

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

O(1)—C(2)	1.366 (4)	C(8)—O(20)	1.232 (6)
O(1)—C(10)	1.439 (4)	C(9)—C(10)	1.518 (6)
C(2)—C(3)	1.374 (6)	C(9)—O(21)	1.427 (5)
C(2)—C(7)	1.391 (6)	C(10)—C(11)	1.494 (5)
C(3)—C(4)	1.379 (5)	C(11)—C(12)	1.359 (5)
C(4)—C(5)	1.393 (6)	C(11)—C(16)	1.389 (6)
C(4)—O(17)	1.354 (5)	C(12)—C(13)	1.363 (6)
C(5)—C(6)	1.381 (7)	C(13)—C(14)	1.344 (8)
C(5)—C(18)	1.502 (5)	C(14)—C(15)	1.363 (7)
C(6)—C(7)	1.418 (5)	C(15)—C(16)	1.378 (6)
C(6)—O(19)	1.358 (5)	O(21)—C(22)	1.344 (5)
C(7)—C(8)	1.442 (6)	C(22)—O(23)	1.189 (5)
C(8)—C(9)	1.502 (6)	C(22)—C(24)	1.477 (7)
C(2)—O(1)—C(10)	115.7 (3)	C(9)—C(8)—O(20)	122.3 (4)
O(1)—C(2)—C(3)	116.4 (4)	C(8)—C(9)—C(10)	108.9 (3)
O(1)—C(2)—C(7)	121.3 (4)	C(8)—C(9)—O(21)	111.2 (3)
C(3)—C(2)—C(7)	122.3 (3)	C(10)—C(9)—O(21)	109.7 (3)
C(2)—C(3)—C(4)	117.5 (4)	O(1)—C(10)—C(9)	106.3 (3)
C(3)—C(4)—C(5)	124.1 (4)	O(1)—C(10)—C(11)	107.9 (3)
C(3)—C(4)—O(17)	120.0 (4)	C(9)—C(10)—C(11)	116.0 (3)
C(5)—C(4)—O(17)	116.0 (3)	C(10)—C(11)—C(12)	122.3 (4)
C(4)—C(5)—C(6)	116.4 (3)	C(10)—C(11)—C(16)	118.8 (3)
C(4)—C(5)—C(18)	121.7 (4)	C(12)—C(11)—C(16)	118.8 (4)
C(6)—C(5)—C(18)	122.0 (4)	C(11)—C(12)—C(13)	121.1 (4)
C(5)—C(6)—C(7)	122.1 (4)	C(12)—C(13)—C(14)	120.8 (4)
C(5)—C(6)—O(19)	118.8 (4)	C(13)—C(14)—C(15)	119.4 (4)
C(7)—C(6)—O(19)	119.1 (4)	C(14)—C(15)—C(16)	121.0 (4)
C(2)—C(7)—C(6)	117.5 (4)	C(11)—C(16)—C(15)	118.9 (4)
C(2)—C(7)—C(8)	120.7 (3)	C(9)—O(21)—C(22)	117.2 (3)
C(6)—C(7)—C(8)	121.6 (4)	O(21)—C(22)—O(23)	122.4 (4)
C(7)—C(8)—C(9)	113.3 (4)	O(21)—C(22)—C(24)	111.7 (3)
C(7)—C(8)—O(20)	124.3 (4)	O(23)—C(22)—C(24)	125.9 (4)

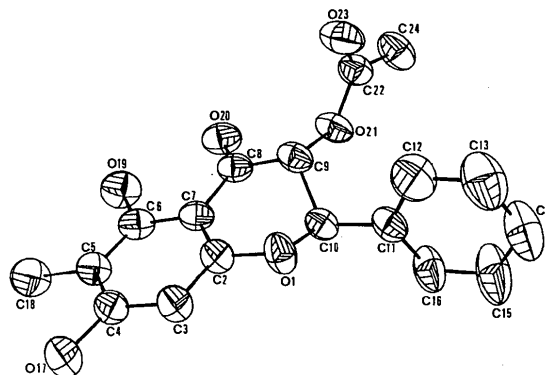


Fig. 1. ORTEP drawing of the title compound with 50% probability ellipsoids.

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*Acta Cryst.* (1990). **C46**, 720–722

## Structure of 6-Azabicyclo[3.2.0]heptan-7-one

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(Received 16 June 1989; accepted 16 October 1989)

**Abstract.**  $C_6H_9NO$ ,  $M_r = 111.15$ , monoclinic,  $P2_1/c$ ,  $a = 10.778$  (3),  $b = 6.147$  (2),  $c = 9.673$  (3) Å,  $\beta = 109.18$  (2)°,  $V = 605.3$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.22$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å (graphite monochromator),  $\mu = 1.02$  cm<sup>-1</sup>,  $F(000) = 240$ ,  $T = 298$  K. Final  $R = 0.050$  for 785 observed reflections

with  $I > 2\sigma(I)$ . The four-membered lactam ring is planar, and with the five-membered ring adopts a nearly ideal envelope conformation. The shared C—C bond is significantly longer than the other C—C bonds in the molecule.  $2_1$ -related molecules are connected by N—H...O bridges forming chains in the [010] direction. The structure forms channels running along the [001] direction, which probably

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allow water molecules to penetrate into the crystal. This may explain the instability of the crystals in the open air.

**Experimental.** Colorless prisms of the title compound were grown from cyclohexane. Crystals were hygroscopic, unstable in the open air and decomposed when an attempt to cut them or fix them in a glass capillary was made. Therefore, a crystal ( $0.9 \times 0.35 \times 0.20$  mm) was quickly plunged into a xylol solution of Canadian balsam and so sealed by a thin film against air moisture, for measurement with an Enraf-Nonius CAD-4 diffractometer. Nevertheless, the intensity of two standard reflections ( $\bar{4}2\bar{5}$ ,  $\bar{2}\bar{3}\bar{5}$ ) monitored every 1 h dropped monotonically. Four corrections of the orientation matrix (monitored every 180 reflections on  $\bar{4}3\bar{4}$ ,  $\bar{2}\bar{2}\bar{5}$  and  $\bar{5}\bar{3}\bar{2}$  reflections) kept the decay to a max. of 17% during 33 h of X-ray exposure time.

The lattice parameters were determined by least-squares fit of 25 reflections ( $9 < 2\theta < 18^\circ$ ),  $\theta$ - $2\theta$  scan ( $3 < 2\theta < 50^\circ$ ) with variable scan rate (min.  $0.7$ , max.  $10^\circ \text{ min}^{-1}$ ) used, max. scan time 120 s. 1319 reflections measured ( $-12 \leq h \leq 12$ ,  $-7 \leq k \leq 0$ ,  $0 \leq l \leq 11$ ), 1070 unique, 785 with  $I > 2\sigma(I)$  were treated as observed. Corrections were made for Lorentz and polarization effects, but not for absorption.

The structure was solved by a straightforward application of *MULTAN82* (Main *et al.*, 1982). All H atoms were located by difference Fourier syntheses. Full-matrix least-squares refinements, non-H atoms anisotropically, H atoms isotropically, gave final  $R = 0.050$  and  $wR = 0.062$  with  $w^{-1} = \sigma^2(F) + 0.0025F^2$ ,  $\sigma(F)$  from counting statistics. The largest peak in the final  $\Delta F$  map was  $0.287 \text{ e } \text{Å}^{-3}$ . Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Calculations were performed using the Enraf-Nonius (1979) *Structure Determination Package* with a PDP 11/34 computer.

The final atomic parameters are given in Table 1,\* selected bond lengths and bond angles are summarized in Table 2. A view of a molecule with atomic numbering is shown in Fig. 1, the stereoplot of the molecular conformation can be seen in Fig. 2.

**Related literature.** The structure of the title compound has been investigated as part of a systematic study of the correlation between stereochemistry and reactivity, by the anionic polymerization of lactams (Šebenda, 1986). Synthesis of the compound was

\* 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 52344 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement factors ( $\times 10^4$ ) for non-H atoms, with e.s.d.'s in parentheses

$$U_{eq} = 1/(6\pi^2) \sum_i \beta_i a_i^2$$

	x	y	z	$U_{eq} (\text{Å}^2)$
O2	3878 (1)	-1440 (2)	6533 (1)	632 (12)
N1	4114 (1)	2129 (3)	5828 (2)	523 (12)
C2	3669 (2)	89 (3)	5681 (2)	442 (10)
C3	2784 (2)	463 (3)	4110 (2)	446 (12)
C4	3320 (2)	2836 (3)	4356 (2)	512 (15)
C5	2161 (2)	4256 (3)	4295 (2)	632 (16)
C6	1193 (2)	2735 (3)	4646 (2)	624 (16)
C7	1329 (2)	617 (3)	3902 (2)	542 (13)

Table 2. Bond distances (Å) and bond angles ( $^\circ$ ), with e.s.d.'s in parentheses

C2—N1	1.333 (2)	C3—C4—C5	105.9 (1)
C2—O2	1.221 (2)	C4—C5—C6	104.8 (1)
C2—C3	1.523 (2)	C5—C6—C7	104.0 (1)
C3—C4	1.558 (2)	C6—C7—C3	104.3 (1)
C3—C7	1.517 (2)	C7—C3—C4	106.1 (1)
C4—N1	1.465 (2)	C3—C2—N1	92.8 (1)
C4—C5	1.510 (2)	C2—N1—C4	95.8 (1)
C5—C6	1.520 (2)	N1—C4—C3	86.5 (1)
C6—C7	1.518 (2)	C4—C3—C2	84.8 (1)
O2—N1*	2.889 (1)	N1—C2—O2	132.2 (1)
O2—H1*	2.068 (16)	C3—C2—O2	134.9 (1)
N1—H1	0.84 (2)	O1—H1*—N1*	165.5 (2)

\*  $2_1$ -related atoms.

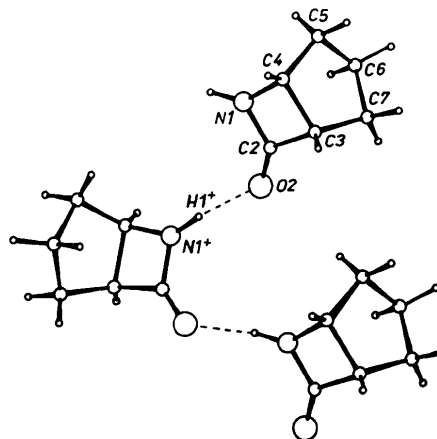


Fig. 1. View of three molecules connected by hydrogen bonds, with the numbering scheme.  $2_1$ -related atoms are denoted by \*.

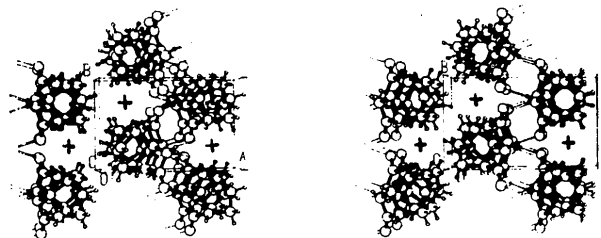


Fig. 2. Stereoview of the molecular packing down  $c$  (range in  $c$  direction:  $-0.5$  to  $1.5$ ). N—H...O bonds are indicated by thin lines, the possible water molecule 'channels' by +.

described by Graf, Lohaus, Börner, Schmidt & Bestian (1962).

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*Acta Cryst.* (1990). **C46**, 722–723

## Structure of a 2-Aza-6,7-benzotricyclo[6.2.1.0<sup>1,5</sup>]undeca-4,6,9-trien-3-one

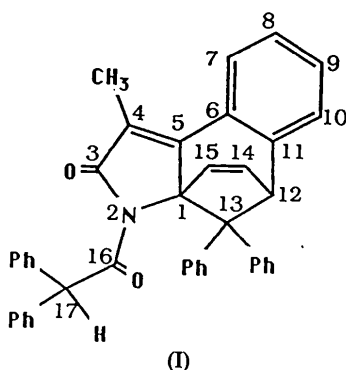
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(Received 19 November 1988; accepted 1 June 1989)

**Abstract.** (1*RS*,8*RS*)-4-Methyl-11,11-diphenyl-2-diphenylacetyl-2-aza-6,7-benzotricyclo[6.2.1.0<sup>1,5</sup>]undeca-4,6,9-trien-3-one.  $C_{41}H_{31}NO_2$ ,  $M_r = 569.71$ , monoclinic,  $P2_1/n$ ,  $a = 14.396$  (4),  $b = 10.375$  (3),  $c = 19.702$  (6) Å,  $\beta = 94.87$  (2)°,  $V = 2932$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.290$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.07$  mm<sup>-1</sup>,  $F(000) = 1200$ ,  $T = 292$  K, final  $R = 0.044$  for 2906 independent observed reflections and 438 least-squares parameters. The title compound was the product of a photolysis reaction, the structure of which could not be elucidated *via* standard spectroscopic techniques.

**Experimental.** A colorless prismatic crystal of (I) with dimensions 0.2 × 0.3 × 0.4 mm (provided by Dr L. S. Trifanov of the Institute of Organic Chemistry, Bulgarian Academy of Sciences) and grown from a 2:1 mixture of chloroform and toluene was used for the data collection.



Accurate cell constants and crystal orientation matrix were determined on a Nicolet R3 diffractometer by a least-squares refinement of the setting angles of 25 reflections with  $2\theta$  in the range 20–24°. Intensity data were collected by the  $\omega$ -scan method and variable scan speed (3–29.3° min<sup>-1</sup>) with graphite-monochromatized radiation in the range  $0 < \theta < 24^\circ$ . The intensities of three reflections (542, 147, 641) were monitored every 150 reflections and showed a random variation of about 1.5%. Intensities of 5725 reflections ( $h$  0→17,  $k$  -1→12,  $l$  -23→23) were measured  $\{[(\sin\theta)/\lambda]_{\max} = 0.57 \text{ \AA}^{-1}\}$  of which 4576 were unique ( $R_{\text{int}} = 0.013$ ) and 2906 observed [ $I > 3\sigma(I)$ ] which were used in the structure solution and refinement. The data were corrected for Lorentz-polarization effects, but not for absorption.

The structure was solved by direct methods, and refined by blocked cascade refinement with about 100 parameters per block. All 44 non-H atoms were revealed in the best  $E$  map. Refinement on  $F$ , initially with isotropic temperature factors and finally with anisotropic displacement parameters for non-H atoms. A difference Fourier map computed on the anisotropic model revealed the 31 H atoms as the largest peaks in the map. The H atoms were included in subsequent cycles riding on the C atoms (C–H 0.95 Å) with individual isotropic temperature factors. Refinement converged with  $R = 0.044$  and  $wR = 0.042$ ; in the final cycle  $(\Delta/\sigma)_{\max} = 0.15$  and  $S = 1.880$ . Weights were derived from counting statistics,  $w = 1/[\sigma^2(F) + 0.00029F^2]$ . A difference map calculated at the conclusion of refinement showed no significant features, the maximum and minimum peaks were 0.19 and  $-0.17 \text{ e \AA}^{-3}$  respectively. All calculations were performed using *SHELXTL*

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