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Naphthalene-2,6-dicarboxylic acid–1-methylpyrrolidin-2-one (1/2)

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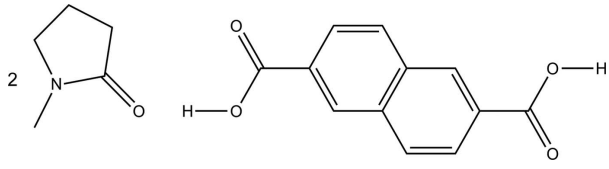
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 7.4.

The asymmetric unit of the title compound, $C_{12}H_8O_4\cdot 2C_5H_9NO$, contains one half-molecule of naphthalene-2,6-dicarboxylic acid (NDA) and one molecule of 1-methylpyrrolidin-2-one (NMP): the NDA molecules lie on the crystallographic twofold rotation axes. In the crystal, the components are linked by strong O—H···O hydrogen bonds and C—H···O interactions.

Related literature

For the crystal structure of naphthalene-2,6-dicarboxylic acid (NDA), see: Kaduk & Golab (1999). For the crystal structure of *N*-methyl-2-Pyrrolidone (NMP), see: Müller *et al.* (1996). For the purification of NDA, see: Nagase *et al.* (2004). For related structures, see: Guo *et al.* (2009); Dale & Elsegood (2004).



Experimental

Crystal data

$C_{12}H_8O_4\cdot 2C_5H_9NO$ M_r = 414.45
Orthorhombic, *Fdd*2
a = 19.7306 (11) Å
b = 28.7632 (19) Å
c = 7.1906 (4) Å
V = 4080.8 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 K
0.30 × 0.11 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur Atlas Gemini ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.987, T_{max} = 0.990
3255 measured reflections
1017 independent reflections
847 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 1.05
1017 reflections
138 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3	0.82	1.75	2.556 (3)	165
C2—H2···O2^{i}	0.93	2.48	3.163 (4)	131
C8—H8A···O2	0.97	2.47	3.311 (4)	145

Symmetry code: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{1}{4}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 12008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2234).

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supporting information

Acta Cryst. (2011). E**67**, o208 [https://doi.org/10.1107/S1600536810052396]

Naphthalene-2,6-dicarboxylic acid–1-methylpyrrolidin-2-one (1/2)

Bianhua Wu, Ge Peng, Youwei Cheng, Xi Li and Jiyong Liu

S1. Comment

Naphthalene-2,6-dicarboxylic acid (NDA) is an important monomer for producing polyester and polyurethane materials and liquid crystal polymers (LCP). During the manufacturing process, the impurities in NDA, such as 6-formyl-2-naphthoic acid (FNA), debase the quality of the products dramatically. So the purification of NDA is very important however, this process is difficult (Nagase *et al.*, 2004). Although many methods have been proposed in this field, they are either too complex or not cost effective. Recently, we have obtained crystals of the title compound, a mixture of NDA and *N*-Methyl Pyrrolidone (NMP). We call this phenomenon adductive crystallization and intend to apply this crystallization technique to the purification of NDA.

NDA crystallizes in the triclinic space group $P\bar{1}$ (Kaduk & Golab, 1999), while NMP crystlalizes in the monoclinc space group $P2_1/c$ (Müller *et al.*, 1996). There have also been some reports on the adductive crystallization of dicarboxylic acids and amides, such as Terephthalic acid (TA) and *N*,*N*-dimethylacetamide (Guo *et al.*, 2009) and TA and *N*,*N*-dimethyl-formamide (Dale & Elsegood, 2004).

The title compound crystallized in the orthorhombic space group Fdd2, and the molecular structure is shown in Fig. 1. The asymmetric unit contains one half-molecule of NDA and one molecule of NMP. The pyrrolidone group has an envelope conformation with atom C9 at the flap. The dihedral angle between the mean planes of the naphthalene ring of the NDA molecule and the pyrrolidone ring of the NMP molecule is 22.39 (15)°.

In the crystal the NDA and NMP molecules are linked by strong O—H···O hydrogen bonds and C-H···O interactions (Fig. 2 and Table 1).

S2. Experimental

The title compound was obtained by putting 0.1 g of Naphthalene-2,6-dicarboxylic acid (NDA) into 1 ml of *N*-Methyl Pyrrolidone (NMP) at room temperature and then leaving the mixture in the freezer, which was maintained at 255 K, for 72 h. During the process, we observed the gradual disappearance of the NDA powder and the appearance of colourless needle-like crystals of the title compound.

S3. Refinement

In the final cycles of refinement, in the absence of significant anomalous scattering effects, Friedel pairs were merged and Δf '' set to zero. The H-atoms were placed in calculated positions and were refined using a riding model: O—H = 0.82 Å, C—$H_{aromatic}$ = 0.93 Å, C—H_{alkyl} = 0.97 Å, C—H_{methyl} = 0.96 Å, with U_{iso}(H) = k × U_{eq}(O or C), where k = 1.5 for CH_3 H-atoms and k = 1.2 for all other H-atoms.

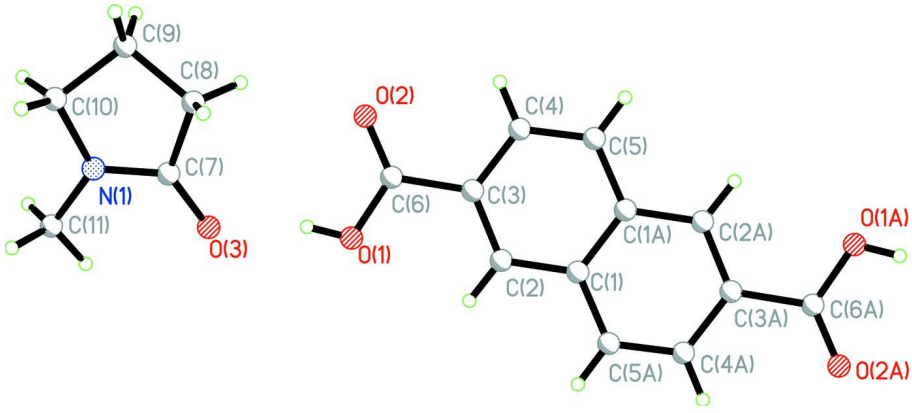

Figure 1

The molecular structure of the Naphthalene-2,6-dicarboxylic acid molecule and one N-Methyl-2-Pyrrolidone molecule of the title compound. Displacement ellipsoids are drawn at the 50% probability level [Symmetry code: A = -x + 0.5, -y + 0.5, z].

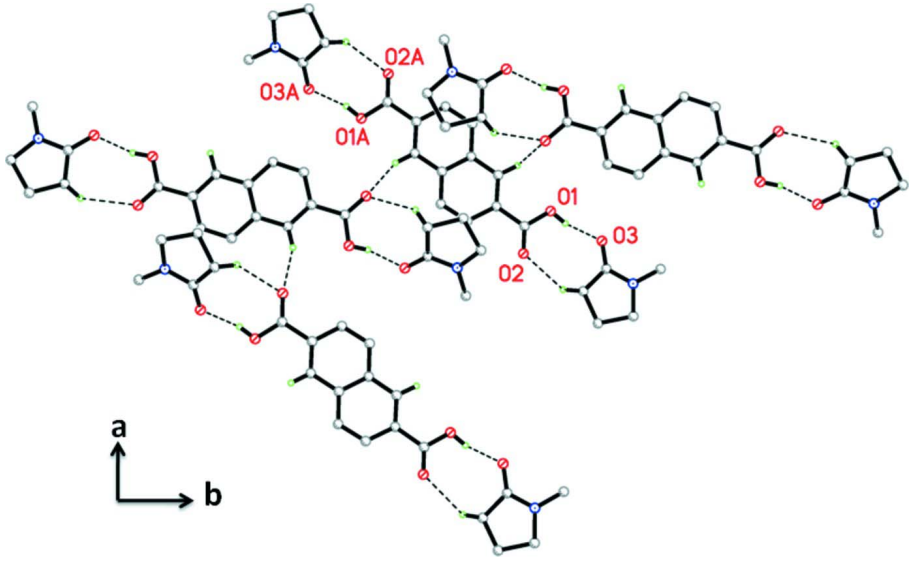

Figure 2

The crystal packing viewed along the c-axis of the title compound, showing the intermolecular O-H···O hydrogen bonds and C-H···O interactions as dashed lines [see Table 1 for details].

Naphthalene-2,6-dicarboxylic acid–1-methylpyrrolidin-2-one (1/2)

Crystal data

$C_{12}H_8O_4·2C_5H_9NO$
$M_r = 414.45$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 19.7306$ (11) Å
$b = 28.7632$ (19) Å
$c = 7.1906$ (4) Å
$V = 4080.8$ (4) Å^3
$Z = 8$
$F(000) = 1760$
$D_x = 1.349$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1127 reflections
$\theta = 3.1$–29.2°
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Needle, colourless
0.30 × 0.11 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur Atlas Gemini ultra diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.3592 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.987, T_{max} = 0.990
3255 measured reflections
1017 independent reflections
847 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.4°, θ_{min} = 3.1°
h = −23→19
k = −22→34
l = −8→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 1.05
1017 reflections
138 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 1.8717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Experimental. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm; CrysAlis PRO (Oxford Diffraction, 2009).

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.24703 (10)	0.09264 (7)	0.7871 (3)	0.0278 (7)
O2	0.13547 (10)	0.10088 (8)	0.7582 (4)	0.0385 (8)
C1	0.27857 (13)	0.23501 (10)	0.7441 (4)	0.0168 (8)
C2	0.26776 (13)	0.18651 (10)	0.7497 (4)	0.0180 (8)
C3	0.20359 (13)	0.16856 (10)	0.7495 (4)	0.0177 (8)
C4	0.14695 (13)	0.19829 (10)	0.7411 (4)	0.0200 (9)
C5	0.15563 (13)	0.24549 (10)	0.7410 (4)	0.0191 (9)
C6	0.19144 (14)	0.11756 (10)	0.7638 (4)	0.0200 (8)
O3	0.23717 (10)	0.00444 (7)	0.8202 (3)	0.0252 (7)
N1	0.18459 (12)	−0.06402 (9)	0.8830 (4)	0.0223 (7)
C7	0.18914 (15)	−0.01786 (11)	0.8877 (4)	0.0219 (9)
C8	0.12795 (15)	0.00109 (11)	0.9868 (5)	0.0252 (9)
C9	0.07849 (14)	−0.03970 (11)	0.9918 (5)	0.0259 (10)
C10	0.12448 (14)	−0.08267 (11)	0.9734 (5)	0.0256 (10)
C11	0.23599 (16)	−0.09402 (11)	0.8060 (5)	0.0294 (10)

H1	0.23680	0.06520	0.80000	0.0420*
H2	0.30480	0.16650	0.75370	0.0220*
H4	0.10350	0.18580	0.73570	0.0240*
H5	0.11800	0.26490	0.73890	0.0230*
H8A	0.10880	0.02720	0.91940	0.0300*
H8B	0.13950	0.01110	1.11160	0.0300*
H9A	0.04650	−0.03800	0.88950	0.0310*
H9B	0.05360	−0.04030	1.10810	0.0310*
H10A	0.10330	−0.10650	0.89760	0.0310*
H10B	0.13540	−0.09560	1.09430	0.0310*
H11A	0.21610	−0.11380	0.71350	0.0440*
H11B	0.27090	−0.07550	0.75000	0.0440*
H11C	0.25520	−0.11270	0.90320	0.0440*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0248 (11)	0.0165 (11)	0.0421 (14)	−0.0012 (9)	0.0016 (10)	0.0040 (11)
O2	0.0264 (12)	0.0216 (12)	0.0675 (17)	−0.0061 (10)	−0.0131 (13)	0.0029 (13)
C1	0.0178 (13)	0.0197 (15)	0.0130 (13)	−0.0010 (12)	0.0015 (13)	0.0011 (14)
C2	0.0217 (14)	0.0186 (15)	0.0138 (14)	0.0029 (12)	0.0002 (13)	−0.0012 (14)
C3	0.0199 (15)	0.0193 (15)	0.0138 (14)	−0.0001 (13)	−0.0024 (12)	−0.0009 (14)
C4	0.0165 (14)	0.0266 (18)	0.0170 (14)	−0.0037 (13)	−0.0015 (14)	0.0019 (14)
C5	0.0178 (14)	0.0213 (17)	0.0183 (14)	0.0027 (12)	−0.0011 (13)	0.0007 (14)
C6	0.0214 (14)	0.0187 (15)	0.0200 (15)	−0.0002 (14)	−0.0025 (13)	0.0012 (14)
O3	0.0240 (12)	0.0195 (11)	0.0322 (13)	−0.0027 (10)	0.0047 (10)	0.0025 (11)
N1	0.0213 (12)	0.0209 (13)	0.0247 (13)	0.0002 (12)	0.0021 (11)	0.0037 (12)
C7	0.0235 (15)	0.0213 (16)	0.0209 (15)	0.0002 (15)	−0.0058 (13)	0.0006 (14)
C8	0.0256 (15)	0.0263 (17)	0.0238 (16)	0.0049 (14)	0.0001 (13)	−0.0018 (17)
C9	0.0229 (15)	0.0301 (19)	0.0246 (16)	0.0012 (14)	0.0011 (15)	0.0034 (16)
C10	0.0250 (16)	0.0281 (18)	0.0237 (17)	−0.0052 (15)	0.0004 (13)	0.0065 (15)
C11	0.0313 (18)	0.0259 (18)	0.0309 (17)	0.0064 (15)	0.0019 (15)	0.0017 (16)

Geometric parameters (Å, º)

O1—C6	1.321 (3)	C4—H4	0.9300
O2—C6	1.205 (3)	C5—H5	0.9300
O1—H1	0.8200	C7—C8	1.504 (4)
O3—C7	1.243 (4)	C8—C9	1.527 (4)
N1—C7	1.331 (4)	C9—C10	1.539 (4)
N1—C10	1.455 (4)	C8—H8A	0.9700
N1—C11	1.442 (4)	C8—H8B	0.9700
C1—C5^{i}	1.414 (4)	C9—H9A	0.9700
C1—C1^{i}	1.419 (4)	C9—H9B	0.9700
C1—C2	1.412 (4)	C10—H10A	0.9700
C2—C3	1.367 (4)	C10—H10B	0.9700
C3—C6	1.490 (4)	C11—H11A	0.9600
C3—C4	1.409 (4)	C11—H11B	0.9600

C4—C5	1.368 (4)	C11—H11C	0.9600
C2—H2	0.9300		

C6—O1—H1	109.00	C7—C8—C9	104.2 (3)
C10—N1—C11	121.6 (3)	C8—C9—C10	103.8 (2)
C7—N1—C10	114.3 (2)	N1—C10—C9	102.9 (2)
C7—N1—C11	124.0 (3)	C7—C8—H8A	111.00
C2—C1—C5^{i}	122.1 (2)	C7—C8—H8B	111.00
C1^{i}—C1—C5^{i}	119.2 (3)	C9—C8—H8A	111.00
C1^{i}—C1—C2	118.7 (2)	C9—C8—H8B	111.00
C1—C2—C3	120.9 (2)	H8A—C8—H8B	109.00
C4—C3—C6	118.2 (2)	C8—C9—H9A	111.00
C2—C3—C6	121.4 (2)	C8—C9—H9B	111.00
C2—C3—C4	120.4 (3)	C10—C9—H9A	111.00
C3—C4—C5	120.2 (2)	C10—C9—H9B	111.00
C1^{i}—C5—C4	120.6 (2)	H9A—C9—H9B	109.00
O1—C6—O2	123.3 (3)	N1—C10—H10A	111.00
O2—C6—C3	122.5 (3)	N1—C10—H10B	111.00
O1—C6—C3	114.2 (2)	C9—C10—H10A	111.00
C3—C2—H2	120.00	C9—C10—H10B	111.00
C1—C2—H2	120.00	H10A—C10—H10B	109.00
C3—C4—H4	120.00	N1—C11—H11A	109.00
C5—C4—H4	120.00	N1—C11—H11B	110.00
C4—C5—H5	120.00	N1—C11—H11C	109.00
C1^{i}—C5—H5	120.00	H11A—C11—H11B	109.00
O3—C7—N1	123.8 (3)	H11A—C11—H11C	110.00
O3—C7—C8	127.6 (3)	H11B—C11—H11C	109.00
N1—C7—C8	108.6 (3)		

C10—N1—C7—O3	179.0 (3)	C1—C2—C3—C4	0.9 (4)
C10—N1—C7—C8	−0.7 (4)	C2—C3—C6—O1	3.7 (4)
C11—N1—C7—O3	2.7 (5)	C2—C3—C4—C5	−2.8 (4)
C11—N1—C7—C8	−177.1 (3)	C6—C3—C4—C5	175.4 (3)
C7—N1—C10—C9	15.8 (4)	C4—C3—C6—O2	4.1 (4)
C11—N1—C10—C9	−167.8 (3)	C2—C3—C6—O2	−177.7 (3)
C2—C1—C5^{i}—C4^{i}	−178.1 (3)	C4—C3—C6—O1	−174.5 (3)
C2—C1—C1^{i}—C5	−2.9 (4)	C3—C4—C5—C1^{i}	1.8 (4)
C1^{i}—C1—C2—C3	1.9 (4)	O3—C7—C8—C9	165.4 (3)
C5^{i}—C1—C2—C3	−178.9 (3)	N1—C7—C8—C9	−14.9 (3)
C2—C1—C1^{i}—C2^{i}	176.3 (3)	C7—C8—C9—C10	23.5 (3)
C5^{i}—C1—C1^{i}—C5	177.9 (3)	C8—C9—C10—N1	−23.5 (3)
C1—C2—C3—C6	−177.3 (3)		

Symmetry code: (i) −*x*+1/2, −*y*+1/2, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3	0.82	1.75	2.556 (3)	165

C2—H2···O2^{ii}	0.93	2.48	3.163 (4)	131
C8—H8*A*···O2	0.97	2.47	3.311 (4)	145

Symmetry code: (ii) $x+1/4, -y+1/4, z+1/4$.