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N,*N*′-Bis(4-cyanopyridylidene)benzil dihydrazone

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.154
Data-to-parameter ratio = 17.1

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

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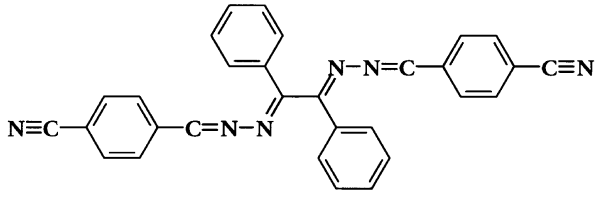
The title compound, $C_{30}H_{20}N_6$, was synthesized and its molecular structure was precisely characterized by low-temperature single-crystal analysis.

Comment

The title compound, (I), is a 1+2 Schiff base ligand of benzil dihydrazone and 4-cyanobenzaldehyde. It finds a wide range of applications in coordination chemistry owing to its polydentate chelating potential. The imino as well as the nitrilo N atoms can act as donor sites to this end. This compound can also be useful in the supramolecular assembly of hybrid organic–inorganic coordination polymers of varied dimensionality.



(I)

Experimental

1.19 g (5 mmol) of benzil dihydrazone, prepared by the method of Busch & Bailor (1956), was dissolved in 100 ml of anhydrous methanol. 1.31 g (10 mmol) of solid 4-cyanobenzaldehyde was added to, and dissolved in, this solution with warming. Then the yellow reaction mixture was refluxed for 8 h, while maintaining dry conditions. The solvent was evaporated to obtain the yellow product (m.p. 442–444 K). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a dilute acetonitrile solution of this compound. Yield 1.75 g (75%). The product was characterized by elemental analyses and ^{1}H NMR spectra. Analysis found (calculated): C 77.68 (77.56%), H 4.33 (4.34%), N 18.07 (18.10%). ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 8.46 (*s*, 2H), 7.89 (*d*, *J* = 8 Hz, 4H), 7.67–7.55 (*m*, 10H), 7.46 (*d*, *J* = 8 Hz, 4H).

Crystal data

$C_{30}H_{20}N_6$
M_r = 464.52
Triclinic, $P\overline{1}$
a = 9.4430 (3) Å
b = 10.1390 (3) Å
c = 14.1380 (7) Å
α = 96.023 (1)°
β = 95.676 (1)°
γ = 112.223 (2)°
V = 1231.94 (8) Å^3
Z = 2
D_x = 1.252 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2889 reflections
θ = 2.4–27.9°
μ = 0.08 mm^{-1}
T = 110 (2) K
Prisms, yellow
0.20 × 0.10 × 0.10 mm

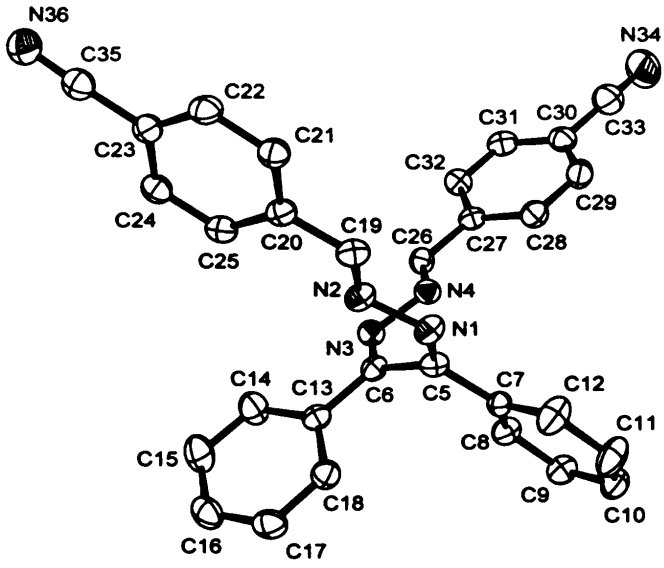


Figure 1
The molecular structure of the title compound. The ellipsoids represent displacement parameters at the 50% probability level at 110 K. Geometric parameters of the central part of this molecule include N—N bond lengths of 1.413 (2) and 1.415 (2) Å, N—Csp^2 bond lengths of 1.275 (2) and 1.277 (2) Å, N—Csp^3 bond lengths of 1.285 (2) and 1.289 (2) Å, and an N1—C5—C6—N3 torsion angle of 102.3 (2)°.

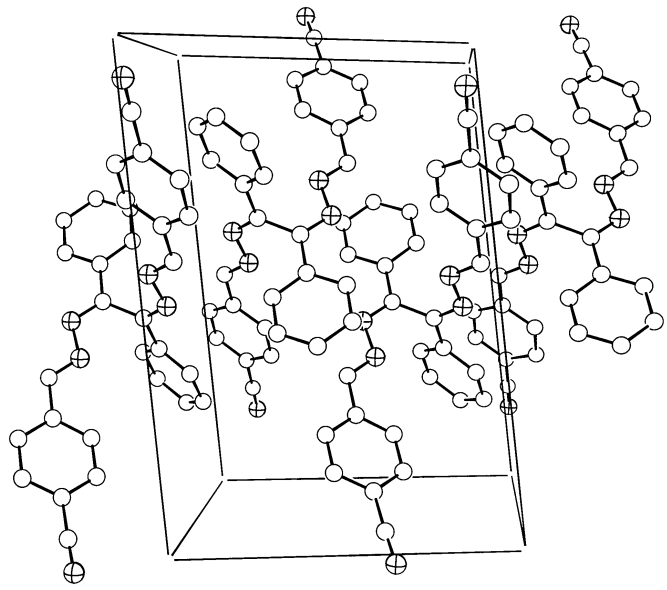


Figure 2
Crystal packing viewed approximately down the *a* axis (*b* is horizontal and *c* is vertical). The contents of two unit cells are shown. The N atoms are indicated by crossed circles. The crystal structure appears to be stabilized by π-stacking between the aryl groups, as well as by dipolar attractions between C—N dipoles of neighboring molecules displaced along *c*, which are arranged in an antiparallel manner with respect to one another.

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.054$
1.0° φ and ω scans	$\theta_{max} = 27.9°$
8099 measured reflections	$h = 0 \rightarrow 12$
5553 independent reflections	$k = -13 \rightarrow 12$
2889 reflections with $I > 2\sigma(I)$	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.154$	
$S = 0.97$	$(\Delta/\sigma)_{max} = 0.016$
5553 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
325 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski, 1985); data reduction: *DENZO*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97.

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References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Busch, D. H. & Bailor, J. C. (1956). *J. Am. Chem. Soc.* **78**, 1137–1142.
Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. (1985). *DENZO*. University of Texas Southwestern Medical Center at Dallas, Texas, USA.
Sheldrick. G. M. (1997). *SHELXL*97. University of Göttingen, Germany.