Table 2. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}/U
C1	0·7763 (6)	0·1259 (2)	0·6200 (4)	0·021 (1)
C2	0·8367 (6)	0·1197 (2)	0·3366 (3)	0·018 (1)
N2	1·0421 (5)	0·1397 (2)	0·6035 (3)	0·017 (1)
O3	1·0804 (5)	0·1343 (2)	0·4171 (3)	0·028 (1)
O1	0·6883 (5)	0·1225 (3)	0·7638 (3)	0·036 (1)
O2	0·7818 (4)	0·1079 (2)	0·1772 (3)	0·026 (1)
N1	0·6507 (5)	0·1165 (2)	0·4601 (3)	0·021 (1)
OW	1·2555 (5)	0·1186 (2)	−0·0115 (3)	0·032 (1)
HN1	0·507 (10)	0·108 (3)	0·433 (5)	0·010 (8)
HN2	1·186	0·151	0·701	

final cycle. $\Delta\rho$ in the final difference map was within +0·84 to −1·43 e Å^{-3}. Atomic scattering factors as supplied in *SHELX*76.

Atomic coordinates and thermal parameters are given in Table 2.* A plot of the molecule with bond lengths and angles is shown in Fig. 1.

Related literature. The crystal structure of the title compound was studied as part of our program of investigations on propellant formulations (Sameena Begum, Jain, Khetrapal & Shiva Prakash, 1987). Characteristic amide bond lengths are given in Venkatesan & Ramakumar (1981) and Sutton (1965).

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44342 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
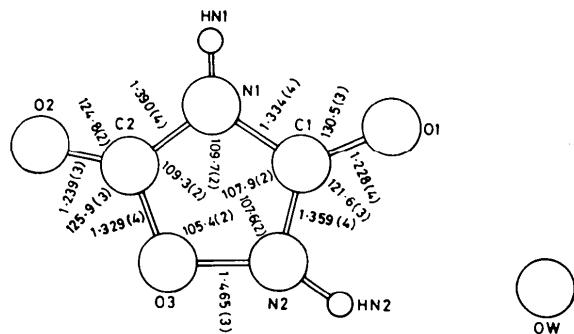
Fig. 1. Bond distances (Å) and angles (°) in 1,2,4-oxadiazolidine-3,5-dione monohydrate.

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Structure of a Mycophenolic Acid Derivative (CAM)

BY YOSHIHARU NAWATA,* YOHKO KURIKI, MASATOSHI HANEDA, KIYOSHIGE OCHI AND TAKASHI MORI

Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, Japan

Abstract. 4-{1,3-Dihydro-5-[(*E*)-5-ethoxycarbonyl-3-methyl-2-pentenyl]-6-methoxy-7-methyl-3-oxo-4-isobenzofuranyloxycarbonylamino}benzoic acid (CAM), $C_{27}H_{29}NO_9$, $M_r = 511·531$, orthorhombic, *Pbca*, $a = 19·010$ (2), $b = 20·604$ (1), $c = 12·917$ (1) Å, $V = 5059·16$ Å^3, $Z = 8$, $D_x = 1·343$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1·5418$ Å, $\mu = 8·04$ cm^{-1}, $F(000) = 2160$, $T = 298$ K, final $R = 0·036$ for 3673 unique reflections [$F_o^2 > 3\sigma(F_o^2)$]. The *trans* zigzag chain of the ethoxycarbonylpentyl moiety and the benzene ring approach each other to maintain intramolecular van der Waals contacts and form a long molecular shape. The least-squares planes of these moieties are approximately perpendicular to the isobenzofuranyl ring.

Experimental. Colorless prisms of CAM were grown from acetone. Crystal size 0·23 × 0·18 × 0·15 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$

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* To whom all correspondence should be addressed.

radiation, graphite monochromator, θ–2θ scan with scan speed 0·55–8·24° min⁻¹ in θ, scan width (0·60 + 0·14tanθ)°. Range of indices, $0 \le h \le 23$, $0 \le k \le 25$, $0 \le l \le 15$ ($2\theta < 140°$). Lattice constants determined based on 25 2θ values ($38 < 2\theta < 104°$). Variation of standard <0·5%; 4786 unique reflections measured; 3673 observed reflections with $F_o^2 > 3\sigma(F_o^2)$. Systematic absences $0kl$, k odd; $h0l$, l odd; $hk0$, h odd. No corrections for absorption. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull,

Lessinger, Germain, Declercq & Woolfson, 1982). Refined by full-matrix least squares. The locations of all the H atoms were found on a difference Fourier map. Non-H atoms refined with anisotropic thermal parameters, and H atoms with isotropic thermal parameters
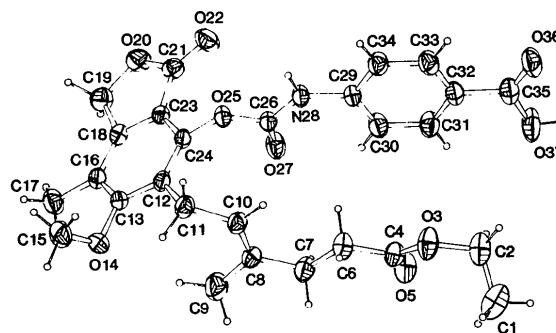

Fig. 1. A perspective view of the molecule with the numbering scheme.
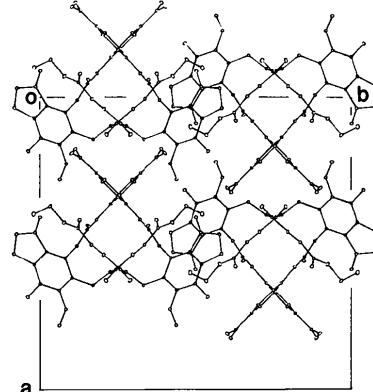
Fig. 2. The crystal structure projected along the c axis.

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-*H* atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j B_{ij}\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	0·3993 (2)	0·5199 (1)	0·0575 (2)	5·79 (6)
C(2)	0·3713 (1)	0·4674 (1)	0·1231 (2)	4·47 (5)
O(3)	0·42829 (8)	0·42320 (7)	0·1546 (1)	4·12 (3)
C(4)	0·4481 (1)	0·37867 (9)	0·0858 (2)	3·05 (4)
O(5)	0·42033 (8)	0·37337 (7)	0·0022 (1)	3·94 (3)
C(6)	0·5049 (1)	0·3356 (1)	0·1268 (2)	3·35 (4)
C(7)	0·5393 (1)	0·2958 (1)	0·0425 (2)	3·62 (4)
C(8)	0·5896 (1)	0·24354 (9)	0·0758 (2)	3·01 (4)
C(9)	0·6270 (1)	0·2121 (1)	−0·0131 (2)	4·67 (5)
C(10)	0·5979 (1)	0·22512 (9)	0·1731 (2)	2·88 (4)
C(11)	0·6434 (1)	0·16882 (9)	0·2078 (2)	3·01 (4)
C(12)	0·61744 (9)	0·10330 (8)	0·1703 (1)	2·37 (3)
C(13)	0·65994 (9)	0·06256 (9)	0·1087 (1)	2·55 (3)
O(14)	0·72618 (6)	0·08408 (6)	0·0824 (1)	3·38 (3)
C(15)	0·7802 (1)	0·0637 (1)	0·1527 (2)	4·23 (5)
C(16)	0·63833 (9)	0·00204 (9)	0·0716 (1)	2·60 (3)
C(17)	0·6835 (1)	−0·0381 (1)	0·0009 (2)	3·72 (4)
C(18)	0·57216 (9)	−0·01800 (8)	0·1027 (1)	2·46 (3)
C(19)	0·5362 (1)	−0·08199 (9)	0·0863 (2)	3·27 (4)
O(20)	0·46893 (7)	−0·07468 (6)	0·1377 (1)	3·48 (3)
C(21)	0·4634 (1)	−0·01533 (9)	0·1825 (2)	2·92 (4)
O(22)	0·41052 (7)	−0·00019 (7)	0·2282 (1)	4·03 (3)
C(23)	0·52845 (9)	0·02052 (8)	0·1621 (1)	2·30 (3)
C(24)	0·55051 (9)	0·08131 (8)	0·1944 (1)	2·25 (3)
O(25)	0·50886 (6)	0·11815 (6)	0·2616 (1)	2·57 (2)
C(26)	0·45314 (9)	0·15125 (8)	0·2193 (1)	2·38 (3)
O(27)	0·44021 (7)	0·15315 (7)	0·1285 (1)	3·38 (3)
N(28)	0·41907 (8)	0·18097 (7)	0·2977 (1)	2·69 (3)
C(29)	0·36392 (9)	0·22632 (8)	0·2898 (1)	2·48 (3)
C(30)	0·3399 (1)	0·2518 (1)	0·1973 (2)	3·32 (4)
C(31)	0·2879 (1)	0·2994 (1)	0·1985 (2)	3·43 (4)
C(32)	0·25929 (9)	0·32073 (9)	0·2904 (2)	2·93 (4)
C(33)	0·2823 (1)	0·2937 (1)	0·3824 (2)	3·50 (4)
C(34)	0·3342 (1)	0·2470 (1)	0·3826 (2)	3·36 (4)
C(35)	0·2045 (1)	0·37198 (9)	0·2962 (2)	3·40 (4)
O(36)	0·17616 (8)	0·38837 (7)	0·3751 (1)	4·61 (3)
O(37)	0·19039 (8)	0·39902 (8)	0·2051 (1)	5·32 (4)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·473 (4)		C(19)–O(20)	1·449 (2)
C(2)–O(3)	1·473 (3)		O(20)–C(21)	1·357 (2)
O(3)–C(4)	1·331 (2)		C(21)–O(22)	1·207 (2)
C(4)–O(5)	1·206 (2)		C(21)–C(23)	1·465 (3)
C(4)–C(6)	1·495 (3)		C(23)–C(24)	1·385 (2)
C(6)–C(7)	1·511 (3)		C(24)–O(25)	1·398 (2)
C(7)–C(8)	1·504 (3)		O(25)–C(26)	1·373 (2)
C(8)–C(9)	1·498 (3)		C(26)–O(27)	1·198 (2)
C(8)–C(10)	1·321 (3)		C(26)–N(28)	1·350 (2)
C(10)–C(11)	1·515 (3)		N(28)–C(29)	1·408 (2)
C(11)–C(12)	1·517 (3)		C(29)–C(30)	1·383 (3)
C(12)–C(13)	1·410 (2)		C(29)–C(34)	1·391 (3)
C(12)–C(24)	1·386 (2)		C(30)–C(31)	1·393 (3)
C(13)–O(14)	1·378 (2)		C(31)–C(32)	1·378 (3)
C(13)–C(16)	1·398 (3)		C(32)–C(33)	1·383 (3)
O(14)–C(15)	1·433 (3)		C(32)–C(35)	1·485 (3)
C(16)–C(17)	1·501 (3)		C(33)–C(34)	1·377 (3)
C(16)–C(18)	1·384 (3)		C(35)–O(36)	1·201 (3)
C(18)–C(19)	1·500 (3)		C(35)–O(37)	1·329 (3)
C(18)–C(23)	1·382 (2)			
C(1)–C(2)–O(3)	110·3 (2)		O(20)–C(21)–O(22)	120·4 (2)
C(2)–O(3)–C(4)	116·8 (2)		O(20)–C(21)–C(23)	108·2 (2)
O(3)–C(4)–O(5)	122·4 (2)		O(22)–C(21)–C(23)	131·4 (2)
O(3)–C(4)–C(6)	112·1 (2)		C(18)–C(23)–C(21)	108·6 (2)
O(5)–C(4)–C(6)	125·4 (2)		C(18)–C(23)–C(24)	120·3 (2)
C(4)–C(6)–C(7)	112·3 (2)		C(21)–C(23)–C(24)	131·1 (2)
C(6)–C(7)–C(8)	117·2 (2)		C(12)–C(24)–C(23)	120·4 (2)
C(7)–C(8)–C(9)	113·1 (2)		C(12)–C(24)–O(25)	118·8 (2)
C(7)–C(8)–C(10)	123·6 (2)		C(23)–C(24)–O(25)	120·4 (2)
C(9)–C(8)–C(10)	123·2 (2)		C(24)–O(25)–C(26)	117·4 (1)
C(8)–C(10)–C(11)	124·7 (2)		O(25)–C(26)–O(27)	124·3 (2)
C(10)–C(11)–C(12)	113·6 (2)		O(25)–C(26)–N(28)	107·2 (2)
C(11)–C(12)–C(13)	121·5 (2)		O(27)–C(26)–N(28)	128·4 (2)
C(11)–C(12)–C(24)	121·2 (2)		C(26)–N(28)–C(29)	127·1 (2)
C(13)–C(12)–C(24)	117·3 (2)		N(28)–C(29)–C(30)	124·1 (2)
C(12)–C(13)–O(14)	118·1 (2)		N(28)–C(29)–C(34)	116·3 (2)
C(12)–C(13)–C(16)	123·8 (2)		C(30)–C(29)–C(34)	119·6 (2)
O(14)–C(13)–C(16)	118·1 (2)		C(29)–C(30)–C(31)	119·5 (2)
C(13)–O(14)–C(15)	113·8 (2)		C(30)–C(31)–C(32)	120·9 (2)
C(13)–C(16)–C(17)	122·1 (2)		C(31)–C(32)–C(33)	119·1 (2)
C(13)–C(16)–C(18)	115·7 (2)		C(31)–C(32)–C(35)	123·1 (2)
C(17)–C(16)–C(18)	122·2 (2)		C(33)–C(32)–C(35)	117·7 (2)
C(16)–C(18)–C(19)	129·4 (2)		C(32)–C(33)–C(34)	120·6 (2)
C(16)–C(18)–C(23)	122·5 (2)		C(29)–C(34)–C(33)	120·2 (2)
C(19)–C(18)–C(23)	108·0 (2)		C(32)–C(35)–O(36)	123·8 (2)
C(18)–C(19)–O(20)	104·2 (2)		C(32)–C(35)–O(37)	113·3 (2)
C(19)–O(20)–C(21)	111·0 (1)		O(36)–C(35)–O(37)	122·9 (2)

(fixed at $B = 5 \cdot 0$ Å^2). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1 \cdot 0$ for $F_o < 598 \cdot 5$, $w = (598 \cdot 5/F_o)^2$ for $F_o \geq 598 \cdot 5$. Final $R = 0 \cdot 036$, $wR = 0 \cdot 034$, $S = 2 \cdot 98$ for 451 variables, secondary-extinction factor (g) $6 \cdot 27\,(7) \times 10^{-7}$ $[|F_o| = |F_c|/(1 + gIc)]$; $\Delta/\sigma < 0 \cdot 25$, largest peak in final ΔF map $+0 \cdot 40$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of CAM is shown in Fig. 1, a packing diagram in Fig. 2; positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles in Table 2.*

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* Lists of structure factors, anisotropic thermal parameters, torsion angles, least-squares planes and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44328 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Title compound has antitumor and immunosuppressive activity (Ohsugi, Suzuki & Takagi, 1976). For the preparation see Mori, Takaku & Suzuki (1983).

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1,2-Dihydro-5,6-dimethoxy-1-benzocyclobutenyl 3,5-Dinitrobenzoate

BY UPALI SIRIWARDANE, R. THIMMA REDDY, SHIRLEY S. C. CHU* AND EDWARD R. BIEHL
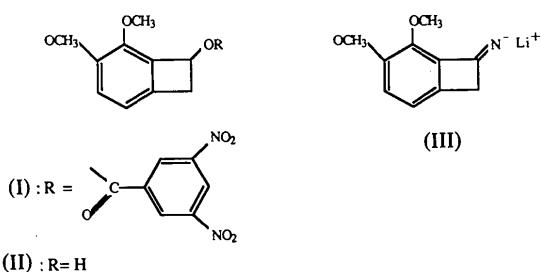
Departments of Chemistry and Electrical Engineering, Southern Methodist University, Dallas, Texas 75275, USA

Abstract. $C_{17}H_{14}N_2O_8$, $M_r = 374 \cdot 31$, monoclinic, $P2_1/n$, $a = 10 \cdot 371\,(3)$, $b = 22 \cdot 759\,(6)$, $c = 7 \cdot 442\,(2)$ Å, $\beta = 106 \cdot 62\,(2)°$, $V = 1683 \cdot 2\,(7)$ Å^3, $Z = 4$, $D_x = 1 \cdot 48$ g cm^{-3}, λ(Mo $K\alpha$) = $0 \cdot 71069$ Å, $\mu = 0 \cdot 76$ cm^{-1}, $F(000) = 776$, $T = 295$ K. Final $R = 0 \cdot 038$ for 1481 observed reflections. The cyclobutene ring is nearly planar. The bond distances are C=C(cyclobutene) = $1 \cdot 386\,(4)$, C—C(cyclobutene, av.) = $1 \cdot 542\,(4)$, C—O(benzo, av.) = $1 \cdot 362\,(4)$, C—O(cyclobutene) = $1 \cdot 445\,(3)$, C—C(benzo, av.) = $1 \cdot 384\,(4)$ and O—CH$_3$(av.) = $1 \cdot 432\,(4)$ Å. The X-ray structure confirms benzocyclobutenimine as an intermediate in a rearrangement reaction.

Experimental. The title compound (I) was prepared as a dinitrobenzoate derivative of (II) to obtain X-ray-quality single crystals. Recently, we have found a novel tandem-addition rearrangement *via* a benzocyclobutenimine intermediate (III) leading to the parent alcohol (II) which was isolated in 25% yield as a fluffy white crystalline material by the reaction of lithio-acetonitrile with 3,4-dimethoxy-1,3-cyclohexadien-5-

yne (generated *in situ* with lithiodiisopropylamine in THF) followed by aqueous work-up.

(I) : R =

(II) : R = H

(III)

Unit-cell parameters by least-squares fit of 15 reflections in the range $10 < 2\theta < 25°$, crystal dimensions $0 \cdot 64 \times 0 \cdot 45 \times 0 \cdot 18$ mm, space group $P2_1/n$ from systematic absences ($0k0$, k odd; $h0l$, $h + l$ odd); automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, variable scan rate ($3 \cdot 0–14 \cdot 7°$ min^{-1}, depending on intensity), 2571 measured reflections, 2208 independent reflections in the range $3 < 2\theta < 45°$, $R_{int} = 0 \cdot 008$, hkl range $h\ 10 \rightarrow -11$, $k\ 0 \rightarrow 23$, $l\ 0 \rightarrow 8$, 1481 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics;

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* To whom correspondence should be addressed.