The Crystal Structure of 1,6-Dinitrobis(ethylenediamine)-cobalt(III) Nitrate

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A preliminary report on the investigation of the crystal structure of 1,6[Co $(NO_2)_2$ $(en)_2]NO_3$ is given in this note.

$1,6[Co(NO_3)_2(en)_2]NO_3$ was prepared by oxidation of a solution of cobalt(II) nitrate, sodium nitrite, and ethylenediamine, partially neutralized by nitric acid, in a stream of air.[1] Analysis of the compound yielded 18.1 % cobalt, the theoretical value being 17.7 %.

Integrated Weissenberg photographs about the *b*-axis ($h0l-h6l$) were recorded with Cu$K\alpha$ radiation. The crystals are of monoclinic symmetry and, from the systematically absent reflections, the space group could be determined to be No. 14 — $P2_1/a$, C_{2h}^5.[2] The dimensions of the unit cell, as calculated from powder diffraction photographs, are $a=13.819$ Å, $b=9.890$ Å, $c=9.095$ Å, and $\beta=105.83°$. The cell volume is 1170.4 Å^3. Assuming a cell content of four formula units, the calculated density is 1.89 g/cm^3. From three-dimensional Patterson calculations, based on the $h0l-h6l$ intensities, the cobalt atoms were found to occupy the general four-fold position 4 (*e*), ± (x, y, z; $\frac{1}{2}+x$, $\frac{1}{2}-y$, z), with $x=0.12$, $y=0.26$, and $z=0.25$. The positions of the light atoms were then deduced from successive electron density calculations. They were all found to occupy the general position 4 (*e*). After a least squares refinement the reliability index converged to $R=0.14$. The resulting parameters are listed in Table 1. The cobalt atom is octahedrally coordinated, through nitrogen atoms, by two NO_2-groups and two $C_2H_8N_2$-groups. The bond distances are Co—N (in NO_2) 1.81 Å and 2.02 Å, and Co—N (in $C_2H_8N_2$) 1.93—1.97 Å. A more detailed presentation of the structure of $1,6[Co(NO_2)_2$ $(en)_2]NO_3$ will be published shortly in this journal.

Table 1.

Atom	x	y	z
Co	0.1196	0.2593	0.2462
N (11)	0.2672	0.2389	0.3436
O (12)	0.3246	0.3378	0.3781
O (13)	0.3106	0.1240	0.3741
N (21)	−0.0137	0.2728	0.1497
O (22)	−0.0673	0.1750	0.1082
O (23)	−0.0515	0.3935	0.1285
N (31)	0.0915	0.1251	0.3856
C (32)	0.0989	0.1762	0.5360
C (33)	0.0723	0.3331	0.5199
N (34)	0.1109	0.3901	0.4020
N (41)	0.1327	0.1206	0.0926
C (42)	0.1923	0.1825	−0.0084
C (43)	0.1538	0.3341	−0.0374
N (44)	0.1586	0.4032	0.1147
N (51)	0.3776	0.2902	0.7582
O (52)	0.4624	0.3554	0.7837
O (53)	0.3042	0.3576	0.7192
O (54)	0.3743	0.1668	0.7605

1. *Inorg. Syn.* McGraw, New York 1953, Vol. IV, p. 177.
2. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1952, Vol. I.

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On the Crystal Structure of 1,2-Bromidopyridinebis(ethylenediamine)cobalt(III) Nitrate

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$1,2[CoBr(C_5H_5N)(C_2H_8N_2)_2](NO_3)_2$ has been prepared and its crystal structure investigated. The preliminary results of the investigation are presented in this note.

Weissenberg photographs of the layers $h0l-h10l$ have been recorded with Cu$K\alpha$ radiation. The crystals are of monoclinic symmetry and, from the systematically absent reflections, the space group could be determined to be No. 14 — $P2_1/n$, C_{2h}^5.[1] The cell dimensions, as calculated

Table 1.

Atom	x	y	z
Co	0.0644	0.2721	0.3170
Br	0.0110	0.1648	0.1263
N (11)	0.1166	0.3635	0.4604
C (12)	0.2222	0.3899	0.4351
C (13)	0.2739	0.3102	0.3773
N (14)	0.2069	0.2585	0.2620
N (21)	0.0403	0.3647	0.1692
C (22)	−0.0712	0.3860	0.1467
C (23)	−0.1225	0.3767	0.2925
N (24)	−0.0780	0.2881	0.3569
N (31)	0.0854	0.1697	0.4598
C (32)	0.0522	0.1864	0.6032
C (33)	0.0574	0.1100	0.7117
C (34)	0.1075	0.0348	0.6793
C (35)	0.1449	0.0318	0.5345
C (36)	0.1347	0.0984	0.4352
N (41)	0.3929	0.0925	0.2002
O (42)	0.4509	0.0432	0.1257
O (43)	0.3557	0.0528	0.3044
O (44)	0.3781	0.1655	0.1724
N (51)	0.1953	0.3332	0.8632
O (52)	0.2459	0.3275	0.9770
O (53)	0.2352	0.3109	0.7511
O (54)	0.1098	0.3541	0.8584

from Weissenberg data, are $a=13.5$ Å, $b=15.0$ Å, $c=9.5$ Å, and $\beta=94.0°$. The volume of the cell is thus approximately 1920 Å^3. Assuming a cell content of four formula units, a calculated density of 1.60 g/cm^3 is obtained which seems reasonable. From three-dimensional Patterson calculations, based on the $h0l-h10l$ intensities, the cobalt and bromine atoms were found to occupy the general four-fold position 4 (*e*), $\pm(x,y,z;\ \frac{1}{2}+x,\ \frac{1}{2}-y,\ \frac{1}{2}+z)$, with $x=0.064$, $y=0.273$, and $z=0.371$ (for cobalt) and $x=0.011$, $y=0.165$, and $z=0.126$ (for bromine). The positions of the light atoms were then deduced from successive electron density calculations and were all found to occupy the general four-fold position 4 (*e*). A preliminary least squares refinement yielded an R-value of 0.14, the resulting parameters being listed in Table 1. The cobalt atoms are octahedrally coordinated by one bromine atom, one C_5H_5N-group, and two $C_2H_8N_2$-groups with bond distances Co−Br 2.49 Å, Co−N (in C_5H_5N) 2.06 Å, and Co−N (in $C_2H_8N_2$) 1.99−2.04 Å.

A complete three-dimensional refinement is in progress, and a detailed presentation of the structure of $1,2[CoBr(C_5H_5N)(C_2H_8N_2)_2](NO_3)_2$ will be published in this journal.

1. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1952, Vol. I.

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