conformation has these dihedral angles at 45 and 0°, respectively (Bernstein, Engel & Hagler, 1981). The four atoms C(16), C(19), N(1) and C(20) are displaced from the least-squares plane defined by them by 0·014 (3), −0·017 (3), −0·010 (3) and 0·015 (3) Å. The conformation of the azomethine bridge is completely described by the torsion angles C(17)–C(16)–C(19)–N(1) 9·3 (4), C(16)–C(19)–N(1)–C(20) −177·4 (3) and C(19)–N(1)–C(20)–C(25) 46·6 (4)°.

A large number of benzylideneanilines, including more than ten mesogenic compounds, have been examined by X-ray analysis. From these results a consistent pattern of bond lengths and angles with the following relatively small ranges was derived by Bryan & Forcier (1980): 1·398–1·460 Å for N(1)–C(20), 1·237–1·287 Å for C(19)–N(1), and 1·430–1·496 Å for C(16)–C(19). As a result of intramolecular steric repulsions, the angles N(1)–C(20)–C(25), C(16)–C(19)–N(1), and C(17)–C(16)–C(19) should be enlarged, and the angles N(1)–C(20)–C(21), C(19)–N(1)–C(20) and C(15)–C(16)–C(19) should be reduced from 120°. The observed bond lengths and angles in CPBBA (Table 2) conform to this pattern.

Fig. 2 illustrates the molecular arrangement in the crystal structure of CPBBA. The packing is characterized by a perfectly parallel alignment of the molecules along their long axes generated by the inversion centres and translations of the space group. Neighbouring molecules are arranged in a head-to-tail fashion. The crystal structure of CPBBA is typical of nematogens (nematic precursors), and could transform to the nematic phase by means of a simple displacive transition (Bryan & Forcier, 1980).

There are no unusually short intermolecular contacts in the structure.

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Structures of (−)-Cytisine and (−)-*N*-Methylcytisine: Tricyclic Quinolizidine Alkaloids

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Abstract. (−)-1,2,3,4,5,6-Hexahydro-1,5-methano-8*H*-pyrido[1,2-*a*][1,5]diazocin-8-one, $C_{11}H_{14}N_2O$, M_r = 190·3, orthorhombic, $P2_12_12_1$, a = 7·178 (2), b = 9·966 (1), c = 26·619 (2) Å, V = 1904·3 Å^3, Z = 8, D_m = 1·28, D_x = 1·33 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 6·55 cm^{-1}, $F(000)$ = 816, T = 291 K, final R = 0·047 for 1999 unique observed reflections. (−)-1,2,3,4,5,6-Hexahydro-3-methyl-1,5-methano-8*H*-pyrido-[1,2-*a*][1,5]diazocin-8-one, $C_{12}H_{16}N_2O$, M_r = 204·3, orthorhombic, $P2_12_12_1$, a = 9·065 (2), b = 10·824 (3), c = 11·206 (2) Å, V = 1099·5 Å^3, Z = 4, D_m = 1·13, D_x = 1·15 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·75 cm^{-1}, $F(000)$ = 440, T = 291 K, final R = 0·035 for 1014 unique observed reflections. The structures of each of the two molecules in the asymmetric unit of cytisine and that of *N*-methylcytisine are almost identical. Both (−)-cytisine (1) and (−)-*N*-methylcytisine (2) adopt the same conformations with ring *A* essentially planar; ring *B* has an envelope conformation with the bridgehead atom, C(8), out of the plane by

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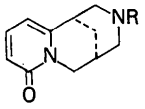
0·764 (4) Å in (1) and 0·734 (4) Å in (2). Ring *C* has a chair conformation. The nitrogen atom N(1) adopts a planar configuration whilst the second nitrogen atom, N(12), forms a flattened pyramid with its lone pair axial to ring *C*.

Introduction. (−)-Cytisine (1) and (−)-*N*-methylcytisine (2) are toxic quinolizidine alkaloids widespread in *Cytisus, Baptisia* and other genera within the family Leguminosae. Cytisine is the constituent responsible for laburnum-seed poisoning.



(1) R = H
(2) R = Me

During the course of our biosynthetic studies (Fraser & Robins, 1986) it became clear that a knowledge of the conformations of these two alkaloids might assist the interpretation of the results of NMR spectroscopic studies.

While the structures (Ing, 1932, 1935) and absolute configurations (Okuda, Tsuda & Kataoka, 1961) of (1) and (2) had been established for many years it was found that determination of the crystal structures had escaped attention.

Experimental. (−)-Cytisine. Colourless cube-shaped crystals grown by slow evaporation from benzene, crystal 0·4 × 0·4 × 0·5 mm used in data collection, CAD-4 diffractometer, Cu radiation. Preliminary Weissenberg photographs indicated crystals to be orthorhombic, $P2_12_12_1$. D_m by flotation. 2163 independent intensities, θ limit 72°, $\omega/2\theta$ scan. Two standard intensities used to monitor variations in intensity data; <3% variation observed. Least-squares technique based on 25 reflections, $\theta > 20°$, used to refine lattice parameters. No absorption correction. h 0 to 8, k 0 to 12 and l 0 to 32. Structure solution by direct methods with *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for all non-H atoms to R and wR of 0·048 and 0·054. $\sum w(|F_o| - |F_c|)^2$ minimized with $w = 1/\sigma^2(F_o)$. H-atom coordinates were located from difference Fourier syntheses and refined isotropically in the final two cycles of least squares. 1999 reflections, $I > 3{\cdot}00\sigma_I$, used. $\Delta_{max}/\sigma = 0{\cdot}55$; max. and min. heights in final difference Fourier synthesis = 0·18 and −0·22 e Å^{-3}.

(−)-*N*-Methylcytisine. Colourless, needle-shaped crystals grown by slow evaporation from benzene, crystal 0·3 × 0·4 × 0·8 mm used in data collection, CAD-4 diffractometer, Mo radiation. Preliminary Weissenberg photographs indicated crystals to be orthorhombic, $P2_12_12_1$. D_m by flotation. 1386 independent intensities, θ limit 27°, $\omega/2\theta$ scan. Two standard intensities used to monitor variations in intensity data; <3% variation observed. Least-squares technique based on 25 reflections, $\theta > 12°$, used to refine lattice parameters. No absorption correction, h 0 to 11, k 0 to 13 and l 0 to 14. Structure solved by direct-phasing techniques using *MITHRIL*. Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters converged to R and wR of 0·035 and 0·035 with $w = 1{\cdot}0$. H-atom coordinates were obtained from difference Fourier syntheses and refined isotropically in the final two cycles of least squares. 1014 reflections, $I \geq 3{\cdot}0\sigma_I$, used. $\Delta_{max}/\sigma =$ 0·09; max. and min. heights in final difference Fourier synthesis = 0·20 and −0·15 e Å^{-3}.

Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using Glasgow *GX* package (Mallinson & Muir, 1985).

Table 1. *Final positional parameters and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
(−)-Cytisine				
O(2)	−0·1895 (4)	−0·1733 (3)	−0·4052 (1)	0·047
N(1)	−0·4779 (4)	−0·1334 (3)	−0·4377 (1)	0·029
N(12)	−0·7250 (5)	0·1281 (4)	−0·4284 (1)	0·034
C(2)	−0·3540 (6)	−0·2089 (4)	−0·4082 (2)	0·037
C(3)	−0·4289 (7)	−0·3260 (5)	−0·3851 (2)	0·041
C(4)	−0·6140 (7)	−0·3604 (5)	−0·3910 (2)	0·043
C(5)	−0·7305 (7)	−0·2782 (4)	−0·4198 (2)	0·039
C(6)	−0·6647 (5)	−0·1666 (4)	−0·4430 (2)	0·031
C(7)	−0·7848 (6)	−0·0799 (4)	−0·4758 (2)	0·034
C(8)	−0·6721 (7)	−0·0265 (5)	−0·5202 (2)	0·040
C(9)	−0·5193 (6)	0·0638 (4)	−0·4979 (2)	0·036
C(10)	−0·3900 (6)	−0·0181 (4)	−0·4640 (2)	0·035
C(11)	−0·8713 (6)	0·0382 (4)	−0·4466 (2)	0·035
C(13)	−0·6113 (7)	0·1806 (4)	−0·4698 (2)	0·038
O(2′)	−1·0896 (4)	−0·1263 (3)	−0·1296 (1)	0·045
N(1′)	−0·8934 (5)	−0·0612 (3)	−0·1926 (1)	0·029
N(12′)	−0·9337 (7)	−0·1110 (4)	−0·3074 (1)	0·044
C(2′)	−0·9316 (7)	−0·1332 (4)	−0·1484 (1)	0·035
C(3′)	−0·7821 (7)	−0·2116 (5)	−0·1290 (2)	0·041
C(4′)	−0·6160 (8)	−0·2157 (6)	−0·1522 (2)	0·051
C(5′)	−0·5851 (7)	−0·1423 (5)	−0·1964 (2)	0·043
C(6′)	−0·7205 (6)	−0·0645 (4)	−0·2157 (2)	0·034
C(7′)	−0·6926 (7)	0·0162 (5)	−0·2633 (2)	0·042
C(8′)	−0·8137 (7)	0·1420 (4)	−0·2628 (2)	0·043
C(9′)	−1·0158 (7)	0·0928 (4)	−0·2607 (2)	0·038
C(10′)	−1·0484 (6)	0·0232 (4)	−0·2110 (2)	0·036
C(11′)	−0·7371 (8)	−0·0687 (5)	−0·3097 (2)	0·048
C(13′)	−1·0577 (8)	0·0053 (5)	−0·3062 (2)	0·046
(−)-*N*-Methylcytisine				
O(2)	−0·8524 (3)	−0·2821 (2)	−0·4935 (2)	0·061
N(1)	−0·9263 (3)	−0·3962 (2)	−0·3336 (2)	0·037
N(12)	−1·1116 (3)	−0·3071 (3)	−0·1192 (2)	0·045
C(2)	−0·8160 (4)	−0·3421 (3)	−0·4039 (3)	0·046
C(3)	−0·6683 (4)	−0·3627 (4)	−0·3653 (4)	0·056
C(4)	−0·6387 (4)	−0·4345 (4)	−0·2699 (4)	0·060
C(5)	−0·7529 (5)	−0·4888 (4)	−0·2039 (3)	0·051
C(6)	−0·8945 (4)	−0·4674 (3)	−0·2345 (3)	0·041
C(7)	−1·0216 (4)	−0·5166 (3)	−0·1622 (3)	0·053
C(8)	−1·1545 (5)	−0·5441 (4)	−0·2398 (4)	0·060
C(9)	−1·1991 (4)	−0·4214 (4)	−0·2957 (3)	0·050
C(10)	−1·0779 (4)	−0·3766 (3)	−0·3777 (3)	0·046
C(11)	−1·0627 (5)	−0·4229 (4)	−0·0651 (3)	0·053
C(13)	−1·2370 (4)	−0·3285 (4)	−0·1976 (3)	0·052
C(14)	−1·1489 (6)	−0·2164 (5)	−0·0276 (4)	0·067

Table 2. *Molecular geometry*

	Cytisine Molecule *A*	Cytisine Molecule *B*	*N*-Methylcytisine
(*a*) Bond distances (Å)			
O(2)–C(2)	1·236 (6)	1·241 (6)	1·240 (5)
N(1)–C(2)	1·405 (6)	1·406 (5)	1·401 (5)
N(1)–C(6)	1·388 (6)	1·385 (6)	1·382 (5)
N(1)–C(10)	1·486 (6)	1·478 (6)	1·475 (5)
N(12)–C(11)	1·463 (6)	1·474 (8)	1·461 (5)
N(12)–C(13)	1·469 (6)	1·462 (7)	1·455 (5)
N(12)–C(14)	–	–	1·461 (6)
C(2)–C(3)	1·424 (7)	1·424 (7)	1·425 (6)
C(3)–C(4)	1·381 (7)	1·344 (8)	1·349 (6)
C(4)–C(5)	1·398 (7)	1·402 (8)	1·401 (6)
C(5)–C(6)	1·358 (7)	1·346 (7)	1·349 (6)
C(6)–C(7)	1·501 (6)	1·514 (7)	1·505 (6)
C(7)–C(8)	1·528 (6)	1·525 (7)	1·516 (6)
C(7)–C(11)	1·543 (7)	1·530 (7)	1·534 (6)
C(8)–C(9)	1·538 (7)	1·533 (7)	1·523 (6)
C(9)–C(10)	1·530 (6)	1·511 (7)	1·513 (6)
C(9)–C(13)	1·533 (7)	1·522 (7)	1·529 (6)
(*b*) Bond angles (°)			
C(2)–N(1)–C(6)	122·7 (4)	122·3 (4)	122·4 (3)
C(2)–N(1)–C(10)	114·1 (4)	114·9 (4)	114·6 (3)
C(6)–N(1)–C(10)	123·2 (4)	122·7 (4)	122·9 (3)
C(11)–N(12)–C(13)	111·7 (4)	110·9 (4)	110·6 (3)
C(11)–N(12)–C(14)	–	–	110·8 (3)
C(13)–N(12)–C(14)	–	–	110·5 (4)
O(2)–C(2)–N(1)	119·2 (4)	119·1 (4)	119·0 (4)
O(2)–C(2)–C(3)	124·6 (5)	124·9 (4)	125·3 (4)
N(1)–C(2)–C(3)	116·2 (4)	115·9 (4)	115·8 (4)
C(2)–C(3)–C(4)	121·2 (5)	121·2 (5)	121·2 (4)
C(3)–C(4)–C(5)	119·5 (5)	120·7 (5)	120·9 (4)
C(4)–C(5)–C(6)	121·5 (5)	120·5 (5)	119·7 (4)
N(1)–C(6)–C(5)	119·0 (4)	119·4 (4)	119·9 (4)
N(1)–C(6)–C(7)	118·5 (4)	118·5 (4)	118·0 (3)
C(5)–C(6)–C(7)	122·5 (4)	122·0 (4)	122·0 (4)
C(6)–C(7)–C(8)	110·3 (4)	110·7 (4)	111·7 (4)
C(6)–C(7)–C(11)	112·2 (4)	110·7 (4)	109·5 (4)
C(8)–C(7)–C(11)	109·7 (4)	110·0 (4)	110·1 (4)
C(7)–C(8)–C(9)	106·5 (4)	106·1 (4)	106·0 (4)
C(8)–C(9)–C(10)	110·4 (4)	109·0 (4)	109·7 (4)
C(8)–C(9)–C(13)	109·0 (4)	109·9 (4)	109·7 (4)
C(10)–C(9)–C(13)	112·3 (4)	113·7 (4)	112·9 (4)
N(1)–C(10)–C(9)	115·6 (4)	115·8 (4)	115·3 (3)
N(12)–C(11)–C(7)	110·2 (4)	109·0 (4)	110·3 (3)
N(12)–C(13)–C(9)	109·6 (4)	110·6 (5)	111·3 (3)
(*c*) Selected torsion angles (°)			
C(6)–N(1)–C(2)–C(3)	2·4 (4)	−0·8 (4)	−1·3 (4)
C(2)–N(1)–C(6)–C(5)	−1·6 (4)	2·2 (4)	−1·3 (4)
C(2)–N(1)–C(6)–C(7)	−179·5 (6)	179·8 (6)	177·4 (5)
C(10)–N(1)–C(2)–C(3)	−175·6 (6)	−177·9 (6)	−178·2 (5)
C(2)–N(1)–C(10)–C(9)	178·1 (5)	−175·3 (6)	−173·2 (5)
C(10)–N(1)–C(6)–C(5)	176·3 (6)	179·1 (6)	175·3 (5)
C(10)–N(1)–C(6)–C(7)	−1·6 (4)	−3·3 (4)	−6·0 (3)
C(6)–N(1)–C(10)–C(9)	0·1 (4)	7·6 (4)	9·9 (3)
C(13)–N(12)–C(11)–C(7)	58·3 (4)	59·6 (5)	58·0 (4)
C(11)–N(12)–C(13)–C(9)	−59·8 (4)	−59·2	−58·1 (4)
C(14)–N(12)–C(11)–C(7)	–	–	−179·1 (5)
C(14)–N(12)–C(13)–C(9)	–	–	178·9 (5)
H(12)–N(12)–C(11)–C(7)	179·6 (33)	175·1 (34)	–
H(12)–N(12)–C(13)–C(9)	178·1 (33)	−174·9 (34)	–
N(1)–C(2)–C(3)–C(4)	−1·3 (5)	−0·4 (5)	2·7 (4)
C(2)–C(3)–C(4)–C(5)	−0·6 (5)	0·2 (5)	−1·5 (4)
C(3)–C(4)–C(5)–C(6)	1·5 (5)	1·2 (5)	−1·3 (4)
C(4)–C(5)–C(6)–N(1)	−0·4 (4)	−2·4 (5)	2·7 (4)
C(4)–C(5)–C(6)–C(7)	177·4 (7)	−180·0 (8)	−176·0 (6)
N(1)–C(6)–C(7)–C(8)	34·3 (4)	31·3 (4)	32·6 (4)
N(1)–C(6)–C(7)–C(11)	−88·3 (5)	−91·0 (5)	−89·6 (4)
C(5)–C(6)–C(7)–C(8)	−143·5 (6)	−151·1 (6)	−148·7 (5)
C(5)–C(6)–C(7)–C(11)	93·9 (5)	86·6 (6)	89·1 (5)
C(6)–C(7)–C(8)–C(9)	−63·9 (4)	−61·8 (4)	−61·3 (4)
C(6)–C(7)–C(11)–N(12)	63·9 (4)	61·1 (5)	62·3 (4)
C(8)–C(7)–C(11)–N(12)	−59·0 (4)	−61·6 (5)	−60·8 (4)
C(11)–C(7)–C(8)–C(9)	60·1 (4)	60·9 (5)	60·5 (4)
C(7)–C(8)–C(9)–C(10)	62·2 (4)	65·9 (4)	64·8 (4)
C(7)–C(8)–C(9)–C(13)	−61·6 (4)	−59·4 (5)	−59·7 (4)
C(8)–C(9)–C(10)–N(1)	−31·1 (4)	−39·2 (4)	−39·8 (4)
C(8)–C(9)–C(13)–N(12)	61·6 (4)	59·7 (5)	59·9 (4)
C(13)–C(9)–C(10)–N(1)	90·7 (5)	83·8 (5)	82·9 (4)
C(10)–C(9)–C(13)–N(12)	−61·0 (4)	−62·8 (5)	−62·7 (4)

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and selected torsion angles with their standard deviations are given in Table 2. An *ORTEP* (Johnson, 1976) diagram, Fig. 1, illustrates the numbering scheme and absolute configuration for the two molecules.

Bond lengths, bond angles and torsion angles show that each of the two molecules in the asymmetric unit of (−)-cytisine and that of (−)-*N*-methylcytisine are almost identical. The pyridone ring, *A*, is essentially planar with maximum deviations of −0·013 (4) and −0·015 (3) Å from the least-squares plane occurring at atom C(2) in (1) and atom C(3) in (2) respectively. The planarity of the pyridone ring confers an envelope conformation on the adjacent ring *B* with the bridgehead atom, C(8), out of the plane defined by atoms N(1), C(6), C(7), C(9) and C(10) by 0·764 (4) Å in (1) and 0·734 (4) Å in (2). Ring *C* adopts a conventional chair conformation as found in *N*-cyanomethylangustifoline (Bratek-Wiewiórowska, Rychlewska & Wiewiórowski, 1979).

* Lists of structure factors, anisotropic temperature factors, H-atom coordinates and bond lengths and angles involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43706 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

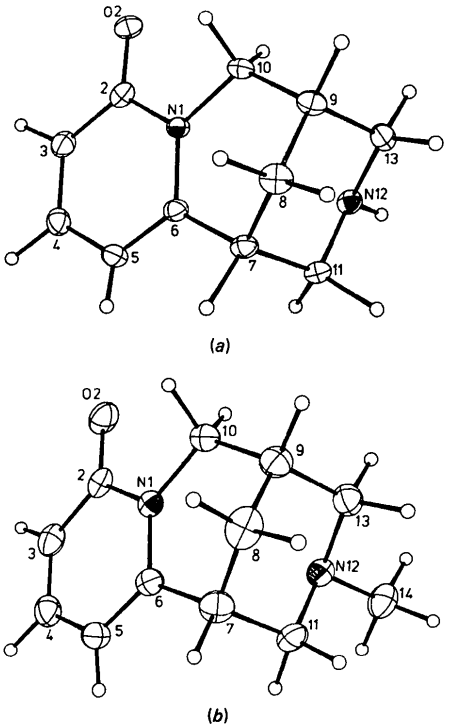


Fig. 1. Perspective views of (*a*) (−)-cytisine and (*b*) (−)-*N*-methylcytisine showing the numbering scheme and vibrational ellipsoids (50% probability level).

The sum (360·0°) of the angles at N(1) indicates that it adopts a planar configuration. The second N atom in (–)-cytisine, N(12), and its three substituents, C(11), C(13) and H(12), form a flattened pyramid with the N lone pair axial relative to ring *C*. A similar conformation exists for C(11), C(13), and C(14) around N(12) in (–)-*N*-methylcytisine. The sum of the three bond angles around N(12) is 331·9° for (–)-*N*-methylcytisine indicating that hybridization of the N atom is close to tetrahedral. A similar situation is observed for (–)-cytisine. The essentially identical conformations adopted by both molecules in the crystal are not consistent with the difference observed in the proton NMR spectra of both alkaloids. The axial protons at positions C(11) and C(13) in *N*-methylcytisine are shielded (δ 1·62 and 1·70) compared with the equatorial protons (δ 2·29 and 2·42). This shielding is due to a well documented combination of effects of the antiperiplanar N lone pair (Hamlow, Okuda & Nakagawa, 1964) and the equatorial methyl substituent (Booth, 1966). However, in cytisine, this axial shielding is absent, all four protons having chemical shifts in the range δ 2·31–2·49. This suggests that, in solution, the conformation of ring *C* of cytisine is not rigidly chair-form.

The packing arrangement of the cytisine molecule, with two molecules in the asymmetric unit, leads to a hydrogen-bonding network between N(12)···O(2′) of neighbouring molecules. These are weak interactions with O(2)···N(12′) and N(12)···O(2′) distances 3·186 (5) and 3·244 (5) Å. Respective hydrogen bonds O(2)···H(12′), 2·55 (5) Å, and O(2′)···H(12), 2·44 (4) Å, subtend intermolecular bond angles of 144 (4) and 146(5)°. *N*-Methylcytisine has no similar intermolecular network.

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Structures of 4-Acetyl-3-(*p*-tolyl)sydnone (1) and 4-Acetyl-3-phenylsydnone Oxime (2)

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Abstract. (1) $C_{11}H_{10}N_2O_3$, $M_r = 218·2$, orthorhombic, $P2_12_12_1$, $a = 10·995$ (4), $b = 15·158$ (2), $c = 6·530$ (3) Å, $V = 1088·3$ (7) Å^3, $Z = 4$, $D_m = 1·3$ (1), $D_x = 1·33$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7093 Å, μ(Mo $K\alpha$) = 0·093 mm^{-1}, $F(000) = 456$, $T = 298$ K, final $R = 0·038$ for 855 observed reflections. (2) $C_{10}H_9N_3O_3$, $M_r = 219·2$, monoclinic, $P2_1/n$, $a = 7·871$ (1), $b = 7·741$ (2), $c = 16·880$ (5) Å, $\beta = 96·20$ (2)°, $V = 1022$ (2) Å^3, $Z = 4$, $D_m = 1·4$ (1), $D_x = 1·42$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 0·87 mm^{-1}, $F(000) = 456$, $T = 298$ K, final $R = 0·041$ for 1553 observed reflections. The bond lengths of the sydnone ring are similar in both structures. The bond lengths N(1)–C(7) and C(7)–C(8) of 3,4-disubstituted sydnone derivatives are longer than the corresponding bond lengths in 3-substituted sydnone derivatives, and the dihedral angles between the sydnone ring and the phenyl ring of (1) and (2) [68·4 (2) and 78·6 (1)° respectively] are larger than those of 3-substituted sydnone derivatives. This may be attributed to steric effects.

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