C1—O1—C6	107.2 (5)	O4—C4—C5	111.2 (6)
C7—O2—C2	113.5 (6)	O4—C4—C3	105.1 (6)
C8—O4—C4	117.6 (5)	C5—C4—C3	108.8 (6)
C1—O5—C5	101.3 (5)	O4—C4—C15	109.5 (5)
O5—C1—O1	105.8 (6)	C5—C4—C15	111.7 (6)
O5—C1—C2	110.4 (6)	C3—C4—C15	110.2 (6)
O1—C1—C2	109.2 (6)	O5—C5—C4	109.6 (6)
O2—C2—C1	104.3 (6)	O5—C5—C6	100.6 (5)
O2—C2—C3	116.0 (6)	C4—C5—C6	115.1 (6)
C1—C2—C3	109.3 (6)	O1—C6—C5	103.3 (5)
C4—C3—C2	114.1 (6)	O4—C8—C9	109.2 (6)
C5—O5—C1—O1	−43.2 (7)	C2—C3—C4—C5	−41.5 (8)
C5—O5—C1—C2	74.8 (7)	C2—C3—C4—C15	−164.3 (6)
C6—O1—C1—O5	22.5 (8)	C1—O5—C5—C4	−76.4 (7)
C6—O1—C1—C2	−96.3 (7)	C1—O5—C5—C6	45.2 (7)
C7—O2—C2—C1	−177.2 (5)	O4—C4—C5—O5	−55.1 (7)
C7—O2—C2—C3	−57.1 (8)	C3—C4—C5—O5	60.2 (7)
O5—C1—C2—O2	66.6 (7)	C15—C4—C5—O5	−177.9 (5)
O1—C1—C2—O2	−177.5 (5)	O4—C4—C5—C6	−167.6 (5)
O5—C1—C2—C3	−58.0 (7)	C3—C4—C5—C6	−52.3 (7)
O1—C1—C2—C3	57.9 (7)	C1—O1—C6—C5	6.3 (7)
O2—C2—C3—C4	−77.5 (8)	O5—C5—C6—O1	−31.8 (7)
C1—C2—C3—C4	40.0 (8)	C4—C5—C6—O1	85.9 (7)
C8—O4—C4—C5	−61.0 (7)	C4—O4—C8—C9	−171.8 (5)
C8—O4—C4—C3	−178.6 (5)	O4—C8—C9—C10	26.2 (9)
C2—C3—C4—O4	77.8 (7)	O4—C8—C9—C14	−152.5 (6)

For both compounds, data collection: *R3m Software* (Siemens, 1983); cell refinement: *R3m Software*; data reduction: *SHELXTL* (Sheldrick, 1983); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1993).

Lists of structure factors, torsion angles, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1065). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Blattner, R., Furneaux, R. H., Mason, J. M. & Tyler, P. C. (1991). *Pestic. Sci.* **31**, 419–435.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Furneaux, R. H., Henzell, R. F. & Tyler, P. C. (1991). US Patent 5 047 518

Furneaux, R. H., Mason, J. M. & Tyler, P. C. (1995). Unpublished results.

Furneaux, R. H., Mason, J. M., Tyler, P. C., Blattner, R., Lawson, K. R., Chrystal, E. J. T., Mitchell, G. & Pilkington, B. L. (1989). European Patent Application 0 302 599.

Gainsford, G. J., Furneaux, R. H., Mason, J. M. & Tyler, P. C. (1995). *Acta Cryst.* **C51**, 2418–2421.

Jeffrey, G. A., Pople, J. A. & Radom, L. (1974). *Carbohydr. Res.* **38**, 81–95.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1983). *SHELXTL User's Manual*. Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1983). *R3m Software*. Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1996). **C52**, 1277–1280

Pyrrolo-Annellated Carbazoles as Potential Antitumour-Active Compounds: Dimethyl 4-Methoxy-9-methyl-9*H*-carbazole-1,2-dicarboxylate and 5-Hydroxy-2-phenyl-10-methyl-1,2,3,10-tetrahydropyrrolo[3,4-*a*]carbazole-1,3-dione Methanol Solvate

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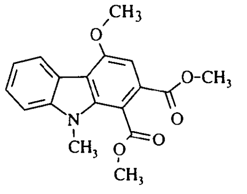
Abstract

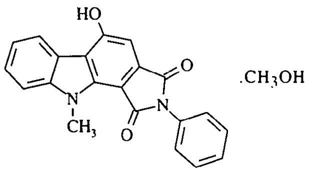
The structures of the title compounds, $C_{18}H_{17}NO_5$ and $C_{21}H_{14}N_2O_3.CH_4O$, were determined. The crystal structure of the first shows significant variations between its two independent molecules; that of the second is characterized by hydrogen bonds to the co-crystallized methanol.

Comment

Selectively functionalized and annellated carbazoles as well as structurally related alkaloids are interesting target compounds for the development of antitumour and antibiotically active drugs (Chakraborty & Roy, 1991; Gribble, 1990; Pindur, 1987; Kansal & Poitier, 1986; Poitier, 1992). Due to the planar chromophore of the carbazoles, the pharmacological activity is based, first of all, on complex formation with human B-DNA (Gribble, 1990). In recent years we have synthesized a variety of functionalized carbazole and annellated carbazole derivatives in order to obtain more details on structure–activity relationships (Pindur, Haber & Erfanian-Abdoust, 1992; Pindur, Pfeuffer, Eitel, Rogge & Haber, 1991; Pindur, 1995). In this context, current molecular-modelling studies of carbazole–DNA interactions were performed (Pindur, Haber & Erfanian-Abdoust, 1992; Dräger, Haber, Erfanian-Abdoust, Pindur & Sattler, 1993; Sattler, 1994; Rehn, 1995) on the basis of molecular mechanics, molecular dynamics and quantum chemistry calculations. To obtain detailed geometrical information on the molecules interacting with DNA, X-ray analyses of the potential drugs are necessary. As a continuation of our investigations in this field, we report the crystal structures of the title compounds, (1) and (2), which will be helpful in designing new antitumour-active drugs.



(1)



(2)

Crystals of (1) have two independent molecules in the unit cell (Fig. 1). The methyl ester group connected to C21 (IUPAC numbering: C1) of the independent molecule *B* (Fig. 1) is disordered with a population ratio of 40:60 by rotating the group about C21—C33 by 180°. Significant deviations ($>3\sigma$) in bond lengths and bond angles between the independent molecules can be found, especially in the methyl ester groups. The 14π carbazole system of (1) is planar.

Crystals of compound (2) contain one methanol molecule per carbazole derivative (Fig. 2), forming typical hydrogen bonds (Brown, 1976) as shown in Fig. 3 [O2···H4O*M* 1.736 (4), O4*M*···O2 2.807 (4) Å, O4*M*—H4O*M*···O2 168.3 (3)°; O4*M*···H3O 1.702 (4), O4*M*···O3 2.645 (4) Å, O4*M*—H3O···O3 165.7 (3)°]. Compound (2) is planar in the pyrrolo-carbazole system; the dihedral angle between its best plane and that of the benzene partial structure is 54.02°.

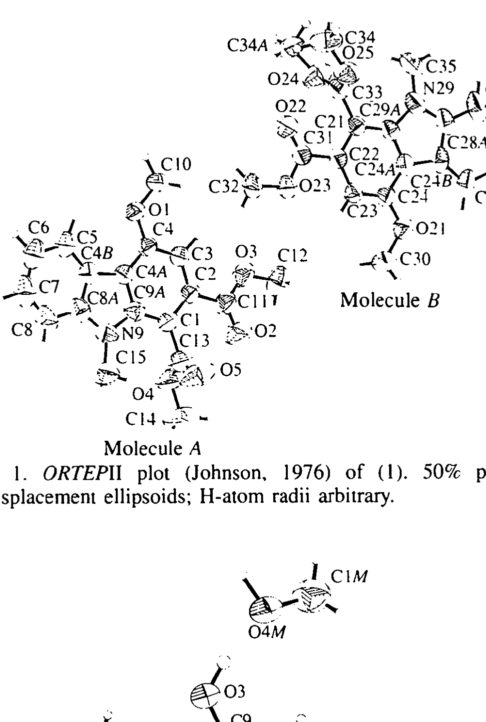


Fig. 1. *ORTEP*II plot (Johnson, 1976) of (1). 50% probability displacement ellipsoids; H-atom radii arbitrary.

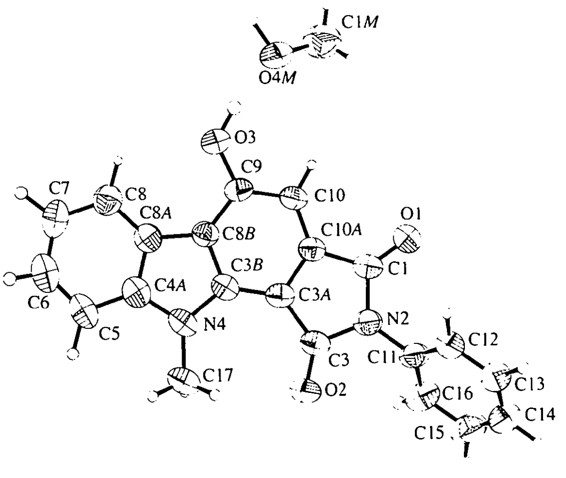


Fig. 2. *ORTEP*II plot (Johnson, 1976) of (2). 50% probability displacement ellipsoids; H-atom radii arbitrary.

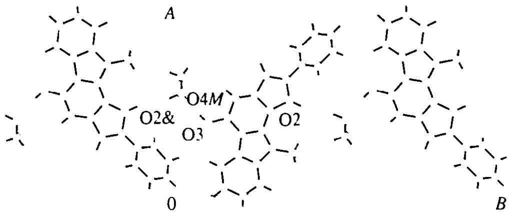


Fig. 3. Part of the crystal packing (Motherwell & Clegg, 1978) of (2) viewed down the *c* axis. The zigzag arrangement of the hydrogen bonding is indicated by broken lines.

Experimental

Compound (1) is readily available from a Diels–Alder reaction of *in situ* generated 1-(*N*-methylindolyl)-1-methoxyethene and dimethyl acetylene dicarboxylate followed by spontaneous dehydrogenation (Pindur, Rogge, Rehn, Massa & Peschel, 1994). The [*a*]-annellated carbazole of compound (2) is readily available from *in situ* generated 1-(*N*-trimethylsilylindol-3-yl)-1-(trimethylsilyloxy)ethene and *N*-phenylmaleimide; the [4+2] cycloadduct primarily formed is dehydrogenated with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (Rogge, Fischer, Schollmeyer & Pindur, 1996). Crystals of (1) were obtained from chloroform by slow evaporation; crystals of (2) by slow diffusion of water in a water–methanol solution (1:10).

Compound (1)

Crystal data

$C_{18}H_{17}NO_5$
$M_r = 327.34$
Monoclinic
$P2_1/c$
$a = 6.8415 (3)$ Å
$b = 24.411 (3)$ Å
$c = 19.377 (1)$ Å
$\beta = 95.888 (6)°$
$V = 3219.1 (3)$ Å^3
$Z = 8$
$D_x = 1.350$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 75 reflections
$\theta = 60$–$70°$
$\mu = 0.734$ mm^{-1}
$T = 298 (2)$ K
Colourless
0.8 × 0.19 × 0.06 mm
Rectangular prism

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6818 measured reflections
5236 independent reflections
4182 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.031$
$\theta_{\rm max} = 70°$

$h = 0 \rightarrow 9$
$k = 0 \rightarrow 30$
$l = -23 \rightarrow 23$
3 standard reflections
frequency: 66.7 min
intensity decay: 5% [corrected with a cubic spline function (*CORINC*; Dräger & Gattow, 1971)]

Refinement

Refinement on F
$R = 0.06$
$wR = 0.07$
$S = 6.88$
4182 reflections
434 parameters
H atoms riding with fixed U_{iso}
$w = 1/[\sigma^2(F_o) + 0.0002F_o^2]$
$(\Delta/\sigma)_{\text{max}} = 0.001$
$\Delta\rho_{\text{max}} = 0.387$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.278$ e Å^{-3}
Extinction correction: *SHELX*76 (Sheldrick, 1976)
Extinction coefficient: 0.0030 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (1)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.4334 (5)	0.6870 (1)	0.8113 (2)	0.060 (1)
C2	0.2739 (5)	0.7208 (1)	0.7910 (1)	0.060 (1)
C3	0.2328 (5)	0.7388 (1)	0.7224 (1)	0.058 (1)
C4	0.3505 (4)	0.7234 (1)	0.6722 (1)	0.0527 (10)
C4*A*	0.5155 (4)	0.6904 (1)	0.6914 (1)	0.0526 (10)
C4*B*	0.6659 (5)	0.6690 (1)	0.6524 (2)	0.058 (1)
C5	0.7029 (5)	0.6715 (1)	0.5822 (2)	0.068 (1)
C6	0.8624 (6)	0.6438 (2)	0.5629 (2)	0.087 (2)
C7	0.9879 (6)	0.6147 (2)	0.6121 (3)	0.098 (2)
C8	0.9576 (6)	0.6130 (1)	0.6802 (2)	0.091 (2)
C8*A*	0.7922 (5)	0.6393 (1)	0.7000 (2)	0.069 (1)
N9	0.7260 (4)	0.6418 (1)	0.7653 (2)	0.071 (1)
C15	0.8286 (7)	0.6164 (2)	0.8268 (2)	0.101 (2)
C9*A*	0.5554 (5)	0.6732 (1)	0.7603 (2)	0.059 (1)
O1	0.3222 (3)	0.73728 (9)	0.6041 (1)	0.0654 (8)
C10	0.1528 (5)	0.7697 (1)	0.5815 (2)	0.071 (1)
C11	0.1439 (6)	0.7378 (2)	0.8440 (2)	0.070 (1)
O2	0.1598 (5)	0.7233 (2)	0.9020 (1)	0.137 (2)
O3	0.0022 (4)	0.7715 (1)	0.8200 (1)	0.092 (1)
C12	−0.1305 (6)	0.7924 (2)	0.8670 (2)	0.096 (2)
C13	0.4665 (6)	0.6636 (2)	0.8829 (2)	0.076 (1)
O4	0.5754 (4)	0.6950 (1)	0.9260 (1)	0.092 (1)
O5	0.4063 (6)	0.6199 (1)	0.8983 (1)	0.124 (2)
C14	0.6105 (9)	0.6727 (2)	0.9955 (2)	0.141 (3)
C21	0.1575 (4)	0.9322 (1)	0.7003 (1)	0.0516 (10)
C22	0.3130 (4)	0.9013 (1)	0.7327 (2)	0.0534 (10)
C23	0.3305 (4)	0.8900 (1)	0.8042 (2)	0.056 (1)
C24	0.1946 (5)	0.9098 (1)	0.8451 (2)	0.057 (1)
C24*A*	0.0370 (4)	0.9408 (1)	0.8148 (2)	0.055 (1)
C24*B*	−0.1263 (5)	0.9667 (1)	0.8431 (2)	0.065 (1)
C25	−0.1802 (6)	0.9721 (1)	0.9107 (2)	0.081 (1)
C26	−0.3518 (7)	1.0017 (2)	0.9183 (3)	0.105 (2)
C27	−0.4610 (6)	1.0245 (2)	0.8621 (3)	0.107 (2)
C28	−0.4103 (5)	1.0204 (1)	0.7961 (3)	0.090 (2)
C28*A*	−0.2394 (5)	0.9910 (1)	0.7868 (2)	0.069 (1)
N29	−0.1523 (4)	0.9818 (1)	0.7261 (2)	0.0657 (10)
C35	−0.2311 (5)	1.0006 (1)	0.6586 (2)	0.085 (1)
C29*A*	0.0186 (4)	0.9513 (1)	0.7430 (2)	0.054 (1)
O21	0.1978 (4)	0.9021 (1)	0.9146 (1)	0.0744 (9)
C30	0.3578 (6)	0.8716 (2)	0.9485 (2)	0.083 (1)
C31	0.4631 (5)	0.8801 (1)	0.6891 (2)	0.056 (1)
O22	0.4616 (4)	0.8873 (1)	0.6288 (1)	0.092 (1)
O23	0.6044 (3)	0.8516 (1)	0.7254 (1)	0.0806 (9)
C32	0.7522 (5)	0.8285 (2)	0.6862 (2)	0.085 (1)
C33	0.1470 (5)	0.9454 (1)	0.6244 (2)	0.063 (1)
O24	0.2304 (5)	0.9880 (1)	0.6068 (1)	0.098 (1)
O25	0.0532 (4)	0.9123 (1)	0.5829 (1)	0.0813 (9)
C34†	0.068 (1)	0.9176 (3)	0.5097 (3)	0.103 (3)
C34*A*†	0.282 (2)	0.9966 (5)	0.5385 (5)	0.120 (5)

† Rotationally disordered (see *Comment*).

Table 2. *Selected geometric parameters (Å, °) for (1)*

C1—C13	1.495 (5)	C21—C33	1.499 (4)
C2—C11	1.486 (5)	C22—C31	1.487 (4)
C4—O1	1.356 (3)	C24—O21	1.358 (4)
C4*A*—C4*B*	1.436 (4)	C24*A*—C24*B*	1.439 (4)
C8*A*—N9	1.388 (5)	C28*A*—N29	1.390 (5)
N9—C15	1.458 (5)	N29—C35	1.438 (5)
N9—C9*A*	1.392 (4)	N29—C29*A*	1.395 (4)
O1—C10	1.435 (4)	O21—C30	1.427 (5)
C11—O2	1.173 (4)	C31—O22	1.182 (4)
C11—O3	1.318 (5)	C31—O23	1.333 (4)
O3—C12	1.443 (5)	O23—C32	1.441 (4)
C13—O4	1.309 (5)	C33—O24	1.251 (4)
C13—O5	1.193 (5)	C33—O25	1.268 (4)
O4—C14	1.449 (5)	O24—C34*A*	1.42 (1)
C21—C29*A*	1.403 (4)	O25—C34	1.438 (6)
C13—C1—C2	121.9 (3)	C33—C21—C22	120.9 (2)
C11—C2—C1	118.5 (3)	C31—C22—C21	118.0 (2)
O1—C4—C3	125.6 (2)	N29—C29*A*—C21	129.9 (2)
N9—C8*A*—C4*B*	110.0 (3)	O21—C24—C23	125.6 (2)
N9—C8*A*—C8	128.5 (3)	N29—C28*A*—C24*B*	109.8 (3)
C15—N9—C8*A*	123.5 (3)	N29—C28*A*—C28	129.0 (3)
C9*A*—N9—C8*A*	108.1 (3)	C35—N29—C28*A*	124.4 (3)
C9*A*—N9—C15	128.4 (3)	C29*A*—N29—C28*A*	108.0 (2)
N9—C9*A*—C1	129.8 (2)	N29—C29*A*—C24*A*	108.5 (2)
N9—C9*A*—C4*A*	108.2 (3)	C30—O21—C24	117.6 (2)
C10—O1—C4	117.8 (2)	O22—C31—C22	125.0 (3)
O2—C11—C2	125.1 (3)	O23—C31—C22	112.8 (2)
O3—C11—C2	113.6 (3)	C32—O23—C31	115.9 (2)
C12—O3—C11	119.0 (3)	O24—C33—C21	118.0 (2)
O4—C13—C1	113.0 (3)	O25—C33—C21	117.1 (2)
O5—C13—C1	123.4 (3)	C34*A*—O24—C33	122.8 (5)
O5—C13—O4	123.6 (3)	C34—O25—C33	119.1 (3)
C14—O4—C13	113.7 (3)	C29*A*—N29—C35	127.6 (2)
C2—C1—C13—O5	92.4 (4)	C1—C2—C11—O2	−3.1 (4)
C9*A*—C1—C13—O4	93.5 (4)	C22—C21—C33—O24	−88.8 (4)
C3—C2—C11—O3	−2.8 (4)	C23—C22—C31—O22	180.0 (3)
C3—C4—O1—C10	−1.5 (3)	C29*A*—C21—C33—O25	−90.9 (3)
C2—C3—C4—O1	178.4 (3)	C21—C22—C31—O23	179.7 (3)
C23—C22—C31—O23	−0.1 (3)	C22—C23—C24—O21	−179.2 (3)
C2—C11—O3—C12	−178.0 (4)	C23—C24—O21—C30	1.1 (4)
C1—C13—O4—C14	−179.4 (4)	C29*A*—C21—C33—O24	89.0 (4)
O5—C13—O4—C14	−2.2 (5)	O25—C33—O24—C34*A*	−19.7 (7)

Compound (2)

Crystal data

$C_{21}H_{14}N_2O_3.CH_4O$
$M_r = 374.38$
Orthorhombic
$Pca2_1$
$a = 22.028$ (2) Å
$b = 11.698$ (1) Å
$c = 7.0116$ (3) Å
$V = 1806.8$ (2) Å^3
$Z = 4$
$D_x = 1.376$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 60 reflections
$\theta = 60$–$70°$
$\mu = 0.786$ mm^{-1}
$T = 293$ (2) K
Rectangular prism
0.58 × 0.10 × 0.06 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3683 measured reflections
3386 independent reflections
2466 observed reflections [$I > 2\sigma(I)$]
$R_{\text{int}} = 0.036$
$\theta_{\text{max}} = 69.94°$
$h = -26 \rightarrow 26$
$k = -14 \rightarrow 14$
$l = -8 \rightarrow 8$
3 standard reflections
frequency: 66.7 min
intensity decay: 5% [corrected with a cubic spline function (*CORINC*; Dräger & Gattow, 1971)]

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0628$
$wR(F^2) = 0.1852$
$S = 1.009$
3386 reflections
267 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.1012P)^2 + 0.6799P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.172$ e Å^{-3}
$\Delta\rho_{\min} = -0.225$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.2668 (2)	0.6717 (3)	0.1634 (6)	0.0502 (10)
N2	0.32849 (14)	0.6482 (3)	0.1852 (5)	0.0507 (8)
C3	0.3383 (2)	0.5302 (3)	0.2154 (6)	0.0475 (9)
C3*A*	0.2777 (2)	0.4765 (3)	0.2183 (6)	0.0454 (8)
C3*B*	0.2556 (2)	0.3651 (3)	0.2452 (6)	0.0455 (8)
N4	0.2844 (2)	0.2621 (3)	0.2776 (5)	0.0520 (8)
C4*A*	0.2396 (2)	0.1783 (3)	0.2976 (6)	0.0535 (10)
C5	0.2477 (3)	0.0608 (4)	0.3339 (8)	0.0675 (13)
C6	0.1962 (3)	−0.0027 (4)	0.3484 (8)	0.0739 (14)
C7	0.1386 (3)	0.0414 (4)	0.3276 (8)	0.0714 (14)
C8	0.1304 (2)	0.1577 (4)	0.2905 (7)	0.0606 (11)
C8*A*	0.1824 (2)	0.2259 (4)	0.2770 (6)	0.0498 (9)
C8*B*	0.1920 (2)	0.3466 (3)	0.2445 (6)	0.0452 (9)
C9	0.1506 (2)	0.4355 (3)	0.2138 (6)	0.0465 (9)
C10	0.1727 (2)	0.5459 (3)	0.1854 (6)	0.0473 (9)
C10*A*	0.2344 (2)	0.5621 (3)	0.1884 (5)	0.0433 (8)
C11	0.3752 (2)	0.7327 (4)	0.1871 (7)	0.0535 (10)
C12	0.3695 (2)	0.8239 (4)	0.3129 (7)	0.0612 (12)
C13	0.4146 (2)	0.9069 (4)	0.3151 (10)	0.0754 (15)
C14	0.4645 (2)	0.8986 (5)	0.1988 (11)	0.080 (2)
C15	0.4701 (2)	0.8080 (6)	0.0764 (10)	0.080 (2)
C16	0.4247 (2)	0.7236 (5)	0.0704 (8)	0.0683 (13)
C17	0.3487 (2)	0.2391 (4)	0.2805 (7)	0.0600 (11)
O1	0.24569 (14)	0.7646 (2)	0.1270 (6)	0.0657 (9)
O2	0.38806 (12)	0.4896 (2)	0.2365 (5)	0.0627 (8)
O3	0.09157 (12)	0.4080 (3)	0.2115 (5)	0.0633 (8)
O4*M*	0.01008 (14)	0.5736 (3)	0.1898 (8)	0.0895 (13)
C1*M*	0.0079 (3)	0.6676 (5)	0.0694 (9)	0.085 (2)

Table 4. *Selected geometric parameters (Å, °) for (2)*

C1—O1	1.210 (5)	C3*B*—N4	1.380 (5)
C1—N2	1.395 (5)	C3*B*—C8*B*	1.416 (5)
C1—C10*A*	1.477 (5)	N4—C4*A*	1.397 (6)
N2—C3	1.413 (5)	N4—C17	1.442 (5)
N2—C11	1.428 (5)	C4*A*—C8*A*	1.385 (6)
C3—O2	1.204 (4)	C8*A*—C8*B*	1.446 (6)
C3—C3*A*	1.474 (5)	C8*B*—C9	1.401 (5)
C3*A*—C10*A*	1.399 (5)	C9—O3	1.339 (4)
C3*A*—C3*B*	1.405 (5)	C10—C10*A*	1.373 (5)
O1—C1—N2	125.1 (4)	N4—C3*B*—C3*A*	132.3 (4)
O1—C1—C10*A*	128.3 (3)	N4—C3*B*—C8*B*	108.7 (3)
N2—C1—C10*A*	106.6 (3)	C3*B*—N4—C4*A*	107.7 (3)
C1—N2—C3	111.0 (3)	C3*B*—N4—C17	128.1 (4)
C1—N2—C11	124.5 (3)	C4*A*—N4—C17	124.1 (3)
C3—N2—C11	124.4 (3)	C8*A*—C4*A*—N4	110.5 (3)
O2—C3—N2	122.9 (4)	N4—C4*A*—C5	127.8 (4)
O2—C3—C3*A*	130.9 (4)	O3—C9—C10	124.0 (3)
N2—C3—C3*A*	106.3 (3)	C16—C11—N2	121.0 (4)
C10*A*—C1—N2—C3	2.2 (5)	C3*B*—N4—C4*A*—C5	−179.4 (5)
C10*A*—C1—N2—C11	−174.7 (4)	C17—N4—C4*A*—C5	3.9 (7)
C1—N2—C3—C3*A*	−1.8 (4)	N4—C4*A*—C8*A*—C8	179.7 (4)
C11—N2—C3—C3*A*	175.1 (4)	N4—C4*A*—C8*A*—C8*B*	−0.7 (4)
O2—C3—C3*A*—C3*B*	0.2 (8)	N4—C3*B*—C8*B*—C9	−179.6 (4)
N2—C3—C3*A*—C3*B*	−178.5 (5)	C3*B*—C8*B*—C9—O3	178.4 (4)
C3—C3*A*—C3*B*—N4	−1.0 (8)	O3—C9—C10—C10*A*	−179.0 (4)
C3—C3*A*—C3*B*—C8*B*	177.8 (4)	O1—C1—C10*A*—C10	−4.0 (7)
C3*A*—C3*B*—N4—C4*A*	178.8 (4)	N2—C1—C10*A*—C10	177.5 (4)
C3*A*—C3*B*—N4—C17	−4.6 (7)	C1—N2—C11—C12	52.9 (6)
C3*B*—N4—C4*A*—C8*A*	0.4 (5)	C3—N2—C11—C12	−123.5 (4)

For both compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CELSIUS* (Svenson, 1974); data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structures: *SHELX*76 (Sheldrick, 1976) for (1); *SHELXL*93 (Sheldrick, 1993) for (2).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1206). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Brown, I. D. (1976). *Acta Cryst.* A**32**, 24–31.

Chakraborty, D. P. & Roy, S. (1991). *Prog. Chem. Organ. Nat. Produkts*, Vol. 57, *Carbazole Alkaloids III*, edited by Z. Walter, G. W. Kirby, W. Steglich & Ch. Tamm, pp. 93–99. Wien, New York: Springer Verlag.

Dräger, M. & Gattow, G. (1971). *Acta Chem. Scand.* **25**, 761. (1994 revised version by L. Wiehl & D. Schollmeyer, Universität Mainz, Germany.)

Dräger, U., Haber, M., Erfanian-Abdoust, H., Pindur, U. & Sattler, K. (1993). *Monatsh. Chem.* **124**, 559–576.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Gribble, G. W. (1990). *The Alkaloids*, Vol. 39, *The Ellipticine Alkaloids*, edited by A. Brossi, pp. 239–343. New York, London: Academic Press.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kansal, V. R. & Poitier, P. (1986). *Tetrahedron*, **42**, 2389–2408.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. University of Cambridge, England.

Pindur, U. (1987). *Pharm. Unserer Zeit*, **16**, 47–52.

Pindur, U. (1995). *Advances in Nitrogen Heterocycles*, Vol. 1, *Cycloadditions with Indole Derivatives*, edited by C. J. Moody, pp. 122–170. Greenwhich: JAI Press.

Pindur, U., Haber, M. & Erfanian-Abdoust, H. (1992). *Heterocycles*, **34**, 781-790.

Pindur, U., Haber, M. & Sattler, K. (1992). *Pharm. Unserer Zeit*, **21**, 21-36.

Pindur, U., Pfeuffer, L., Eitel, M., Rogge, M. & Haber, M. (1991). *Monatsh. Chem.* **122**, 291-298.

Pindur, U., Rogge, M., Rehn, C., Massa, W. & Peschel, B. (1994). *J. Heterocycl. Chem.* **31**, 981-988.

Poitier, P. (1992). *Chem. Soc. Rev.* **21**, 113-119.

Rehn, C. (1995). Thesis, University of Mainz, Germany.

Rogge, M., Fischer, G., Schollmeyer, D. & Pindur, U. (1996). *Monatsh. Chem.* **127**, 97-102.

Sattler, K. (1994). Thesis, University of Mainz, Germany.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. University of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Reinement of Crystal Structures*. University of Göttingen, Germany.

Svenson, C. (1974). *CELSIUS. Program for Refinement of Lattice Parameters*. Lund, Sweden.