The Crystal Structure of 1,2-Dinitrobis(ethylenediamine)-cobalt(III) Nitrate

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In connection with studies of nitro and nitrito compounds of transition metals, $1,2[Co(NO_2)_2(en)_2]NO_3$ has been prepared and its crystal structure investigated, a preliminary report being given in this note.

$1,2[Co(NO_2)_2(en)_2]NO_3$ was prepared by heating ethylenediamine with potassium hexanitrocobaltate(III) to give $1,2[Co(NO_2)_2(en)_2]NO_2$, followed by conversion to the nitrate with nitric acid.[1] Analysis of the compound yielded 17.6 % cobalt, the theoretical value being 17.7 %.

Weissenberg photographs of the layers $hk0-hk5$ have been recorded, using 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. $14-P2_1/n$, $C_{2h}{}^5$.[2] The unit cell dimensions, as calculated from powder diffraction data, are $a=11.745$ Å, $b=16.090$ Å, $c=6.512$ Å, and $\beta=98.92°$. The volume of the cell is thus 1215.8 Å.[3] Assuming a cell content of four formula units, the calculated density is 1.82 g/cm³. From three-dimensional Patterson calculations, based on the $hk0-hk5$ intensities, the cobalt atoms were found to occupy the general four-fold position $4(e)$, $\pm(x,y,z \quad \frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$, with $x=0.30$, $y=0.11$, and $z=0.17$. The positions of the nitrogen, oxygen, and carbon atoms were then deduced from successive electron density calculations and were all found to occupy the general four-fold position. A three-dimensional least squares refinement of the structure was performed yielding an R-value of 0.09. 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) 1.89—1.91 Å and Co—N (in $C_2H_8N_2$) 1.94—1.99 Å. A more detailed presentation of the structure of $1,2[Co(NO_2)_2(en)_2]NO_3$ will be published shortly in this journal.

Table 1.

Atom	x	y	z
Co	0.2988	0.1054	0.1671
N(11)	0.3271	0.1671	0.4162
O(12)	0.3197	0.2432	0.4203
O(13)	0.3576	0.1328	0.5848
N(21)	0.4621	0.0925	0.1769
O(22)	0.5146	0.0388	0.2849
O(23)	0.5162	0.1442	0.0961
N(31)	0.3033	0.2954	0.0018
C(32)	0.1842	0.2371	−0.0755
C(33)	0.1067	0.2166	0.0740
N(34)	0.1298	0.1268	0.1446
N(41)	0.2868	0.0017	0.3232
C(42)	0.2917	−0.0736	0.1970
C(43)	0.2323	−0.0507	−0.0244
N(44)	0.2745	0.0330	−0.0837
N(51)	0.4963	0.4102	0.1089
O(52)	0.5944	0.4092	0.0648
O(53)	0.4158	0.3831	−0.0177
O(54)	0.4766	0.4447	0.2653

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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