# Crystal structure of bis(2-pyridin-2-yl-ethanone)cobalt(II) dithiocyanate — 2-nitrophenylamine, $Co(C_7H_7ON)_2(SCN)_2 \cdot C_6H_6N_2O_2$

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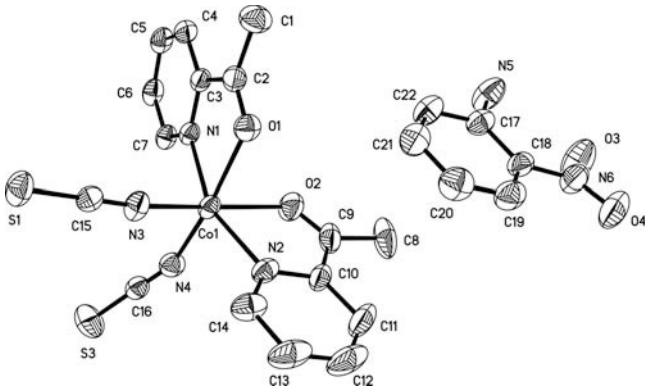


**Abstract**

$C_{22}H_{20}CoN_6O_4S_2$, triclinic, $P\bar{1}$ (no. 2), $a$ = 9.004(1) Å, $b$ = 11.727(1) Å, $c$ = 13.835(2) Å, $\alpha$ = 95.303(1)°, $\beta$ = 108.603(2)°, $\gamma$ = 111.017(2)°, $V$ = 1256.6 Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.092, $T$ = 298 K.

**Source of material**

2-Acetylpyridine (0.1 mmol, 12.1 mg), $NH_4SCN$ (0.1 mmol, 7.6 mg), $Co(CH_3COO)_2$ (0.1 mmol, 17.7 mg) and *o*-nitroaniline (0.1 mmol, 13.8 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at room temperature to give a clear brown solution. The title compound were obtained from the solution after 4 d. Crystals of the title compound suitable for X-ray diffraction experiment were recrystallized from methanol after one week at room temperature.

**Discussion**

The design of metal-organic coordination polymers is of current interest in the fields of supramolecular chemistry and crystal engineering because of their potential applications as functional materials [1,2]. It is well known that organic ligands containing O or N donors play crucial roles in the design and construction of desirable frameworks and they are widely used as building blocks to link metal ions and produce interesting metal-organic framework structures and properties [3].

The title crystal structure consists of $Co(C_7H_7ON)_2(SCN)_2$ molecules and uncoordinated *o*-nitroaniline molecules. 2-Acetylpyridine ligand bonds the metal ion through its carbonylic oxygen O1 and pyridine nitrogen N, two $SCN^-$ bond the metal through their nitrogen atoms, the $Co^{2+}$ is six-coordinated in form of a distorted octahedron. The $\{Co(C_7H_7NO)_2(SCN)_2\}$ molecule is asymmetric with respect to Co—N and Co—O bonds. The corresponding distances are $d$(Co—N1) = 2.107(2) Å, $d$(Co—O1) = 2.205(2) Å, $d$(Co—N3) = 2.031(2) Å, $d$(Co—N2) = 2.123(2) Å, $d$(Co—N4) = 2.046(3) Å, $d$(Co—O2) = 2.173(2) Å. The acute angles (O1–Co–N1 and O2–Co–N2) in the five-membered chelate ring are 74.73(8)° and 74.87(9)°, respectively, and other acute angle around Co (N3–Co–N4) is 94.9(1)°. The pyridine rings of 2-acetylpyridine ligands and the five-membered chelate ring are nearly coplanar, the torsion angles of C7–N1–C3–C2 and C14–N2–C10–C9 are 179.3(3) Å and –174.9(2) Å, respectively. To avoide the conflicts, the two rigid ring systems, ring A (N1/C3-C7) and ring B (N2/C10-C14) are rotated by 86.90°. A $\pi$-$\pi$ interaction dimer is formed between ring B and the phenyl ring with an parallel manner, of which the dihedral angle being 2.02°, the intercentroid distances being 4.719 Å and interplanar distances being 3.373 Å. All the intercentroid and interplanar distances are comparable to the reported previously [4]. There are some intramolecular (N5–H5B···O3) and intermolecular (N5–H5A···N4 ($x$–1,$y$,$z$) and N5–H5B···S3(–$x$+1,–$y$+1,–$z$+1) hydrogen bonds in the complex. These hydrogen bonds and $\pi$-$\pi$ stacking interactions link the 2-acetylpyridine ligands, $SCN^-$ ligands and an isolated *o*-nitroaniline molecule together, and play very important roles in the formation and stability of the title compound.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | brown block, size 0.35 × 0.37 × 0.45 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 8.89 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 50.02° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 6576, 4365 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 3422 |
| $N(param)_{refined}$: | 318 |
| Programs: | SHELXS-97 [5], SHELXL-97 [6], SHELXTL [7] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(5A) | 2*i* | 0.1523 | 0.5723 | 0.6385 | 0.117 |
| H(5B) | 2*i* | 0.1958 | 0.7095 | 0.6624 | 0.117 |
| H(1A) | 2*i* | 0.3425 | 0.2081 | 0.8359 | 0.118 |
| H(1B) | 2*i* | 0.2545 | 0.0783 | 0.7548 | 0.118 |
| H(1C) | 2*i* | 0.2064 | 0.1897 | 0.7249 | 0.118 |
| H(4) | 2*i* | 0.1668 | 0.0122 | 0.5693 | 0.070 |
| H(5) | 2*i* | 0.1392 | –0.0579 | 0.3993 | 0.073 |
| H(6) | 2*i* | 0.3629 | 0.0372 | 0.3469 | 0.067 |
| H(7) | 2*i* | 0.6120 | 0.1945 | 0.4653 | 0.059 |
| H(8A) | 2*i* | 0.7619 | 0.7377 | 0.6394 | 0.167 |
| H(8B) | 2*i* | 0.6640 | 0.7178 | 0.7162 | 0.167 |
| H(8C) | 2*i* | 0.5718 | 0.6372 | 0.6004 | 0.167 |

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**Table 2.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(11) | 2*i* | 0.9683 | 0.7982 | 0.8396 | 0.122 |
| H(12) | 2*i* | 1.2069 | 0.8267 | 0.9864 | 0.141 |
| H(13) | 2*i* | 1.2647 | 0.6580 | 1.0219 | 0.118 |
| H(14) | 2*i* | 1.0887 | 0.4620 | 0.9104 | 0.088 |
| H(19) | 2*i* | 0.6741 | 0.8677 | 0.9701 | 0.094 |
| H(20) | 2*i* | 0.7222 | 0.6943 | 1.0038 | 0.103 |
| H(21) | 2*i* | 0.5398 | 0.5007 | 0.8907 | 0.098 |
| H(22) | 2*i* | 0.3135 | 0.4806 | 0.7461 | 0.093 |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Co(1) | 2*i* | 0.76808(4) | 0.33841(3) | 0.70040(3) | 0.0494(2) | 0.0352(2) | 0.0422(2) | 0.0139(2) | 0.0100(2) | 0.0021(1) |
| N(1) | 2*i* | 0.5403(3) | 0.2002(2) | 0.5845(2) | 0.046(1) | 0.036(1) | 0.041(1) | 0.017(1) | 0.009(1) | 0.0056(9) |
| N(2) | 2*i* | 0.9306(3) | 0.5124(2) | 0.8115(2) | 0.061(2) | 0.045(1) | 0.051(1) | 0.009(1) | 0.026(1) | 0.002(1) |
| N(3) | 2*i* | 0.8611(3) | 0.2285(2) | 0.7837(2) | 0.070(2) | 0.048(1) | 0.054(1) | 0.025(1) | 0.013(1) | 0.011(1) |
| N(4) | 2*i* | 0.9135(3) | 0.3552(2) | 0.6108(2) | 0.058(2) | 0.056(2) | 0.058(2) | 0.017(1) | 0.021(1) | 0.005(1) |
| N(5) | 2*i* | 0.2171(4) | 0.6463(3) | 0.6782(2) | 0.102(2) | 0.061(2) | 0.083(2) | 0.017(2) | –0.000(2) | 0.005(2) |
| N(6) | 2*i* | 0.4429(4) | 0.8925(3) | 0.8186(3) | 0.093(2) | 0.057(2) | 0.098(2) | 0.016(2) | 0.045(2) | 0.006(2) |
| O(1) | 2*i* | 0.5751(2) | 0.2966(2) | 0.7730(1) | 0.062(1) | 0.055(1) | 0.049(1) | 0.018(1) | 0.020(1) | 0.0001(9) |
| O(2) | 2*i* | 0.6694(3) | 0.4723(2) | 0.6399(2) | 0.076(1) | 0.056(1) | 0.087(2) | 0.032(1) | 0.023(1) | 0.025(1) |
| O(3) | 2*i* | 0.3423(5) | 0.8908(3) | 0.7343(3) | 0.188(4) | 0.081(2) | 0.107(2) | 0.058(2) | 0.030(2) | 0.028(2) |
| O(4) | 2*i* | 0.5261(4) | 0.9876(3) | 0.8883(3) | 0.121(2) | 0.066(2) | 0.168(3) | 0.025(2) | 0.029(2) | –0.027(2) |
| S(1) | 2*i* | 0.9457(1) | 0.04636(8) | 0.87015(7) | 0.0839(6) | 0.0579(5) | 0.0717(5) | 0.0347(4) | 0.0145(5) | 0.0212(4) |
| S(3) | 2*i* | 1.1030(1) | 0.2924(1) | 0.51150(9) | 0.1013(8) | 0.1245(9) | 0.1055(8) | 0.0665(7) | 0.0595(7) | 0.0292(7) |
| C(1) | 2*i* | 0.2978(4) | 0.1677(3) | 0.7630(3) | 0.073(2) | 0.083(2) | 0.078(2) | 0.022(2) | 0.041(2) | 0.010(2) |
| C(2) | 2*i* | 0.4366(4) | 0.2094(3) | 0.7216(2) | 0.055(2) | 0.049(2) | 0.056(2) | 0.023(1) | 0.018(1) | 0.010(1) |
| C(3) | 2*i* | 0.4070(3) | 0.1477(2) | 0.6136(2) | 0.045(1) | 0.037(1) | 0.049(2) | 0.018(1) | 0.010(1) | 0.006(1) |
| C(4) | 2*i* | 0.2559(3) | 0.0494(3) | 0.5468(2) | 0.046(2) | 0.046(2) | 0.072(2) | 0.015(1) | 0.015(1) | 0.007(1) |
| C(5) | 2*i* | 0.2400(4) | 0.0077(3) | 0.4459(2) | 0.051(2) | 0.044(2) | 0.059(2) | 0.016(1) | –0.004(1) | –0.007(1) |
| C(6) | 2*i* | 0.3725(4) | 0.0631(2) | 0.4151(2) | 0.065(2) | 0.047(2) | 0.042(2) | 0.026(2) | 0.003(1) | 0.000(1) |
| C(7) | 2*i* | 0.5209(4) | 0.1580(2) | 0.4864(2) | 0.058(2) | 0.043(1) | 0.041(2) | 0.023(1) | 0.012(1) | 0.007(1) |
| C(8) | 2*i* | 0.6795(6) | 0.6768(4) | 0.6592(4) | 0.176(4) | 0.105(3) | 0.160(4) | 0.111(3) | 0.120(4) | 0.088(3) |
| C(9) | 2*i* | 0.7431(5) | 0.5798(3) | 0.6927(3) | 0.099(3) | 0.059(2) | 0.105(3) | 0.049(2) | 0.071(2) | 0.040(2) |
| C(10) | 2*i* | 0.8952(4) | 0.6113(3) | 0.7897(3) | 0.079(2) | 0.038(2) | 0.078(2) | 0.020(2) | 0.051(2) | 0.009(1) |
| C(11) | 2*i* | 0.9954(6) | 0.7307(3) | 0.8546(4) | 0.134(4) | 0.040(2) | 0.139(4) | 0.014(2) | 0.094(3) | –0.003(2) |
| C(12) | 2*i* | 1.1366(7) | 0.7474(4) | 0.9424(4) | 0.115(4) | 0.076(3) | 0.109(4) | –0.023(3) | 0.064(3) | –0.043(3) |
| C(13) | 2*i* | 1.1709(5) | 0.6484(4) | 0.9631(3) | 0.083(3) | 0.094(3) | 0.062(2) | –0.014(2) | 0.027(2) | –0.023(2) |
| C(14) | 2*i* | 1.0642(4) | 0.5305(3) | 0.8954(2) | 0.069(2) | 0.075(2) | 0.049(2) | 0.007(2) | 0.020(2) | –0.001(2) |
| C(15) | 2*i* | 0.8957(3) | 0.1528(2) | 0.8193(2) | 0.048(2) | 0.044(2) | 0.040(1) | 0.011(1) | 0.009(1) | –0.001(1) |
| C(16) | 2*i* | 0.9900(4) | 0.3284(2) | 0.5695(2) | 0.053(2) | 0.045(2) | 0.050(2) | 0.015(1) | 0.012(1) | 0.009(1) |
| C(17) | 2*i* | 0.3525(4) | 0.6633(3) | 0.7653(2) | 0.069(2) | 0.057(2) | 0.055(2) | 0.014(2) | 0.025(2) | 0.010(1) |
| C(18) | 2*i* | 0.4658(4) | 0.7796(3) | 0.8360(2) | 0.069(2) | 0.049(2) | 0.066(2) | 0.010(2) | 0.038(2) | 0.007(1) |
| C(19) | 2*i* | 0.6018(5) | 0.7897(3) | 0.9244(3) | 0.074(2) | 0.071(2) | 0.067(2) | 0.011(2) | 0.024(2) | –0.006(2) |
| C(20) | 2*i* | 0.6302(5) | 0.6870(4) | 0.9448(3) | 0.078(2) | 0.095(3) | 0.068(2) | 0.028(2) | 0.019(2) | 0.009(2) |
| C(21) | 2*i* | 0.5210(5) | 0.5716(3) | 0.8770(3) | 0.090(3) | 0.071(2) | 0.084(3) | 0.032(2) | 0.033(2) | 0.017(2) |
| C(22) | 2*i* | 0.3856(5) | 0.5597(3) | 0.7901(3) | 0.085(2) | 0.053(2) | 0.071(2) | 0.014(2) | 0.020(2) | 0.003(2) |

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