

observation has recently been made in the structure of a 3,8-diethyl-5,10-dimethyl-1,6-dioxapyrene complex with 7,7,8,8-tetracyano-*p*-quinodimethane (Thorup, Hjorth, Christensen & Bechgaard, 1992). The organic cations form segregated stacks (Fig. 2) along the short *a* axis, and two neighbouring cations are related by a glide operation ( $x + \frac{1}{2}$ ,  $y$ ,  $\frac{1}{2} - z$ ). Interplanar distances are identical and equal to  $\frac{1}{2}a = 3.314$  Å. The only S...S distance shorter than the van der Waals sum of 3.60 Å is a close S(1)...S(1) contact of 3.348 (2) Å between two neighbouring cations within the stack. The organic stacks are surrounded by PF<sub>6</sub><sup>-</sup> anions. Some weak C—H...F interactions may exist, but a detailed analysis is hardly justifiable owing to the anion disorder.

**Related literature.** This work is part of a broad study of organic conducting materials. Owing to the small size of the present crystals the single-crystal conductivity was difficult to measure, but it was established that the crystals exhibit semiconducting activated behaviour with  $\sigma(300\text{ K}) = 2.9\text{ S m}^{-1}$ . Previously, we have studied a charge-transfer complex of 1,6-dithiapyrene with 7,7,8,8-tetracyano-*p*-quinodimethane (Thorup, Rindorf, Jacobsen, Bechgaard, Johannsen & Mortensen, 1985) with segregated stacks and metallic behaviour. In that compound the interplanar spacing of the cations was

3.39 Å, *i.e.* slightly longer than in the present structure. A number of 2,7-substituted 1,6-dithiapyrene compounds have been studied by Nakasuji and co-workers (Nakasuji, Sasaki, Kotani, Murata, Enoki, Imaeda, Inokuchi, Kawamoto & Tanaka, 1987; Kawamoto, Tanaka, Oda, Mizumura, Murata & Nakasuji, 1990).

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## (5 $\alpha$ ,6 $\alpha$ ,7 $\alpha$ ,11 $\beta$ )- $\Delta^{13,14}$ -Sophocarpine Monohydrate

BY MUSHTAQ A. KHAN

*Department of Chemistry, Oklahoma State University, Stillwater, Oklahoma 74078, USA*

GEORGE E. BURROWS

*Department of Physiological Sciences, College of Veterinary Medicine, Oklahoma State University, Stillwater, Oklahoma 74078, USA*

AND ELIZABETH M. HOLT

*Department of Chemistry, Oklahoma State University, Stillwater, Oklahoma 74078, USA*

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**Abstract.** 13,14-Didehydromatridin-15-one, C<sub>15</sub>H<sub>22</sub>N<sub>2</sub>O.H<sub>2</sub>O, *M*<sub>r</sub> = 264.37, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 10.667 (2), *b* = 16.552 (5), *c* = 8.116 (1) Å, *V* = 1433.0 (6) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.225 g cm<sup>-3</sup>, Mo *K* $\alpha$ <sub>1</sub>,  $\lambda$  = 0.71069 Å,  $\mu$  = 0.761 cm<sup>-1</sup>, *F*(000) = 576, *T* = 294 K, *R* = 0.062 for 850 reflections [*I* > 3 $\sigma$ (*I*)]. The

matrine-type alkaloid, isolated from the epigeal part of *Sophora nuttalliana* (syn. *sericea*) displays the 5 $\alpha$ ,6 $\alpha$ ,7 $\alpha$ ,11 $\beta$  stereochemistry reported for (–)-sophocarpine. Rings *A*, *B* and *C* are in chair conformation. The water molecule is hydrogen bonded to the carbonyl O atom.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^2$ )
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
O1	-0.2914 (5)	0.1254 (3)	1.0848 (7)	6.0
OW1	0.4998 (6)	0.6808 (4)	0.2327 (8)	8.3
N1	0.1213 (5)	0.1591 (4)	0.6780 (8)	4.9
N16	-0.1789 (6)	0.1059 (3)	0.8510 (8)	4.0
C2	0.2387 (7)	0.1681 (6)	0.7681 (12)	6.3
C3	0.2200 (8)	0.1553 (6)	0.9548 (12)	6.3
C4	0.1620 (8)	0.0708 (6)	0.9857 (11)	7.0
C5	0.0402 (7)	0.0604 (4)	0.8838 (9)	4.8
C6	0.0662 (6)	0.0793 (5)	0.7007 (9)	4.5
C7	-0.0571 (7)	0.0677 (4)	0.6051 (9)	4.2
C8	-0.0316 (8)	0.0869 (5)	0.4207 (10)	5.9
C9	0.0268 (9)	0.1684 (5)	0.4028 (10)	6.6
C10	0.1475 (8)	0.1740 (6)	0.5047 (11)	6.4
C11	-0.1693 (7)	0.1188 (4)	0.6713 (9)	4.4
C12	-0.2889 (8)	0.0910 (5)	0.5872 (12)	6.1
C13	-0.4024 (8)	0.1206 (6)	0.6736 (12)	6.7
C14	-0.4013 (8)	0.1344 (5)	0.8332 (12)	6.1
C15	-0.2847 (8)	0.1210 (4)	0.9317 (10)	4.4
C17	-0.0619 (7)	0.1143 (5)	0.9502 (9)	5.4

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

N1—C2	1.46 (1)	C2—N1—C6	112.0 (6)
N1—C6	1.46 (1)	C2—N1—C10	107.6 (6)
N1—C10	1.45 (1)	C6—N1—C10	110.7 (6)
C2—C3	1.54 (1)	N1—C2—C3	111.6 (6)
C3—C4	1.55 (1)	C2—C3—C4	109.5 (7)
C4—C5	1.55 (1)	C3—C4—C5	110.4 (7)
C5—C6	1.54 (1)	C4—C5—C6	109.9 (6)
C5—C17	1.51 (1)	C4—C5—C17	110.4 (6)
C6—C7	1.54 (1)	C6—C5—C17	110.8 (6)
C7—C8	1.55 (1)	C5—C6—N1	112.2 (6)
C7—C11	1.56 (1)	C5—C6—C7	107.8 (6)
C8—C9	1.49 (1)	N1—C6—C7	113.1 (6)
C9—C10	1.53 (1)	C6—C7—C8	108.1 (6)
C11—C12	1.52 (1)	C6—C7—C11	114.5 (6)
C11—N16	1.48 (1)	C8—C7—C11	110.8 (6)
C12—C13	1.48 (1)	C7—C8—C9	110.5 (6)
C13—C14	1.32 (1)	C8—C9—C10	110.6 (7)
C14—C15	1.49 (1)	C9—C10—N1	110.5 (7)
C15—O1	1.25 (1)	C7—C11—C12	109.0 (6)
C15—N16	1.33 (1)	C7—C11—N16	108.3 (6)
N16—C17	1.49 (1)	C12—C11—N16	110.0 (6)
		C11—C12—C13	111.9 (8)
		C12—C13—C14	121.1 (8)
		C13—C14—C15	120.5 (8)
		C14—C15—O1	118.4 (7)
		C14—C15—N16	118.2 (7)
		O1—C15—N16	123.4 (8)
		C15—N16—C11	121.2 (6)
		C15—N16—C17	115.3 (6)
		C11—N16—C17	117.4 (6)
		N16—C17—C5	110.8 (6)
O1...H#2'	1.78		

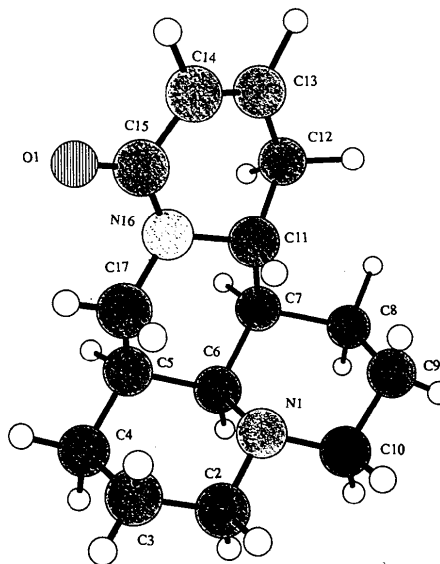
Symmetry code: (')  $-x, -\frac{1}{2} + y, \frac{3}{2} - z$ .

**Experimental.** Acid extraction of the methanolic extract of epigeal *Sophora nuttalliana* followed by chromatography on silica gel yielded the title compound as the least polar component; m.p. 325–327 K;  $[\alpha]_D^{21} - 26.8^\circ$  ( $c = 0.1$  g ml, EtOH, 297 K). A chunk of dimensions  $0.1 \times 0.1 \times 0.1$  mm, cleaved from a colorless needle, was used for data collection on a Syntex P3 automated diffractometer with monochromated Mo  $K\alpha$  radiation. Lattice parameters were determined from 15 reflections with  $\theta = 17$ – $33^\circ$ . Intensities for  $\theta \leq 50^\circ$ ,  $h$  0 to 15,  $k$  0 to 21,  $l$  0 to 13, were measured using  $\theta$ – $2\theta$  scans with scan width of  $1.2^\circ$  below  $K\alpha_1$  and  $1.2^\circ$  above  $K\alpha_2$  and a variable scan rate; backgrounds measured at each side of the scan for a combined time equal to the

total scan time. Three standard reflections showed no significant variation. Of 1113 unique reflections, 850 had  $I \geq 3.0\sigma(I)$ . The structure was solved for non-H positions using *MULTAN80* (Main *et al.*, 1980), and refined by full-matrix least-squares procedures (*XRAY*; Stewart, 1978). H atoms were located from difference Fourier synthesis. Refinement was based on  $F$  with  $w = 1/\sigma^2(F)$ . Scattering factors were those of Cromer & Mann (1968). Refinements using anomalous-dispersion corrections did not establish the absolute configuration. The absolute configuration was assigned from agreement of physical constants with those of (–)-sophocarpine and from the observation that all known matrine alkaloids display the  $11\beta$  configuration (Kinghorn & Balandrin, 1984; Aslanov, Kushmuradov & Sadykov, 1987; Southon & Buckingham, 1989). Anisotropic refinement for non-H atoms (H atoms with fixed isotropic thermal parameters were held fixed) converged to give final  $R = 6.2\%$ ,  $wR = 8.0\%$  for 172 parameters;  $(\Delta/\sigma)_{\max} = 0.103$ ; maximum  $\Delta\rho = 0.34$  e  $\text{\AA}^{-3}$ . Positional parameters are listed in Table 1, bond lengths and angles in Table 2. A projection of the molecule is given in Fig. 1.\*

**Related literature.** Crystal structure of (5 $\beta$ ,6 $\alpha$ ,7 $\alpha$ ,11 $\beta$ )- $\Delta^{13,14}$ -sophocarpine monohydrate (m.p. 356–357 K;  $[\alpha]_D - 77^\circ$ ; ring *A*, chair; rings *B*

\* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55318 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0413]

Fig. 1. Projection of (5 $\alpha$ ,6 $\alpha$ ,7 $\alpha$ ,11 $\beta$ )- $\Delta^{13,14}$ -sophocarpine monohydrate.

and C, boat) isolated from epigeal *Sophora flavescens*, and isolation of a  $C_{15}H_{22}N_2O$  compound, de-*N*-oxidized derivative of  $\Delta^{13,14}$ -dehydroso-phoridine *N*-oxide (apparently  $5\beta,6\alpha,7\beta,11\beta$ ; m.p. 356–357 K;  $[\alpha]_D^{25}$  76.8°) have been reported (Morinaga, Ueno, Fukushima, Namikoshi, Iitaka & Okuda, 1978; Ueno, Morinaga, Fukushima & Okuda, 1976), as well as isolation and physical constants of ( $5\alpha,6\alpha,7\alpha,11\beta$ )- $\Delta^{13,14}$ -(-)-sophocarpine monohydrate (m.p. 327 K;  $[\alpha]_D^{25}$  -29.4°) and its chemical conversion to (+)-matrine (Okuda, Kamata, Tsuda & Murakoshi, 1962).

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## Structure Cristalline d'un *N*-Azaarylphthalimide à Activité Anti-inflammatoire, le 2-(4,6-Diméthylpyridin-2-yl)-2,3-dihydro-1*H*-isoindole-1,3-dione

PAR N. RODIER

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, 5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

ET J.-M. ROBERT, D. LEBLOIS ET G. LE BAUT

Laboratoire de Chimie thérapeutique, Faculté de Pharmacie, 1 Rue Gaston Veil, 44035 Nantes CEDEX, France

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**Abstract.**  $C_{15}H_{12}N_2O_2$ ,  $M_r = 252.3$ , triclinic,  $P\bar{1}$ ,  $a = 7.7464$  (4),  $b = 12.214$  (1),  $c = 7.8813$  (9) Å,  $\alpha = 88.78$  (1),  $\beta = 119.255$  (6),  $\gamma = 103.803$  (7)°,  $V = 627.8$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.334$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.70$  mm<sup>-1</sup>,  $F(000) = 264$ ,  $T = 294$  (1) K,  $R = 0.031$  for 1413 independent observed reflections. The pyridyl ring is planar and the isoindolyl group approximately planar. The least-squares planes of the two rings make an angle of 69.17 (5)°; the whole molecule is therefore not planar as was suggested by <sup>1</sup>H NMR study. The bond lengths and angles agree with published values for similar compounds. The crystal structure was solved in order to obtain an exact knowledge of the molecular geometry.

**Partie expérimentale.** Cristal obtenu par attaque nucléophile de la 6-amino-2,4-lutidine sur l'anhydride phthalique, au reflux de l'acide acétique, en

présence d'anhydride acétique et d'acétate de sodium anhydre. Produit recristallisé dans l'éthanol. Cristal approximativement parallélépipédique: 0,30 × 0,25 × 0,14 mm. Dimensions de la maille déterminées avec 25 réflexions telles que  $13,91 \leq \theta \leq 47,31^\circ$ . Diffractomètre Enraf-Nonius CAD-4. Balayage  $\theta/2\theta$  d'amplitude  $s$  (°) =  $1,10 + 0,14\text{tg}\theta$ .  $0,023 \leq (\sin\theta)/\lambda \leq 0,609$  Å<sup>-1</sup>,  $0 \leq h \leq 9$ ,  $-14 \leq k \leq 14$ ,  $-9 \leq l \leq 8$ . Réflexions de contrôle de l'intensité:  $\bar{1}0,0,1$ , 403 et 40 $\bar{3}$ . Variations non significatives de  $I$ . 2361 réflexions indépendantes mesurées, 948 inobservées [ $I < 3\sigma(I)$ ]. Méthodes directes, programme MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H des CH<sub>3</sub>, série de Fourier des  $\Delta F$ ; autres H: positions calculées. Affinement basé sur les  $F$ , programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974, Tome IV, pp. 99, 149). Paramètres affinés:  $x$ ,  $y$ ,  $z$  de tous les