As in (I) and (V), the ring C atoms to which the methylene bridges are bonded are bent towards the Cr atom (Table 8). Although the conformation of the [3.3]paracyclophane skeleton in (I) is unequivocally the chair conformation, in (II) owing to the statistical distribution of C(4) we cannot say whether we have the chair and/or the boat form.

The average C—C distance within the aromatic ring, 1·39 (2) Å, agrees with that found in (I) and (V). 2·07 (2) Å is the corresponding value for the Cr—$C_{ring}$ distances, which is only within 2$\sigma$ significantly shorter than the 2·12 (1) Å in (I). Table 8 shows the distance from the Cr atom to the least-squares planes *P*1 and *P*2 to be 1·57 and 1·59 Å respectively, which compare well with the parameters for (I) and (V).

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*Acta Cryst.* (1980). B**36**, 2059–2063

# The Structure of Tris(2,4-pentanedionato)indium(III)

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(*Received* 19 *April* 1979; *accepted* 5 *March* 1980)

## Abstract

The crystal and molecular structure of tris(2,4-pentanedionato)indium(III), or [In(acac)$_3$], has been determined by three-dimensional X-ray diffraction methods. Crystals of orthorhombic [In(acac)$_3$], [In($C_5H_7O_2$)$_3$], $C_{15}H_{21}InO_6$, space group *Pbca*, have unit-cell dimensions $a$ = 15·576 (4), $b$ = 13·724 (5) and $c$ = 16·855 (5) Å. Since the compound is isomorphous with [Fe(acac)$_3$], the parameters for the latter compound were used as starting parameters for a least-squares refinement. The final *R* was 0·034 for the 1516 reflections used in the analysis. The average In—O distance is 2·132 (8) Å. The dimensions of the acac ligands are similar to those reported for other acac complexes. The O···O bite of 2·905 (8) Å is less than the O···O nonbonded contact of 3·053 (85) Å, the usual case in orthorhombic [*M*(acac)$_3$] complexes. Plots of *M*—O distance *versus* ligand bite, *M*—O distance *versus* ionic radius and *M*—O distance *versus* the O···O nonbonded distance are given. The best correlation of the *M*—O distance is with ionic radius, which is not surprising if the interactions between *M* and ligand are mainly electrostatic.

## Introduction

Acetylacetone (acac) forms complexes with most metallic elements. The crystal structures of a number of acac complexes have been determined by X-ray diffraction techniques. The tris complexes of trivalent metal ions are usually octahedral species and fall into either of two isomorphous groups. The [*M*(acac)$_3$] complexes with *M* = Al (Hon & Pfluger, 1973), Co (Kruger & Reynhardt, 1974; Hon & Pfluger, 1973),

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Ga (Dymock & Palenik, 1974), Cr (Morosin, 1965), Mn (Fackler & Avdeef, 1974), Rh (Morrow & Parker, 1973), and Ru (Chao, Sime & Sime, 1973) are monoclinic, space group $P2_1/c$. If $M$ = Sc (Anderson, Neuman & Melson, 1973), V (Morosin & Montgomery, 1969), Fe (Iball & Morgan, 1967), Ga and In (Astbury, 1926), the complexes are orthorhombic with space group *Pbca*. Both Hon & Pfluger (1973) and Lingafelter & Braun (1966) have tried to correlate the metal—oxygen distances, the ligand bite, and the O$\cdots$O non-bonded contacts with each other or with the metal-ion radius. With the exception of the Rh and Ru complexes, all the data have involved metal ions in the same row of the Periodic Table. Therefore, after our study of the $Ga^{3+}$ complex (Dymock & Palenik, 1974), we decided to extend our investigations to the spherical $d^{10}$ $In^{III}$ ion. The $In^{III}$ ion is in the same row as the Rh and Ru ions, and a comparison with the more extensively studied ions of the preceding row will help in understanding the structural chemistry of acac complexes. Furthermore, recently Rodríguez, Cano & García-Blanco (1979) reported a new monoclinic form of [In(acac)$_3$] which would provide an additional comparison with our results.

Table 2. *Final positional parameters for tris*(2,4-*pentanedionato*)*indium*(III)

The parameters are × $10^4$ except for those of indium which are × $10^5$.

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| In | 14259 (3) | 26891 (3) | 24244 (3) |
| O(1) | 711 (4) | 3578 (4) | 3224 (3) |
| O(2) | 376 (3) | 1690 (3) | 2505 (4) |
| O(3) | 921 (3) | 3420 (4) | 1412 (3) |
| O(4) | 2025 (3) | 1736 (4) | 1585 (3) |
| O(5) | 2439 (3) | 3730 (3) | 2503 (4) |
| O(6) | 2076 (3) | 1915 (4) | 3330 (3) |
| C(1) | −24 (5) | 3393 (6) | 3513 (4) |
| C(2) | −511 (5) | 2553 (6) | 3387 (5) |
| C(3) | −303 (5) | 1776 (5) | 2915 (4) |
| C(4) | −350 (7) | 4169 (8) | 4061 (6) |
| C(5) | −896 (5) | 903 (6) | 2873 (6) |
| C(6) | 967 (5) | 3177 (6) | 698 (5) |
| C(7) | 1407 (6) | 2355 (6) | 412 (4) |
| C(8) | 1897 (5) | 1712 (6) | 847 (4) |
| C(9) | 518 (7) | 3825 (8) | 117 (5) |
| C(10) | 2378 (7) | 908 (7) | 418 (5) |
| C(11) | 3080 (5) | 3708 (5) | 2961 (5) |
| C(12) | 3282 (5) | 2960 (6) | 3471 (5) |
| C(13) | 2797 (5) | 2131 (6) | 3611 (4) |
| C(14) | 3667 (6) | 4592 (6) | 2908 (6) |
| C(15) | 3188 (7) | 1354 (8) | 4196 (6) |

## Experimental

The title compound was prepared according to the procedure given by Morgan & Moss (1914). Colorless crystals of [In(acac)$_3$] were grown by slow evaporation of an absolute methanol solution. Preliminary precession photographs indicated that the crystals were orthorhombic with the space group *Pbca*. The unit-cell dimensions and intensity measurements were made using a Syntex $P\bar{1}$ diffractometer with a graphite monochromator and Mo radiation. The details are similar to those reported by Dymock & Palenik (1974), and the pertinent crystal data are given in Table 1. The four standard reflections which were measured after every 96 reflections showed a maximum variation of ±3%. The polarization corrections were calculated assuming that the monochromator was a 50% perfect and 50% mosaic crystal.

### *Solution and refinement of the structure*

A comparison of reflections with large structure amplitudes for [In(acac)$_3$] with those of [Fe(acac)$_3$] showed an approximately constant ratio, as expected for an isomorphous pair. A Fourier synthesis was calculated using the coordinates of the non-hydrogen atoms in the [Fe(acac)$_3$] structure. The usual residual $R$ $(=\sum||F_o|-|F_c||/\sum|F_o|)$ was 0·14. Three full-matrix least-squares cycles with individual isotropic thermal parameters reduced $R$ to 0·060. Three additional least-squares cycles with anisotropic thermal parameters for all atoms reduced $R$ to 0·042. A difference Fourier synthesis was computed and used to locate all the H atoms. Each of the five H atoms was assigned an isotropic thermal parameter which was one unit larger than the isotropic value for the atom to which the H was bonded. The H atom contributions were included but not varied in three subsequent least-squares cycles which reduced $R$ to 0·034. The shifts in all the variable parameters were less than 0·1 of their estimated standard deviations, and therefore the refinement was considered complete. The final positional parameters for the non-hydrogen atoms are given in Table 2.*

Table 1. *Crystal data and experimental parameters*

$C_{15}H_{21}InO_6$, $M_r$ = 412·15, space group *Pbca*, $a$ = 15·576 (4),
$b$ = 13·724 (5), $c$ = 16·855 (5) Å, $V$ = 3603 (2) Å$^3$, $Z$ = 8,
$D_c$ = 1·521, $D_m$ = 1·52 Mg m$^{-3}$, $\mu$(Mo) = 1·33 mm$^{-1}$,
$\lambda$(Mo $K\alpha_1$) = 0·70926, $\lambda$(Mo $K\alpha_2$) = 0·71354 Å, $2\theta$ limit = 45°
2370 reflections with 1516 reflections ≥ $2\sigma(I)$,
crystal size 0·17 × 0·13 × 0·09 mm.

The quantity minimized in the least-squares refinement was $\sum w(|F_o|-|F_c|)^2$ where $w^{1/2}=|F_o|/54{\cdot}0$ if $|F_o|<54{\cdot}0$, $w^{1/2}=1$ if $54{\cdot}0\le|F_o|\le162{\cdot}0$, or $w^{1/2}=162/|F_o|$ if $|F_o|>162{\cdot}0$. The scattering factors for In, C and O were taken from Hanson, Herman, Lea &

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* Lists of structure factors, anisotropic thermal parameters for the non-hydrogen atoms and the hydrogen atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35256 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Skillman (1964) and those for H from Stewart, Davidson & Simpson (1965).

## Results and discussion

An *ORTEP* (Johnson, 1965) drawing illustrating the thermal ellipsoids and atomic numbering is given in Fig. 1. The six O atoms are at the vertices of a slightly distorted octahedron. The molecular symmetry is nearly $D_3$ with the threefold axis normal to the O(2)–O(4)–O(6) face. The two triangular faces are tipped by 2·4° relative to each other. The twist angles are defined in terms of the dihedral angles of the two centroids and two atoms in the opposite faces, as discussed by Dymock & Palenik (1975). The angular twists are 67·6° for the O(1)–O(6) pair, 68·4° for the O(5)–O(4) pair and 68·8° for the O(3)–O(2) pair. These angles are larger than the ideal 60° because the angles involving the ligand are all less than 60° (53·4, 51·6 and 50·4° for the three ligands), presumably because of the restrictions on the ligand bite.

Table 3. *Bond lengths, non-bonded contacts and bond angles for tris*(2,4-*pentanedionato*)*indium*(III) *with their estimated standard deviations in parentheses*

(*a*) Bond lengths (Å)

| | | | |
|---|---|---|---|
| In–O(1) | 2·132 (5) | O(3)–C(6) | 1·252 (9) |
| In–O(2) | 2·138 (4) | C(6)–C(9) | 1·495 (13) |
| In–O(3) | 2·130 (5) | C(6)–C(7) | 1·406 (12) |
| In–O(4) | 2·141 (5) | C(7)–C(8) | 1·379 (11) |
| In–O(5) | 2·133 (4) | C(8)–C(10) | 1·517 (13) |
| In–O(6) | 2·118 (5) | C(8)–O(4) | 1·260 (9) |
| O(1)–C(1) | 1·270 (10) | O(5)–C(11) | 1·263 (9) |
| C(1)–C(4) | 1·499 (13) | C(11)–C(14) | 1·522 (11) |
| C(1)–C(2) | 1·397 (12) | C(11)–C(12) | 1·375 (11) |
| C(2)–C(3) | 1·370 (11) | C(12)–C(13) | 1·386 (11) |
| C(3)–C(5) | 1·514 (11) | C(13)–C(15) | 1·575 (14) |
| C(3)–O(2) | 1·269 (9) | C(13)–O(6) | 1·254 (9) |

(*b*) Non-bonded contacts (Å)

| | | | |
|---|---|---|---|
| O(1)–O(2) | 2·908 (7) | O(2)–O(6) | 3·008 (7) |
| O(1)–O(3) | 3·079 (7) | O(3)–O(4) | 2·895 (7) |
| O(1)–O(5) | 2·960 (7) | O(3)–O(5) | 3·025 (7) |
| O(1)–O(6) | 3·125 (7) | O(4)–O(5) | 3·209 (7) |
| O(2)–O(3) | 3·122 (7) | O(4)–O(6) | 2·954 (7) |
| O(2)–O(4) | 3·000 (7) | O(5)–O(6) | 2·911 (7) |

(*c*) Bond angles (°)

| | | | |
|---|---|---|---|
| O(1)–In–O(2) | 85·9 (2) | C(5)–C(3)–O(2) | 114·2 (6) |
| O(1)–In–O(3) | 92·5 (2) | C(2)–C(3)–O(2) | 125·8 (7) |
| O(1)–In–O(4) | 174·3 (2) | C(3)–O(2)–In | 127·8 (5) |
| O(1)–In–O(5) | 87·9 (2) | In–O(3)–C(6) | 128·6 (5) |
| O(1)–In–O(6) | 94·6 (2) | O(3)–C(6)–C(7) | 124·9 (7) |
| O(2)–In–O(3) | 94·0 (2) | O(3)–C(6)–C(9) | 116·4 (7) |
| O(2)–In–O(4) | 89·1 (2) | C(9)–C(6)–C(7) | 118·7 (8) |
| O(2)–In–O(5) | 172·5 (2) | C(6)–C(7)–C(8) | 127·0 (8) |
| O(2)–In–O(6) | 89·9 (2) | C(7)–C(8)–O(4) | 126·6 (7) |
| O(3)–In–O(4) | 85·4 (2) | C(7)–C(8)–C(10) | 119·1 (7) |
| O(3)–In–O(5) | 90·4 (2) | C(10)–C(8)–O(4) | 114·3 (7) |
| O(3)–In–O(6) | 172·1 (2) | C(8)–O(4)–In | 126·9 (5) |
| O(4)–In–O(5) | 97·4 (2) | In–O(5)–C(11) | 127·4 (5) |
| O(4)–In–O(6) | 87·8 (2) | O(5)–C(11)–C(12) | 125·5 (7) |
| O(5)–In–O(6) | 86·4 (2) | O(5)–C(11)–C(14) | 114·8 (7) |
| In–O(1)–C(1) | 126·8 (5) | C(14)–C(11)–C(12) | 119·7 (7) |
| O(1)–C(1)–C(4) | 113·6 (7) | C(11)–C(12)–C(13) | 126·5 (7) |
| C(4)–C(1)–C(2) | 119·7 (7) | C(12)–C(13)–O(6) | 128·2 (7) |
| C(1)–C(2)–C(3) | 127·0 (7) | C(12)–C(13)–C(15) | 116·9 (7) |
| C(2)–C(3)–C(5) | 119·9 (7) | C(15)–C(13)–O(6) | 115·0 (7) |
| O(1)–C(1)–C(2) | 126·7 (7) | C(13)–O(6)–In | 125·6 (5) |

The average distances and angles in the acac ligand [C–O 1·261 (7), C–$CH_3$ 1·525 (30), C–CH 1·386 (14) Å; O–C–CH 126·2 (12), C–CH–C 126·8 (3), O–C–$CH_3$ 114·7 (10), CH–C–$CH_3$ 119·0 (11)°] are similar to the average values in other acac complexes, summarized by Fackler & Avdeef (1974). However, as noted by Chao, Sime & Sime (1973), the O···O bite is greater than the O···O non-bite for the isomorphous $P2_1/c$ series while the reverse is true for the complexes crystallizing in *Pbca*. Alternatively, the O–*M*–O angles are greater than 90° and the *M*–O–C angles are less than about 127° in the $P2_1/c$ series. However, in the isomorphous monoclinic $P2_1/n$ forms of [In(acac)$_3$] and *β*-[V(acac)$_3$], the O–*M*–O angles, *etc*., are identical to those found in the orthorhombic *Pbca* forms. That the O–*M*–O angle is independent of the *M*–O distance can be seen by comparing [Fe(acac)$_3$] [Fe–O is 1·992 Å, O–Fe–O 87·1°] with [Rh(acac)$_3$] [Rh–O is 1·992 Å, O–Rh–O 95·3°]. Finally, In and Ga have the same electronic configuration, but the corresponding acac complexes crystallize in different space groups. Consequently, one is forced to conclude that in [*M*(acac)$_3$] complexes the molecular dimensions and crystal packing are probably not a simple function of any one parameter.

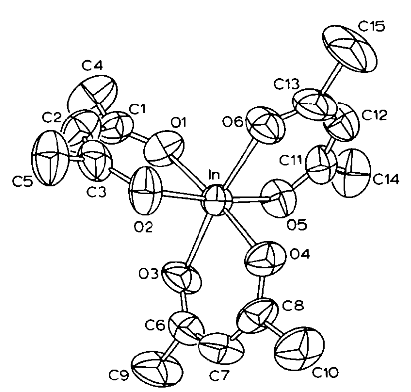

Fig. 1. An *ORTEP* drawing of tris(2,4-pentanedionato)indium(III) illustrating the thermal ellipsoids and atomic numbering.

The bond distances and angles are given in Table 3. The In–O distances range from 2·118 to 2·141 Å, with an average of 2·132 (8) Å. The average value is virtually identical to that of 2·128 (12) Å found by Rodríguez, Cano & García-Blanco (1979) for the monoclinic form [In(acac)$_3$], although their values range from 2·108 to 2·146 Å. In tris[4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato]indium(III) (Soling, 1976) the In–O distances average 2·131 (19) Å, but the six distances appear to fall into two groups. The In—O bonds on the $CF_3$ side are 2·099, 2·118 and 2·132 Å with an average of 2·116 Å, while on the thienyl side the distances are 2·144, 2·140 and 2·151 Å with an average of 2·145 Å. The difference of the In—O bonds in this case reflects the differences in the electronic and steric properties of the two sides of the ligand. The fact that the average value is virtually identical to that of the unsubstituted acac complexes may be a manifestation of the electroneutrality principle (Pauling, 1960). In $[In(acac)(bipy)Cl_2]$ the two In—O distances were reported to be 2·124 and 2·164 Å (Contreras, Einstein & Tuck, 1974). The longer In—O bond is *trans* to a Cl atom which suggests a *trans* effect. A comparison of the In—Cl and In—O distances in the series $[InCl_3(H_2O)_3]$, $[InCl_3(H_2O)_3]^-$, and $[InCl_5(H_2O)]^{2-}$, however, is not completely in agreement with a *trans* effect. The average In—Cl distance increases from 2·422 Å in $[InCl_3(H_2O)_3]$ (Whitlow & Gabe, 1975), to 2·455 Å in $[InCl_4(H_2O)_2]^-$ (Ziegler, Schlimper, Nuber, Weiss & Ertl, 1975), to 2·479 Å in $[InCl_5(H_2O)]^{2-}$ (Wignacourt, Mairesse & Barbier, 1976). The average In—O bond distances are 2·244 (42) Å in $[InCl_3(H_2O)_3]$, 2·264 (7) Å in $[InCl_4(H_2O)_2]^-$, and 2·224 (9) Å in $[InCl_5(H_2O)]^{2-}$. Unfortunately, the large e.s.d.'s of the In—O bonds in $[InCl_3(H_2O)_3]$ make a discussion of any trends meaningless. However, the most important observation is that the In—O and In—Cl bonds are influenced not only by the *trans* atom, but also by the other atoms in the coordination sphere.

Attempts have been made by Hon & Pfluger (1973) and Lingafelter & Braun (1966) to correlate the *M*—O distance with ligand bite, ionic radius, or non-bonded contacts. The relationship between the *M*—O distance and the ligand bite is illustrated in Fig. 2, which includes nine additional points for structures which were not available to Hon & Pfluger (1973). Although the data for the elements from Sc to Co are scattered near a linear fit (the goodness of fit is 0·71), the values for the Al, Ru, Rh and In complexes deviate rather markedly. Consequently, a linear relationship between the *M*—O distance and the ligand bite does not exist for all $[M(acac)_3]$ complexes.

However, there is a linear relationship (goodness of fit 0·99) between the *M*—O distance and the $M^{3+}$ ionic radius, as shown in Fig. 3. The values of the ionic radii for coordination number six were taken from the compilation of Shannon (1976). The intercept of the least-squares line is 1·40 Å, which is the ionic radius of oxygen, and the slope of 0·91 is close to the expected value of 1·00. The linearity of the plot is in agreement with the assumption that the *M*—O bond is largely ionic in character. Therefore, although the acac ligand can be considered as a delocalized system, the metal–ligand interaction is predominantly ionic.

A plot of the metal–oxygen distance *versus* the O···O non-bonded contact is shown in Fig. 4. Excluding the values for Al, Rh, Ru and In, the remaining values fall on a straight line (goodness of fit 0·99). The values for the heavier elements Rh, Ru and In all fall above the line while the Al values are below the line. In the case of six independent O atoms octahedrally surrounding a metal ion, the O···O non-bonded contact would be the $\sqrt{2}$ times the *M*—O distance. The observed straight line has a slope of only 0·46 with an intercept of 0·67. Obviously, the six O atoms in the $[M(acac)_3]$ complexes are not independent; nevertheless, maintaining an appropriate

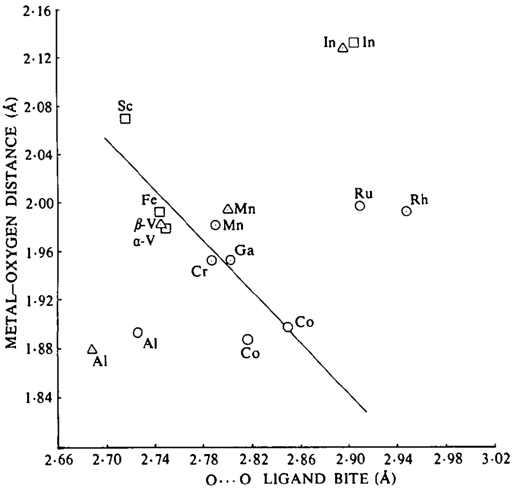

Fig. 2. A plot of the metal–oxygen distance *versus* the ligand bite for various $[M(acac)_3]$ complexes. The circles indicate isomorphous complexes with the space group $P2_1/c$, while the squares are those with space group *Pbca*. The triangles are complexes with space groups other than the above two. The straight line is the least-squares fit for the first-row transition-metal complexes Sc to Co.

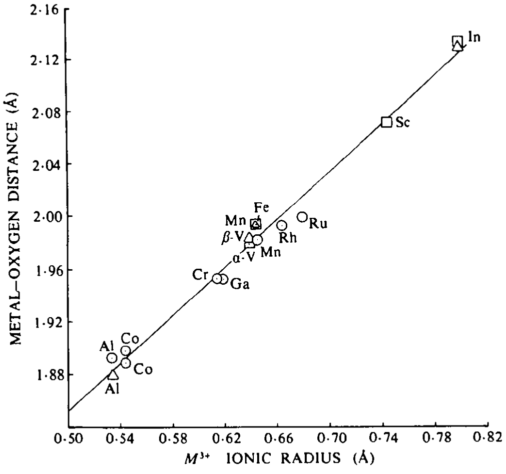

Fig. 3. A plot of the metal—oxygen distance *versus* the $M^{3+}$ ionic radius. The circles, squares and triangles are explained in the caption for Fig. 2. The straight line has a slope of 0·91 with an intercept of 1·40 Å.

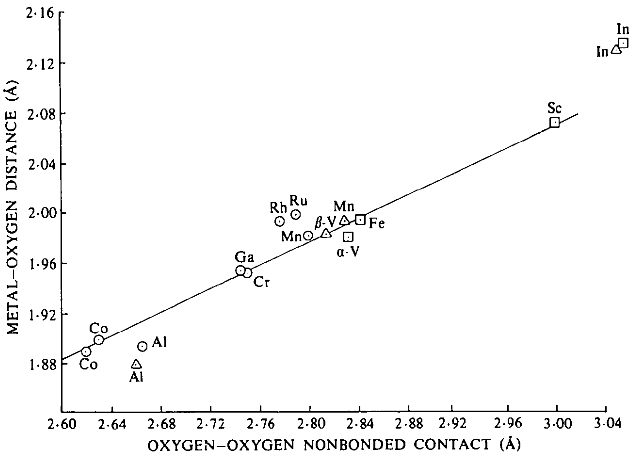

Fig. 4. A plot of the metal–oxygen radius *versus* the O···O nonbonded distance. See caption for Fig. 2 for a description of the circles, squares and triangles and the straight line.

O···O non-bonded distance must be an important factor. In the relatively large Sc and In atoms the O···O contact appears to be larger than the expected van der Waals distance of about 2·8 Å. Although the acac ligand is somewhat flexible, the bite cannot increase sufficiently to maintain van der Waals contact as the *M*–O distance increases. Therefore, the observation that the large tripositive lanthanide ions form seven- and eight-coordinate complexes, either by dimerization (de Villiers & Boyens, 1971) or addition of other bases (Wasson, Sands & Wagner, 1973), is not surprising.

We are grateful for a Graduate School Fellowship (KRD) and a grant of computer time from the Center for Instructional Research Computing Activities, University of Florida (GJP).

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