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N,N'-Bis(4-pyridyl)naphthalene-3,4:7,8-dicarboximide dimethylformamide disolvate

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Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.006 Å
R factor = 0.097
wR factor = 0.309
Data-to-parameter ratio = 12.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{24}H_{12}N_4O_4\cdot 2C_3H_7NO$, the naphthalene imide, which is an organic pigment utilized for H_2 gas sensors, has inversion symmetry. The pyridyl rings are twisted, in the same direction, out of the plane of the naphthalene imide skeleton by 69.5 (2)°.

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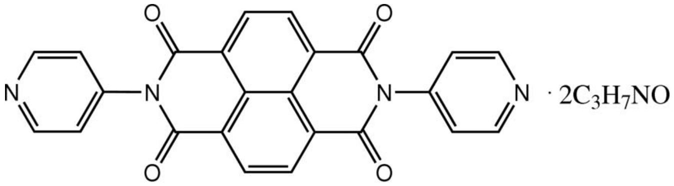
Comment

Naphthalene imide compounds are industrially important pigments on the market, side-by-side with perylene imides (Herbst & Hunger, 1993). The title compound, (I), is a *para*-naphthalene imide (p-NI) dimethylformamide (DMF) disolvate. We have carried out a series of investigations on H_2 gas sensors utilizing the high proton affinity of organic pigments that have pyridyl rings connected directly to the chromophore (Takahashi & Mizuguchi, 2005). The N atom of the pyridyl ring acts as a strong proton acceptor and plays a decisive role for signal detection. Among these, we have so far determined the structure of *ortho*-dipyridylpyrrolopyrrole (Imoda *et al.*, 2005) and the polymorph of the *para* derivative (Mizuguchi *et al.*, 2002, 2005), as well as *ortho*, *meta* and *para* derivatives of dipyridylperylene imides (Mizuguchi *et al.*, 2005*a*,*b*; Hino *et al.*, 2005, respectively). The present paper reports the structure of p-NI·2DMF, (I).



(I)

Fig. 1 shows the *ORTEP* (Burnett & Johnson, 1996) plot of (I). The skeleton of the centrosymmetric p-NI is entirely planar, as indicated by a small mean deviation of 0.006 Å from the least-squares plane of the rings defined by atoms C1–C7/N1. The pyridyl rings are twisted by 69.5 (2)°, in the same direction, out of the plane of the naphthalene imide skeleton. There is a short intermolecular contact of 2.802 (8) Å between the carbonyl C atom of the skeleton (C4) and the carbonyl O atom of DMF (O3; symmetry code: $1 - x, 1 - y, 1 - z$). This is due to an electrostatic interaction between positively polarized C4 and negatively polarized O3. Because of this, two DMF molecules are bound to the p-NI skeleton as shown in Fig. 1.

Fig. 2 shows the molecular arrangement of (I). There are two kinds of stacking columns along the a axis, composed of p-NI and DMF molecules. Between two DMF columns, the polar DMF molecules are arranged in one column in such a way as to cancel the dipole moment of DMFs in the neighboring one, leading to the stabilization of the crystal structure.

Experimental

p-NI was synthesized by reaction of naphthalenetetracarboxylic dianhydride with 4-aminopyridine in dimethylnaphthalene at 473 K for 3 h according to the method in the literature (Herbst & Hunger, 1993). The product was then purified three times by sublimation at 760 K, using a two-zone furnace (Mizuguchi, 1981). Single crystals of (I) were grown from a mixed solvent of acetone and dimethylformamide by slow evaporation.

Crystal data

$C_{24}H_{12}N_4O_4 \cdot 2C_3H_7NO$
$M_r = 566.57$
Monoclinic, $P2_1/c$
$a = 5.3890\ (8)$ Å
$b = 12.2012\ (17)$ Å
$c = 19.998\ (3)$ Å
$\beta = 91.764\ (10)°$
$V = 1314.3\ (3)$ Å^3
$Z = 2$
$D_x = 1.432$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 8845 reflections
$\theta = 3.6\text{–}68.1°$
$\mu = 0.85$ mm^{-1}
$T = 93.1$ K
Needle, gold
0.50 × 0.10 × 0.05 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.625$, $T_{\max} = 0.958$
12221 measured reflections
2241 independent reflections
1357 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.048$
$\theta_{\max} = 68.2°$
$h = -5 \rightarrow 5$
$k = -14 \rightarrow 14$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.097$
$wR(F^2) = 0.309$
$S = 1.11$
2241 reflections
187 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.05$ e Å^{-3}
$\Delta\rho_{\min} = -0.73$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.246 (5)	N1—C3	1.412 (5)
O2—C4	1.226 (5)	N1—C4	1.434 (5)
C8—N1—C3	114.4 (3)	O1—C3—N1	121.2 (3)
C8—N1—C4	119.0 (3)	O2—C4—N1	120.7 (3)
C3—N1—C4	126.6 (3)		

The H atoms, except for the methyl H atoms, were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The methyl H atoms were constrained to an ideal geometry, with C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), but each group was allowed to rotate freely about its C—C bond. The highest peak of electron density is located 0.94 Å from H15*C*. The DMF molecule shows relatively large atomic displacement parameters (Fig. 1), suggesting positional disorder.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

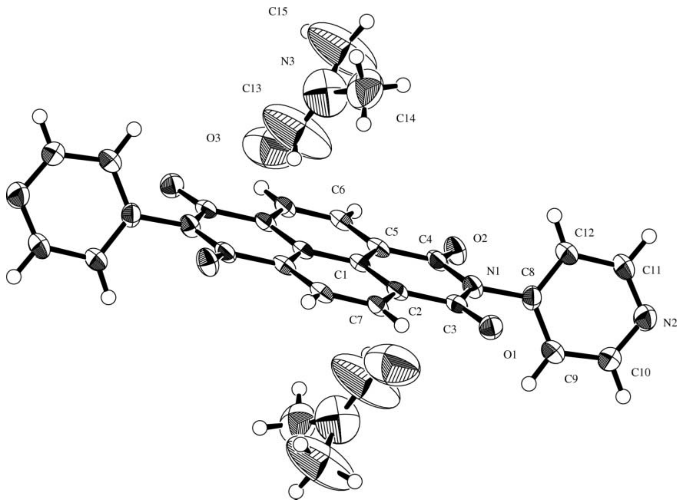


Figure 1
A view of the structure of (I), showing 50% probability displacement ellipsoids. The unlabelled atoms are related to labelled atoms by the symmetry code (1 − *x*, 1 − *y*, 1 − *z*).

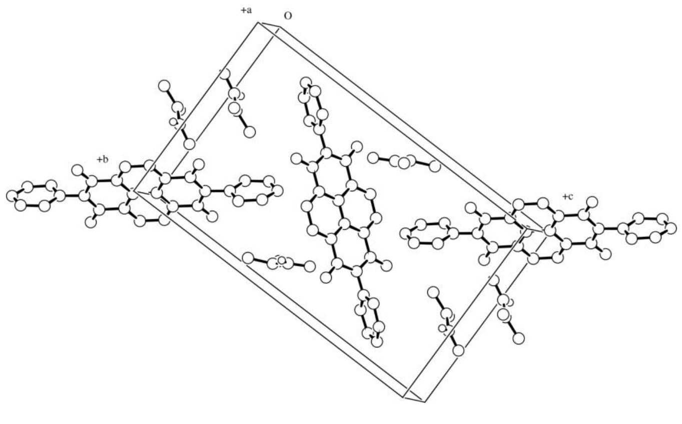


Figure 2
The molecular arrangement of (I).

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