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# 2,2′-[Pyridine-2,6-diylbis(carbonylhydrazono)]dipropanoic acid *N*,*N*-dimethylformamide disolvate

**Yanling Qiao, Jichun Cui, Longhua Ding and Handong Yin***

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: handongyin@163.com

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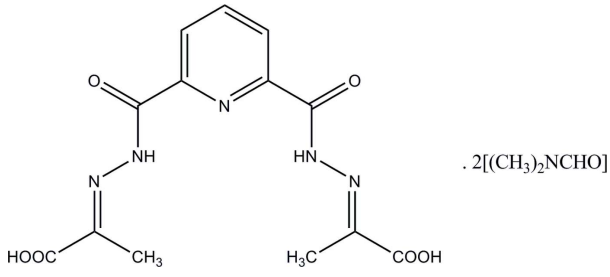
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.045; $wR$ factor = 0.137; data-to-parameter ratio = 12.8.

The complete molecule of the title compound, $C_{13}H_{13}N_5O_6\cdot 2C_3H_7NO$, is generated by crystallographic twofold rotation with an N and a C atom lying on the axis. The structure is stabilized by intermolecular O—H···O hydrogen bonds.

## Related literature

For the synthesis and structures of some organotin(IV) complexes of related tridentate hydrazone ligands see: Yin *et al.* (2008).




. 2[$(CH_3)_2NCHO$]

## Experimental

### Crystal data

$C_{13}H_{13}N_5O_6\cdot 2C_3H_7NO$ $M_r$ = 481.48
Monoclinic, $C2/c$
$a$ = 19.5743 (17) Å
$b$ = 10.4041 (11) Å
$c$ = 11.7924 (12) Å
$\beta$ = 107.684 (1)°
$V$ = 2288.1 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 298 K
0.40 × 0.39 × 0.17 mm

### Data collection

Siemens CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.957, $T_{max}$ = 0.981
5528 measured reflections
2017 independent reflections
1102 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.137
$S$ = 1.00
2017 reflections
158 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O4 | 0.82 | 1.75 | 2.574 (3) | 178 |

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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*.

We acknowledge the National Natural Foundation of China (20771053), the Scientific Research Fund of Liaocheng University (X081006) and the Students Science and Technology Innovation Fund of Liaocheng University (SRT08031HX2).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2660).

## References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yin, H., Cui, J. & Qiao, Y. (2008). *Polyhedron*, **27**, 2157–2166.

# supporting information

*Acta Cryst.* (2009). E**65**, o2652 [https://doi.org/10.1107/S160053680903997X]

## 2,2′-[Pyridine-2,6-diylbis(carbonylhydrazono)]dipropanoic acid *N*,*N*-dimethylformamide disolvate

**Yanling Qiao, Jichun Cui, Longhua Ding and Handong Yin**

### S1. Comment

Recently, we have reported some organotin(IV) complexes with hydrazone ligands (Yin *et al.*, 2008). As an extension of our work on the structural characterization of hydrazone compounds, the title compound, (I), is reported here.

The two halves of the main molecule are symmetrically related, Fig. 1, with the N1, C4 and H4 atoms of the pyridine ring lying on the two-fold axis to form a helical species. The N3═C6 bond length of 1.283 (3) Å (Table 1) conforms to the value for a double bond, while the N2—C1 [1.361 (3) Å] and N2—N3 [1.369 (3) Å] bonds are intermediate between a double bond and a single bond because of conjugation effects in the molecule.

### S2. Experimental

Compound (I) was synthesized by the reaction of pyridine-2,6-dihydrazide (5 mmol) with pyruvic acid (10 mmol). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an N,N-dimethylformamide solution.

### S3. Refinement

The H atoms were positioned geometrically, with methyl C—H distances of 0.96 Å, aromatic and aldehydic C—H distances of 0.93 Å, N—H distances of 0.86 Å and O—H distances of 0.82 Å, and refined as riding on their parent atoms, with $U_{iso}$(H) = 1.2 $U_{eq}$(C, N) or 1.5 $U_{eq}$(C, O) for the methyl groups.

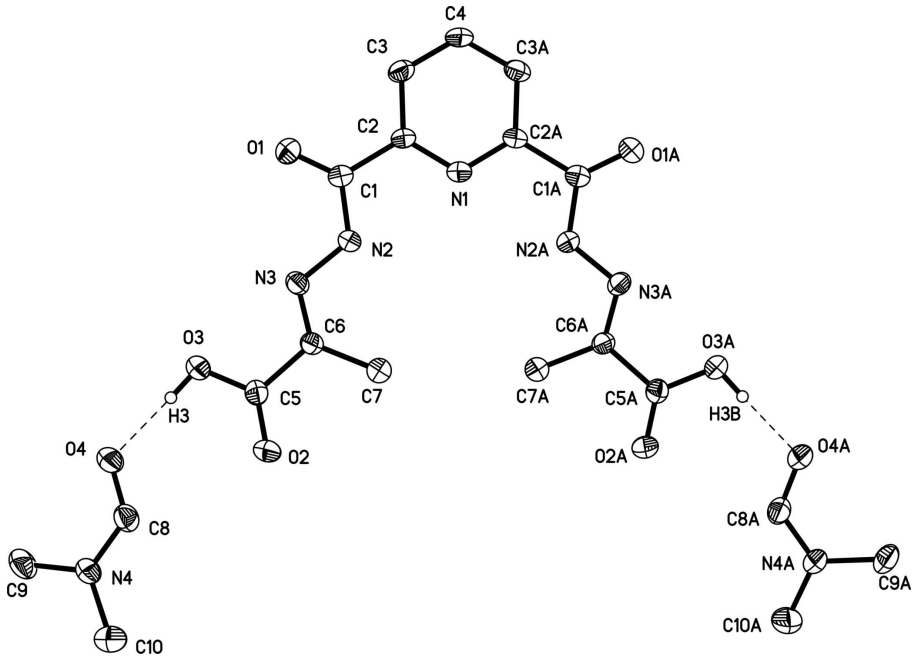


**Figure 1**

The structure of the title compound, with 50% probability displacement ellipsoids and hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

## 2,2′-[Pyridine-2,6-diylbis(carbonylhydrazono)]dipropanoic acid *N*,*N*-dimethylformamide disolvate

*Crystal data*

$C_{13}H_{13}N_5O_6\cdot 2C_3H_7NO$
$M_r = 481.48$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 19.5743\ (17)$ Å
$b = 10.4041\ (11)$ Å
$c = 11.7924\ (12)$ Å
$\beta = 107.684\ (1)°$
$V = 2288.1\ (4)$ Å$^3$
$Z = 4$
$F(000) = 1016$
$D_x = 1.398$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1027 reflections
$\theta = 2.2–21.9°$
$\mu = 0.11$ mm$^{-1}$
$T = 298$ K
Block, colorless
0.40 × 0.39 × 0.17 mm

*Data collection*

Siemens CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.957$, $T_{max} = 0.981$
5528 measured reflections
2017 independent reflections
1102 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -23\rightarrow23$
$k = -6\rightarrow12$
$l = -12\rightarrow14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.137$
$S = 1.00$
2017 reflections
158 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.8585P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.20$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.5000 | 0.2225 (3) | 0.7500 | 0.0370 (8) |
| N2 | 0.56173 (12) | 0.3597 (2) | 0.6173 (2) | 0.0440 (6) |
| H2 | 0.5454 | 0.3894 | 0.6721 | 0.053* |
| N3 | 0.58981 (11) | 0.4428 (2) | 0.55325 (18) | 0.0414 (6) |
| N4 | 0.71491 (12) | 1.0005 (2) | 0.3217 (2) | 0.0492 (7) |
| O1 | 0.58033 (11) | 0.18075 (19) | 0.52026 (18) | 0.0608 (6) |
| O2 | 0.62171 (12) | 0.76940 (19) | 0.54017 (18) | 0.0615 (6) |
| O3 | 0.64206 (11) | 0.60873 (17) | 0.43031 (16) | 0.0539 (6) |
| H3 | 0.6570 | 0.6669 | 0.3972 | 0.081* |
| O4 | 0.69084 (11) | 0.78824 (19) | 0.32604 (17) | 0.0546 (6) |
| C1 | 0.55919 (15) | 0.2310 (3) | 0.5960 (2) | 0.0414 (7) |
| C2 | 0.52726 (14) | 0.1563 (2) | 0.6765 (2) | 0.0365 (7) |
| C3 | 0.52768 (15) | 0.0235 (3) | 0.6730 (2) | 0.0472 (7) |
| H3A | 0.5462 | −0.0199 | 0.6199 | 0.057* |
| C4 | 0.5000 | −0.0427 (4) | 0.7500 | 0.0550 (12) |
| H4 | 0.5000 | −0.1320 | 0.7500 | 0.066* |
| C5 | 0.62039 (14) | 0.6566 (3) | 0.5162 (2) | 0.0417 (7) |
| C6 | 0.59176 (14) | 0.5610 (3) | 0.5853 (2) | 0.0394 (7) |
| C7 | 0.56634 (16) | 0.6134 (3) | 0.6825 (3) | 0.0569 (9) |
| H7A | 0.5151 | 0.6058 | 0.6610 | 0.085* |
| H7B | 0.5797 | 0.7023 | 0.6950 | 0.085* |
| H7C | 0.5878 | 0.5660 | 0.7544 | 0.085* |
| C8 | 0.69523 (15) | 0.8983 (3) | 0.3682 (3) | 0.0502 (8) |
| H8 | 0.6836 | 0.9088 | 0.4385 | 0.060* |
| C9 | 0.73224 (17) | 0.9912 (3) | 0.2111 (3) | 0.0645 (10) |
| H9A | 0.7245 | 0.9046 | 0.1818 | 0.097* |
| H9B | 0.7816 | 1.0141 | 0.2246 | 0.097* |
| H9C | 0.7021 | 1.0486 | 0.1536 | 0.097* |
| C10 | 0.7204 (2) | 1.1255 (3) | 0.3783 (3) | 0.0787 (11) |
| H10A | 0.6987 | 1.1220 | 0.4412 | 0.118* |
| H10B | 0.6961 | 1.1884 | 0.3206 | 0.118* |
| H10C | 0.7700 | 1.1488 | 0.4106 | 0.118* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0357 (18) | 0.0301 (17) | 0.0426 (18) | 0.000 | 0.0078 (15) | 0.000 |
| N2 | 0.0566 (16) | 0.0331 (13) | 0.0493 (14) | −0.0003 (12) | 0.0264 (12) | 0.0034 (11) |
| N3 | 0.0420 (14) | 0.0390 (14) | 0.0456 (14) | 0.0005 (12) | 0.0170 (11) | 0.0080 (12) |
| N4 | 0.0583 (16) | 0.0443 (14) | 0.0490 (15) | −0.0039 (13) | 0.0222 (13) | 0.0069 (13) |
| O1 | 0.0845 (16) | 0.0466 (13) | 0.0638 (14) | −0.0028 (12) | 0.0410 (12) | −0.0082 (11) |
| O2 | 0.0899 (17) | 0.0351 (12) | 0.0710 (15) | −0.0122 (12) | 0.0419 (13) | −0.0040 (11) |
| O3 | 0.0756 (14) | 0.0412 (12) | 0.0559 (13) | −0.0021 (11) | 0.0360 (11) | 0.0046 (10) |
| O4 | 0.0693 (14) | 0.0457 (13) | 0.0542 (13) | −0.0067 (11) | 0.0268 (11) | 0.0046 (11) |
| C1 | 0.0444 (17) | 0.0342 (16) | 0.0452 (17) | 0.0023 (14) | 0.0129 (14) | −0.0011 (14) |
| C2 | 0.0395 (16) | 0.0276 (15) | 0.0402 (16) | 0.0001 (13) | 0.0089 (13) | −0.0028 (13) |
| C3 | 0.0542 (19) | 0.0338 (16) | 0.0558 (19) | 0.0004 (15) | 0.0202 (15) | −0.0081 (15) |
| C4 | 0.075 (3) | 0.025 (2) | 0.073 (3) | 0.000 | 0.034 (3) | 0.000 |
| C5 | 0.0407 (17) | 0.0444 (19) | 0.0420 (17) | −0.0004 (15) | 0.0154 (14) | 0.0015 (14) |
| C6 | 0.0404 (17) | 0.0343 (16) | 0.0459 (16) | 0.0010 (14) | 0.0169 (13) | 0.0038 (14) |
| C7 | 0.075 (2) | 0.0429 (18) | 0.065 (2) | −0.0003 (17) | 0.0401 (18) | −0.0006 (16) |
| C8 | 0.055 (2) | 0.054 (2) | 0.0442 (17) | −0.0059 (17) | 0.0193 (15) | 0.0034 (16) |
| C9 | 0.081 (2) | 0.062 (2) | 0.057 (2) | −0.0082 (19) | 0.0322 (18) | 0.0150 (17) |
| C10 | 0.108 (3) | 0.048 (2) | 0.093 (3) | −0.009 (2) | 0.049 (2) | −0.008 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C2$^{i}$ | 1.339 (3) | C3—C4 | 1.375 (3) |
| N1—C2 | 1.339 (3) | C3—H3A | 0.9300 |
| N2—C1 | 1.361 (3) | C4—C3$^{i}$ | 1.375 (3) |
| N2—N3 | 1.369 (3) | C4—H4 | 0.9300 |
| N2—H2 | 0.8600 | C5—C6 | 1.499 (4) |
| N3—C6 | 1.283 (3) | C6—C7 | 1.485 (4) |
| N4—C8 | 1.308 (3) | C7—H7A | 0.9600 |
| N4—C9 | 1.447 (3) | C7—H7B | 0.9600 |
| N4—C10 | 1.451 (4) | C7—H7C | 0.9600 |
| O1—C1 | 1.211 (3) | C8—H8 | 0.9300 |
| O2—C5 | 1.205 (3) | C9—H9A | 0.9600 |
| O3—C5 | 1.310 (3) | C9—H9B | 0.9600 |
| O3—H3 | 0.8200 | C9—H9C | 0.9600 |
| O4—C8 | 1.241 (3) | C10—H10A | 0.9600 |
| C1—C2 | 1.503 (4) | C10—H10B | 0.9600 |
| C2—C3 | 1.382 (4) | C10—H10C | 0.9600 |
| | | | |
| C2$^{i}$—N1—C2 | 118.0 (3) | N3—C6—C7 | 126.3 (2) |
| C1—N2—N3 | 121.2 (2) | N3—C6—C5 | 117.3 (2) |
| C1—N2—H2 | 119.4 | C7—C6—C5 | 116.4 (2) |
| N3—N2—H2 | 119.4 | C6—C7—H7A | 109.5 |
| C6—N3—N2 | 115.1 (2) | C6—C7—H7B | 109.5 |
| C8—N4—C9 | 120.2 (3) | H7A—C7—H7B | 109.5 |
| C8—N4—C10 | 121.7 (3) | C6—C7—H7C | 109.5 |

| | | | |
|---|---|---|---|
| C9—N4—C10 | 118.0 (3) | H7A—C7—H7C | 109.5 |
| C5—O3—H3 | 109.5 | H7B—C7—H7C | 109.5 |
| O1—C1—N2 | 124.0 (3) | O4—C8—N4 | 125.1 (3) |
| O1—C1—C2 | 123.0 (2) | O4—C8—H8 | 117.4 |
| N2—C1—C2 | 113.0 (2) | N4—C8—H8 | 117.4 |
| N1—C2—C3 | 122.8 (3) | N4—C9—H9A | 109.5 |
| N1—C2—C1 | 117.8 (2) | N4—C9—H9B | 109.5 |
| C3—C2—C1 | 119.4 (2) | H9A—C9—H9B | 109.5 |
| C4—C3—C2 | 118.3 (3) | N4—C9—H9C | 109.5 |
| C4—C3—H3A | 120.9 | H9A—C9—H9C | 109.5 |
| C2—C3—H3A | 120.9 | H9B—C9—H9C | 109.5 |
| C3—C4—C3$^{i}$ | 119.9 (4) | N4—C10—H10A | 109.5 |
| C3—C4—H4 | 120.1 | N4—C10—H10B | 109.5 |
| C3$^{i}$—C4—H4 | 120.1 | H10A—C10—H10B | 109.5 |
| O2—C5—O3 | 124.2 (3) | N4—C10—H10C | 109.5 |
| O2—C5—C6 | 120.3 (3) | H10A—C10—H10C | 109.5 |
| O3—C5—C6 | 115.5 (2) | H10B—C10—H10C | 109.5 |

Symmetry code: (i) $-x+1, y, -z+3/2$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O4 | 0.82 | 1.75 | 2.574 (3) | 178 |