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4-Nitrobenzoic acid–2,2′-biimidazole (2/1)

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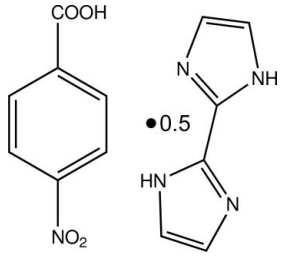
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.134; data-to-parameter ratio = 11.7.

In the title adduct, $C_7H_5NO_4{\cdot}0.5C_6H_6N_4$, the complete biimidazole molecule is generated by a crystallographic inversion centre. In the crystal, N—H···O and O—H···N hydrogen bonds connects the 4-nitrobenzoic acid and 2,2′-biimidazole units, affording multi-dimensional frameworks with graph-set descriptor $R_2^2(9)$.

Related literature

For the potential applications of coordination complexes as functional materials and enzymes, see: Zhang *et al.* (2003) For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_7H_5NO_4{\cdot}0.5C_6H_6N_4$
M_r = 234.19
Monoclinic, $P2_1/c$
a = 4.852 (1) Å
b = 10.9245 (10) Å
c = 19.7981 (10) Å
β = 90.496 (1)°
V = 1049.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.986, T_{max} = 0.991
5185 measured reflections
1849 independent reflections
1264 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 1.00
1849 reflections
158 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N1	0.85 (1)	1.75 (1)	2.580 (2)	168 (3)
N2—H2···O1^{i}	0.86	1.89	2.742 (2)	173

Symmetry code: (i) $-x+2, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2278).

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supporting information

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4-Nitrobenzoic acid–2,2′-biimidazole (2/1)

Xin Liu and Weiqun Zhu

S1. Comment

Recently, the design and synthesis of coordination complexes have attracted much attention due to their diversity structures as well as potential applications as functional materials and enzymes (Zhang *et al.* , 2003). Here, we report one by-product of the hydrothermal reaction of $FeCl_3$ with 4-nitrobenzoic acid and biimidazole. The asymmetric unit of (I) consists of a 4-nitrobenzoic acid molecule and half biimidazole molecule, Fig 1. In the 4-nitrobenzoic acid molecule, the nitro group is rotated 10.6 (3)° from aromatic ring. N—H···O and O—H···N hydrogen bonds connects the $C_7H_5NO_4$. $0.5C_6H_6N_4$ units to affords a macrocycle with graph-set descriptor $R^2_2(9)$ (Bernstein *et al., 1995), Fig2.*

S2. Experimental

A mixture of 4-nitrobenzoic acid (1 mmoL, 0.17 g), biimidazole (1 mmoL, 0.14 g), and iron trichloride (1 mmoL, 0.27 g) in 12 ml distilled water sealed in a 25 ml Teflon-lined stainless steel autoclave was kept at 433 K for three days. Colorless crystals suitable for the single X-ray diffraction were obtained.

S3. Refinement

All H atoms were placed in calculated positions with C—H = 0.93Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier). The lengths of bond H—O were constrained with 0.82 Å .

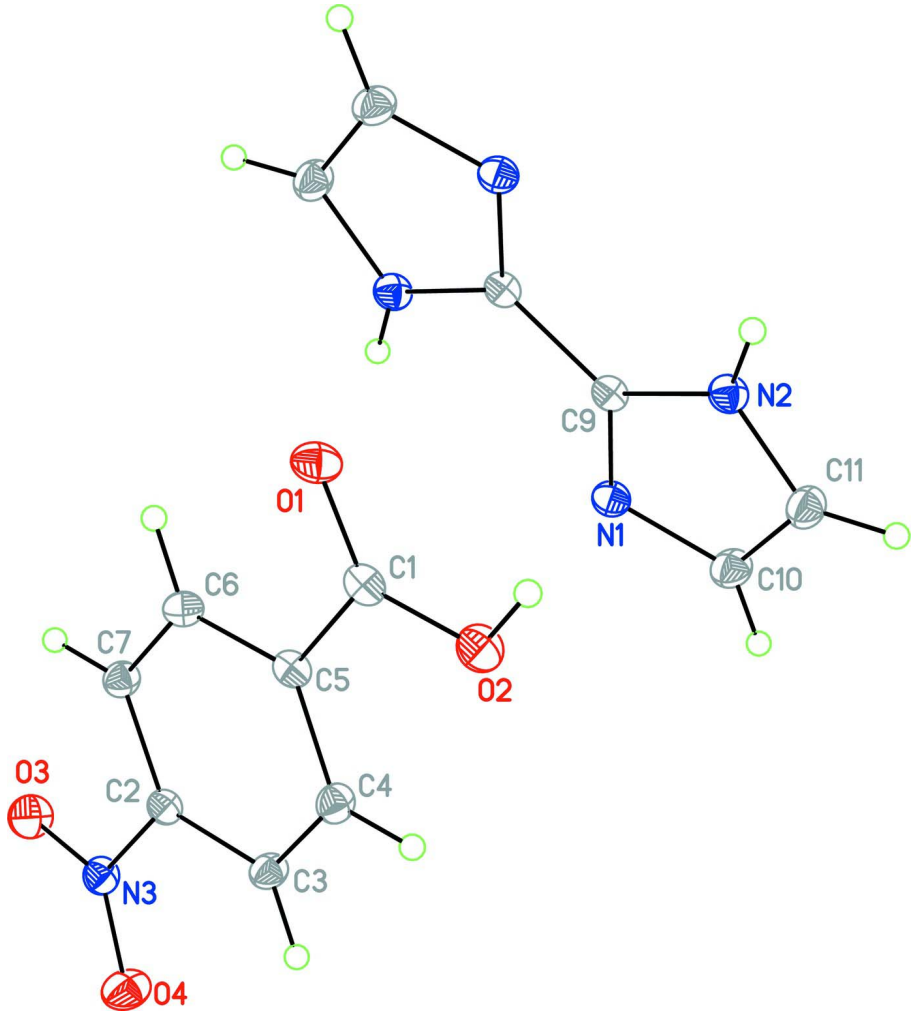

Figure 1

The molecular structure of (I), showing the atom-labelling scheme and with displacement ellipsoids at the 30% probability level. Unlabeled atoms are related to labeled atoms by the symmetry code (-x, 2-y, 1-z).

4-Nitrobenzoic acid–2,2′-biimidazole (2/1)

Crystal data

$C_7H_5NO_4·0.5C_6H_6N_4$
$M_r = 234.19$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.852$ (1) Å
$b = 10.9245$ (10) Å
$c = 19.7981$ (10) Å
$\beta = 90.496$ (1)°
$V = 1049.4$ (2) Å^3
$Z = 4$
$F(000) = 484$
$D_x = 1.482$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1230 reflections
$\theta = 2.8$–22.0°
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Block, colourless
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.986, T_{max} = 0.991
5185 measured reflections
1849 independent reflections
1264 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.0°, θ_{min} = 2.1°
h = −5→5
k = −12→9
l = −23→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 1.00
1849 reflections
158 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.078P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.013 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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 $F^2 > 2$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}
C1	0.3475 (4)	0.7795 (2)	0.58109 (11)	0.0548 (6)
C2	−0.2424 (4)	0.54924 (18)	0.67279 (9)	0.0493 (5)
C3	−0.1097 (4)	0.5072 (2)	0.61681 (10)	0.0577 (6)
H3	−0.1475	0.4298	0.5995	0.069*
C4	0.0825 (4)	0.5826 (2)	0.58629 (11)	0.0586 (6)
H4	0.1753	0.5557	0.5482	0.070*
C5	0.1368 (4)	0.69760 (19)	0.61233 (10)	0.0502 (5)
C6	−0.0063 (4)	0.7371 (2)	0.66845 (10)	0.0565 (6)
H6	0.0283	0.8148	0.6858	0.068*
C7	−0.1990 (4)	0.66362 (19)	0.69917 (11)	0.0551 (6)
H7	−0.2963	0.6908	0.7366	0.066*
C9	0.9965 (4)	0.95887 (18)	0.47185 (10)	0.0468 (5)
C10	0.8856 (5)	0.8140 (2)	0.40287 (11)	0.0676 (7)
H10	0.7961	0.7472	0.3836	0.081*
C11	1.0980 (5)	0.8751 (2)	0.37532 (11)	0.0678 (7)

H11	1.1813	0.8580	0.3342	0.081*
H2A	0.566 (4)	0.7886 (19)	0.5083 (11)	0.080*
N1	0.8218 (3)	0.86605 (16)	0.46396 (9)	0.0549 (5)
N2	1.1672 (3)	0.96604 (16)	0.41881 (8)	0.0550 (5)
H2	1.2968	1.0188	0.4135	0.066*
N3	−0.4451 (4)	0.46869 (19)	0.70582 (9)	0.0596 (5)
O1	0.4057 (3)	0.87721 (14)	0.60756 (8)	0.0689 (5)
O2	0.4566 (3)	0.73838 (16)	0.52653 (8)	0.0739 (5)
O3	−0.5946 (4)	0.50989 (16)	0.74887 (10)	0.0873 (6)
O4	−0.4548 (4)	0.36262 (17)	0.68800 (10)	0.0926 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0442 (12)	0.0579 (14)	0.0621 (14)	−0.0011 (10)	−0.0014 (10)	0.0114 (11)
C2	0.0449 (11)	0.0507 (12)	0.0522 (12)	−0.0036 (9)	0.0044 (9)	0.0051 (10)
C3	0.0628 (14)	0.0509 (12)	0.0595 (13)	−0.0100 (10)	0.0119 (11)	−0.0058 (10)
C4	0.0577 (14)	0.0624 (14)	0.0559 (13)	−0.0052 (11)	0.0124 (10)	−0.0028 (11)
C5	0.0427 (12)	0.0505 (13)	0.0574 (12)	−0.0028 (9)	−0.0024 (10)	0.0076 (10)
C6	0.0561 (13)	0.0481 (13)	0.0653 (13)	−0.0028 (10)	0.0007 (11)	−0.0026 (10)
C7	0.0543 (13)	0.0554 (13)	0.0556 (12)	0.0002 (10)	0.0060 (10)	−0.0031 (10)
C9	0.0399 (11)	0.0483 (12)	0.0523 (11)	0.0004 (9)	0.0024 (9)	0.0056 (9)
C10	0.0701 (16)	0.0621 (15)	0.0705 (15)	−0.0161 (12)	0.0037 (12)	−0.0095 (12)
C11	0.0719 (16)	0.0709 (16)	0.0609 (14)	−0.0068 (13)	0.0132 (12)	−0.0095 (13)
N1	0.0520 (11)	0.0516 (10)	0.0610 (11)	−0.0074 (8)	0.0014 (8)	0.0017 (9)
N2	0.0500 (10)	0.0544 (11)	0.0609 (11)	−0.0053 (8)	0.0088 (9)	0.0025 (9)
N3	0.0581 (12)	0.0634 (13)	0.0573 (11)	−0.0071 (10)	0.0092 (9)	0.0029 (9)
O1	0.0602 (10)	0.0563 (10)	0.0904 (11)	−0.0113 (8)	0.0095 (8)	0.0033 (9)
O2	0.0713 (12)	0.0737 (12)	0.0769 (11)	−0.0217 (9)	0.0177 (9)	0.0076 (9)
O3	0.0907 (13)	0.0871 (13)	0.0849 (12)	−0.0056 (10)	0.0419 (10)	0.0034 (10)
O4	0.1113 (16)	0.0649 (12)	0.1021 (13)	−0.0320 (10)	0.0356 (11)	−0.0089 (10)

Geometric parameters (Å, º)

C1—O1	1.222 (3)	C7—H7	0.9300
C1—O2	1.288 (3)	C9—N1	1.330 (2)
C1—C5	1.496 (3)	C9—N2	1.345 (2)
C2—C3	1.366 (3)	C9—C9^{i}	1.432 (4)
C2—C7	1.370 (3)	C10—C11	1.347 (3)
C2—N3	1.476 (3)	C10—N1	1.374 (3)
C3—C4	1.387 (3)	C10—H10	0.9300
C3—H3	0.9300	C11—N2	1.355 (3)
C4—C5	1.382 (3)	C11—H11	0.9300
C4—H4	0.9300	N2—H2	0.8600
C5—C6	1.384 (3)	N3—O3	1.211 (2)
C6—C7	1.377 (3)	N3—O4	1.212 (2)
C6—H6	0.9300	O2—H2A	0.845 (10)

O1—C1—O2	124.7 (2)	C2—C7—H7	121.1
O1—C1—C5	120.1 (2)	C6—C7—H7	121.1
O2—C1—C5	115.2 (2)	N1—C9—N2	110.41 (18)
C3—C2—C7	122.95 (19)	N1—C9—C9^{i}	125.5 (2)
C3—C2—N3	118.65 (19)	N2—C9—C9^{i}	124.1 (2)
C7—C2—N3	118.40 (18)	C11—C10—N1	109.3 (2)
C2—C3—C4	118.5 (2)	C11—C10—H10	125.3
C2—C3—H3	120.7	N1—C10—H10	125.3
C4—C3—H3	120.7	C10—C11—N2	106.98 (19)
C5—C4—C3	120.3 (2)	C10—C11—H11	126.5
C5—C4—H4	119.9	N2—C11—H11	126.5
C3—C4—H4	119.9	C9—N1—C10	105.70 (18)
C4—C5—C6	119.17 (19)	C9—N2—C11	107.60 (17)
C4—C5—C1	121.2 (2)	C9—N2—H2	126.2
C6—C5—C1	119.7 (2)	C11—N2—H2	126.2
C7—C6—C5	121.3 (2)	O3—N3—O4	122.56 (19)
C7—C6—H6	119.3	O3—N3—C2	119.7 (2)
C5—C6—H6	119.3	O4—N3—C2	117.72 (18)
C2—C7—C6	117.76 (19)	C1—O2—H2A	113.3 (17)

C7—C2—C3—C4	1.6 (3)	C5—C6—C7—C2	0.7 (3)
N3—C2—C3—C4	−179.36 (18)	N1—C10—C11—N2	−0.4 (3)
C2—C3—C4—C5	−0.1 (3)	N2—C9—N1—C10	−0.6 (2)
C3—C4—C5—C6	−1.1 (3)	C9^{i}—C9—N1—C10	178.9 (2)
C3—C4—C5—C1	178.72 (18)	C11—C10—N1—C9	0.6 (3)
O1—C1—C5—C4	−175.01 (18)	N1—C9—N2—C11	0.4 (2)
O2—C1—C5—C4	4.7 (3)	C9^{i}—C9—N2—C11	−179.1 (2)
O1—C1—C5—C6	4.8 (3)	C10—C11—N2—C9	0.0 (2)
O2—C1—C5—C6	−175.56 (18)	C3—C2—N3—O3	−168.9 (2)
C4—C5—C6—C7	0.7 (3)	C7—C2—N3—O3	10.2 (3)
C1—C5—C6—C7	−179.06 (18)	C3—C2—N3—O4	10.8 (3)
C3—C2—C7—C6	−1.9 (3)	C7—C2—N3—O4	−170.2 (2)
N3—C2—C7—C6	179.04 (18)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1	0.85 (1)	1.75 (1)	2.580 (2)	168 (3)
N2—H2···O1^{i}	0.86	1.89	2.742 (2)	173

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