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

C6—Cl6	1.730 (2)		
C6—N1—C2	117.38 (14)	N1—C6—C5	121.06 (14)
N3—C2—N1	127.9 (2)	C8—N7—C5	105.05 (14)
C2—N3—C4	113.44 (14)	N9—C8—N7	114.9 (2)
N3—C4—C5	123.84 (14)	C8—N9—C4	104.24 (13)
C6—C5—C4	116.36 (15)		
C8—N7—C10—C11	106.1 (2)	N7—C10—C11—O13	−19.7 (2)
N7—C10—C11—O12	161.3 (2)	C11—O13—C14—C15	76.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2⋯O12^i	0.94 (2)	2.51 (2)	3.324 (2)	145 (2)
C8—H8⋯N3^{ii}	0.96 (2)	2.49 (2)	3.442 (2)	173 (2)

Symmetry codes: (i) $x, y − 1, z$; (ii) $1 − x, \frac{1}{2} + y, −\frac{1}{2} − z$.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1208). Services for accessing these data are described at the back of the journal.

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2,6-Dimethyl-3,7-diphenyl-2,6-naphthyridine-1,5(2H,6H)-dione

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Abstract

The title compound, $C_{22}H_{18}N_2O_2$, has a center of symmetry and the parameters of half of each of the two independent molecules have been determined. The naphthyridine ring is planar; the coplanarity of the naphthyridine ring and the phenyl rings is hindered by the N-methyl groups. The dihedral angles between the rings are 51.8 (2) and 61.5 (2)° in the two independent molecules.

Comment

It has been reported that an aminolysis product of a Pechmann dye [(E)-5,5'-diphenyl-3,3'-bifuranylidene-2,2'-dione] is supposed to be a γ-dilactam or a naphthyridinedione (Klingsberg, 1954). A γ-dilactam has been obtained from a Pechmann dye (Kollenz et al., 1996), but the formation of the naphthyridinedione has not been reported. In the present study, the title compound, (I), was prepared from 3,7-diphenylpyrano[4,3-c]pyran-1,5-dione, since aminolysis of the Pechmann dye gave the target compound in very poor yield.
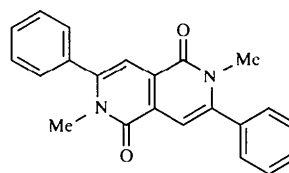
(I)

Although there are four molecules in the unit cell, each molecule has a center of symmetry and two of the four molecules are independent from the others. A drawing of the two independent molecules is shown in Fig. 1. The bond distances and angles of the two independent molecules are very similar except for the dihedral angles between the phenyl ring and the central naphthyridine ring. The central rings are planar within 0.03 Å. The phenyl rings are also planar but the coplanarity of the naphthyridine ring and the phenyl rings is hindered by the N-methyl groups. The dihedral angle between the rings is 51.8 (2)° in one molecule and 61.5 (2)° in the other. The N-methyl C atom deviates 0.2 Å out of the naphthyridine plane. The length of the central C4=C4^i double bond [1.373 (6) Å] is a little longer than a typical C=C double-bond distance (1.33 Å), and the neighboring C1—C4^i [1.457 (7) Å] and C3—C4 [1.417 (7) Å] single bonds are shorter than a typical C—C single bond (1.54 Å), reflecting the conjugation in the naphthyridine ring [symmetry code: (i) $-x$, $-y$, $1 - z$].
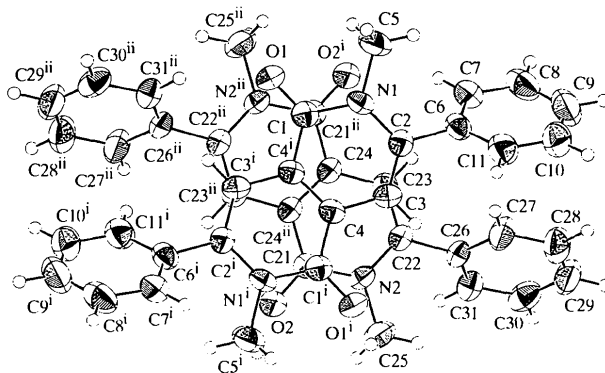

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the two independent molecules of 2,6-dimethyl-3,7-diphenyl-2,6-naphthyridine-1,5(2H,6H)-dione with the atomic numbering scheme. Displacement ellipsoids of non-H atoms are shown at the 50% probability level. H atoms are shown as spheres of arbitrary radii. Symmetry codes are as in Table 1.

The two naphthyridine rings of adjacent molecules are almost parallel and cross rectangularly with respect to each other, with non-bonded distances of more than 3.3 Å (a van der Waals contact) between them. The phenyl rings in the two molecules are rotated in opposite directions in order to reduce the repulsion between them.

Experimental

Crystal data

$C_{22}H_{18}N_2O_2$
$M_r = 342.4$
Monoclinic
$P2_1/n$
$a = 23.138 (5)$ Å
$b = 11.220 (2)$ Å
$c = 6.882 (1)$ Å
$\beta = 93.94 (2)°$
$V = 1782.4 (6)$ Å^3
$Z = 4$
$D_x = 1.276$ Mg m^{-3}
$D_m = 1.26$ Mg m^{-3}
D_m measured by flotation in aqueous KI solution

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 21 reflections
$\theta = 4.5$–$12.3°$
$\mu = 0.083$ mm^{-1}
$T = 298$ K
Needle
$0.30 \times 0.15 \times 0.10$ mm
Colorless

Data collection

MacScience MXC3 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
4646 measured reflections
4081 independent reflections
1598 reflections with
$I > 2\sigma(I)$

$R_{int} = 0.036$
$\theta_{max} = 26.43°$
$h = -30 \rightarrow 29$
$k = -14 \rightarrow 0$
$l = 0 \rightarrow 8$
3 standard reflections
every 100 reflections
intensity decay: <1%

Refinement

Refinement on F
$R = 0.067$
$wR = 0.069$
$S = 1.041$
2515 reflections ($I > 0$)
235 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

C1—N1	1.389 (7)	C21—N2	1.389 (6)
C1—C4^i	1.457 (7)	C21—C24^{ii}	1.469 (7)
C1—O1	1.238 (6)	C21—O2	1.230 (6)
N1—C2	1.397 (7)	N2—C22	1.391 (7)
N1—C5	1.481 (7)	N2—C25	1.476 (7)
C2—C3	1.353 (7)	C22—C23	1.351 (7)
C2—C6	1.491 (7)	C22—C26	1.493 (7)
C3—C4	1.417 (7)	C23—C24	1.412 (7)
C4—C4^i	1.373 (6)	C24—C24^{ii}	1.383 (7)
C6—C7	1.387 (8)	C26—C27	1.388 (8)
C6—C11	1.389 (8)	C26—C31	1.371 (7)
C7—C8	1.386 (9)	C27—C28	1.394 (9)
C8—C9	1.364 (10)	C28—C29	1.360 (10)
C9—C10	1.365 (11)	C29—C30	1.360 (10)
C10—C11	1.383 (9)	C30—C31	1.387 (8)
N1—C1—C4^i	116.0 (5)	N2—C21—C24^{ii}	115.7 (5)
N1—C1—O1	120.8 (5)	N2—C21—O2	121.2 (5)
C4^i—C1—O1	123.1 (5)	C24^{ii}—C21—O2	123.2 (5)
C1—N1—C2	122.8 (4)	C21—N2—C22	123.6 (4)
C1—N1—C5	115.2 (4)	C21—N2—C25	114.9 (4)
C2—N1—C5	121.8 (4)	C22—N2—C25	121.4 (4)
N1—C2—C3	119.9 (5)	N2—C22—C23	119.8 (5)
N1—C2—C6	119.7 (5)	N2—C22—C26	119.2 (4)
C3—C2—C6	120.4 (5)	C23—C22—C26	121.0 (5)
C2—C3—C4	120.5 (5)	C22—C23—C24	120.3 (5)
C1^i—C4—C3	119.3 (4)	C21^{ii}—C24—C23	119.5 (4)
C1^i—C4—C4^i	120.7 (5)	C21^{ii}—C24—C24^{ii}	119.6 (5)
C3—C4—C4^i	120.0 (5)	C23—C24—C24^{ii}	120.8 (5)
C2—C6—C7	122.4 (5)	C22—C26—C27	118.5 (5)
C2—C6—C11	118.5 (5)	C22—C26—C31	122.4 (5)

C7—C6—C11	118.8 (5)	C27—C26—C31	119.0 (5)
C6—C7—C8	119.6 (6)	C26—C27—C28	120.1 (6)
C7—C8—C9	120.9 (6)	C27—C28—C29	119.8 (6)
C8—C9—C10	120.2 (7)	C28—C29—C30	120.4 (6)
C9—C10—C11	119.9 (7)	C29—C30—C31	120.4 (6)
C6—C11—C10	120.6 (6)	C26—C31—C30	120.3 (5)

Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $-x, -y, -z$.

The title structure was solved by direct methods and refined by full-matrix least-squares calculations. H atoms were placed at geometrically idealized positions with C—H bond lengths of 0.96 Å and isotropic displacement parameters fixed at 0.06 Å².

Data collection: MXC diffractometer system (MacScience, 1989). Cell refinement: MXC diffractometer system (MacScience, 1989). Data reduction: CRYSTAN (Gilmore & Brown, 1988). Program(s) used to solve structure: CRYSTAN. Program(s) used to refine structure: CRYSTAN. Molecular graphics: ORTEP (Johnson, 1965) in CRYSTAN. Software used to prepare material for publication: CRYSTAN.

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Two Diastereoisomers of 2-(Benzenesulfonyl)-5-benzoyl-1-oxo-3-phenyl-2,5-diazaspiro[3.4]octan-7-yl Acetate

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Abstract

The structures of two diastereoisomers of the title compound, $C_{27}H_{24}N_2O_6S$, were determined by single-crystal diffractometry. The two molecules are very similar as far as bond distances and angles are concerned, but more substantial differences are observed in some torsion angles.

Comment

Two diastereoisomers of the title compound were analysed by single-crystal diffractometry, that is, the 3S,4S,7R, (I), and 3R,4S,7R, (II), isomers. Both compounds were synthesized starting from the natural product 4-hydroxy-L-proline (La Rosa & Dalla Croce, 1998), hence the stereochemistry at the C7 atom is known. The X-ray analysis of these compounds was strongly suggested by the difficulty in assigning the relative configurations to the other two asymmetric centres (C3 and C4) by NMR spectroscopy. A complete knowledge of the stereochemistry of β-lactams is required in order to study the biological activity of this class of compounds.

(I)   (II)
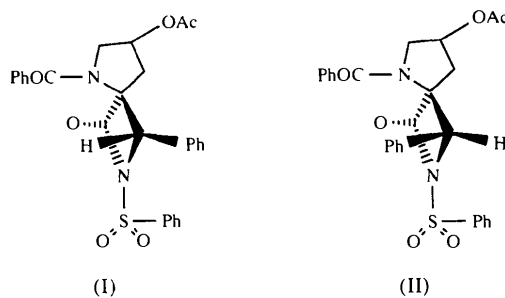
The geometries of the two molecules are very similar as far as bond distances and angles are concerned, the greatest differences being 0.042 (6) Å in the distances (C1—N2) and 5.3 (4)° in the angles (C3—N2—S22).

The geometric parameters of the two molecules (Figs. 1 and 2) are not very different from the values found in the literature for other β-lactam derivatives. We only mention that the bonds involving the N atom in the four-membered ring of compound (II) are slightly more elongated [C1—N2 1.422 (5) and N2—C3 1.516 (5) Å (Table 4) compared with C_{sp^2}—N 1.385 (19) and N—C_{sp^3} 1.464 (12) Å reported for β-lactams in International Tables for Crystallography (1995, Vol. C)]. This indicates, in compound (II), a reduced conjugation of the N2 atom with the C1=O21 group, and it is accompanied by some degree of pyramidalization of N2 which is out of the plane formed by C1, C3 and S22 by 0.344 (4) Å [sum of angles at N2 is 344.4 (5)°]. In compound (I), this distance decreases to 0.159 (2) Å and the angles at N2 sum to 356.5 (3)°. In this case, the four-membered ring deviates from planarity, having an average torsion angle of 5.5 (1)° and a maximum deviation from the least-squares plane of the ring of 0.046 (3) Å for C1, while in compound (II), the ring is nearly planar, the average torsion angle being 0.6 (1)°. The O atom (O21) of the β-lactam is also in the least-squares plane of the ring, the atom–plane distances being 0.071 (2) Å for (I) and 0.128 (4) Å for (II).

The four-membered ring is nearly perpendicular to the least-squares plane of the five-membered ring, the two planes forming a dihedral angle of 86 (1)° in (I) and 82 (1)° in (II). The puckering analysis (Cremer & Pople,