Absolute Configuration of Sodium $(-)_{546}$-Bis[*trans*-*N*-methyl-(*S*)-alaninato]-oxalatocobaltate(III) Dihydrate by an *X*-Ray Crystal Structure Determination

By G. Wm. Svetich,* A. A. Voge, J. G. Brushmiller, and E. A. Berends
(*Chemistry Department, University of North Dakota, Grand Forks, North Dakota* 58201)

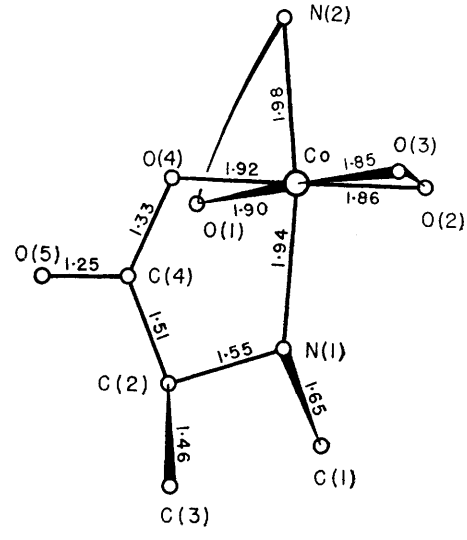
Summary An *X*-ray crystal structure of sodium $(-)_{546}$-bis[*trans*-*N*-methyl-(*S*)-alaninato]oxalatocobaltate(III) dihydrate (**1**) confirms the *R*–*S* configuration of the *N*-methyl-*S*-alanine ligands and the absolute configuration of the anion as *Λ*.

N-METHYL-*S*-ALANINE (L), when co-ordinated as a bidentate ligand in Co^{III} complexes, exists possibly in an *SS* or an *RS* configuration (the configuration at the asymmetric nitrogen atom is given first, the configuration at the alpha carbon atom is given second). Saburi and Yoshikawa[1] suggested, from n.m.r. and c.d. data, that L co-ordinates *RS* in $[Co(NH_3)_4L]^{2+}$. Berends and Brushmiller[2] determined, from n.m.r., the absolute configuration of sodium $(-)_{546}$-bis-[*trans*-*N*-methyl-(*S*)-alaninato]oxalatocobaltate(III) dihydrate (**1**) to be *Λ*. A structurally similar diamine, *N*-methyl-*S*-propylenediamine has been studied by Saburi[3] *et al.* They present additional evidence for the *RS* configuration of the co-ordinated diamine.

The c.d. spectrum of (**1**) has 3 bands: 390 nm, $\Delta\epsilon = -0{\cdot}300$; 555 nm, $\Delta\epsilon = +0{\cdot}645$; and 620 nm (shoulder), $\Delta\epsilon = +0{\cdot}370$.

Crystal data: $M = 412{\cdot}198$, hexagonal, $a = 13{\cdot}752$, c = 15·546 Å, $D_m = 1{\cdot}65$, $Z = 6$, $D_c = 1{\cdot}61$. Space group $P6_5$, Cu-K_α radiation. Data were collected by multiple film equi-inclination Weissenberg methods. A total of 675 reflections were investigated on layers *hk*0 through *hk*8 with 607 observed and 68 treated as unobserved. The structure was solved by Patterson and heavy atom Fourier methods. Refinement by full-matrix least-squares yielded a final *R* value of 0·078.

The absolute configuration of the anion of (**1**) is *Λ* with the nitrogens of the *N*-methyl-(*S*)-alanines situated *trans*. Both alanine rings are co-ordinated in the *RS* configuration (See Figure for one illustrated alanine ring). The dihedral angle between C(1)–N(1)–C(2) and C(3)–C(2)–N(1) is 69·6° for one alanine and 79·9° for the second alanine. The difference in the angles is attributed to crystal packing forces.



FIGURE

Pertinent bond lengths in the cobalt octahedron are presented in the Figure. Full details of the structure will be published elsewhere.

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