

Table 1. Atom coordinates and *e.s.d.*'s ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ )
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
C(1)	5633 (10)	4850 (10)	5103 (5)	455
C(2)	7183 (11)	5070 (10)	3784 (6)	488
C(3)	8754 (10)	7870 (9)	3987 (5)	446
C(4)	6813 (10)	8918 (9)	4485 (5)	443
C(5)	5250 (10)	7261 (9)	5494 (5)	458
C(6)	6520 (10)	7572 (9)	7217 (5)	424
C(7)	7472 (11)	5607 (9)	6693 (6)	489
C(8)	11015 (11)	11349 (10)	8290 (6)	461
C(9)	12252 (11)	13297 (10)	9744 (6)	516
C(10)	9979 (12)	12960 (10)	10599 (6)	543
C(11)	7632 (10)	10605 (9)	9687 (6)	472
N(1)	8317	9851	8321	431
O(1)	4815 (9)	8627 (8)	3165 (5)	546
O(2)	9611 (8)	8629 (9)	2630 (5)	582
O(3)	9190 (10)	5060 (9)	7248 (5)	656
O(4)	12120 (9)	11019 (9)	7238 (5)	582
O(5)	5437 (9)	9510 (8)	10037 (5)	569

Table 2. Bond lengths ( $\text{\AA}$ ) with *e.s.d.*'s

C(1)–C(2)	1.530 (6)	C(7)–O(3)	1.193 (5)
C(2)–C(3)	1.529 (6)	C(6)–N(1)	1.448 (5)
C(3)–C(4)	1.521 (5)	N(1)–C(8)	1.391 (5)
C(3)–O(2)	1.430 (5)	N(1)–C(11)	1.390 (5)
C(4)–C(5)	1.525 (5)	C(8)–C(9)	1.489 (5)
C(4)–O(1)	1.440 (4)	C(8)–O(4)	1.216 (5)
C(5)–C(6)	1.560 (5)	C(9)–C(10)	1.502 (7)
C(5)–C(1)	1.555 (6)	C(10)–C(11)	1.501 (6)
C(1)–C(7)	1.522 (5)	C(11)–O(5)	1.221 (5)
C(6)–C(7)	1.520 (6)		

**Related literature.** This structure is one of a series of  $\beta$ -lactam analogues. Previous structures of the series are listed in Sheldrick, Akrigg, Page & Agathocleous (1985).

We thank Francis Keenan for taking a series of X-ray photographs and the University of Leeds Computing Service for the provision of computing facilities.

#### References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting crystal and molecular structures. Univ. of Cambridge, England.
- SHAKKED, Z. & RABINOVICH, D. (1977). Proc. Fourth Eur. Crystallogr. Meet., Oxford, pp. 142–143.
- SHELDRIK, B., AKRIGG, D., PAGE, M. I. & AGATHOCLEOUS, D. (1985). *Acta Cryst.* **C41**, 942–944.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

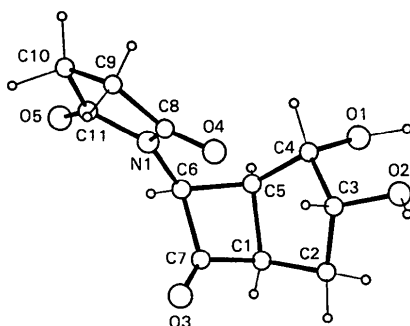


Fig. 1. Diagram of the molecule showing the numbering scheme.

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## Structures of Carbocyclic Analogues of Penicillin. 3. *N*-(Bicyclo[3.2.0]hept-2-ene-6-spiro-2'-dioxolane-7-yl)phthalimide

BY B. SHELDRIK AND D. AKRIGG

*Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England*

AND M. I. PAGE AND G. COX

*Department of Chemical Sciences, The Polytechnic, Queensgate, Huddersfield HD1 3DH, England.*

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**Abstract.**  $C_{17}H_{15}NO_4$ ,  $M_r = 297.31$ , triclinic,  $P\bar{1}$ ,  $a = 7.562$  (2),  $b = 8.663$  (2),  $c = 10.979$  (2)  $\text{\AA}$ ,  $\alpha = 102.14$  (1),  $\beta = 96.86$  (3),  $\gamma = 92.51$  (3) $^\circ$ ,  $V = 696.35$   $\text{\AA}^3$ ,  $Z = 2$ ,  $D_x = 1.418$   $\text{Mg m}^{-3}$ ,  $\lambda(\text{Cu K}\alpha) =$

1.5418  $\text{\AA}$ ,  $\mu = 0.74$   $\text{mm}^{-1}$ ,  $F(000) = 312$ ,  $T = 293$  K,  $R = 0.056$  for 2464 unique observed reflections. The determination confirms the chemical structure. The four-membered ring is twisted by  $15.7$  (1) $^\circ$  and the

least-squares best plane through it makes an angle of 115.5(5)° with the pentene ring. Bond lengths and angles are normal.

**Experimental.** Material prepared by M. I. Page and G. Cox and crystallized from ethanol. Tabular crystal 0.20 × 0.15 × 0.40 mm. Enraf-Nonius CAD-4F diffractometer. No correction for absorption.  $2\theta_{\max} = 140^\circ$ ,  $+/-h,k,l$ ; 4714 reflections measured and 567 classed as unobserved ( $I < 0$ ). Check reflection 206: average count = 884, calculated  $\sigma$  (of the distribution) = 30 (3.4%). Cell dimensions from  $\theta$  measurements of 20 reflections ( $16 < \theta < 35^\circ$ ). Data merged using *SHELX76* (Sheldrick, 1976) giving 2476 unique reflections, index range  $h +/- 9$ ,  $k +/- 10$ ,  $l/13$ ; merging  $R_{\text{int}} = 0.021$ . *SHELX76* used to solve structure, by direct methods. Least-squares refinement; positional parameters of all atoms and anisotropic thermal vibration parameters for non-H atoms refined;  $U_{\text{iso}}$  for H fixed at value of  $U_{\text{eq}}$  of bonded atom;  $\sum w(\Delta F)^2$  minimized with  $w = 1/[\sigma^2(F) + 0.0114F^2]$ . 12

reflections thought to suffer from extinction were omitted from the refinement. H atoms from difference Fourier syntheses. In final cycle max.  $\Delta/\sigma = 0.040$ , average = 0.005.  $\Delta\rho$  in final difference Fourier map within +0.14 and -0.27 e Å<sup>-3</sup>. Scattering factors from *International Tables for X-ray Crystallography* (1974).  $R = 0.056$ ,  $wR = 0.103$  for 2464 observed reflections [ $F_o > 3\sigma(F_o)$ ].

Atom parameters are given in Table 1,\* with bond distances in Table 2. Figs. 1 and 2 drawn with *PLUTO78* (Motherwell & Clegg, 1978) show the molecule numbering scheme and a stereoview of the packing.

**Related literature.** This structure is one of a series of  $\beta$ -lactam analogues. Previous structures of the series are listed in Sheldrick, Akrigg, Page & Agathocleous (1985) and Sheldrick, Akrigg, Page & Cox (1987).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond angles, torsion angles and best planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43472 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atom coordinates ( $\times 10^4$ ) with e.s.d.'s and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
O(1)	4004 (2)	-1095 (2)	7605 (2)	755
O(2)	-1584 (2)	531 (2)	6844 (1)	571
O(3)	146 (2)	2648 (2)	5093 (1)	554
O(4)	833 (2)	3470 (1)	7215 (1)	496
N(1)	1440 (2)	165 (2)	7180 (1)	465
C(1)	3368 (3)	3127 (2)	5951 (2)	541
C(2)	4249 (3)	4622 (3)	6852 (2)	639
C(3)	4975 (3)	4003 (3)	7977 (2)	628
C(4)	4832 (3)	2441 (2)	7785 (2)	570
C(5)	4031 (2)	1676 (2)	6474 (2)	521
C(6)	2071 (2)	965 (2)	6255 (1)	465
C(7)	1433 (2)	2616 (2)	6127 (1)	456
C(8)	2469 (2)	-839 (2)	7749 (2)	505
C(9)	-350 (2)	-30 (2)	7356 (1)	466
C(10)	-392 (3)	-1084 (2)	8268 (2)	487
C(11)	-1791 (3)	-1559 (2)	8838 (2)	595
C(12)	-1377 (4)	-2501 (2)	9711 (2)	664
C(13)	328 (4)	-2947 (2)	9964 (2)	675
C(14)	1721 (3)	-2506 (2)	9357 (2)	601
C(15)	1306 (2)	-1546 (2)	8516 (2)	503
C(16)	-1366 (3)	3388 (2)	5549 (2)	586
C(17)	-618 (3)	4335 (3)	6832 (2)	609

Table 2. Bond lengths ( $\text{\AA}$ )

C(1)—C(2)	1.525 (3)	C(16)—C(17)	1.504 (3)
C(2)—C(3)	1.502 (3)	N(1)—C(8)	1.387 (2)
C(3)—C(4)	1.322 (3)	N(1)—C(9)	1.398 (2)
C(4)—C(5)	1.494 (3)	C(8)—O(1)	1.213 (2)
C(5)—C(1)	1.566 (2)	C(8)—C(15)	1.484 (3)
C(5)—C(6)	1.551 (2)	C(9)—O(2)	1.205 (2)
C(6)—C(7)	1.558 (2)	C(9)—C(10)	1.492 (2)
C(6)—N(1)	1.457 (2)	C(10)—C(15)	1.375 (3)
C(7)—C(1)	1.557 (2)	C(10)—C(11)	1.382 (3)
C(7)—O(3)	1.410 (2)	C(11)—C(12)	1.400 (3)
C(7)—O(4)	1.403 (2)	C(12)—C(13)	1.376 (4)
O(3)—C(16)	1.427 (3)	C(13)—C(14)	1.392 (3)
O(4)—C(17)	1.427 (2)	C(14)—C(15)	1.386 (2)

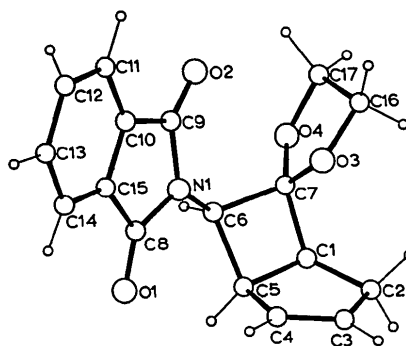


Fig. 1. Diagram of the molecule showing the numbering scheme.

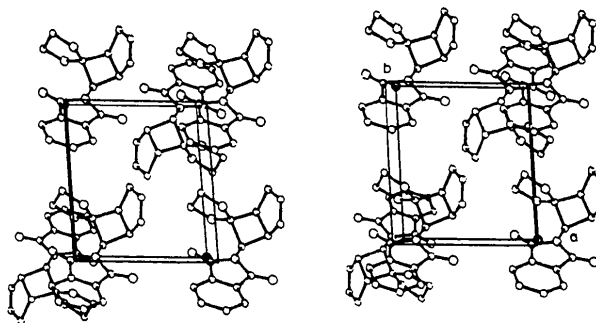


Fig. 2. Stereoscopic view along the c axis showing the cell packing.

We thank the University of Leeds Computing Service for the provision of facilities.

#### References

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting crystal and molecular structures. Univ. of Cambridge, England.

SHELDRIK, B., AKRIGG, D., PAGE, M. I. & AGATHOCLEOUS, D. (1985). *Acta Cryst.* **C41**, 942–944.

SHELDRIK, B., AKRIGG, D., PAGE, M. I. & COX, G. (1987). *Acta Cryst.* **C43**, 594–595.

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

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## The Structure of a Condensation Product of Isobutylidenecyanoacetic Acid Ester with Cyclohexanone

BY YOSHIHARU NAKANO,\* MASAOKI YATSU AND MINORU IGARASHI

*Department of Chemistry, Ibaraki University, Bunkyo 2-1-1, Mito 310, Japan*

AND SHOICHI SATO

*The Institute for Solid State Physics, The University of Tokyo, Roppongi 7-22-1, Tokyo 106, Japan*

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**Abstract.** 4-Ethyl-2-oxo-1-oxaspiro[5.5]undec-3-ene-3-carbonitrile,  $C_{13}H_{17}NO_2$ ,  $M_r = 219.3$ , orthorhombic,  $Pnca$ ,  $a = 17.454$  (3),  $b = 17.650$  (4),  $c = 7.882$  (1) Å,  $V = 2428.1$  (7) Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.20$ ,  $D_x = 1.18$  g cm<sup>-3</sup>,  $\lambda$ (Mo  $K\alpha$ , graphite monochromated) = 0.71069 Å,  $\mu = 0.871$  cm<sup>-1</sup>,  $F(000) = 944$ , room temperature, final  $R(F) = 0.065$  for 971 reflections. The molecular structure of a condensation product of isobutylidenecyanoacetic acid ester with cyclohexanone was determined by the X-ray diffraction method and the product confirmed as a spiro compound. There are no unusual bond angles or distances.

**Experimental.** The title compound was prepared as described earlier (Igarashi, Nakano & Yatsu, 1984).  $D_m$  was measured by flotation in aqueous potassium iodide. The intensity data were collected on an automated Rigaku AFC-3 four-circle diffractometer at the Institute for Solid State Physics. A specimen with approximate dimensions 0.1 × 0.2 × 0.2 mm (from ethanolic solution) was used. Reflections within the range  $2\theta < 55^\circ$  were collected by the  $\theta$ - $2\theta$  scan technique with a scan speed of 4° min<sup>-1</sup> in  $2\theta$  and scan width  $\Delta\omega$  (°) = 1.1 + 0.5 tan  $\theta$ .  $h0 \rightarrow 22$ ,  $k0 \rightarrow 22$ ,  $l0 \rightarrow 10$ . In total 1833 unique reflections were collected, 971 reflections with  $|F_o| > 2.5\sigma(|F_o|)$  considered as observed. No corrections for absorption or extinction. Three standard reflections showed <1.3% variation.

Cell dimensions determined by a least-squares method from setting angles of 38 accurately centered reflections ( $13 < \theta < 20^\circ$ ). The structure was determined by direct-methods *MULTAN* (Main, Woolfson & Germain, 1971). An *E* map computed from the phase set

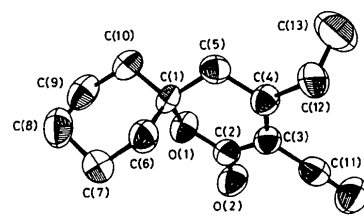


Fig. 1. Perspective view and numbering scheme of the spiro compound.

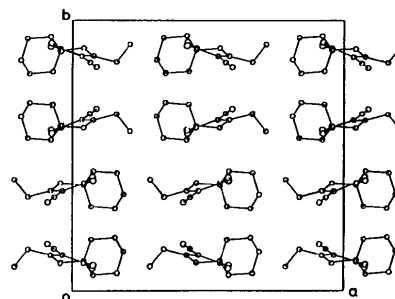


Fig. 2. The arrangement of the compound in the unit cell viewed along *c*.

\* To whom correspondence should be addressed.