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Structure of a 2-Aza-6,7-benzotricyclo[6.2.1.0^{1,5}]undeca-4,6,9-trien-3-one

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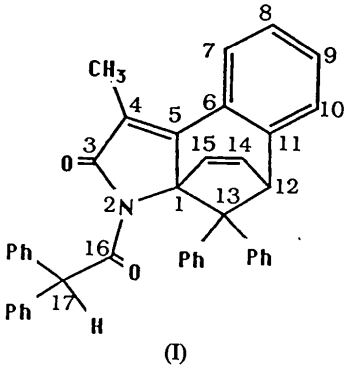
Abstract. (1*RS*,8*RS*)-4-Methyl-11,11-diphenyl-2-diphenylacetyl-2-aza-6,7-benzotricyclo[6.2.1.0^{1,5}]undeca-4,6,9-trien-3-one. $C_{41}H_{31}NO_2$, $M_r = 569{\cdot}71$, monoclinic, $P2_1/n$, $a = 14{\cdot}396$ (4), $b = 10{\cdot}375$ (3), $c = 19{\cdot}702$ (6) Å, $\beta = 94{\cdot}87$ (2)°, $V = 2932$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}290$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}07$ mm^{-1}, $F(000) = 1200$, $T = 292$ K, final $R = 0{\cdot}044$ 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.



(I)

Accurate cell constants and crystal orientation matrix were determined on a Nicolet *R*3 diffractometer by a least-squares refinement of the setting angles of 25 reflections with 2θ in the range 20–24°. Intensity data were collected by the ω-scan method and variable scan speed (3–29·3° min^{-1}) with graphite-monochromatized radiation in the range $0 < \theta < 24°$. 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 {$[(\sin\theta)/\lambda]_{max} = 0{\cdot}57$ Å^{-1}} of which 4576 were unique ($R_{int} = 0{\cdot}013$) 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 $R = 0{\cdot}044$ and $wR = 0{\cdot}042$; in the final cycle $(\Delta/\sigma)_{max} = 0{\cdot}15$ and $S = 1{\cdot}880$. Weights were derived from counting statistics, $w = 1/[\sigma^2(F) + 0{\cdot}00029F^2]$. A difference map calculated at the conclusion of refinement showed no significant features, the maximum and minimum peaks were 0·19 and −0·17 e Å^{-3} respectively. All calculations were performed using *SHELXTL*

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Table 1. *Atom coordinates and temperature factors* ($Å^2$)

	x	y	z	U_{eq}*
C(1)	0·9026 (2)	0·2216 (2)	0·1824 (1)	0·033 (1)
N(2)	0·9858 (1)	0·2959 (2)	0·2079 (1)	0·034 (1)
C(3)	1·0328 (2)	0·3379 (3)	0·1514 (1)	0·038 (1)
C(4)	0·9898 (2)	0·2739 (3)	0·0905 (1)	0·039 (1)
C(5)	0·9186 (2)	0·1998 (2)	0·1078 (1)	0·033 (1)
C(6)	0·8571 (2)	0·1079 (2)	0·0692 (1)	0·036 (1)
C(7)	0·8677 (2)	0·0751 (3)	0·0017 (1)	0·046 (1)
C(8)	0·8108 (2)	−0·0163 (3)	−0·0311 (1)	0·054 (1)
C(9)	0·7419 (2)	−0·0759 (3)	0·0018 (1)	0·053 (1)
C(10)	0·7307 (2)	−0·0456 (3)	0·0687 (1)	0·044 (1)
C(11)	0·7876 (2)	0·0446 (2)	0·1027 (1)	0·035 (1)
C(12)	0·7778 (2)	0·0782 (2)	0·1766 (1)	0·034 (1)
C(13)	0·8776 (2)	0·0881 (2)	0·2152 (1)	0·032 (1)
C(14)	0·7421 (2)	0·2148 (3)	0·1789 (1)	0·040 (1)
C(15)	0·8115 (2)	0·2961 (3)	0·1821 (1)	0·038 (1)
C(16)	0·9928 (2)	0·3580 (2)	0·2721 (1)	0·035 (1)
C(17)	1·0899 (2)	0·4007 (2)	0·2999 (1)	0·037 (1)
C(4*m*)	1·0286 (2)	0·2996 (3)	0·0233 (1)	0·062 (1)
O(1)	1·0972 (1)	0·4145 (2)	0·1542 (1)	0·054 (1)
O(2)	0·9249 (1)	0·3672 (2)	0·3039 (1)	0·044 (1)
C(18)	0·8730 (2)	0·0923 (2)	0·2930 (1)	0·032 (1)
C(19)	0·9529 (2)	0·0705 (2)	0·3361 (1)	0·040 (1)
C(20)	0·9514 (2)	0·0743 (3)	0·4061 (1)	0·050 (1)
C(21)	0·8694 (2)	0·0999 (3)	0·4349 (1)	0·054 (1)
C(22)	0·7892 (2)	0·1199 (3)	0·3934 (1)	0·049 (1)
C(23)	0·7906 (2)	0·1153 (2)	0·3232 (1)	0·040 (1)
C(24)	0·9394 (2)	−0·0247 (2)	0·1982 (1)	0·032 (1)
C(25)	1·0339 (2)	−0·0127 (3)	0·1890 (1)	0·039 (1)
C(26)	1·0869 (2)	−0·1194 (3)	0·1755 (1)	0·044 (1)
C(27)	1·0481 (2)	−0·2400 (3)	0·1708 (1)	0·045 (1)
C(28)	0·9547 (2)	−0·2536 (3)	0·1803 (1)	0·047 (1)
C(29)	0·9021 (2)	−0·1476 (2)	0·1942 (1)	0·040 (1)
C(30)	1·1260 (2)	0·3146 (3)	0·3594 (1)	0·043 (1)
C(31)	1·1880 (2)	0·2174 (3)	0·3485 (2)	0·061 (1)
C(32)	1·2275 (2)	0·1413 (4)	0·4021 (2)	0·088 (2)
C(33)	1·2034 (2)	0·1652 (4)	0·4664 (2)	0·101 (2)
C(34)	1·1405 (2)	0·2593 (3)	0·4786 (2)	0·084 (2)
C(35)	1·1014 (2)	0·3341 (3)	0·4250 (1)	0·063 (1)
C(36)	1·0964 (2)	0·5426 (2)	0·3192 (1)	0·038 (1)
C(37)	1·0272 (2)	0·6107 (3)	0·3488 (1)	0·053 (1)
C(38)	1·0405 (2)	0·7377 (3)	0·3681 (2)	0·063 (1)
C(39)	1·1228 (2)	0·7994 (3)	0·3590 (2)	0·061 (1)
C(40)	1·1917 (2)	0·7343 (3)	0·3299 (2)	0·057 (1)
C(41)	1·1784 (2)	0·6070 (3)	0·3096 (1)	0·046 (1)

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

C(1)—N(2)	1·475 (3)	C(1)—C(5)	1·524 (3)
C(1)—C(13)	1·583 (3)	C(1)—C(15)	1·523 (3)
N(2)—C(3)	1·419 (3)	N(2)—C(16)	1·416 (3)
C(3)—C(4)	1·463 (3)	C(3)—O(1)	1·219 (3)
C(4)—C(5)	1·348 (3)	C(4)—C(4*m*)	1·504 (4)
C(5)—C(6)	1·468 (3)	C(13)—C(12)	1·570 (3)
C(13)—C(18)	1·540 (3)	C(13)—C(21)	1·525 (3)
C(12)—C(11)	1·515 (3)	C(12)—C(14)	1·509 (4)
C(11)—C(10)	1·379 (3)	C(11)—C(6)	1·408 (3)
C(7)—C(8)	1·377 (4)	C(7)—C(6)	1·393 (3)
C(8)—C(9)	1·376 (4)	C(9)—C(10)	1·379 (4)
C(14)—C(15)	1·305 (3)	C(16)—C(17)	1·522 (3)
C(16)—O(2)	1·208 (3)	C(17)—C(31)	1·530 (3)
C(17)—C(41)	1·521 (3)		
N(2)—C(1)—C(5)	102·8 (2)	N(2)—C(1)—C(13)	121·6 (2)
C(5)—C(1)—C(13)	108·8 (2)	N(2)—C(1)—C(15)	114·2 (2)
C(5)—C(1)—C(15)	105·8 (2)	C(13)—C(1)—C(15)	102·7 (2)
C(1)—N(2)—C(3)	108·7 (2)	C(1)—N(2)—C(16)	122·4 (2)
C(3)—N(2)—C(16)	123·9 (2)	N(2)—C(3)—C(4)	107·9 (2)
N(2)—C(3)—O(1)	125·2 (2)	C(4)—C(3)—O(1)	126·9 (2)
C(3)—C(4)—C(5)	109·3 (2)	C(3)—C(4)—C(4*m*)	118·6 (2)
C(5)—C(4)—C(4*m*)	132·1 (2)	C(1)—C(5)—C(4)	109·8 (2)
C(1)—C(5)—C(6)	117·6 (2)	C(4)—C(5)—C(6)	132·6 (2)
C(1)—C(13)—C(12)	95·1 (2)	C(1)—C(13)—C(18)	114·3 (2)
C(12)—C(13)—C(18)	111·7 (2)	C(1)—C(13)—C(24)	115·1 (2)
C(12)—C(13)—C(24)	111·7 (2)	C(18)—C(13)—C(24)	108·5 (2)
C(13)—C(12)—C(11)	109·0 (2)	C(13)—C(12)—C(14)	103·0 (2)
C(11)—C(12)—C(14)	107·7 (2)	C(12)—C(11)—C(10)	121·5 (2)
C(12)—C(11)—C(6)	118·1 (2)	C(10)—C(11)—C(6)	120·4 (2)
C(8)—C(7)—C(6)	120·5 (2)	C(7)—C(8)—C(9)	120·8 (2)
C(8)—C(9)—C(10)	119·7 (3)	C(11)—C(10)—C(9)	120·3 (2)
C(5)—C(6)—C(11)	118·8 (2)	C(5)—C(6)—C(7)	122·9 (2)
C(11)—C(6)—C(7)	118·2 (2)	C(12)—C(14)—C(15)	110·3 (2)
C(1)—C(15)—C(14)	109·1 (2)	N(2)—C(16)—C(17)	116·6 (2)
N(2)—C(16)—O(2)	119·9 (2)	C(17)—C(16)—O(2)	123·3 (2)
C(16)—C(17)—C(30)	110·3 (2)	C(16)—C(17)—C(36)	114·0 (2)
C(30)—C(17)—C(36)	111·3 (2)		

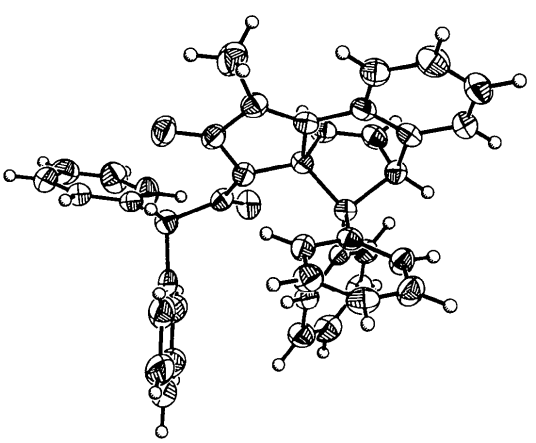

Fig. 1. Perspective view of the molecule drawn with 50% thermal ellipsoids.

version 5·1 (Sheldrick, 1986). Scattering factors as implemented in that program. An empirical extinction correction was made using the modified form of the Larson (1967) technique available in *SHELXTL*. The value of x was refined to 0·00051 (8).*

Atomic parameters are in Table 1, and selected bond lengths and angles in Table 2.† Fig. 1 is a view of the molecule. The numbering scheme is indicated in the structural diagram.

Related literature. The photolysis reaction which gave rise to this product will be discussed elsewhere (Trifanov, Dimitrov & Orahovats, 1989).

* $F^* = F_c/[1{\cdot}0 + 0{\cdot}002xF^2/\sin 2\theta]^{0{\cdot}25}$.

† Lists of structure factors, anisotropic displacement parameters, H-atom positions and complete listings of bond lengths, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52291 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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