube at 413–423 K for 12 h. The succinate dimer is obtained in 60% yield as a 1:1 *meso*:*dl* mixture. Repeated crystallization (dichloromethane/diethyl ether) gives the pure *dl* isomer (m.p. 397 K) and the *meso* compound (m.p. 468 K) studied here.



Parallelepiped crystal with dimensions 0·23 × 0·25 × 0·30 mm. Lattice parameters refined using 15 reflections in the range $5 \le 2\theta \le 25°$. Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 1317 h, k, $\pm l$ independent reflections with $(\sin\theta)/\lambda \le 0{\cdot}595$ Å^{-1}; $0 \le h \le 12$, $0 \le k \le 11$, $-8 \le l \le 8$, 958 with $I \ge 2{\cdot}5\sigma(I)$. Standard reflection ($2\bar{4}1$) checked every 50 reflections: no significant deviation. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Five H atoms from difference Fourier synthesis, those of the ethyl group calculated (C—H = 1·08 Å). Anisotropic least-squares refinement (*SHELX*76, Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0{\cdot}00071F^2)$, $R = 0{\cdot}061$, $wR = 0{\cdot}074$ for 958 observed reflections. $S = 2{\cdot}39$. Final $(\Delta/\sigma)_{\max} = 0{\cdot}52$ [y of atom C(5)]. Maximum and minimum heights in final difference Fourier synthesis = 0·31 and −0·30 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
C1	0·9501 (3)	0·0597 (3)	0·4777 (4)	2·83 (4)
C2	0·9034 (3)	0·0913 (3)	0·6428 (4)	3·56 (4)
O3	0·8308 (3)	0·0174 (3)	0·6939 (4)	6·53 (4)
O4	0·9559 (3)	0·2075 (3)	0·7244 (4)	5·39 (4)
C5	0·9282 (5)	0·2461 (6)	0·8929 (7)	8·02 (5)
C6	1·0359 (6)	0·2920 (8)	1·0201 (7)	11·75 (6)
N7	0·8431 (2)	0·0158 (2)	0·3262 (3)	3·10 (3)
C8	0·7688 (3)	0·1129 (3)	0·2198 (4)	3·15 (4)
O9	0·7896 (2)	0·2410 (2)	0·2438 (3)	4·62 (4)
C10	0·6616 (3)	0·0568 (4)	0·0687 (5)	4·91 (5)

Table 2. *Bond distances* (Å) *and angles* (°)

C2—C1	1·519 (4)	N7—C1	1·457 (3)
C1—C1	1·530 (5)	O3—C2	1·193 (4)
O4—C2	1·309 (4)	C5—O4	1·453 (5)
C6—C5	1·367 (6)	C8—N7	1·336 (4)
O9—C8	1·229 (4)	C10—C8	1·493 (4)
N7—C1—C2	110·5 (2)	O3—C2—C1	124·2 (3)
O4—C2—C1	111·3 (3)	O4—C2—O3	124·5 (3)
C5—O4—C2	117·8 (3)	C6—C5—O4	111·9 (4)
C8—N7—C1	120·5 (2)	O9—C8—N7	121·5 (3)
C10—C8—N7	116·2 (3)	C10—C8—O9	122·2 (3)

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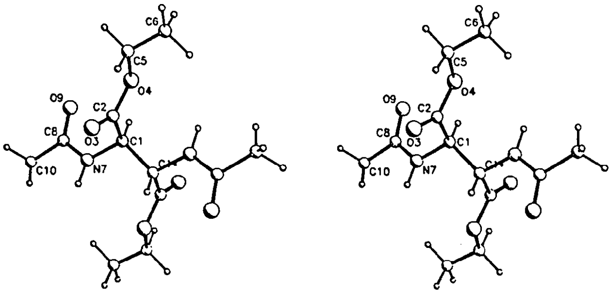

Fig. 1. Stereoscopic view of the molecule (program *PLUTO*, Motherwell & Clegg, 1978).

Table 1 gives atomic parameters,* Table 2 bond distances and angles. Fig. 1 shows a stereoscopic view of the molecule.

Related literature. This analysis is part of a study of the effects of *gem* electron-donor and -acceptor substitution on molecular deformation in dehydrodimers $(R_1R_2R_3)_2$. It was thought that the title compound could be a possible candidate for an electron deformation study but crystal quality was not sufficiently good. 1,1,2,2-Ethanetetracarbonitrile (Declercq, Tinant, Parfonry, Van Meerssche, Legrand & Lehmann, 1983), 2,3-bis(dimethylamino)-butanedinitrile (Parfonry, Declercq, Tinant, Van Meerssche & Schweiss, 1988), and 2,3-dimethyl-2,3-diphenyl-1,4-butanedinitrile (Tinant, Parfonry & Declercq, 1988) were chosen for deformation density analysis.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp.S1–S19.

Declercq, J.-P., Tinant, B., Parfonry, A., Van Meerssche, M., Legrand, E. & Lehmann, M. S. (1983). *Acta Cryst.* C39, 1401–1405.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. A program for plotting crystal and molecular structures. Univ. of Cambridge, England.

Obata, N. & Niimura, K. (1977). *J. Chem. Soc. Chem. Commun.* pp. 238–239.

Parfonry, A., Declercq, J.-P., Tinant, B., Van Meerssche, M. & Schweiss, P. (1988). *Acta Cryst.* B44, 435–440.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Tinant, B., Parfonry, A. & Declercq, J.-P. (1988). *Bull. Soc. Chim. Belg.* 97, 883–892.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52573 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4,4-Difluoro-1,3,5,7,8-pentamethyl-3a,4a-diaza-4-bora-*s*-indacene

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Abstract. $C_{14}H_{17}BF_2N_2$, $M_r = 262{\cdot}1$, orthorhombic, *Pnma*, $a = 11{\cdot}349$ (2), $b = 7{\cdot}191$ (2), $c = 16{\cdot}016$ (3) Å, $V = 1307{\cdot}1$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}332$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}927$ cm^{-1}, $F(000) = 552$, $T = 293$ K, final $R = 0{\cdot}039$ for 597 reflections with $I > 3\sigma(I)$. The molecule is located on a crystallographic mirror plane passing through all of the C, N and B atoms. The C(5)—C(12) bond distance [1·512 (6) Å] is somewhat longer than the average of the other four C—CH_3 distances [1·495 (3) Å], suggesting a crowding effect. There are no unusual intermolecular distances.

Experimental. The title compound was synthesized following the procedure of Treibs & Kreuzer (1968) and recrystallized from ethyl acetate. An orange needle-shaped crystal with approximate dimensions 0·6 × 0·2 × 0·2 mm was glued to a glass fiber and mounted on an Enraf–Nonius CAD-4 diffractometer with graphite crystal monochromator. Unit-cell parameters were determined from least-squares refinement of the setting angles of 25 reflections with $18 < 2\theta < 40°$ using Mo $K\alpha$ radiation. Systematic absences, $0kl$: $k + l = 2n + 1$, $hk0$: $h = 2n + 1$, indicating either space group *Pnma* or $Pn2_1a$. *Pnma* was chosen on the basis of intensity statistics and confirmed by successful determination of the struc-

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