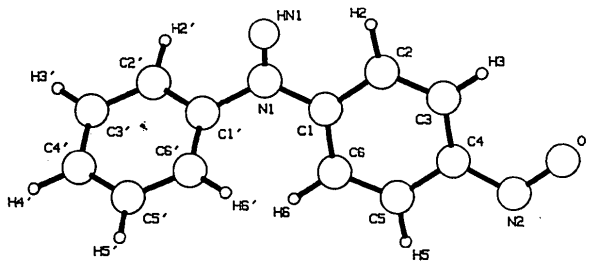

Fig. 1. Perspective view of the molecule.

Related literature. Average C—C distances of the two aromatic rings differ slightly [1·390 (5) and 1·378 (5) Å], as observed in *p*-(*p*-nitroanilino)phenyl isothiocynate (Hardgrove, Einstein & Wei, 1983). The lengths of the two C—N bonds connecting the aromatic rings are different, as observed in 6-methyl-4-(*p*-methylphenylamino)-5,6-dihydro-2-pyrone (Laidoudi, Boubekeur, Nedjar & Brianso, 1980) and in three derivatives of 9,10-anthracenedione (Foitzik, Paulus & Haase, 1986; Foitzik, Paulus & Quotschalla, 1987).

We thank Dr S. Krishnan for the computer drawing of the molecule.

References

FOITZIK, J. K., PAULUS, H. & HAASE, W. (1986). *Acta Cryst.* C**42**, 106–107, 108–109.

FOITZIK, J. K., PAULUS, H. & QUOTSCHALLA, U. (1987). *Acta Cryst.* C**43**, 1166–1168.

GANTZEL, P. K., SPARKS, R. A. & TRUEBLOOD, K. N. (1961). *LALS. A Program for the Full-Matrix Least-Squares Refinement of Positional and Thermal Parameters and Scale Factors.* Univ. of California Program, UCLALS1.

HARDGROVE, G. L. JR, EINSTEIN, J. R. & WEI, C. H. (1983). *Acta Cryst.* C**39**, 616–620.

LAIDOUDI, A., BOUBEKEUR, K., NEDJAR, B. & BRIANSO, M. C. (1980). *Acta Cryst.* B**36**, 2852–2854.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

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

Acta Cryst. (1991). C**47**, 218–220

Structure of Myrotoxin B Hydrate

BY CLIFFORD GEORGE, RICHARD GILARDI AND JUDITH L. FLIPPEN-ANDERSON

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington DC 20375, *USA*

(*Received* 24 *April* 1990; *accepted* 18 *June* 1990)

Abstract. Myrotoxin B hydrate ethyl acetate solvate, $C_{29}H_{36}O_{12}.C_4H_8O_2$, $M_r = 664{\cdot}71$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}911$ (1), $b = 15{\cdot}366$ (2), $c = 22{\cdot}548$ (3) Å, $V = 3434{\cdot}1$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}286$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 0{\cdot}81$ mm^{-1}, $F(000) = 1416$, $T = 295$ K, final $R = 0{\cdot}048$, $wR = 0{\cdot}063$ for 3131 independent reflections with $F_o > 3\sigma F_o$. In the macrocyclic ring the two hydroxyls of the tetrahydropyranyl ring are *cis* to each other with the hydroxyl on C(12)′ axial to the ring. The solvate molecules pack in channels parallel to the *a* axis, and there is an intermolecular hydrogen bond between the two hydroxyls with the axial hydroxyl acting as a donor [H···O = 1·83 (5), O···O = 2·735 (7) Å, and ∠O—H···O = 170 (3)°].

Experimental. A clear colorless 0·30 × 0·45 × 0·64 mm data crystal was provided by Bruce Jarvis of the University of Maryland. Automated Siemens *R*3*m*/*V* diffractometer with incident beam monochromator. 25 centered reflections within $39 \le 2\theta \le 78°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}59$ Å^{-1}, range of *hkl*: $0 \le h \le 11$, $0 \le k \le 18$, $0 \le l \le 25$. Standards 4,0,0; 0,8,0; 0,0,10, monitored every 60 reflections with random variation of 2·7% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (2·0° min^{-1} minimum, 15·0° min^{-1} maximum) in ω, 3482 reflections measured, 3300 unique, $R_{int} = 1{\cdot}1\%$, 3131 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz, polarization and absorption effects, max. and min. transmission 0·84 and 0·76. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00023$. Secondary-extinction parameter $p = 0{\cdot}0028$ (5) in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin(2\theta)]^{0{\cdot}25}$. There were 448 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included

0108-2701/91/010218-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

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

	x	y	z	U_{eq}
O(1)	4287 (3)	2269 (2)	3039 (1)	71 (1)
O(2)	1593 (3)	3894 (3)	2997 (2)	88 (1)
O(3)	6194 (2)	4567 (1)	1781 (1)	51 (1)
O(4)	4864 (2)	5771 (2)	−428 (1)	63 (1)
O(5)	2019 (2)	3634 (2)	1606 (1)	66 (1)
O(6)	7875 (2)	4291 (2)	1144 (1)	67 (1)
O(7)	7857 (2)	6168 (2)	885 (1)	59 (1)
O(8)	6479 (3)	7227 (2)	−75 (1)	77 (1)
O(9)	3372 (3)	3996 (2)	845 (1)	72 (1)
O(10)	3901 (3)	7072 (2)	−256 (1)	70 (1)
O(11)	8257 (3)	4182 (2)	2801 (1)	64 (1)
O(12)	8218 (4)	5362 (2)	3384 (2)	94 (1)
C(2)	2948 (4)	2563 (3)	2884 (2)	74 (2)
C(3)	2577 (5)	2406 (3)	2240 (2)	84 (2)
C(4)	3134 (4)	3210 (3)	1906 (2)	62 (1)
C(5)	3740 (3)	3817 (2)	2387 (2)	52 (1)
C(6)	5258 (3)	3538 (2)	2496 (2)	49 (1)
C(7)	5860 (3)	4015 (2)	3034 (2)	52 (1)
C(8)	7286 (3)	3743 (3)	3186 (2)	55 (1)
C(9)	7544 (4)	2786 (3)	3129 (2)	57 (1)
C(10)	6636 (4)	2266 (3)	2893 (2)	61 (1)
C(11)	5323 (4)	2549 (2)	2629 (2)	58 (1)
C(12)	2929 (4)	3525 (3)	2924 (2)	66 (1)
C(13)	2587 (5)	4012 (4)	3451 (2)	86 (2)
C(14)	3550 (4)	4789 (2)	2255 (2)	57 (1)
C(15)	6146 (3)	3661 (2)	1945 (2)	49 (1)
C(16)	8880 (4)	2461 (3)	3369 (2)	74 (1)
C(17)	8621 (4)	4983 (3)	2952 (2)	67 (1)
C(18)	9556 (4)	5375 (3)	2505 (3)	85 (2)
C(1′)	7048 (3)	4778 (2)	1345 (1)	44 (1)
C(2′)	6765 (3)	5689 (2)	1153 (1)	44 (1)
C(3′)	6708 (3)	5930 (2)	517 (2)	49 (1)
C(4′)	6979 (4)	5308 (3)	25 (2)	61 (1)
C(5′)	5622 (4)	5038 (3)	−237 (2)	66 (1)
C(6′)	4524 (3)	6357 (2)	35 (2)	54 (1)
C(7′)	3497 (3)	5989 (2)	465 (2)	52 (1)
C(8′)	2345 (3)	5510 (3)	143 (2)	63 (1)
C(9′)	1205 (3)	5260 (3)	540 (2)	68 (1)
C(10′)	1168 (3)	4615 (3)	935 (2)	65 (1)
C(11′)	2306 (3)	4061 (3)	1100 (2)	59 (1)
C(12′)	5823 (3)	6683 (2)	347 (2)	55 (1)
C(101)	9118 (33)	2167 (14)	936 (16)	582 (24)
C(102)	8023 (20)	1603 (15)	1203 (11)	367 (20)
O(103)	6993 (21)	1792 (8)	734 (5)	292 (8)
C(104)	5771 (19)	1497 (14)	872 (7)	487 (24)
C(105)	4645 (26)	1739 (18)	480 (9)	512 (25)
O(106)	5781 (18)	1171 (12)	1361 (8)	379 (11)

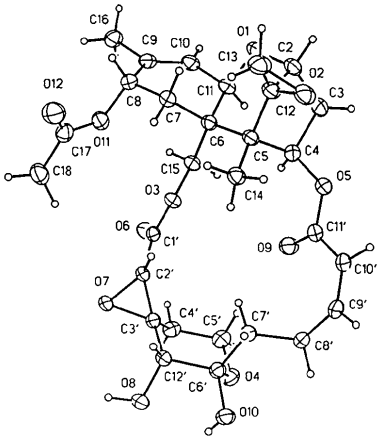

Fig. 1. Thermal ellipsoid plot of Myrotoxin B hydrate drawn at the 20% probability level. The ethyl acetate solvate molecule is omitted for clarity. The chirality is chosen to conform to the absolute configuration of Verrucarin A.

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

O(1)—C(2)	1·445 (5)	O(1)—C(11)	1·446 (5)
O(2)—C(12)	1·449 (5)	O(2)—C(13)	1·433 (6)
O(3)—C(15)	1·443 (4)	O(3)—C(1′)	1·338 (4)
O(4)—C(5′)	1·420 (5)	O(4)—C(6′)	1·420 (4)
O(5)—C(4)	1·451 (5)	O(5)—C(11′)	1·345 (5)
O(6)—C(1′)	1·199 (4)	O(7)—C(2′)	1·442 (4)
O(7)—C(3′)	1·456 (4)	O(8)—C(12′)	1·423 (5)
O(9)—C(11′)	1·208 (4)	O(10)—C(6′)	1·421 (5)
O(11)—C(8)	1·461 (4)	O(11)—C(17)	1·327 (5)
O(12)—C(17)	1·202 (6)	C(2)—C(3)	1·518 (7)
C(2)—C(12)	1·481 (6)	C(3)—C(4)	1·548 (6)
C(4)—C(5)	1·552 (5)	C(5)—C(6)	1·583 (5)
C(5)—C(12)	1·522 (5)	C(5)—C(14)	1·535 (5)
C(6)—C(7)	1·539 (5)	C(6)—C(11)	1·550 (5)
C(6)—C(15)	1·534 (5)	C(7)—C(8)	1·513 (5)
C(8)—C(9)	1·497 (5)	C(9)—C(10)	1·316 (5)
C(9)—C(16)	1·515 (6)	C(10)—C(11)	1·496 (5)
C(12)—C(13)	1·444 (7)	C(17)—C(18)	1·496 (7)
C(1′)—C(2′)	1·493 (5)	C(2′)—C(3′)	1·482 (5)
C(3′)—C(4′)	1·488 (5)	C(3′)—C(12′)	1·502 (5)
C(4′)—C(5′)	1·527 (6)	C(6′)—C(7′)	1·514 (5)
C(6′)—C(12′)	1·550 (5)	C(7′)—C(8′)	1·539 (5)
C(8′)—C(9′)	1·492 (5)	C(9′)—C(10′)	1·332 (6)
C(10′)—C(11′)	1·461 (5)	C(101)—C(102)	1·513 (37)
C(102)—O(103)	1·499 (27)	O(103)—C(104)	1·330 (27)
C(104)—C(105)	1·472 (30)	C(104)—O(106)	1·212 (25)

C(2)—O(1)—C(11)	113·9 (3)	C(12)—O(2)—C(13)	60·2 (3)
C(15)—O(3)—C(1′)	116·3 (2)	C(5′)—O(4)—C(6′)	113·9 (3)
C(4)—O(5)—C(11′)	117·0 (3)	C(2′)—O(7)—C(3′)	61·5 (2)
C(8) O(11) C(17)	117·0 (3)	O(1)—C(2)—C(3)	113·8 (4)
O(1)—C(2)—C(12)	108·0 (3)	C(3)—C(2)—C(12)	102·4 (4)
C(2)—C(3)—C(4)	104·6 (4)	O(5)—C(4)—C(3)	108·3 (3)
O(5)—C(4)—C(5)	110·6 (3)	C(3)—C(4)—C(5)	106·1 (3)
C(4)—C(5)—C(6)	108·3 (3)	C(4)—C(5)—C(12)	100·1 (3)
C(6)—C(5)—C(12)	107·4 (3)	C(4)—C(5)—C(14)	113·7 (3)
C(6)—C(5)—C(14)	114·2 (3)	C(12)—C(5)—C(14)	112·1 (3)
C(5)—C(6)—C(7)	111·2 (3)	C(5)—C(6)—C(11)	109·6 (3)
C(7)—C(6)—C(11)	107·3 (3)	C(5)—C(6)—C(15)	112·8 (3)
C(7)—C(6)—C(15)	110·9 (3)	C(11)—C(6)—C(15)	104·7 (3)
C(6)—C(7)—C(8)	114·2 (3)	O(11)—C(8)—C(7)	110·7 (3)
O(11)—C(8)—C(9)	106·9 (3)	C(7)—C(8)—C(9)	114·3 (3)
C(8)—C(9)—C(10)	121·0 (3)	C(8)—C(9)—C(16)	116·3 (3)
C(10)—C(9)—C(16)	122·8 (4)	C(9)—C(10)—C(11)	125·3 (4)
O(1)—C(11)—C(6)	112·8 (3)	O(1)—C(11)—C(10)	106·1 (3)
C(6)—C(11)—C(10)	113·5 (3)	O(2)—C(12)—C(2)	114·2 (3)
O(2)—C(12)—C(5)	117·2 (3)	C(2)—C(12)—C(5)	103·8 (3)
O(2)—C(12)—C(13)	59·4 (3)	C(2)—C(12)—C(13)	124·8 (4)
C(5)—C(12)—C(13)	128·7 (4)	O(2)—C(13)—C(12)	60·5 (3)
O(3)—C(15)—C(6)	110·2 (3)	O(11)—C(17)—O(12)	124·5 (4)
O(11)—C(17)—C(18)	111·5 (4)	O(12)—C(17)—C(18)	123·9 (4)
O(3)—C(1′)—O(6)	124·0 (3)	O(3)—C(1′)—C(2′)	108·7 (2)
O(6)—C(1′)—C(2′)	127·2 (3)	O(7)—C(2′)—C(1′)	117·3 (2)
O(7)—C(2′)—C(3′)	59·7 (2)	C(1′)—C(2′)—C(3′)	121·4 (3)
O(7)—C(3′)—C(2′)	58·8 (2)	O(7)—C(3′)—C(4′)	116·5 (3)
C(2′)—C(3′)—C(4′)	123·6 (3)	O(7)—C(3′)—C(12′)	114·1 (3)
C(2′)—C(3′)—C(12′)	117·4 (3)	C(4′)—C(3′)—C(12′)	114·3 (3)
C(3′)—C(4′)—C(5′)	107·7 (3)	O(4)—C(5′)—C(4′)	111·6 (3)
O(4)—C(6′)—O(10)	104·7 (3)	O(4)—C(6′)—C(7′)	113·1 (3)
O(10)—C(6′)—C(7′)	106·9 (3)	O(4)—C(6′)—C(12′)	110·0 (3)
O(10)—C(6′)—C(12′)	108·7 (3)	C(7′)—C(6′)—C(12′)	112·9 (3)
C(6′)—C(7′)—C(8′)	112·1 (3)	C(7′)—C(8′)—C(9′)	113·7 (3)
C(8′)—C(9′)—C(10′)	127·8 (3)	C(9′)—C(10′)—C(11′)	125·6 (3)
O(5)—C(11′)—O(9)	123·3 (3)	O(5)—C(11′)—C(10′)	109·7 (3)
O(9)—C(11′)—C(10′)	127·0 (4)	O(8)—C(12′)—C(3′)	110·8 (3)
O(8)—C(12′)—C(6′)	105·4 (3)	C(3′)—C(12′)—C(6′)	110·5 (3)
C(101)—C(102)—O(103)	95·5 (19)	C(102)—O(103)—C(104)	112·9 (14)
O(103)—C(104)—C(105)	117·7 (17)	O(103)—C(104)—O(106)	110·3 (17)
C(105)—C(104)—O(106)	131·1 (19)		

using riding model (coordinate shifts of C applied to attached H atoms), C—H = 0·96 Å, H angles idealized, U(H) = 1·1 U_{eq}(C), hydroxyl hydrogens refined isotropically, ethyl acetate solvate refined with bond distance and next nearest neighbor distance constraints. $(\Delta/\sigma)_{max}$ = 1·1 in the solvent, ratio of observations to parameters 7·0:1, R = 0·048, wR = 0·063, S = 2·78. R = 0·050 for all data. Final difference Fourier excursions 0·21 and −0·26 e $Å^{-3}$. Atomic

scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 shows the atomic coordinates, Table 2 lists bond distances and angles, and the molecule is plotted in Fig. 1.*

Related literature. A number of macrocyclic trichothecenes have been reported. These include Verrucarin A (McPhail & Sim, 1966), Verrucarin B (Breitenstein, Tamm, Arnold & Clardy, 1979), Baccharin (Kupchan, Jarvis, Dailey, Bright, Bryan & Yoshikazu, 1976) and Roridan A (Jarvis, Midiwo, Flippen-Anderson & Mazzola, 1982). The structures of Myrotoxin A and C (Jarvis, Cömezoğlu, Lee, Flippen-Anderson, Gilardi & George, 1986) which were produced from the same *myrothecium roridum* culture as Myrotoxin B hydrate share a nearly identical conformation for the trichothecene ring. The conformation of the common portion of the macrocyclic ring has its largest differences in the area of the tetrahydropyranyl ring due to differences in hydrogen bonding and packing.

This work was supported in part by the Office of Naval Research.

* 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 53301 (18pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BREITENSTEIN, W., TAMM, C., ARNOLD, E. V. & CLARDY, J. (1979). *Helv. Chim. Acta*, **62**, 2067–2073.

JARVIS, B. B., CÖMEZOĞLU, F. T., LEE, Y. M., FLIPPEN-ANDERSON, J. L., GILARDI, R. D. & GEORGE, C. (1986). *Bull. Soc. Chim. Belg.* **95**, 681–697.

JARVIS, B. B., MIDIWO, J. O., FLIPPEN-ANDERSON, J. L. & MAZZOLA, E. P. (1982). *J. Nat. Prod.* **45**, 440–448.

KUPCHAN, S. M., JARVIS, B. B., DAILEY, R. G. JR, BRIGHT, W., BRYAN, R. F. & YOSHIKAZU, S. (1976). *J. Am. Chem. Soc.* **98**, 7092–7093.

MCPHAIL, A. T. & SIM, G. A. (1966). *J. Chem. Soc. C*, pp. 1394–1396.

SHELDRICK, G. M. (1980). *SHELXTL*80. *An Integrated System for Solving, Refining, and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1991). C**47**, 220–221

1-Fluoro-2,4-dinitrobenzene

BY A. WILKINS* AND R. W. H. SMALL

The Chemistry Department, The University, Lancaster, England

(*Received* 15 *February* 1990; *accepted* 25 *June* 1990)

Abstract. $C_6H_3FN_2O_4$, $M_r = 186{\cdot}0$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}210\ (5)$, $b = 9{\cdot}45\ (1)$, $c = 12{\cdot}85\ (1)$ Å, $V = 754{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}638$, $D_m = 1{\cdot}63$ g cm^{-3}, $F(000) = 376$, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}106$ mm^{-1}, $T = 293$ K. Diffractometer data, $R = 0{\cdot}057$ for 611 unique observed reflexions. The nitro group *ortho* to F is twisted 16·0 (7)° relative to the plane of the benzene ring giving F···O of 2·575 (7) Å; the *para* nitro group is twisted through 8·4 (7)°.

Experimental. Commercial sample, recrystallized from ethanol, pale yellow prisms, melting point 299 K, density by flotation, crystal size 0·5 × 0·4 × 0·4 mm. Crystal in capillary tube on STADI-2 two-circle diffractometer, cell dimensions from setting angles of 24 reflexions in the range $20 < 2\theta < 40°$. Intensities for layers $0kl$ to $7kl$; k, 0 to 10; l, 0 to 17; standard every 20 reflexions, maximum $\sin\theta/\lambda$ 0·70 Å^{-1}, measured on the STADI-2, variable ω scan, $2\theta'$ fixed, stationary background count. Lp corrections but no absorption correction, 1185 unique measured reflexions of which 611 with $I >$

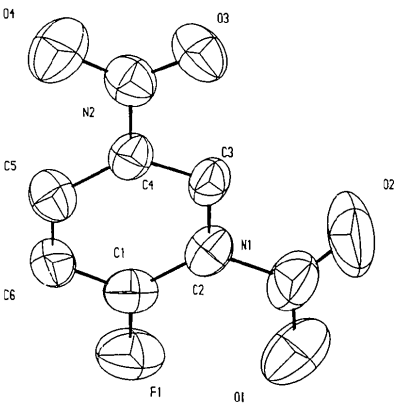

Fig. 1. Molecule of 1-fluoro-2,4-dinitrobenzene, showing atomic labelling and 50% thermal ellipsoids.

* Present address: Logica Energy & Industry Systems Ltd, Regal House, Duke St., Stockport, England.

0108-2701/91/010220-02$03.00