program(s) used to solve structures: *NRCVAX SOLVER*; program(s) used to refine structures: *NRCVAX LSTSQ*; molecular graphics: *NRCVAX ORTEP*; software used to prepare material for publication: *NRCVAX TABLES*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: SZ1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Berthiaume, G. & Deslongchamps, P. (1995). *Bull. Soc. Chim. Fr.* **132**, 371–383.
Deslongchamps, P. & Roy, B. (1986). *Can. J. Chem.* **64**, 2068–2075.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Girard, S. & Deslongchamps, P. (1992). *Can. J. Chem.* **70**, 1265–1273.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

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# Tricoccin $S_{43}$

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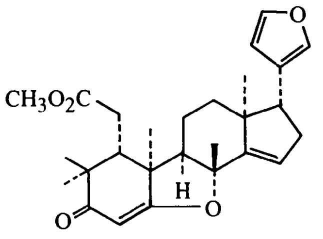
## Abstract

In methyl (1*S*,2*S*,8*S*,9*S*,10*S*,13*S*,17*S*)-17-(3-furyl)-2,2,8-trimethyl-3-oxo-6-oxa-7-norandrosta-4,14-dien-1-ylmethylcarboxylate, $C_{27}H_{34}O_5$, several C—C bonds and C—C—C angles deviate significantly from their expected values. The two six-membered rings, *A* and *C*, have slightly distorted sofa and boat conformations. Two of the five-membered rings, *B* and *D*, have half-chair and envelope conformations, and the terminal five-membered ring is nearly planar. The structure is stabilized by van der Waals interactions.

## Comment

The title compound, (I), was isolated by Epe & Mondon (Herz, Grisebach & Kirby, 1983) from *Cneorum tricoccin L.*, a shrub with hairless leaves, yellow blossoms and red fruits, native to coastal areas of the western Mediterranean. Crystals were grown from ethanol–acetone solution at room temperature.



(I)

Several C—C bonds and C—C—C angles involved in the fused-ring systems deviate by more than 3σ from their expected values, presumably as a result of the strain introduced by axial substitutions with bulky groups (Hall & Maslen, 1965; Gzella, Zaprutko, Wrezciono & Jaskolski, 1987; Sekar, Parthasarathy, Kundu & Barik, 1992, 1993). Ring *A* has a slightly distorted sofa conformation with a mean torsion angle of 23.5 (7)°. The ring puckering is pronounced near atom C5 and less so near C2. Ring *B* has a half-chair conformation and ring *C* has a slightly distorted boat conformation with a mean torsion angle of 37.2 (6)°. The puckering is high at atom C11 and low at C14. Ring *D* has an envelope conformation. Ring *E* is planar within the 4σ level. The structure is stabilized by van der Waals forces.

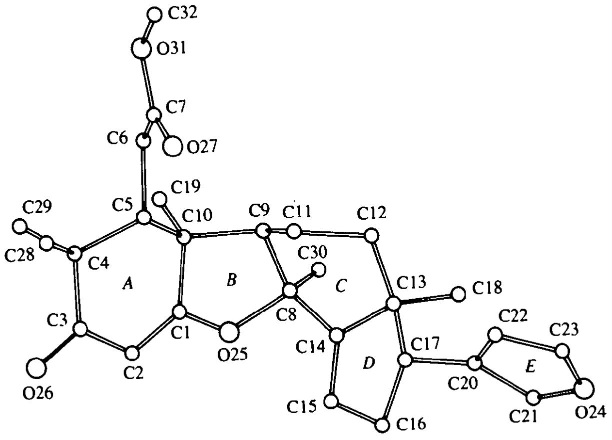


Fig. 1. A perspective view of the title molecule with the atom numbering and ring labels indicated.

## Experimental

*Crystal data*

$C_{27}H_{34}O_5$
$M_r = 438.56$
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å

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‡ Contribution No. 850.

Orthorhombic
$P2_12_12_1$
$a = 10.181\ (2)$ Å
$b = 14.635\ (2)$ Å
$c = 16.076\ (2)$ Å
$V = 2395.3\ (7)$ Å$^3$
$Z = 4$
$D_x = 1.216$ Mg m$^{-3}$

Cell parameters from 30 reflections
$\theta = 20$–$30°$
$\mu = 0.628$ mm$^{-1}$
$T = 292$ K
Needle
$0.25 \times 0.25 \times 0.20$ mm
Colourless

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$–$2\theta$ scans
Absorption correction: empirical ($\psi$ scan)
$T_{\min} = 0.959$, $T_{\max} = 0.988$
2518 measured reflections
2502 independent reflections

2212 observed reflections $[I \geq 3\sigma(I)]$
$\theta_{\max} = 70.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.064$
$wR = 0.078$
$S = 1.442$
2212 reflections
425 parameters
$w = 1/[\sigma^2(F) + 0.005237F^2]$

$(\Delta/\sigma)_{\max} = 0.06$
$\Delta\rho_{\max} = 0.441$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.367$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| C1 | −0.0104 (5) | 0.9572 (3) | 0.7041 (4) | 0.048 (2) |
| C2 | 0.0120 (5) | 1.0218 (3) | 0.6483 (4) | 0.054 (2) |
| C3 | −0.0305 (5) | 1.0154 (4) | 0.5638 (4) | 0.058 (2) |
| C4 | −0.1131 (6) | 0.9321 (4) | 0.5376 (4) | 0.058 (2) |
| C5 | −0.0836 (4) | 0.8482 (3) | 0.5936 (3) | 0.043 (2) |
| C6 | −0.1696 (5) | 0.7646 (3) | 0.5747 (3) | 0.053 (2) |
| C7 | −0.1138 (6) | 0.6979 (4) | 0.5130 (4) | 0.059 (2) |
| C8 | 0.0551 (5) | 0.8691 (4) | 0.8124 (3) | 0.055 (2) |
| C9 | −0.0001 (5) | 0.8033 (3) | 0.7441 (3) | 0.048 (2) |
| C10 | −0.0820 (4) | 0.8699 (3) | 0.6863 (3) | 0.046 (2) |
| C11 | 0.1102 (5) | 0.7500 (3) | 0.7009 (3) | 0.049 (2) |
| C12 | 0.1969 (5) | 0.7013 (3) | 0.7645 (4) | 0.055 (2) |
| C13 | 0.2701 (5) | 0.7699 (3) | 0.8198 (3) | 0.046 (2) |
| C14 | 0.2034 (5) | 0.8622 (4) | 0.8165 (3) | 0.047 (2) |
| C15 | 0.2877 (6) | 0.9308 (3) | 0.8084 (4) | 0.055 (2) |
| C16 | 0.4275 (5) | 0.8972 (4) | 0.8033 (4) | 0.056 (2) |
| C17 | 0.4088 (5) | 0.7950 (3) | 0.7823 (3) | 0.044 (2) |
| C18 | 0.2854 (6) | 0.7346 (4) | 0.9102 (4) | 0.068 (2) |
| C19 | −0.2246 (5) | 0.8787 (4) | 0.7253 (4) | 0.067 (2) |
| C20 | 0.5209 (5) | 0.7348 (3) | 0.8072 (3) | 0.045 (2) |
| C21 | 0.5983 (6) | 0.7431 (4) | 0.8812 (3) | 0.052 (2) |
| C22 | 0.5709 (7) | 0.6631 (4) | 0.7647 (4) | 0.077 (3) |
| C23 | 0.6771 (5) | 0.6268 (3) | 0.8046 (3) | 0.051 (2) |
| O24 | 0.6892 (6) | 0.6740 (4) | 0.8771 (4) | 0.131 (3) |
| O25 | 0.0261 (4) | 0.9617 (2) | 0.7836 (2) | 0.061 (1) |
| O26 | −0.0056 (5) | 1.0764 (3) | 0.5142 (3) | 0.096 (2) |
| O27 | −0.0041 (5) | 0.6952 (3) | 0.4863 (3) | 0.097 (2) |
| C28 | −0.0778 (8) | 0.9089 (4) | 0.4456 (4) | 0.084 (3) |
| C29 | −0.2578 (6) | 0.9664 (4) | 0.5386 (5) | 0.088 (3) |
| C30 | −0.0070 (7) | 0.8567 (6) | 0.8974 (4) | 0.087 (3) |
| O31 | −0.2043 (4) | 0.6356 (3) | 0.4933 (3) | 0.087 (2) |
| C32 | −0.1617 (8) | 0.5608 (5) | 0.4404 (6) | 0.096 (3) |

Table 2. *Bond lengths (Å), angles (°) and torsion angles (°) within each ring*

| | | | |
|---|---|---|---|
| C4—C5 | 1.552 (8) | C9—C10 | 1.585 (7) |
| C4—C28 | 1.560 (9) | C10—C19 | 1.588 (7) |
| C4—C29 | 1.558 (9) | C13—C17 | 1.580 (7) |
| C8—C9 | 1.565 (7) | C13—C18 | 1.551 (8) |
| C8—O25 | 1.462 (7) | O31—C32 | 1.453 (9) |
| C3—C4—C29 | 105.1 (5) | C14—C8—O25 | 106.1 (4) |
| C3—C4—C28 | 107.8 (5) | C9—C8—O25 | 106.0 (4) |
| C3—C4—C5 | 111.2 (5) | C9—C8—C14 | 110.4 (4) |
| C28—C4—C29 | 107.4 (5) | C9—C10—C19 | 107.5 (4) |
| C5—C4—C28 | 109.5 (5) | C1—C10—C19 | 107.5 (4) |
| O25—C8—C30 | 108.2 (5) | C14—C13—C17 | 100.4 (4) |
| C14—C8—C30 | 111.6 (4) | | |
| Ring *A* | | Ring *C* | |
| C1—C2—C3—C4 | 3.7 (8) | C8—C9—C11—C12 | 53.1 (5) |
| C2—C3—C4—C5 | −26.8 (7) | C9—C11—C12—C13 | −64.9 (5) |
| C3—C4—C5—C10 | 46.7 (6) | C11—C12—C13—C14 | 19.3 (6) |
| C4—C5—C10—C1 | −43.3 (6) | C12—C13—C14—C8 | 37.4 (6) |
| C5—C10—C1—C2 | 20.1 (7) | C13—C14—C8—C9 | −48.0 (6) |
| C10—C1—C2—C3 | 0.2 (9) | C14—C8—C9—C11 | 0.4 (6) |
| Ring *B* | | Ring *D* | |
| C8—C9—C10—C1 | 25.7 (4) | C13—C14—C15—C16 | 0.9 (7) |
| C9—C10—C1—O25 | −37.0 (5) | C14—C15—C16—C17 | 17.1 (6) |
| C10—C1—O25—C8 | 32.8 (5) | C15—C16—C17—C13 | −27.1 (5) |
| C1—O25—C8—C9 | −12.8 (5) | C16—C17—C13—C14 | 27.2 (5) |
| O25—C8—C9—C10 | −9.8 (5) | C17—C13—C14—C15 | −18.0 (6) |
| Ring *E* | | | |
| C20—C21—O24—C23 | 2.8 (7) | | |
| C21—O24—C23—C22 | −4.0 (7) | | |
| O24—C23—C22—C20 | 3.7 (7) | | |
| C23—C22—C20—C21 | −2.0 (7) | | |
| C22—C20—C21—O24 | −0.5 (6) | | |

Data collection: Enraf–Nonius CAD-4 diffractometer software. Program used to solve structure: *SHELX*86 (Sheldrick, 1990). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Program used to calculate the molecular parameters: *PARST* (Nardelli, 1983). Program used to draw molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Refinement was by full-matrix least-squares methods. The H atoms were refined isotropically taking the starting values of the displacement factors to be those of the heavy atoms to which they were covalently linked.

Thanks are due to the Council of Scientific and Industrial Research, India, for the award of a Senior Research Fellowship to KS.

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data, H-atom coordinates and complete geometry, including bond distances and angles involving H atoms, have been deposited with the IUCr (Reference: VJ1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Gzella, A., Zaprutko, L., Wrezciono, U. & Jaskolski, M. (1987). *Acta Cryst.* C**43**, 759–762.

Hall, S. R. & Maslen, E. N. (1965). *Acta Cryst.* **18**, 265–279.

Herz, W., Grisebach, H. & Kirby, G. W. (1983). *Progress in the Chemistry of Organic Natural Products*, Vol. 44, pp. 101–187. Vienna: Springer.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. University of Cambridge, England.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Sekar, K., Parthasarathy, S., Kundu, A. B. & Barik, B. R. (1992). *Acta Cryst.* C**48**, 2251–2253.
Sekar, K., Parthasarathy, S., Kundu, A. B. & Barik, B. R. (1993). *Acta Cryst.* C**49**, 616–618.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* University of Cambridge, England.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

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# *N*-Acetyliminodiacetate Dimethyl Ester

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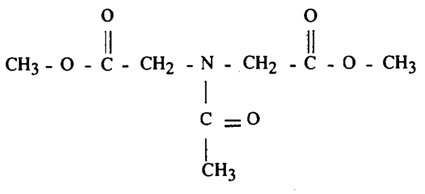
## Abstract

*N*-Acetyliminodiacetate dimethyl ester, $C_8H_{13}NO_5$, was prepared for the synthesis of 3,4-diphenylpyrrole in a multi-step synthetic route to a model porphyrin. The technique used in this preparation yielded colorless crystals of high purity. Site geometry around the central N atom is nearly trigonal planar.

## Comment

In the search for synthetic routes for porphyrins, the title compound, (I) (Fig. 1), was synthesized. The crystal structure determination was undertaken to verify the synthetic procedure and to ascertain the geometry around the N atom.



$$\mathrm{CH_3-O-\overset{\displaystyle O}{\overset{\|}{C}}-CH_2-\underset{\underset{\displaystyle CH_3}{|}}{\underset{\displaystyle C=O}{\underset{|}{N}}}-CH_2-\overset{\displaystyle O}{\overset{\|}{C}}-O-CH_3}$$

(I)

A unique feature of the title structure is the trigonal planar site geometry around the central N atom. The sum of the three angles around N(1) is 360° and the mean deviation of the plane formed by atoms N(1), C(1), C(4) and C(7) is 0.0080 Å [deviations with respect to the other atoms are N(1) −0.0160, C(1) 0.0050, C(4) 0.0050, C(7) 0.0060 Å]. Bond lengths N(1)—C(1) and N(1)—C(4) of the diester portions of the structure are comparable to those found in dithiocarbamates, 1.460 (5), 1.467 (6) and 1.475 (8), 1.477 (8) Å, respectively (Heinemann, Dölling & Hartung, 1992), and in *N*-methylnitrilotriacetamide, 1.448 (4), 1.466 (3) and 1.477 (4) Å (Skrzypczak-Jankun & Smith, 1994). The N—C bond of the amide linkage, N(1)—C(7), is shorter by 0.1 Å and is similar to values found in linuron, 1.361 (6) and 1.395 (7) Å (Cadiergue, Pèpe, Astier, Boistelle & Fiard, 1993), and in *N*-methylnitrilotriacetamide, 1.307 (3), 1.318 (4) and 1.321 (4) Å (Skrzypczak-Jankun & Smith, 1994). This shorter N(1)—C(7) bond length indicates some double-bond character which is characteristic of zwitterion formation in N(1)—C(7)—O(5).

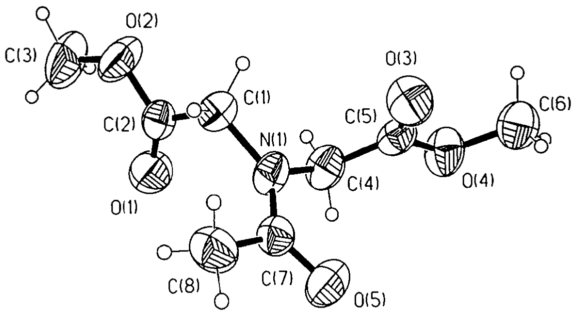


Fig. 1. View of $C_8H_{13}NO_5$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

## Experimental

Iminodiacetate dimethyl ester hydrochloride was isolated from a refluxing solution of iminodiacetic acid (Jongkees, 1907) which had been saturated with hydrogen chloride gas. Conversion to the *N*-acetyliminodiacetate dimethyl ester was achieved by neutralizing 10 g of the ester hydrochloride with 15 ml of 3 M ammonium carbonate followed by the addition of 8 ml of acetic anhydride and extraction with chloroform. The solution was evaporated to give a white solid. The resulting material was washed with hexane and recrystallized from acetone to give clear colorless crystals of the title compound, yield 60.0% (Prayzner, 1994).

*Crystal data*

$C_8H_{13}NO_5$
$M_r = 203.2$
Monoclinic
$P2_1/c$
$a = 9.411$ (2) Å
$b = 14.457$ (3) Å
$c = 7.853$ (2) Å
$\beta = 105.95$ (3)°
$V = 1027.2$ (4) Å$^3$
$Z = 4$
$D_x = 1.314$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.0$–$12.5°$
$\mu = 0.110$ mm$^{-1}$
$T = 297$ K
Wedge
0.40 × 0.35 × 0.20 mm
Colorless

*Data collection*

Siemens *R*3*m*/*V* diffractometer

$R_{\rm int} = 0.022$
$\theta_{\rm max} = 23.5°$