

Figure 3. Generalized eight-coordination adapted from ref 5.

Our O...O separation (2.484 (4) Å) falls in the region where there is overlap between both curves. While our O-H distance of 1.17 (6) Å is within the limits of error of either the symmetric or the asymmetric curve, we consider the data from the tropolone rings indicate very strongly an asymmetric proton position.

Infrared stretching frequencies as a function of distances in hydrogen-bonded systems have been reviewed,¹⁴ and for an O...O distance of 2.48 Å, an O-H stretching frequency of 1975 cm⁻¹ is predicted on the basis of other results. The infrared spectrum of the acid dimer presented here shows a

(14) K. Nakamoto, M. Margoshes, and R. E. Rundle, *J. Amer. Chem. Soc.*, **77**, 6480 (1955).

small, broad band at 1976 cm⁻¹ in agreement with this prediction.

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Addendum

The structure of this compound has also been determined independently by Anderson, Neuman, and Melson (following paper).¹⁵ Our unit cell is related to theirs by the transformation

$$\begin{pmatrix} 0 & 0 & -1 \\ -1 & 0 & -1 \\ 0 & 1 & 0 \end{pmatrix}$$

The results of the two crystal structures are the same within the limits of the combined errors. The main differences are (1) the alternation of bonds in the C₇ ring of ligand 2 (their ligand 4) is not so pronounced and (2) the reduced thermal motion of the tropolonate ligands involved in hydrogen bonding is not so apparent from their rigid-body analysis.

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Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. (Unobserved reflections are indicated by asterisks.) Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for \$5.00 for photocopy or \$2.00 for microfiche, referring to code number INORG-74-1880.

(15) T. J. Anderson, M. A. Neuman, and G. A. Melson, *Inorg. Chem.*, **13**, 1884 (1974).

Contribution from the Department of Chemistry,
Michigan State University, East Lansing, Michigan 48824

Coordination Chemistry of Scandium. VII.¹ Crystal and Molecular Structure of Hydrogentetrakis(tropolonato)scandium(III), a Scandium(III) Complex with Approximate *D*_{2d} Dodecahedral Stereochemistry

THOMAS J. ANDERSON, MELVIN A. NEUMAN, and GORDON A. MELSON*

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The crystal and molecular structure of hydrogentetrakis(tropolonato)scandium(III), HSc(C₇H₅O₂)₄, has been determined by a single-crystal X-ray diffraction technique. The compound crystallizes in the space group *P*1, with cell constants *a* = 10.022 (4), *b* = 11.515 (3), and *c* = 12.004 (4) Å; α = 72.74°, β = 84.58°, and γ = 65.04°; *Z* = 2. Least-squares refinement of the counter data give a final *R* factor of 0.027 for 2539 significant reflections. The compound exists as a hydrogen-bonded dimer with each scandium atom coordinated by eight oxygen atoms. The coordination environment about the scandium ion is close to that of a *D*_{2d} dodecahedron. In the crystal, the two dodecahedra are held together about the center of symmetry by two almost linear hydrogen bonds, with an O-H distance of 1.00 Å, a hydrogen-bonded O...H distance of 1.49 Å, and an O-H...O angle of 175.9°.

Introduction

As part of our program concerning the structural characterization of scandium coordination compounds, we recently reported the crystal and molecular structures of two six-coordinated scandium(III) species, *viz.*, tris(acetylacetonato)scandium(III),² Sc(acac)₃ (acac = acetylacetonato anion,

C₅H₇O₂⁻), and tris(tropolonato)scandium(III),¹ Sc(trop)₃ (trop = tropolonato anion C₇H₅O₂⁻). In Sc(acac)₃, the coordination environment for the scandium(III) ion is close to trigonal antiprismatic, whereas in Sc(trop)₃, the stereochemistry is almost midway between trigonal antiprismatic and trigonal prismatic. From these structures, we concluded that in the solid state, the coordination environment of the scandium(III) ion (d⁰ configuration) is influenced by the nature of the ligands coordinated to it and by interactions between the ligands.

(1) Part VI: T. J. Anderson, M. A. Neuman, and G. A. Melson, *Inorg. Chem.*, **13**, 158 (1974).

(2) T. J. Anderson, M. A. Neuman, and G. A. Melson, *Inorg. Chem.*, **12**, 927 (1973).

Muettterties and Wright suggested³ that the tropolonato ion is ideally suited to the formation of complexes of high coordination number due to the rigidity and planarity of the ligand and in a later paper,⁴ these authors reported syntheses of the scandium complexes $MSc(trop)_4$, $M = H, Na$. Although few physical measurements were made on these compounds, it was proposed that they contain eight-coordinated scandium(III). The possibility that $HSc(trop)_4$ may exist as a 1:1 adduct between tris(tropolonato)scandium(III) and tropolone, *i.e.*, $Sc(trop)_3 \cdot Htrop$, similar to the species $ScQ_3 \cdot HQ^{5,6}$ ($HQ = 8$ -hydroxyquinoline), was discounted by infrared and X-ray photoelectron spectroscopy.⁷ It was also concluded from the ESCA data that the eight oxygen atoms surrounding the scandium(III) ion are equivalent within measurement limits. The scandium(III) ion is considerably smaller than any of the lanthanides which often exhibit high coordination numbers. Although no molecular species containing scandium(III) with coordination numbers greater than 6 have been confirmed, the structures of some compounds with eight-coordinated scandium(III) in polymeric networks have been described. These include $ScPO_4$,⁸ $ScVO_4$,^{9,10} $ScAsO_4$,¹⁰ $Na_5[Sc(CO_3)_4] \cdot 6H_2O$,¹¹ and $Sc_2(C_2O_4)_3 \cdot 6H_2O$.¹²

The determination of the crystal and molecular structure of $HSc(trop)_4$ reported here was initiated in order to confirm the conclusions reached on the basis of spectroscopic information and to determine the coordination environment of the scandium(III) ion. A preliminary account of this structure has been published.⁷

Experimental Section

Scandium(III) oxide, Sc_2O_3 (99.9%), was purchased from Research Organic/Inorganic Corp. Tropolone, $C_7H_6O_2$ (98%), was purchased from Aldrich Chemical Co., Inc., and was used without further purification.

Hydrogentetrakis(tropolonato)scandium(III), $HSc(trop)_4$. Hydrated scandium nitrate (0.34 g), prepared by evaporation of a solution of Sc_2O_3 and dilute nitric acid, was dissolved in water (30 ml) and added to a solution of tropolone (0.46 g) in methanol (5 ml). The solution was stirred at room temperature for 2–3 hr and then filtered. The residue was dried over P_2O_5 *in vacuo* at room temperature and consisted of reddish brown crystals of $Sc(trop)_3$ and pale yellow crystals of $HSc(trop)_4$. Crystals of $HSc(trop)_4$ suitable for single-crystal X-ray analysis were readily separated from this mixture. The procedure reported by Muettterties and Wright⁴ for the preparation of $HSc(trop)_4$ also produced suitable crystals.

Measurement of Cell Dimensions and Intensity Data. Preliminary investigations were conducted using the precession technique with $Mo K\alpha$ radiation. The highest Laue symmetry we could determine was $P\bar{1}$; thus the space group is either $P1$ or $P\bar{1}$. Complete three-dimensional single-crystal X-ray diffraction data were obtained on a Picker four-circle automatic diffractometer controlled by a PDP-8/1 computer. A graphite monochromator in perpendicular geometry with the (002) plane in diffracting orientation was used to obtain monochromatic $Mo K\alpha$ radiation ($\lambda(Mo K\alpha_1) 0.70926 \text{ \AA}$). A takeoff angle of 3° was used. The radiation was detected using a scintillation

Table I. Crystal Data^a

Molecular formula	$HSc(C_7H_5O_2)_4$	α , deg	72.74 (1)
Mol wt	530.43	β , deg	84.58 (1)
Crystal habit	Triclinic	γ , deg	65.04 (1)
Crystal color	Pale yellow	V , \AA^3	1198.2 (1)
Space group	$P\bar{1}$	d_{exptl}^b , g/ml	1.47
a , \AA	10.022 (4)	d_{calcd} , g/ml	1.47
b , \AA	11.515 (3)	Z	2
c , \AA	12.004 (4)	μ , cm^{-1}	3.67

^a Matrix of transformation from our cell parameters to those of Davis and Einstein is

$$\begin{pmatrix} 1 & -1 & 0 \\ 0 & 0 & 1 \\ -1 & 0 & 0 \end{pmatrix}$$

See A. R. Davis and F. W. B. Einstein, *Inorg. Chem.*, 13, 1880 (1974).

^b Measured by flotation in chloroform.

counter with pulse height discrimination. To avoid counter overload, attenuators were used when the count rate exceeded 10,000 counts/sec. The crystal ($0.4 \times 0.25 \times 0.18 \text{ mm}$) was mounted with the a^* axis approximately coincident with the ϕ axis of the diffractometer. Lattice parameters (Table I) were determined from 12 hand-centered reflections at ambient temperature ($22 \pm 1^\circ$). Intensities were measured by an ω -scan technique with ω scanned over a width of 0.8° at a rate of $0.5^\circ/\text{min}$. Data for the hemisphere ($h, \pm k, \pm l$) were measured between 2.8 and 45° for 2θ . Backgrounds were estimated by two 10-sec counts made one at each end of the scan using stationary-crystal, stationary-counter measurements. Three monitor reflections [(111), (014), (2 $\bar{1}$ 3)] were measured every 100 reflections. No decomposition nor motion of the crystal was noticed, the average deviation of the intensities being $\pm 1\%$. A total of 3144 independent reflections were measured of which 2539 were found to be above background using the criterion $I/\sigma(I) > 3.0$.

Solution and Refinement of the Structure. All calculations were performed at the Michigan State University Computer Center.¹³ Scattering factors for the neutral Sc, O, and C atoms were obtained from ref 14 and those for H from ref 15. The normal Lorentz and polarization corrections to the raw data were made, but no correction for anomalous dispersion nor absorption was employed.

After data correction,^{13a} the unscaled $|F|$ values were used to generate a three-dimensional point-sharpened Patterson map.^{13d} However it was not obvious from the map where the scandium was located, possibly due to coincidental vector overlap. Because of this problem, direct methods were used for at least a partial solution of the structure. A set of 275 $|E|$ values was calculated using the program FAME.^{13b} However, after assignment of seven symbols, the programs MAGIC, LINK, and SYMPL^{13b} failed to yield a nontrivial solution. At this time, MULTAN^{13c} became available to us. Using the $|E|$'s calculated from FAME, a Σ_2 listing was determined from which an initial set of three origin reflections and three symbols were determined by hand for a total of 29 reflections. These reflections were then used with the tangent formula to calculate phases for the complete molecule. The third combination of signs (Table II) yielded an E map which gave atomic positions for the scandium and for six oxygen atoms. Upon returning to the Patterson map, peaks corresponding to these positions were found. The coordinates of the other two oxygens were then determined from the map.

Calculations of structure factors and subsequent least-squares refinement were based upon the cell being centrosymmetric; *i.e.*, the space group is $P\bar{1}$. This conclusion arose from the statistics obtained from FAME (Table III). A structure factor calculation based on the determined atomic coordinates and isotropic temperature factors was used to obtain a Fourier map from which some of the

(3) E. L. Muettterties and C. M. Wright, *J. Amer. Chem. Soc.*, 86, 5132 (1964).

(4) E. L. Muettterties and C. M. Wright, *J. Amer. Chem. Soc.*, 87, 4706 (1965).

(5) T. J. Cardwell and R. J. Magee, *Anal. Chim. Acta*, 36, 180 (1966).

(6) A. Corsini, F. Toneguzzo, and M. Thompson, *Can. J. Chem.*, 51, 1248 (1973).

(7) D. J. Olszanski, T. J. Anderson, M. A. Neuman, and G. A. Melson, *Inorg. Nucl. Chem. Lett.*, 10, 137 (1974).

(8) R. C. L. Mooney, *Acta Crystallogr.*, 9, 677 (1956).

(9) W. O. Milligan and L. W. Vernon, *J. Phys. Chem.*, 56, 145 (1952).

(10) H. Schwarz, *Z. Anorg. Allg. Chem.*, 323, 44 (1963).

(11) T. A. Zhdanov, A. A. Voronkov, L. N. Komissarova, and Yu. A. Pyatenko, *Dokl. Akad. Nauk SSSR*, 196, 1076 (1971); *Sov. Phys.-Dokl.*, 16, 70 (1971).

(12) E. Hansson, *Acta Chem. Scand.*, 26, 1337 (1972).

(13) Computer programs used in the analysis: (a) DATCOR, a data correction routine, M. A. Neuman; (b) FAME, MAGIC, LINK, and SYMPL, R. B. K. Dewar and A. L. Stone; (c) MULTAN; (d) CONNIE, a Fourier summation program written by M. A. Neuman; (e) WRFLS, a full-matrix least-squares refinement program based on ORFLS of W. Busing, K. Martin, and H. Levy; (f) ORFFE, a Fortran function and error analysis program by Busing, Martin, and Levy; (g) BLANDA, a Fortran bond distance and angle program by R. Shiono which includes standard deviations based only on errors in coordinates and not correlations between parameters; (h) ORTEP, a Fortran thermal-ellipsoid plot program for crystal structure illustrations by C. K. Johnson.

(14) "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, England, 1962.

(15) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 42, 3172 (1965).

Table II. Starting Phases from MULTAN

Reflection			E	Symbol ^a	Phase, deg
h	k	l			
1	-1	2	3.36	+	0
2	-1	3	2.68	+	0
6	-2	-1	3.37	+	0
1	3	12	2.48	A	0
2	-3	-4	2.87	B	0
4	3	-1	2.56	C	180

^a + is an origin reflection

Table III. Statistical Analysis of |E| Values from FAME ($\langle |E|^2 \rangle$ Rescaled to 1.00)

	Theoret values		Calcd values
	Centric	Acentric	
Av magnitude of E's	0.798	0.886	0.806
$\langle E ^2 - 1 \rangle$	0.968	0.736	0.950
Percentage of E 's > 1	32.00	37.00	32.49
Percentage of E 's > 2	5.00	1.80	3.99
Percentage of E 's > 3	0.33	0.01	0.26

carbon positions were located. Repeating this procedure twice more using the coordinates of all atoms determined enabled the positions of all nonhydrogen atoms to be obtained. Using these coordinates as initial values and isotropic temperature factors of 2.5 \AA^2 for all atoms, four cycles of full-matrix least-squares refinement of coordinates and temperature factors lowered the unweighted *R* factor from 0.467 to 0.096. At this time, a difference Fourier was calculated and the positions of all the ring hydrogen atoms were found. To avoid errors in coordinates, they were placed 0.97 \AA from their respective carbon atoms along the line of the appropriate angle bisectors. The "acid" hydrogen was placed between O1(1) and O1(3). It was thought at this time that the hydrogen may be bridging between these oxygens because of the short contacts between them (2.5 Å) and the slightly longer scandium-oxygen bond distances compared with those for other oxygen atoms. Further refinement of coordinates and isotropic temperature factors continued, to a converged *R* = 0.064. At this time the acid hydrogen had moved $\sim 1 \text{ \AA}$. A difference Fourier at final convergence (Figure 1) further suggested the new hydrogen position between O1(1) and O1(4) across the center of inversion (see description of hydrogen bonding). Further refinement of coordinates and anisotropic temperature factors for all nonhydrogen atoms and isotropic temperature factors for the hydrogens was performed in two parts. At this time, there appeared to be mutually perpendicular quasi-mirror planes in the structure, and atoms of each quasi-mirror plane were refined separately. To check on correlations, the rings were refined in pairs not contained by this quasi-mirror plane for one cycle and were not found to correlate significantly; thus the final cycles were not overlapped and the final *R* factor was 0.027 while R_w was 0.029.¹⁶ The final refinement obviously justifies the choice of $P\bar{1}$ as the space group. A listing of final observed and calculated structure factor amplitudes ($\times 10$) is available as supplementary material.

No parameter shift was found to be greater than 0.08 of its estimated standard deviation during the last cycle. A final difference Fourier showed no peaks larger than 0.2 e/\AA^3 .

Description of Structure and Discussion

Hydrogentetrakis(tropolonato)scandium(III) crystallizes from aqueous methanol solution in the space group $P\bar{1}$. The crystal data are recorded in Table I. Final atomic coordinates and thermal parameters for scandium, oxygen, and carbon are listed in Tables IV and V and for hydrogen in Table VI. The compound exists as a hydrogen-bonded dimer located about the center of inversion of the cell. A view of half of the dimer, excluding ring hydrogens, is shown in Figure 2. The shortest nonbonding separations are listed in Table VII. These are classified as intramolecular (within the dimer) or intermolecular (between dimers). For the intramolecular separations, the shortest O...O distance (2.49 Å) involves the hydrogen (H) of the hydrogen bond,

(16) $R = [\sum ||F_o| - |F_c||] / \sum |F_o|$; $R_w = [(\sum w|F_o| - |F_c|)^2 / \sum w \cdot (|F_o|)^2]^{1/2}$, where $w = 1$.

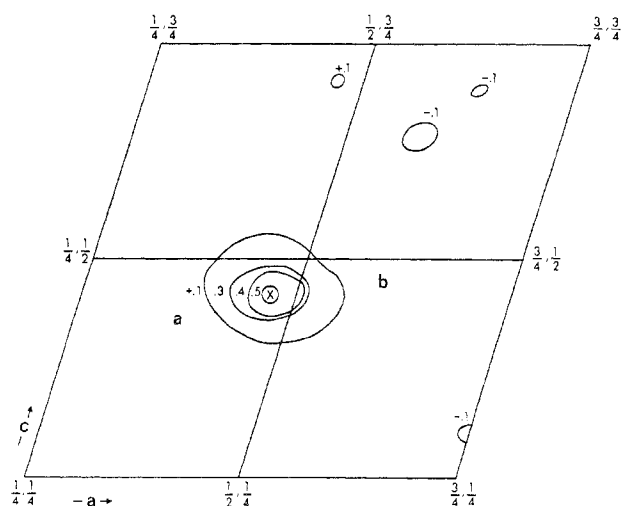


Figure 1. Difference Fourier showing hydrogen (H) position, x, located between oxygen atoms O1(4) and O1(1) (represented by a and b). Section of map at $b = 0.45$. Atomic coordinates are related to those in Tables IV and VI by $1-x, 1-y, 1-z$.

Table IV. Final Atomic Coordinates of Sc, O, and C

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>
Sc	0.32858 (5) ^a	0.62313 (4)	0.66803 (4)
O1(1) ^b	0.3071 (2)	0.5869 (1)	0.4923 (1)
O2(1)	0.1914 (2)	0.8086 (2)	0.5363 (1)
C1(1)	0.2161 (2)	0.6843 (2)	0.4092 (2)
C2(1)	0.1906 (3)	0.6597 (3)	0.3092 (2)
C3(1)	0.1016 (3)	0.7458 (3)	0.2102 (2)
C4(1)	0.0139 (3)	0.8797 (3)	0.1844 (2)
C5(1)	-0.0069 (3)	0.9623 (3)	0.2544 (2)
C6(1)	0.0538 (3)	0.9336 (3)	0.3621 (2)
C7(1)	0.1531 (3)	0.8112 (2)	0.4377 (2)
O1(2)	0.1389 (2)	0.5736 (2)	0.6861 (2)
O2(2)	0.1841 (2)	0.7091 (2)	0.7953 (1)
C1(2)	0.0427 (3)	0.6087 (2)	0.7610 (2)
C2(2)	-0.0784 (3)	0.5739 (3)	0.7743 (3)
C3(2)	-0.1904 (4)	0.5942 (3)	0.8507 (3)
C4(2)	-0.2163 (4)	0.6548 (3)	0.9391 (3)
C5(2)	-0.1371 (4)	0.7146 (3)	0.9675 (3)
C6(2)	-0.0131 (3)	0.7301 (3)	0.9180 (2)
C7(2)	0.0710 (3)	0.6852 (2)	0.8271 (2)
O1(3)	0.4299 (2)	0.4098 (2)	0.6853 (1)
O2(3)	0.4703 (2)	0.5152 (2)	0.8256 (1)
C1(3)	0.4947 (3)	0.3265 (2)	0.7810 (2)
C2(3)	0.5352 (3)	0.1895 (3)	0.7997 (3)
C3(3)	0.6035 (4)	0.0821 (3)	0.8953 (3)
C4(3)	0.6520 (4)	0.0792 (3)	1.0004 (3)
C5(3)	0.6461 (3)	0.1860 (3)	1.0337 (3)
C6(3)	0.5909 (3)	0.3195 (3)	0.9744 (2)
C7(3)	0.5200 (3)	0.3876 (2)	0.8636 (2)
O1(4)	0.5342 (2)	0.6160 (2)	0.5693 (1)
O2(4)	0.4013 (2)	0.7742 (2)	0.6878 (1)
C1(4)	0.5890 (3)	0.7043 (2)	0.5625 (2)
C2(4)	0.7036 (3)	0.7073 (3)	0.4912 (2)
C3(4)	0.7779 (3)	0.7900 (3)	0.4729 (3)
C4(4)	0.7586 (4)	0.8874 (3)	0.5230 (3)
C5(4)	0.6591 (3)	0.9276 (3)	0.6078 (3)
C6(4)	0.5521 (3)	0.8875 (3)	0.6556 (3)
C7(4)	0.5103 (3)	0.7906 (2)	0.6371 (2)

^a Numbers in parentheses refer to the estimated standard deviations (esd) of the last decimal place. ^b The numbering system given refers first to the atom of a given ring and then to the ring; e.g., O1(1) is the first oxygen on ring 1. O1 is bonded to C1 and O2 is bonded to C7.

with the shortest separation not involving H being 3.13 \AA (O1(3)-O1(4)). (The shortest O...O distance within one $\text{HSc}(\text{trop})_4$ unit is the ligand "bite" distance which averages 2.505 \AA .) For separations between other atoms, no unusually short distances are found. The closest intermolecu-

Table V. Final Anisotropic Temperature Factors^a

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sc	0.00704 (6)	0.00456 (5)	0.00386 (4)	-0.00215 (5)	0.00061 (4)	-0.00196 (3)
O1(1)	0.0081 (2)	0.0050 (2)	0.0040 (1)	-0.0015 (2)	-0.0005 (1)	-0.0018 (1)
O2(1)	0.0110 (3)	0.0059 (2)	0.0052 (2)	-0.0023 (2)	-0.0066 (2)	-0.0026 (1)
C1(1)	0.0059 (3)	0.0058 (3)	0.0043 (2)	-0.0024 (2)	0.0009 (2)	-0.0017 (2)
C2(1)	0.0085 (4)	0.0065 (3)	0.0055 (2)	-0.0023 (3)	0.0000 (2)	-0.0028 (2)
C3(1)	0.0111 (4)	0.0097 (3)	0.0053 (2)	-0.0029 (3)	-0.0011 (2)	-0.0034 (2)
C4(1)	0.0118 (4)	0.0092 (4)	0.0052 (2)	-0.0021 (3)	-0.0024 (3)	-0.0013 (2)
C5(1)	0.0108 (4)	0.0060 (3)	0.0060 (3)	-0.0015 (3)	-0.0011 (3)	-0.0005 (2)
C6(1)	0.0102 (4)	0.0056 (3)	0.0059 (2)	-0.0027 (3)	0.0007 (2)	-0.0021 (2)
C7(1)	0.0073 (3)	0.0057 (3)	0.0047 (2)	-0.0031 (2)	0.0008 (2)	-0.0017 (2)
O1(2)	0.0114 (3)	0.0114 (2)	0.0082 (2)	-0.0069 (2)	0.0033 (2)	-0.0058 (2)
O2(2)	0.0091 (3)	0.0090 (2)	0.0060 (2)	-0.0048 (2)	0.0020 (2)	-0.0039 (1)
C1(2)	0.0084 (4)	0.0066 (3)	0.0055 (2)	-0.0028 (3)	0.0004 (2)	-0.0012 (2)
C2(2)	0.0115 (5)	0.0116 (4)	0.0089 (3)	-0.0068 (3)	0.0012 (3)	-0.0034 (3)
C3(2)	0.0106 (5)	0.0131 (4)	0.0109 (4)	-0.0071 (4)	0.0019 (3)	-0.0026 (3)
C4(2)	0.0098 (5)	0.0137 (4)	0.0096 (3)	-0.0049 (4)	0.0039 (3)	-0.0017 (3)
C5(2)	0.0130 (5)	0.0124 (4)	0.0069 (3)	-0.0045 (4)	0.0034 (3)	-0.0031 (3)
C6(2)	0.0159 (4)	0.0101 (3)	0.0057 (2)	-0.0043 (3)	0.0016 (3)	-0.0029 (2)
C7(2)	0.0077 (4)	0.0057 (3)	0.0041 (2)	-0.0019 (3)	0.0001 (2)	-0.0010 (2)
O1(3)	0.0128 (3)	0.0055 (2)	0.0046 (1)	-0.0027 (2)	-0.0018 (2)	-0.0014 (1)
O2(3)	0.0128 (3)	0.0060 (2)	0.0061 (2)	-0.0021 (2)	-0.0019 (2)	-0.0026 (1)
C1(3)	0.0077 (3)	0.0062 (3)	0.0053 (2)	-0.0030 (3)	0.0007 (2)	-0.0021 (2)
C2(3)	0.0142 (5)	0.0067 (3)	0.0069 (3)	-0.0040 (3)	-0.0023 (3)	-0.0018 (2)
C3(3)	0.0168 (5)	0.0063 (3)	0.0100 (3)	-0.0042 (3)	-0.0038 (3)	-0.0007 (3)
C4(3)	0.0150 (5)	0.0082 (4)	0.0075 (3)	-0.0040 (3)	-0.0032 (3)	0.0010 (3)
C5(3)	0.0099 (4)	0.0109 (4)	0.0050 (3)	-0.0027 (3)	-0.0013 (3)	-0.0013 (3)
C6(3)	0.0090 (4)	0.0086 (3)	0.0054 (2)	-0.0019 (3)	-0.0004 (2)	-0.0029 (2)
C7(3)	0.0067 (3)	0.0072 (3)	0.0046 (2)	-0.0022 (3)	0.0009 (2)	-0.0026 (2)
O1(4)	0.0078 (2)	0.0057 (2)	0.0060 (1)	-0.0030 (2)	0.0013 (1)	-0.0033 (1)
O2(4)	0.0094 (2)	0.0072 (2)	0.0063 (2)	-0.0044 (2)	0.0022 (2)	-0.0037 (1)
C1(4)	0.0073 (3)	0.0056 (3)	0.0049 (2)	-0.0027 (2)	-0.0003 (2)	-0.0018 (2)
C2(4)	0.0097 (4)	0.0087 (3)	0.0073 (3)	-0.0047 (3)	0.0027 (3)	-0.0045 (2)
C3(4)	0.0114 (5)	0.0128 (4)	0.0105 (3)	-0.0078 (4)	0.0053 (3)	-0.0064 (3)
C4(4)	0.0145 (5)	0.0135 (4)	0.0138 (4)	-0.0109 (4)	0.0059 (4)	-0.0074 (4)
C5(4)	0.0145 (5)	0.0110 (4)	0.0119 (4)	-0.0086 (4)	0.0042 (3)	-0.0071 (3)
C6(4)	0.0117 (4)	0.0089 (3)	0.0084 (3)	-0.0055 (3)	0.0026 (3)	-0.0053 (3)
C7(4)	0.0076 (3)	0.0057 (3)	0.0049 (3)	-0.0026 (2)	0.0002 (2)	-0.0020 (2)

^a Anisotropic temperature factors are of the form $\exp[-\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl]$.

Table VI. Final Hydrogen Coordinates and Isotropic Temperature Factors

	x/a	y/b	z/c	$B, \text{Å}^2$
H ^a	0.595 (4)	0.536 (4)	0.542 (3)	7.30 (93)
H2(1) ^b	0.240 (3)	0.570 (2)	0.309 (2)	2.41 (52)
H3(1)	0.101 (3)	0.704 (3)	0.149 (2)	3.63 (60)
H4(1)	-0.040 (3)	0.922 (3)	0.110 (2)	3.78 (62)
H5(1)	-0.068 (3)	1.051 (3)	0.225 (2)	3.22 (59)
H6(1)	0.027 (3)	1.009 (3)	0.391 (2)	2.82 (55)
H2(2)	-0.081 (3)	0.533 (3)	0.715 (3)	4.87 (74)
H3(2)	-0.264 (4)	0.564 (3)	0.838 (3)	5.38 (78)
H4(2)	-0.303 (4)	0.661 (3)	0.979 (3)	5.49 (79)
H5(2)	-0.162 (4)	0.748 (3)	1.034 (3)	6.19 (88)
H6(2)	0.030 (3)	0.774 (3)	0.954 (2)	3.83 (64)
H2(3)	0.507 (3)	0.170 (3)	0.732 (2)	3.85 (62)
H3(3)	0.613 (3)	-0.004 (3)	0.891 (3)	5.99 (82)
H4(3)	0.687 (3)	-0.006 (3)	1.061 (3)	5.46 (77)
H5(3)	0.677 (3)	0.169 (3)	1.108 (2)	3.39 (61)
H6(3)	0.601 (3)	0.374 (3)	1.018 (2)	3.96 (65)
H2(4)	0.737 (3)	0.645 (2)	0.449 (2)	2.43 (52)
H3(4)	0.852 (3)	0.771 (3)	0.419 (2)	3.88 (65)
H4(4)	0.817 (3)	0.934 (3)	0.499 (3)	5.07 (75)
H5(4)	0.665 (3)	1.000 (3)	0.634 (2)	4.49 (67)
H6(4)	0.494 (3)	0.927 (3)	0.713 (3)	4.42 (69)

^a Hydrogen attached to O1(4). ^b First number refers to the carbon atom to which the hydrogen is attached; the second, to the ring.

lar contacts involve the tropolonate ring hydrogens, the shortest distance being 2.31 Å (H2(1)-H5(2)).

Coordination Polyhedron. The scandium(III) ions are each coordinated by eight oxygen atoms from four bidentate ligands. Interatomic distances and angles within the coordination polyhedron are given in Table VIII. The average Sc-O distance calculated from bond lengths involving oxygen

Table VII. Shortest Nonbonding Separations

Intramolecular ^a			
Type	Distance, Å	Type	Distance, Å
H-H	2.73	C2(1)-O1(4)	3.23
H1(1)-H	2.24	C1(1)-C1(3)	3.29
O1(3)-H	2.62	O1(1)-O1(4) ^b	2.49
O1(3)-O1(4)	3.13		
Intermolecular			
Type	Distance, Å	Atoms related by	
H2(1)-H5(2)	2.31	$x, y, z - 1$	
H1(1)-H1(2)	2.43	$-x, 1 - y, 1 - z$	
H1(3)-H4(4)	2.48	$x, y - 1, z$	

^a Atoms are from opposite halves of the dimer. ^b Hydrogen H is located between these atoms.

atoms of ligands 2 and 3, which are not part of the hydrogen-bonding system, is 2.18 Å from which an ionic radius for eight-coordinated scandium(III) of approximately 0.78 Å is estimated. This value is similar to that obtained from the Sc-O distance in ScPO₄,⁸ Na₅[Sc(CO₃)₄]·2H₂O,¹¹ and Sc₂(C₂O₄)₃·6H₂O.¹² For a coordination number of 8, there are two important idealized coordination polyhedra, the dodecahedron and the square antiprism, although the polyhedron found in a structure is often distorted from one of these types.^{17,18} We employed the method of Lippard and Russ¹⁸ in order to distinguish between these two possibilities. Of particular interest concerning the choice of the polyhedron is the identification of the trapezoidal best

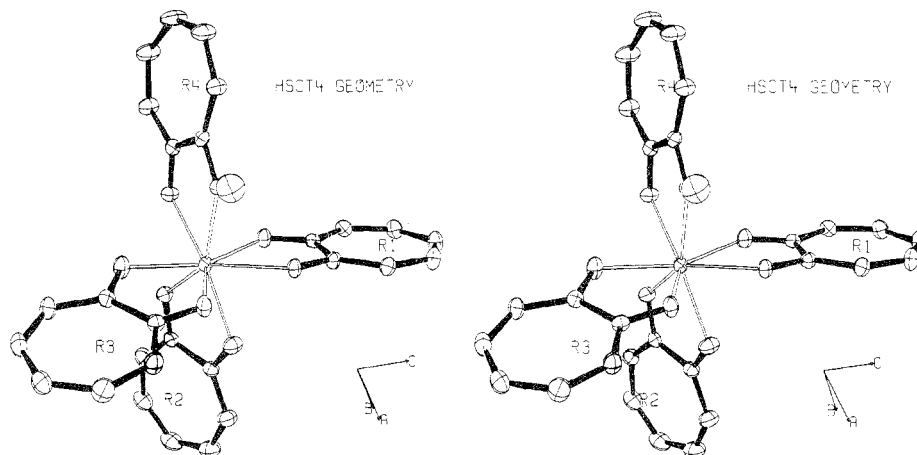


Figure 2. Stereoscopic view of half of the $\text{HSc}(\text{trop})_4$ dimer showing 25% probability of the thermal ellipsoids. Ring hydrogens omitted for clarity.

Table VIII. Interatomic Distances (Å) and Angles (deg) within the Coordination Polyhedron^a

Distances ^b			
Sc-O1(1)	2.314 (2)	O1(1)-O1(2)	2.747 (3)
Sc-O2(1)	2.209 (2)	O1(1)-O1(3)	2.518 (3)
Sc-O1(2)	2.180 (2)	O1(1)-O1(4)	2.715 (3)
Sc-O2(2)	2.164 (2)	O2(1)-O1(2)	2.995 (3)
Sc-O1(3)	2.178 (2)	O2(1)-O1(4)	3.192 (3)
Sc-O2(3)	2.183 (2)	O2(1)-O2(2)	2.983 (3)
Sc-O2(4)	2.260 (2)	O2(1)-O2(4)	2.736 (3)
Sc-O2(4)	2.228 (2)	O1(2)-O1(3)	2.726 (4)
H-O1(4)	1.00 (4)	O1(2)-O2(3)	3.577 (4)
H...O1(1)	1.49 (4)	O2(2)-O2(3)	2.767 (4)
O1(1)-O2(1)	2.516 (2) ^c	O2(2)-O2(4)	2.768 (4)
O1(2)-O2(2)	2.496 (2) ^c	O1(3)-O1(4)	2.920 (4)
O1(3)-O2(3)	2.507 (2) ^c	O2(3)-O1(4)	3.059 (4)
O1(4)-O2(4)	2.500 (2) ^c	O2(3)-O2(4)	2.691 (4)
Av	2.505 (5) ^d		
Angles			
O1(1)-Sc-O2(1)	67.53 (6)	O1(3)-Sc-O2(3)	69.84 (7)
Sc-O1(1)-C1(1)	119.9 (1)	Sc-O1(3)-C1(3)	120.2 (1)
Sc-O2(1)-C7(1)	123.6 (1)	Sc-O2(3)-C7(3)	120.3 (1)
O1(2)-Sc-O2(2)	70.50 (7)	O1(4)-Sc-O2(4)	67.69 (6)
Sc-O1(2)-C1(2)	120.2 (2)	Sc-O1(4)-C1(4)	121.8 (1)
Sc-O2(2)-C7(2)	120.9 (1)	Sc-O2(4)-C7(4)	122.9 (1)
		O1(1)-H-O1(4)	175.9 (33)
		H-O1(1)-Sc	137.5 (21)
		H-O1(4)-Sc	119.0 (20)
		H-O1(4)-C1(4)	118.3 (20)

^a Errors referred to the last significant digit are in parentheses.

^b Corrections for librational motion of Sc-O bonds are less than one standard deviation. ^c Ligand bite. ^d Errors for average are computed using the method of small sample statistics: W. Blaedel and V. Meloche, "Elementary Quantitative Analysis," Row, Peterson and Co., Evanston, Ill., 1957, p 557.

planes for a dodecahedron and the calculation of the angle of intersection between these planes. Table IX and Figure 3 identify the planes T_1 and T_2 . The angle between them, $\alpha_{T_1 T_2}$, is calculated to be 89.5° (Table X). For an idealized dodecahedron, this value should be 90° whereas for a square antiprism it should be 77.4° . The values of Θ_A and Θ_B are also close to those expected for dodecahedral stereochemistry. We therefore conclude that the coordination polyhedron in $\text{HSc}(\text{trop})_4$ is close to that of a dodecahedron. Table VIII lists the individual O...O nonbonding distances which comprise the edges of the coordination polyhedron, and Figure 4 shows the polyhedron with the edges a , b , m , and g identified. Table X records the average normalized values of these edges. The bidentate ligands span equivalent m edges of the polyhedron and thus the dodecahedron has

Table IX. Least-Squares Planes^a

Trapezoids						
Plane	a	b	c	d	Atom	Dev from plane, Å
T_1	0.8337	0.3754	-0.4050	-3.4044	O1(1)*	0.154 (2)
					O2(1)*	-0.109 (2)
					O1(3)*	-0.190 (2)
					O2(3)*	0.125 (2)
					Sc	-0.133 (1)
T_2	-0.5291	0.3826	-0.7574	2.9261	O1(2)*	0.141 (2)
					O2(2)*	-0.182 (2)
					O1(4)*	-0.105 (2)
					O2(4)*	0.175 (2)
					Sc	-0.118 (1)
Tropolonato Rings ^a						
Ring	a	b	c	d		
1	0.7828	0.4542	-0.4253	-4.0678		
2	-0.5389	0.5289	-0.6557	0.4985		
3	0.9040	0.1692	-0.3926	-1.7281		
4	-0.6187	0.2501	-0.7448	4.7944		
Deviations from Planes, Å						
Atom	Ring 1	Ring 2	Ring 3	Ring 4		
O1	0.174 (2)	-0.089 (2)	-0.012 (2)	-0.077 (2)		
O2	-0.026 (2)	0.026 (2)	-0.044 (2)	0.110 (2)		
C1*	0.002 (3)	-0.024 (3)	-0.004 (2)	-0.022 (3)		
C2*	0.000 (3)	0.015 (4)	0.011 (3)	-0.010 (3)		
C3*	0.000 (3)	0.023 (5)	0.005 (3)	0.024 (4)		
C4*	0.001 (3)	-0.018 (5)	-0.016 (3)	0.023 (4)		
C5*	-0.006 (3)	-0.018 (4)	-0.002 (3)	-0.034 (4)		
C6*	0.008 (3)	-0.015 (4)	0.018 (3)	-0.014 (3)		
C7*	-0.005 (3)	0.009 (3)	-0.010 (3)	0.035 (3)		
Sc	-0.227 (1)	-0.194 (1)	-0.428 (1)	0.153 (1)		

^a General equation for planes is $ax + by + cz + d = 0$. x , y , and z are coordinates (Å) in an orthogonal system defined by $b \times c$, b , and c^* , respectively. Atoms defining planes are signified by asterisks.

approximate D_{2d} symmetry. This mode of chelation is quite common for bidentate ligands,^{19,20} although in $\text{Sc}_2(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$ the three oxalato ligands span two m edges and one a edge of the dodecahedron¹² and in $\text{Cs}[\text{Y}(\text{Hf}(\text{ac})_4)]$, $\text{hf}(\text{ac}) = \text{hexafluoroacetylacetonate}$ ion, the diketone ligands span g edges resulting in D_2 symmetry.²¹ In an idealized D_{2d} dodecahedron, there may be a difference in the bond lengths between the A and B type Sc-O bonds as defined by Hoard and Silverton.¹⁷ However, for $\text{HSc}(\text{trop})_4$, no signifi-

(19) E. L. Muetterties and C. M. Wright, *Quart. Rev., Chem. Soc.*, **21**, 109 (1967).

(20) S. J. Lippard, *Progr. Inorg. Chem.*, **8**, 109 (1967).

(21) M. J. Bennett, F. A. Cotton, P. Legzdins, and S. J. Lippard, *Inorg. Chem.*, **7**, 1770 (1968).

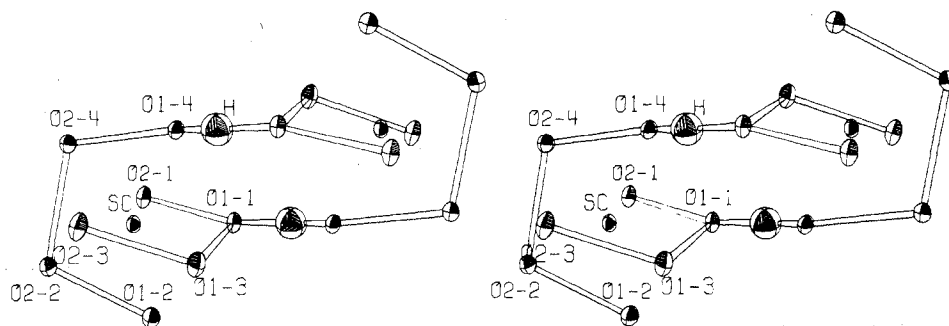


Figure 3. Interpenetrating trapezoids and hydrogen bonding in $\text{HSc}(\text{trop})_4$.

Table X. Shape Parameters^a for $\text{HSc}(\text{trop})_4$

Parameter ^b	$\text{HSc}(\text{trop})_4$	Dodecahedron
a	1.19	1.17
m^c	1.13	1.17
b	1.45	1.49
g	1.26	1.24
θ_A , deg	36.4	35.2
θ_B , deg	74.7	73.5
$\alpha_{T_1 T_2}$, ^d deg	89.5	90.0
d_{T_1} , ^e Å	0.005	0.0
d_{T_2} , ^e Å	0.007	0.0

^a See ref 17 and 18 for definition of shape parameters. ^b Polyhedron edges normalized by dividing by the average Sc-O bond distance (2.215 Å). ^c m is the normalized ligand "bite" distance. ^d $\alpha_{T_1 T_2}$ is the angle between the best trapezoidal planes T_1 and T_2 . ^e d_{T_1} and d_{T_2} are mean displacements of ligand atoms from best trapezoidal planes T_1 and T_2 .

cant difference is observed for the averages of these bond lengths.

Hydrogen Bonding and the Ligands. In the crystal, the two dodecahedra are held together about the center of inversion by two almost linear hydrogen bonds (see Figures 2 and 3). The hydrogens attached to the O1(4) oxygens (O-H bond length 1.00 Å) are hydrogen bonded to the O1(1) oxygens across the center. The hydrogen-bonded O...H distance is 1.49 Å and the O-H...O angle is 175.9°. The atoms Sc, O1(4), O1(1), and H and the related atoms across the center form a chair-shaped arrangement with the O1(4), H, and O1(1) atoms almost planar. The scandium atoms are located such that they make an angle of 132° with this plane.

During the refinement of the structure, the hydrogen was placed initially between O1(1) and O1(3) of one dodecahedron, because of the short O...O separation between these atoms (2.518 Å). However, after refinement, the hydrogen was located in the position described above, with H...O1(3) across the center equal to 2.62 Å. The position of O1(3) is probably influenced by the hydrogen, but the hydrogen bond is not bifurcated as in tropolone itself.²² Any effect that the hydrogen produces on ring 3 is of much smaller magnitude than that produced on rings 1 and 4.

Further information concerning the hydrogen bonding is obtained from consideration of the nature of the ligands coordinated to the scandium(III) ion. Table XI records the interatomic distances and angles for these ligands. The C_7H_5 rings are almost planar, there being only slight deviations of the carbons from the planes defined by C1-C7. Table IX lists these deviations and the equations of the planes. However, O1 and O2 for the four rings show a complex pattern of deviations from the best carbon planes. Rings 2 and 3 show much smaller deviations than do rings

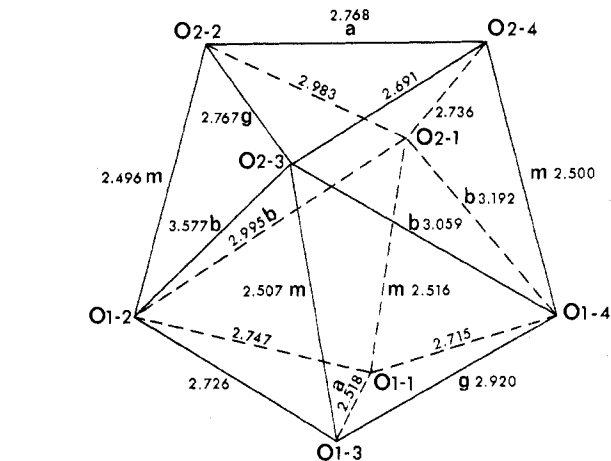


Figure 4. Coordination polyhedron for the scandium(III) ion in $\text{HSc}(\text{trop})_4$.

1 and 4, with total out-of-plane distances being 0.20 Å for ring 1, 0.11 Å for ring 2, 0.03 Å for ring 3, and 0.19 Å for ring 4. The considerable twisting of rings 1 and 4 may be related to the nature of these ligands (see later).

The bond lengths and angles within the four rings all show systematic variations (see Table XI). Rings 2 and 3 conform to the patterns observed in $\text{Sc}(\text{trop})_3$ ¹ and $\text{Al}(\text{trop})_3$.²³ These complexes each contain three coordinated tropolonato ligands with the C-C bond lengths decreasing as one proceeds from the carbon atoms attached to the oxygens to the C4 ring atom. Rings 1 and 4 do not exhibit this pattern however. The bond lengths in ring 4 are very similar to those found in tropolone²² where a more localized positioning of double bonds than in tropolonato ions is to be expected. The placement of the hydrogen atom on O1(4) is consistent with the increased C1-O1 bond length and the corresponding decrease in the C2-O2 bond length (1.324 vs. 1.257 Å, respectively). Ring 4 is thus more of a "tropolone" ligand than a "tropolonato" ligand. Ring 1 is affected by the hydrogen atom placement since the C-C bond length pattern is similar to that observed in ring 4, and C-O bond lengths for ring 1 are unequal at 1.315 and 1.267 Å. Thus, the hydrogen bond influences the electronic distribution within the ligands 1 and 4, such that they appear similar to tropolone molecules. However, the difference Fourier (Figure 1) does not indicate an "averaging" of the hydrogen position.

The hydrogen bonding in $\text{HSc}(\text{trop})_4$ also affects the Sc-O bond lengths involving ligands 1 and 4. These bond lengths (2.314, 2.209, 2.260, and 2.228 Å) are significantly longer than those involving ligands 2 and 3 (average value 2.18 Å).

(22) H. Shimanouchi and Y. Sasada, *Acta Crystallogr., Sect. B*, 29, 81 (1973).

(23) E. L. Muetterties and L. J. Guggenberger, *J. Amer. Chem. Soc.*, 94, 8046 (1972).

Table XI. Interatomic Distances (Å) and Angles (deg) for the Tropolonato Ligands^a

	Ring 1	Ring 2	Ring 3	Ring 4
	Distances ^b			
O1-C1	1.315 (3)	1.275 (3)	1.274 (3)	1.324 (3)
O2-C7	1.267 (3)	1.276 (3)	1.281 (3)	1.257 (3)
C1-C7	1.459 (3)	1.467 (4)	1.468 (3)	1.465 (3)
C1-C2	1.381 (3)	1.413 (4)	1.403 (4)	1.373 (3)
C2-C3	1.395 (4)	1.366 (4)	1.381 (4)	1.398 (4)
C3-C4	1.368 (4)	1.384 (5)	1.380 (4)	1.360 (4)
C4-C5	1.389 (4)	1.369 (5)	1.379 (4)	1.394 (4)
C5-C6	1.370 (4)	1.383 (4)	1.373 (4)	1.355 (4)
C6-C7	1.424 (4)	1.400 (4)	1.407 (4)	1.425 (4)
C2-H2	0.94 (2)	0.97 (3)	1.00 (3)	0.93 (2)
C3-H3	0.99 (3)	0.97 (3)	0.97 (3)	0.93 (3)
C4-H4	0.96 (3)	0.94 (3)	0.98 (3)	0.93 (3)
C5-H5	0.92 (3)	0.95 (3)	0.91 (3)	1.00 (3)
C6-H6	0.96 (3)	0.98 (3)	0.97 (3)	0.95 (3)
	Angles			
O1-C1-C2	120.2 (2)	120.0 (2)	120.0 (2)	120.6 (2)
O1-C1-C7	112.1 (2)	114.3 (2)	114.1 (2)	111.0 (2)
C7-C1-C2	127.7 (2)	125.7 (2)	125.9 (2)	128.5 (2)
C1-C2-C3	130.5 (2)	130.9 (3)	130.5 (3)	129.5 (3)
C1-C2-H2	114.5 (14)	111.5 (18)	112.3 (15)	115.4 (15)
H2-C2-C3	115.1 (14)	117.5 (18)	117.2 (15)	115.1 (15)
C2-C3-C4	129.9 (3)	129.4 (3)	129.7 (3)	129.5 (3)
C2-C3-H3	115.5 (15)	114.2 (19)	115.4 (19)	112.1 (17)
H3-C3-C4	114.6 (15)	116.4 (18)	114.7 (19)	118.4 (17)
C3-C4-C5	126.7 (13)	127.5 (3)	127.4 (3)	127.7 (3)
C3-C4-H4	117.6 (16)	115.0 (20)	117.2 (18)	118.0 (19)
H4-C4-C5	115.7 (16)	117.3 (19)	115.2 (18)	114.2 (19)
C4-C5-C6	130.0 (3)	130.1 (3)	130.1 (3)	129.7 (3)
C4-C5-H5	116.8 (16)	119.3 (20)	117.0 (17)	114.9 (16)
H5-C5-C6	113.1 (16)	110.4 (20)	112.8 (17)	115.3 (16)
C5-C6-C7	131.3 (3)	130.4 (3)	130.2 (3)	130.9 (3)
C5-C6-H6	114.5 (15)	117.5 (16)	113.5 (16)	117.6 (17)
H6-C6-C7	114.1 (15)	112.0 (16)	116.2 (16)	111.5 (17)
C6-C7-H1	123.9 (2)	125.8 (2)	126.2 (2)	123.9 (2)
C6-C7-O2	119.9 (2)	120.4 (2)	120.5 (2)	119.8 (2)
O2-C7-C1	116.2 (2)	113.8 (2)	113.3 (2)	116.3 (2)

^a Errors referred to last significant digit are in parentheses. ^b Corrections for librational motion of C-O bonds are about one standard deviation; those for C-C bonds, in the range of one to two standard deviations.

The differences are considerably greater than expected errors in the observed distances. However, although the hydrogen bond obviously weakens the bonding between the Sc and the oxygens of ligands 1 and 4, these ligands are still considered to be coordinated in a bidentate manner.

The carbon-hydrogen distances in the ligands average to 0.95 Å, very close to the optimal C-H distance discussed recently by Churchill.²⁴ The average ligand "bite" distance (O1...O2, Table VIII), 2.505 Å, falls within the narrow range observed for other compounds containing chelating tropolonato ligands.^{1,23} The average O1-Sc-O2 angle is 69.9°, significantly smaller than the 73.8° found in Sc(trop)₃.¹ Thus the change in effective ionic radius of the scandium(III) ion on increasing the coordination number from 6 to 8 results in a closing of the O-Sc-O angle rather than an increase in the ligand bite distance.

The thermal amplitudes of the ligand groups can be interpreted in terms of rigid-body motions. The rms deviations of the observed U_{ij} from that for a rigid body corrected for the total number of degrees of freedom, $[\sum \Delta U_{ij}^2 / (n-s)]^{1/2}$

(24) M. R. Churchill, *Inorg. Chem.*, **12**, 1213 (1973).

($U_{ij} = \beta_{ij} / 2\pi^2 a_i^* a_j^*$, $\Delta U_{ij} = U_{ij}(\text{found}) - U_{ij}(\text{rigid body})$), of the four ligands range from 0.0016 to 0.0019 Å² and $\sigma(U_{ij}) \approx 0.0020$ Å².²⁵ Analysis of the librational motion of each ligand indicates the angular movement of each ligand to be approximately of equal magnitude.

Conclusions

Hydrogentetrakis(tropolonato)scandium(III) has been shown to exist as a hydrogen-bonded dimer with the coordination environment of each scandium ion close to that of a D_{2d} dodecahedron. Thus, the prediction made by Muetterties and Wright^{3,4} that the tropolone system may be used to form complexes of high coordination number has been confirmed for the scandium(III) ion. Blight and Kepert²⁶ have discussed the effect of bidentate ligands on the stereochemistry of eight-coordination. For the $M(\text{bidentate})_4$ case, these authors conclude that as the normalized ligand bite distance increases, there is a change in preferred stereochemistry from the D_{2d} dodecahedron to the D_2 square antiprism. For $\text{HSc}(\text{trop})_4$, the normalized ligand bite is 1.13 Å, which is in the region where an intermediate stereochemistry can arise. Presumably the steric requirements of the tropolonato ligands are an important factor in the determination of the stereochemistry adopted. It is interesting to speculate at this point about the stereochemistry of the species $M[\text{Sc}(\text{hfac})_4]$, hfac = hexafluoroacetylacetonate ion, reported by Gurevich, *et al.*^{27,28} If we assume the ligand bite distance obtained by Bennett, *et al.*,²¹ 2.77 Å and an eight-coordinate Sc-O bond length of 2.2 Å, a normalized ligand bite of approximately 1.25 Å is obtained. Blight and Kepert²⁶ suggested that three stereochemistries are possible for values of this magnitude, *viz.*, the D_2 square antiprism, the D_4 square antiprism, and the D_2 dodecahedron. There is little difference between these stereochemistries and it is impossible to predict which one will be preferred. It is anticipated, however, that the stereochemistry of the $[\text{Sc}(\text{hfac})_4]^-$ ion will be something other than the D_{2d} dodecahedron found for $\text{HSc}(\text{trop})_4$.

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Supplementary Material Available. A listing of structure factor amplitudes ($\times 10$) will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 \times 148 mm, 24 \times reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for \$4.00 for photocopy or \$2.00 for microfiche, referring to code number INORG-74-1884.

(25) V. Schomaker and K. N. Trueblood, *Acta Crystallogr., Sect. B*, **24**, 63 (1968); TLS modified for mass weighted displacement coordinates by M. A. Neuman, American Crystallographic Society Meeting, Ames, Iowa, 1971.

(26) D. G. Blight and D. L. Kepert, *Inorg. Chem.*, **11**, 1556 (1972).

(27) M. Z. Gurevich, B. D. Stepin, and V. V. Zelentsov, *Zh. Neorg. Khim.*, **15**, 890 (1970).

(28) M. Z. Gurevich, B. D. Stepin, L. N. Komissarova, N. E. Lebedeva, and T. M. Sas, *Zh. Neorg. Khim.*, **16**, 93 (1971).