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Tetrakis(4-acetylpyridine)diisothiocyanatocobalt(II)

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.115
Data-to-parameter ratio = 19.1

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

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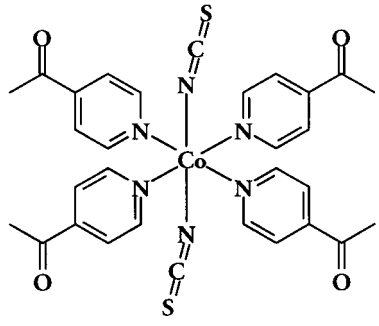
The title compound, $[Co(NCS)_2(C_7H_7NO)_4]$, was synthesized and its molecular structure was precisely characterized by low-temperature single-crystal analysis. The central cobalt ion has an octahedral coordination environment.

Comment

It has been demonstrated during the last few decades that halide or pseudo-halide complexes of cobalt(II) that contain pyridines and substituted pyridines undergo a series of interesting solid-state reactions (Agnew *et al.*, 1974; Liptay *et al.*, 1992). These complexes also exhibited selective enclathration of other species (Schaeffer *et al.*, 1957). Extensive differential scanning calorimetry and optical microscopic studies were carried out on the solid-state reactions of cobalt(II) pseudo-halide complexes containing substituted pyridines (Agnew & Brown, 1974). In the observed structure, the central Co atom is six-coordinate to four equatorial pyridyl donors and two axial *N*-bound thiocyanates, forming an octahedral structure.



(I)

Experimental

Ethylenediamine, 4-acetyl pyridine and cobalt(II) thiocyanate were obtained from Aldrich. The *N,N′*-bis(4-acetylpyrilidene)ethylenediamine component was prepared in the following manner: 1 ml (15 mmol) of distilled ethylenediamine and 3.32 ml (30 mmol) of freshly distilled 4-acetylpyridine were refluxed in 50 ml of anhydrous methanol for 6 h. On evaporation of the solvent, a yellow semi-solid was obtained, which on recrystallization from *n*-hexane gave colorless needles of *N,N′*-bis(4-acetylpyrilidene)ethylenediamine. Yield: 2.30 g (58%); m.p. 446–448 K. Analysis found (calculated): C 72.29 (72.14), H 6.92 (6.82), N 20.97% (21.04%). EI–MS: m/z 267.1 (MH^+, 90%), 237.1 ($MH^+ - 2CH_3$, 12%). ^{1}H NMR (200 MHz, $CDCl_3$, TMS): δ 8.64 (*d*, J = 4 Hz, 4H), 7.60 (*d*, J = 4 Hz, 4H), 3.94 (*s*, methylene, 4H), 2.28 (*s*, methyl, 6H). ^{13}C NMR (200 MHz, $CDCl_3$, TMS): δ 164.54, 150.14, 147.74, 121.19, 53.35, 15.66. 0.13 g (0.5 mmol) of the latter product was dissolved in 30 ml methanol, and 10 ml aqueous solution of 0.09 g

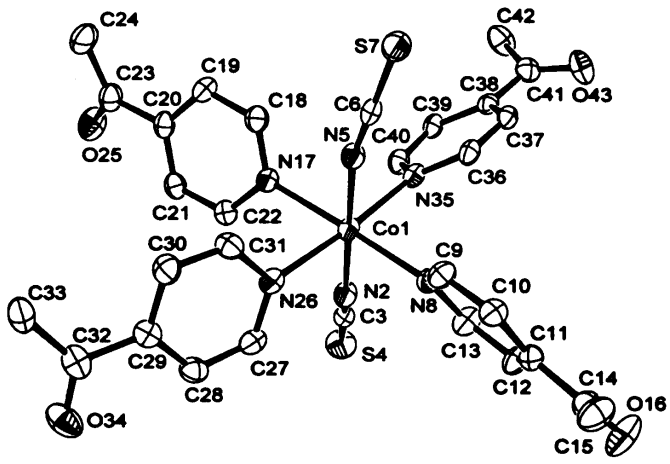


Figure 1
The molecular structure of the title compound. Ellipsoids are shown at the 50% probability level at 110 K. The coordination distances to the metal ion are: Co1—N2 2.067 (2), Co1—N5 2.072 (2), Co1—N8 2.183 (2), Co1—N17 2.224 (2), Co1—N26 2.205 (2) and Co1—N35 2.153 (2) Å.

(0.5 mmol) cobalt(II) thiocyanate was added with constant stirring. The resulting red–yellow solution was kept in the refrigerator for three days until yellow–red crystals of the title compound separated out. They were filtered off and dried in air. Yield: 0.15 g (45%).

Crystal data

$[Co(NCS)_2(C_7H_7NO)_4]$
M_r = 659.63
Orthorhombic, *Pbca*
a = 11.9180 (2) Å
b = 15.9700 (2) Å
c = 33.1930 (6) Å
V = 6317.64 (17) Å^3
Z = 8
D_x = 1.387 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4541 reflections
θ = 2.8–27.9°
μ = 0.72 mm^{-1}
T = 110 (2) K
Prism, yellow–red
0.25 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
1.0° φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.841, T_{max} = 0.932
14 852 measured reflections
7506 independent reflections
4541 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.9°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 43$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.115
S = 1.02
7506 reflections
392 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.1175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 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.

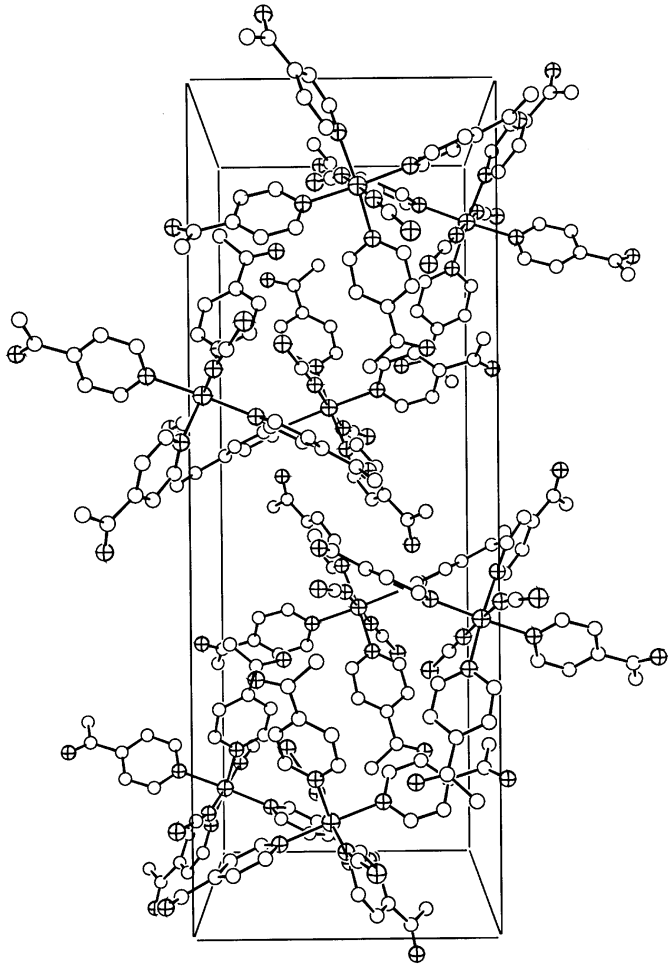


Figure 2
The crystal packing viewed down the *b* axis of the crystal (*a* is horizontal and *c* is vertical). Atoms are shown as arbitrarily sized spheres; all non-C atoms are denoted by crossed circles.

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