Table 2. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°) *involving non-*H *atoms, with e.s.d.'s in parentheses*

O1—C2	1·424 (5)	C5—C6	1·510 (5)	C8—C9	1·373 (6)
C2—C3	1·515 (5)	C6—C7	1·394 (5)	C9—C10	1·391 (6)
C3—N4	1·457 (4)	C6—C11	1·384 (5)	C10—C11	1·387 (6)
C3—C5	1·537 (5)	C7—C8	1·379 (6)		

O1—C2—C3	110·3 (3)	C7—C6—C11	118·1 (3)
C2—C3—N4	108·6 (3)	C6—C7—C8	120·7 (4)
C2—C3—C5	109·5 (3)	C7—C8—C9	120·9 (4)
N4—C3—C5	110·6 (3)	C8—C9—C10	119·3 (4)
C3—C5—C6	113·8 (3)	C9—C10—C11	119·7 (4)
C5—C6—C7	121·0 (3)	C6—C11—C10	121·3 (3)
C5—C6—C11	120·9 (3)		

ψO1—C2—C3—N4	57·11 (0·37)
O1—C2—C3—C5	177·91 (0·27)
C2—C3—C5—C6	173·03 (0·29)
χ₁N4—C3—C5—C6	−67·36 (0·38)
χ₂C3—C5—C6—C7	94·06 (0·42)
C3—C5—C6—C11	−86·02 (0·42)
C5—C6—C7—C8	−179·14 (0·38)
C11—C6—C7—C8	0·93 (0·60)
C5—C6—C11—C10	179·42 (0·38)
C7—C6—C11—C10	−0·65 (0·60)
C6—C7—C8—C9	−1·19 (0·67)
C7—C8—C9—C10	1·12 (0·68)
C8—C9—C10—C11	−0·83 (0·66)
C9—C10—C11—C6	0·61 (0·64)

Table 3. *Comparison of torsion angles* (°) (*defined in Table* 2)

	ψ	χ_1	χ_2	Reference
Phenylalaninol	57·1 (4)	−67·4 (4)	94·1 (4)	(a)
Boc-glycyl-L-phenylalanine	170·3	52·0	85·8	(b)
L-Tyrosyl-L-phenylalanine	146·6	−75·1	75·9	(c)
L-Phenylalanine-L-proline H₂O	−42·5 (7)	−170·6 (5)	81·6 (7)	(d)

References: (a) Present study; (b) Murali & Subramanian (1986); (c) Murali & Subramanian (1987); (d) Panneerselvam & Chacko (1989).

Related literature. In the unit cell the molecules are stabilized by N—H···O-type intermolecular hydrogen bonds with N···O distance of 2·764 (2) Å with sym-
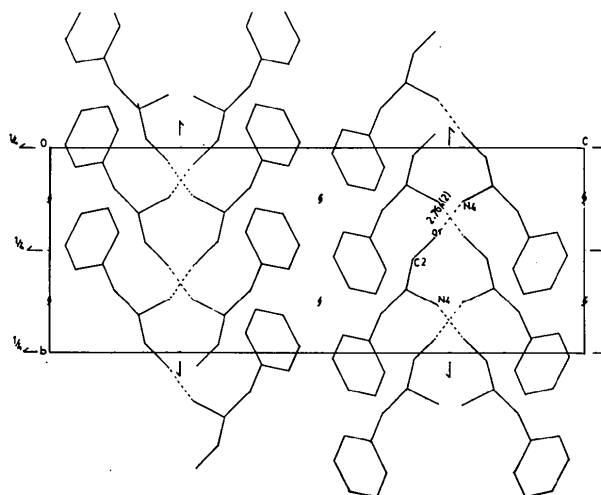

Fig. 2. Packing of the molecules in the unit cell projected down the *a* axis.

metry $1-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$. The backbone and side-chain torsion angles are comparable with the values observed in related structures containing a phenyl group (Murali & Subramanian, 1986, 1987; Panneerselvam & Chacko, 1989) (Table 3).

References

FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP - A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Determination.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.

MURALI, R. & SUBRAMANIAN, E. (1986). *Int. J. Peptide Protein Res.* **27**, 478–482.

MURALI, R. & SUBRAMANIAN, E. (1987). *Int. J. Peptide Protein Res.* **29**, 187–192.

PANNEERSELVAM, K. & CHACKO, K. K. (1989). *Acta Cryst.* C45, 106–109.

1:2 Adduct of 1-Ethoxy-2-methylisoindole with *N*-Methylmaleimide

BY HANS PREUT, FRANK JELITTO AND RICHARD P. KREHER

Fachbereich Chemie, Universität Dortmund, Postfach 500500, D-4600 Dortmund 50, Federal Republic of Germany

**Abstract.** *endo*-1-Ethoxy-*N*-methyl-1,4-(*N*-methyl-imino)-4-(*N*-methylsuccinimido)-1,2,3,4-tetrahydro-naphthalene-2,3-dicarboximide hemimethanol solvate, $C_{21}H_{23}N_3O_5 \cdot \frac{1}{2}CH_3OH$, $M_r = 413 \cdot 46$, triclinic, $P\bar{1}$, $a = 8 \cdot 623$ (4), $b = 9 \cdot 876$ (6), $c = 14 \cdot 235$ (10) Å, $\alpha = 108 \cdot 96$ (5), $\beta = 97 \cdot 40$ (5), $\gamma = 102 \cdot 28$ (4)°, $V =$

1094 (1) Å³, $Z = 2$, $D_x = 1\cdot255$ Mg m⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot09$ mm⁻¹, $F(000) = 438$, $T = 291$ (1) K, final $R = 0\cdot092$ for 2392 unique observed $|F \geq 4\cdot0\sigma(F)|$ diffractometer data. The structure determination has established the configuration of the 1:2 adduct as *endo*; the *syn* orientation of the benzenoid moiety and the dienophile segment is clearly demonstrated. The methyl group at the N atom of the imino group obviously prefers to be *anti* to the benzenoid system. The angles at the bridging positions – C(2)– C(1)–C(8a) and C(3)–C(4)–C(4a) – are not significantly distorted by the substituents. The five-membered ring (succinimido group) – introduced by Michael addition – takes up a preferred conformation relative to the azanorbornene system. Therefore H(3) is influenced by the neighbourhood of the carbonyl group; the deshielding effect becomes evident in the ¹H NMR spectra of the 1:2 adduct.

Experimental. The formation of the 1:2 adduct (2) of 1-ethoxy-2-methylisoindole (1) with *N*-methylmaleimide was observed by Kreher & Hennige (1973) and explained as the result of subsequent Michael addition and Diels–Alder addition. These consecutive reactions are of special interest because the ambivalent reactivity of the *o*-quinonoid hetarene (1) is demonstrated convincingly. The preliminary results were confirmed by Jelitto (1988). In order to establish the constitution and configuration an X-ray analysis of the crystalline 1:2 adduct (2) (m.p. 481–483 K from ether) was
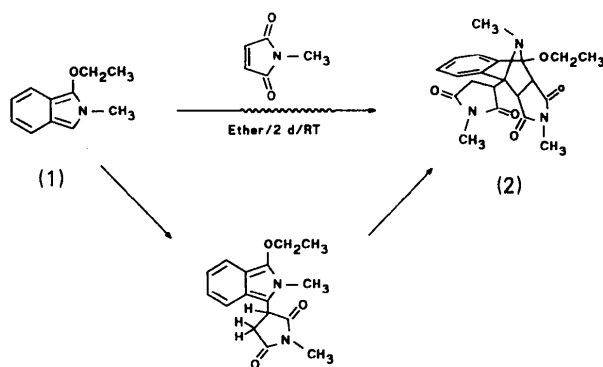
performed; colourless crystals from ether by slow concentration (2 d) at 298 K. Crystal size $\sim 0\cdot32 \times 0\cdot28 \times 0\cdot08$ mm, $\omega/2\theta$ scan, scan speed 1·5° min⁻¹ in θ, Nicolet $R3m$/V diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 17 reflections up to $2\theta = 29\cdot9°$; six standard reflections recorded every 2·5 h, only random deviations; 7926 reflections measured, $1\cdot5 \leq \theta \leq 25\cdot0°$, $-11 \leq h \leq 11$, $-12 \leq k \leq 12$, $-17 \leq l \leq 17$; after averaging ($R_{int} = 0\cdot019$): 3882 unique reflections, 2394 with $F \geq 4\cdot0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans, max./min. transmission 1·00/0·92; structure solution *via* direct

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å² × 10³)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
O(1)	0·7986 (5)	−0·0769 (5)	0·0919 (3)	54
O(2)	1·1138 (5)	0·1588 (5)	0·3026 (3)	56
O(3)	0·7968 (5)	0·0871 (5)	0·5233 (3)	53
O(4)	0·0302 (5)	−0·3430 (5)	0·2381 (3)	50
O(5)	0·5383 (5)	−0·3204 (5)	0·3869 (4)	67
N(1)	0·5922 (5)	−0·1475 (5)	0·1825 (4)	39
N(2)	0·9735 (5)	0·1537 (5)	0·4277 (4)	38
N(3)	0·2750 (6)	−0·3641 (5)	0·3080 (4)	42
C(1)	0·7456 (7)	−0·0415 (6)	0·1822 (5)	42
C(2)	0·8718 (6)	−0·0481 (6)	0·2708 (4)	39
C(3)	0·7673 (6)	−0·0711 (6)	0·3458 (4)	36
C(4)	0·5933 (6)	−0·0719 (6)	0·2920 (4)	31
C(4a)	0·6084 (6)	0·0856 (6)	0·2957 (4)	33
C(5)	0·5596 (7)	0·2025 (6)	0·3562 (5)	40
C(6)	0·6062 (8)	0·3403 (7)	0·3448 (5)	53
C(7)	0·7011 (8)	0·3585 (8)	0·2761 (6)	66
C(8)	0·7520 (8)	0·2419 (8)	0·2156 (5)	58
C(8a)	0·7015 (6)	0·1046 (6)	0·2257 (4)	39
C(9)	0·5970 (8)	−0·3040 (6)	0·1511 (5)	50
C(10)	0·6865 (9)	−0·079 (1)	0·0062 (5)	79
C(11)	0·752 (1)	−0·110 (1)	−0·0832 (7)	136
C(12)	1·0014 (7)	0·0976 (6)	0·3305 (5)	43
C(13)	0·8414 (7)	0·0615 (6)	0·4427 (5)	39
C(14)	1·0706 (7)	0·2960 (7)	0·5028 (5)	49
C(15)	0·4484 (6)	−0·1331 (6)	0·3314 (4)	34
C(16)	0·2825 (6)	−0·1522 (6)	0·2670 (5)	45
C(17)	0·1757 (7)	−0·2933 (6)	0·2681 (5)	40
C(18)	0·4348 (7)	−0·2811 (6)	0·3452 (5)	44
C(19)	0·2125 (8)	−0·5078 (7)	0·3175 (6)	68
O(6)	0·954 (2)	0·381 (2)	1·008 (1)	170
C(20)	0·886 (3)	0·506 (2)	0·984 (2)	139

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

O(1)–C(1)	1·382 (8)	C(2)–C(3)	1·523 (9)
O(1)–C(10)	1·45 (1)	C(2)–C(12)	1·522 (7)
O(2)–C(12)	1·212 (8)	C(3)–C(4)	1·591 (8)
O(3)–C(13)	1·223 (8)	C(3)–C(13)	1·506 (7)
O(4)–C(17)	1·209 (7)	C(4)–C(4a)	1·516 (9)
O(5)–C(18)	1·209 (9)	C(4)–C(15)	1·523 (8)
N(1)–C(1)	1·503 (7)	C(4a)–C(5)	1·381 (8)
N(1)–C(4)	1·491 (7)	C(4a)–C(8a)	1·391 (9)
N(1)–C(9)	1·474 (8)	C(5)–C(6)	1·40 (1)
N(2)–C(12)	1·387 (8)	C(6)–C(7)	1·38 (1)
N(2)–C(13)	1·386 (8)	C(7)–C(8)	1·39 (1)
N(2)–C(14)	1·454 (6)	C(8)–C(8a)	1·39 (1)
N(3)–C(17)	1·390 (9)	C(10)–C(11)	1·43 (1)
N(3)–C(18)	1·379 (7)	C(15)–C(16)	1·536 (8)
N(3)–C(19)	1·464 (9)	C(15)–C(18)	1·520 (9)
C(1)–C(2)	1·584 (9)	C(16)–C(17)	1·504 (9)
C(1)–C(8a)	1·524 (9)	O(6)–C(20)	1·59 (3)

|			
C(4)–N(1)–C(9)	116·2 (5)	C(4)–C(4a)–C(8a)	106·1 (5)
C(1)–N(1)–C(9)	114·2 (5)	C(4)–C(4a)–C(5)	133·0 (6)
C(1)–N(1)–C(4)	95·6 (5)	C(5)–C(4a)–C(8a)	120·7 (6)
C(13)–N(2)–C(14)	124·8 (5)	C(4a)–C(5)–C(6)	118·2 (6)
C(12)–N(2)–C(14)	122·7 (6)	C(5)–C(6)–C(7)	120·6 (7)
C(12)–N(2)–C(13)	112·4 (5)	C(6)–C(7)–C(8)	121·7 (8)
C(18)–N(3)–C(19)	123·1 (6)	C(7)–C(8)–C(8a)	117·2 (7)
C(17)–N(3)–C(19)	122·9 (6)	C(4a)–C(8a)–C(8)	121·7 (6)
C(17)–N(3)–C(18)	113·8 (6)	C(1)–C(8a)–C(8)	132·3 (6)
O(1)–C(1)–N(1)	115·6 (5)	C(1)–C(8a)–C(4a)	105·5 (6)
N(1)–C(1)–C(8a)	99·2 (5)	O(1)–C(10)–C(11)	111·1 (7)
N(1)–C(1)–C(2)	102·1 (5)	N(2)–C(12)–C(2)	108·6 (5)
O(1)–C(1)–C(8a)	120·5 (6)	O(2)–C(12)–C(2)	128·3 (6)
O(1)–C(1)–C(2)	111·3 (5)	O(2)–C(12)–N(2)	123·1 (6)
C(2)–C(1)–C(8a)	106·1 (5)	N(2)–C(13)–C(3)	109·1 (5)
C(1)–C(2)–C(12)	114·6 (5)	O(3)–C(13)–C(3)	127·2 (6)
C(1)–C(2)–C(3)	102·7 (5)	O(3)–C(13)–N(2)	123·7 (6)
C(3)–C(2)–C(12)	104·7 (5)	C(4)–C(15)–C(18)	117·9 (5)
C(2)–C(3)–C(13)	105·2 (5)	C(4)–C(15)–C(16)	114·4 (5)
C(2)–C(3)–C(4)	102·6 (5)	C(16)–C(15)–C(18)	104·6 (5)
C(4)–C(3)–C(13)	113·3 (5)	C(15)–C(16)–C(17)	104·7 (5)
N(1)–C(4)–C(3)	101·7 (5)	N(3)–C(17)–C(16)	107·8 (6)
C(3)–C(4)–C(15)	116·8 (5)	O(4)–C(17)–C(16)	128·3 (6)
C(3)–C(4)–C(4a)	105·8 (5)	O(4)–C(17)–N(3)	123·9 (7)
N(1)–C(4)–C(15)	117·0 (5)	N(3)–C(18)–C(15)	107·6 (6)
N(1)–C(4)–C(4a)	100·0 (5)	O(5)–C(18)–C(15)	128·4 (6)
C(4a)–C(4)–C(15)	113·4 (5)	O(5)–C(18)–N(3)	123·8 (7)
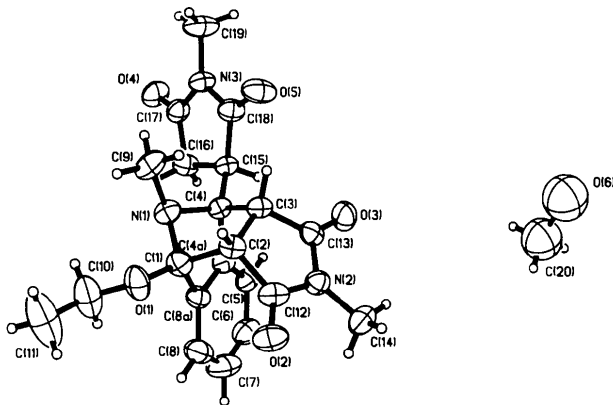
Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme.

methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å). After locating the atoms of the molecule $C_{21}H_{23}N_3O_5$ the ΔF synthesis still showed rather high peaks. The two highest peaks were refined as O and C respectively with an occupancy factor 0·5. Refinement on F with 2392 reflections and 271 refined parameters; $w = 1\cdot00/[\sigma^2(F) + 0\cdot0005F^2]$; $S = 2\cdot51$, $R = 0\cdot092$, $wR = 0\cdot088$. The weakly diffracting crystal and the incompletely described disorder around the solvent molecule could be a reason for the rather high R value. $(\Delta/\sigma)_{max} = 0\cdot38$, no extinction correction; largest peak in final ΔF map $\pm1\cdot0$ (2) e Å^{-3}

near the probably disordered methanol molecule and $\pm0\cdot2$ (2) e Å^{-3} in remaining parts of the unit cell, atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Related literature. For preparation and spectroscopic data see Kreher & Hennige (1973) and Jelitto (1988).

References

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distibutor Kluwer Academic Publishers, Dordrecht.)
JELITTO, F. (1988). Part of dissertation (in progress). Univ. of Dortmund, Federal Republic of Germany.
KREHER, R. & HENNIGE, H. (1973). *Tetrahedron Lett.* p. 1911.
NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
SHELDRICK, G. M. (1987). *SHELXTL-Plus*, release 2. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* For Nicolet *R*3m/V. Univ. of Göttingen, Federal Republic of Germany.
WILLIAMS, D. E. (1984). *QCPE Bull.* **4**.

Structures of Some Lanosterol Derivatives

BY E. E. CASTELLANO AND J. ZUKERMAN-SCHPECTOR*

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 São Carlos, SP, Brazil

AND VERA L. G. REHDER AND A. J. MARSAIOLI

Instituto de Química, Universidade Estadual de Campinas, Caixa Postal 6154, 13081 Campinas, SP, Brazil

Abstract. (I) $C_{32}H_{54}O_4$: 3β-acetoxy-8,9-secolanostane-8,9-dione, $M_r = 502\cdot78$, monoclinic, $P2_1$, $a = $

* Permanent address: Laboratório de Cristalografia e Físico-Química dos Materiais, CCEN, Universidade Federal de Alagoas, 57000 – Maceió, AL, Brazil.

11·456 (3), $b = 7\cdot400$ (1), $c = 17\cdot843$ (5) Å, $\beta = 95\cdot69$ (2)°, $V = 1505$ (1) Å^3, $Z = 2$, $D_x = 1\cdot109$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot04$ mm^{-1}, $F(000) = 556$, $T = 296$ K, $R = 0\cdot089$ for 1061 independent reflections $[I > 3\sigma(I)]$. The ten-membered ring is in a crown conformation, the