bond character of the azo group is best preserved in the Ni(tan)$_2$ complex (Kurahashi, 1974*b*) [N—N = 1·26 (6) Å] while a very long N—N length exists in PdCl(tan) [1·39 (3) Å (Kurahashi, 1974*a*)], similar to those in the present structure.

The amide H atoms originally attached to N(1) and N(6) help to enolize the keto group so that the C—N and C—O bonds show partial double-bond character due to the delocalization of π electrons in this group.

There is one loosely bound water molecule of crystallization which is not hydrogen bonded. The H atoms attached to O(2) and O(4) are not well resolved; but judging from the O···N distances, two intramolecular hydrogen bonds may be assumed to exist between O(2) and N(1), and O(4) and N(6) at distances of 2·55 (2) and 2·53 (2) Å respectively. Fig. 2 illustrates the packing of the molecules in the unit cell; the structure consists essentially of a series of layers parallel to *ab* and the molecules are displaced with respect to one another to relieve short intermolecular contacts. Since all other H atoms are attached to C, it is unlikely that they are involved in hydrogen bonding; therefore, intermolecular bonding is probably of the van der Waals type. There are no unusually short intermolecular contacts so that packing forces are unlikely to influence the geometry of the molecule to an appreciable extent.

The authors are glad to have this opportunity of thanking Dr Siddhartha Ray, IACS, Jadavpur, Calcutta, for valuable suggestions and stimulating discussions and Professor R. L. Dutta of the Department of Chemistry, University of Burdwan, for providing the sample used in this investigation. We thank the Regional Sophisticated Instrumentation Centre, Calcutta, for the diffractometer-data collection. Financial support of this work by the University Grants Commission, New Delhi, is gratefully acknowledged.

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Acta Cryst. (1984). C**40**, 1533–1536

Organomercury Medicinal Chemistry. Synthesis and Structure of a (β-Methoxyethyl)mercury(II) Derivative of N(7)-Deprotonated Theophylline,* [Hg(C_3H_7O)($C_7H_7N_4O_2$)]

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(*Received* 6 *March* 1984; *accepted* 24 *April* 1984)

Abstract. $M_r = 438\cdot84$, monoclinic, $P2_1/c$, $a = 8\cdot305\,(2)$, $b = 9\cdot349\,(3)$, $c = 16\cdot370\,(4)$ Å, $\beta = 97\cdot37\,(1)°$, $U = 1260\cdot5$ Å^3, $Z = 4$, density not measured as material very soluble, $D_x = 2\cdot31$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot7107$ Å, $\mu = 12\cdot176$ mm^{-1}, $F(000) = 824$, $T = 293$ K. Final $R = 0\cdot057$ for 1461 observed counter reflections. The structure consists of molecules of [Hg($CH_2CH_2OCH_3$)($C_7H_7N_4O_2$)] linked into a three-dimensional network by weak intermolecular Hg···O (methoxyethyl) and Hg···N(9) (theophylline) interactions of 2·89 (1) and 2·91 (1) Å respectively.

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* (2-Methoxyethyl)(theophyllinato-N^7)mercury(II).

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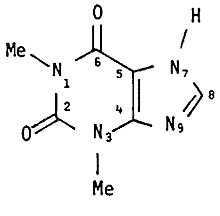
The mercury atom is bound to the deprotonated nitrogen atom N(7), with Hg—N(7) 2·08 (1), Hg—C (methoxyethyl) 2·13 (2) Å and C—Hg—N(7) 173·3 (6)°.

Introduction. Organomercury compounds are widely used in medicinal applications (*Martindale, the Extra Pharmacopoeia,* 1977; *British Pharmacopoeia,* 1980), *e.g.* phenylmercury(II) compounds and the ethylmercury complex thimerosal as preservatives in some eyedrop and topically applied preparations, and in some injection solutions, respectively. Alkoxyalkylmercurials have been used as fungicides and bacteriocides (Vostal & Clarkson, 1973) and, in addition, a range of diuretics containing substituted (β-methoxyethyl)mercury(II) groups $R\mathrm{CH(OMe)CH_2Hg^{II}}$ and $\mathrm{CH_2(OMe)CH(}R\mathrm{)Hg^{II}}$, have been extensively used since the 1920's (*Martindale, the Extra Pharmacopoeia,* 1977; Howard, 1973). Theophylline (Htheo) is often administered in conjunction with the mercurial diuretics as its presence greatly enhances diuretic effectiveness, but the nature of any chemical reaction between organomercurials and theophylline has not been established, although it has been proposed that a complex is formed with mercury bound to N(7) of a deprotonated theophylline.



Htheo

We report here the isolation of a crystalline complex from the reaction of (β-methoxyethyl)mercury(II) acetate and theophylline, and a crystallographic determination of the binding site for mercury in the complex. The complex may be regarded as a model for the species formed on co-administration of an organomercury diuretic with theophylline.

Experimental. (β-Methoxyethyl)mercury(II) acetate was prepared as described (Schoeller, Schrauth & Essers, 1913; Cotton & Leto, 1958), and theophylline was obtained from BDH. Theophylline in boiling methanol was added dropwise to an equimolar quantity of (β-methoxyethyl)mercury(II) acetate in methanol, and the resulting solution stirred for 2–3 h at ambient temperature. On evaporation to dryness a white solid was collected and recrystallized from hot benzene. The crystals were washed with cold benzene and dried over silica gel under vacuum {yield 85%; m.p. 439–439·5 K; composition: found: C 27·6, H 3·2, N 12·7%; calculated for [$\mathrm{Hg(CH_2CH_2OCH_3)(C_7H_7N_4O_2)}$]: C 27·4, H 3·2, N 12·8%}. Microanalyses were by the Australian Microanalytical Service, Melbourne.

Space group $P2_1/c$ from systematic absences, unit-cell parameters determined with a Philips PW 1100 automatic four-circle diffractometer equipped with a graphite monochromator as described previously (Gatehouse & Miskin, 1974), and are the means of eight refinements of the 24 reflections used to monitor crystal stability during data collection; three standard reflections measured at 2 h intervals indicated that decomposition had occurred during data collection, correction made for a 6% decomposition during data-collection period; data collected using $\omega/2\theta$ scans, a symmetric scan width of $\pm1{\cdot}40°$ in ω from the calculated Bragg angle, with an allowance for dispersion, at a scan rate of $0{\cdot}04°\,\mathrm{s}^{-1}$; no reflection sufficiently intense to require attenuation; data processed using a program written specifically for the PW 1100 diffractometer (Hornstra & Stubbe, 1972); values of I and $\sigma(I)$ corrected for Lorentz and polarization effects; absorption correction applied based on indexed crystal faces, max. and min. values of transmission factors 0·4348 and 0·2103; 2210 reflections measured to $2\theta = 55°$ from a crystal 0·05 × 0·05 × 0·063 mm and, of these, 1461 unique reflections [$I \geq 3\sigma(I)$] used in the analysis, index range: $h \pm 9$, k 0/11, l 0/18. Hg atom located from Patterson synthesis; Hg-atom position refined and all non-H atoms located in subsequent difference Fourier syntheses; function minimized in full-matrix least-squares refinement $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F_o)$; Hg atom refined anisotropically and other non-H atoms refined isotropically, final $R = 0{\cdot}057$, $R_w = 0{\cdot}053$ (for observed reflections); max. shift/error = 0·001; inclusion of H atoms not considered warranted; 78 variables refined; residual electron density in final difference synthesis within −1·8 and +1·15 e Å^{-3}. Scattering factors for neutral atoms and corrections for anomalous dispersion from Cromer & Mann (1968) and Cromer & Liberman

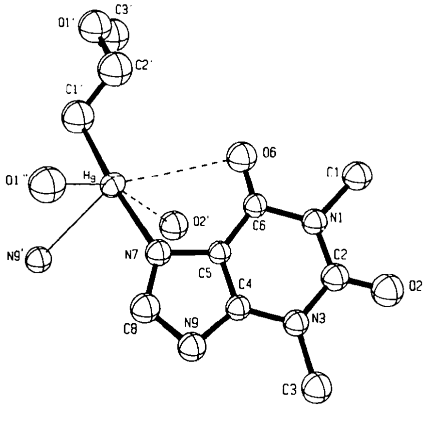


Fig. 1. View of the structure of [$\mathrm{Hg(CH_2CH_2OMe)(theo)}$] showing intermolecular Hg···O(1′$^{\mathrm{II}}$) and Hg···N(9$^{\mathrm{I}}$) interactions.

Table 1. *Fractional coordinates* (×10^4) *and thermal parameters* (×10^3) *with e.s.d.'s in parentheses*

	x	y	z	$U_{\rm iso}$(Å^2)
Hg	7654 (1)	2099 (1)	2132 (1)	*
C(1)	7588 (20)	−915 (19)	−919 (10)	50 (5)
C(2)	5680 (19)	−2214 (18)	−182 (9)	41 (4)
C(3)	3690 (19)	−3614 (20)	517 (10)	47 (4)
C(4)	5426 (17)	−1695 (15)	1199 (8)	32 (4)
C(5)	6423 (17)	−568 (15)	1171 (8)	29 (3)
C(6)	7215 (17)	−205 (16)	505 (8)	33 (4)
C(8)	5668 (20)	−720 (18)	2377 (10)	47 (4)
C(1′)	8739 (24)	4160 (21)	2160 (11)	65 (6)
C(2′)	9862 (25)	4225 (23)	1544 (12)	73 (6)
C(3′)	9425 (26)	6601 (24)	922 (13)	76 (6)
N(1)	6782 (14)	−1142 (14)	−170 (7)	36 (3)
N(3)	4989 (15)	−2529 (13)	529 (7)	35 (3)
N(7)	6583 (14)	91 (14)	1956 (7)	38 (3)
N(9)	4891 (15)	−1841 (14)	1958 (7)	42 (3)
O(2)	5248 (14)	−2920 (13)	−809 (7)	56 (3)
O(6)	8175 (14)	777 (12)	442 (6)	51 (3)
O(1′)	10478 (15)	5706 (14)	1480 (7)	68 (4)

* Anisotropic thermal parameters (Å^2 ×10^4) are of the type given by: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg	566 (4)	401 (4)	425 (4)	−142 (4)	114 (3)	−56 (4)

Table 2. *Interatomic distances* (Å) *and angles* (°) *in* [Hg(CH_2CH_2OMe)(*theo*)], *with e.s.d.'s in parentheses*

(*a*) Mercury environment			
Hg–N(7)	2·08 (1)	Hg···N(9$^{\rm i}$)	2·91 (1)
Hg–C(1′)	2·13 (2)	Hg···O(1$'^{\rm ii}$)	2·89 (1)
C(1′)–Hg–N(7)	173·3 (6)	N(7)–Hg–N(9$^{\rm i}$)	93·0 (4)
C(1′)–Hg–N(9$^{\rm i}$)	90·8 (6)	N(7)–Hg–O(1$'^{\rm ii}$)	83·1 (4)
C(1′)–Hg–O(1$'^{\rm ii}$)	102·0 (6)	N(9$^{\rm i}$)–Hg–O(1$'^{\rm ii}$)	95·9 (3)
Non-bonding neighbouring atoms			
Hg···O(6)	3·11 (1)	Hg···O(2$^{\rm i}$)	3·12 (1)
C(1′)–Hg–O(6)	105·8 (6)	O(2$^{\rm i}$)–Hg–O(6)	70·4 (3)
C(1′)–Hg–O(2$^{\rm i}$)	94·6 (5)	O(2$^{\rm i}$)–Hg–N(7)	81·2 (4)
O(6)–Hg–O(1$'^{\rm ii}$)	113·5 (3)	O(6)–Hg–N(7)	67·9 (4)
O(1$'^{\rm ii}$)–Hg–O(2$^{\rm i}$)	160·8 (3)	O(6)–Hg–N(9$^{\rm i}$)	141·6 (3)
O(2$^{\rm i}$)–Hg–N(9$^{\rm i}$)	74·0 (3)		
(*b*) (Methoxyethyl)mercury group			
C(1′)–C(2′)	1·46 (3)	C(3′)–O(1′)	1·45 (2)
C(2′)–O(1′)	1·48 (3)		
Hg–C(1′)–C(2′)	109 (1)	C(1′)–C(2′)–O(1′)	110 (2)
C(2′)–O(1′)–C(3′)	113 (1)		
(*c*) Theophylline group			
C(1)–N(1)	1·49 (2)	C(4)–N(9)	1·38 (2)
C(2)–N(1)	1·36 (2)	C(5)–C(6)	1·39 (2)
C(2)–N(3)	1·39 (2)	C(5)–N(7)	1·42 (2)
C(2)–O(2)	1·23 (2)	C(6)–N(1)	1·42 (2)
C(3)–N(3)	1·48 (2)	C(6)–O(6)	1·23 (2)
C(4)–N(3)	1·36 (2)	C(8)–N(7)	1·33 (2)
C(4)–C(5)	1·34 (2)	C(8)–N(9)	1·37 (2)
C(1)–N(1)–C(2)	118 (1)	C(4)–C(5)–C(6)	125 (1)
C(1)–N(1)–C(6)	117 (1)	C(4)–C(5)–N(7)	107 (1)
C(2)–N(1)–C(6)	125 (1)	C(6)–C(5)–N(7)	128 (1)
N(1)–C(2)–N(3)	120 (1)	C(5)–C(6)–N(1)	111 (1)
N(1)–C(2)–O(2)	122 (1)	C(5)–C(6)–O(6)	129 (1)
N(3)–C(2)–O(2)	118 (1)	N(1)–C(6)–O(6)	120 (1)
C(2)–N(3)–C(3)	121 (1)	C(5)–N(7)–C(8)	103 (1)
C(2)–N(3)–C(4)	117 (1)	C(5)–N(7)–Hg	120·4 (9)
C(3)–N(3)–C(4)	122 (1)	C(8)–N(7)–Hg	135 (1)
N(3)–C(4)–C(5)	122 (1)	N(7)–C(8)–N(9)	116 (1)
N(3)–C(4)–N(9)	126 (1)	C(4)–N(9)–C(8)	101 (1)
C(5)–C(4)–N(9)	112 (1)		

Symmetry code: (i) $1-x, -y, -z$; (ii) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

(1970); calculations carried out using the Monash University B6700 computer. Major programs used: *SHELX*76 (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964) and *MEANPL* (Blow, 1960); figure drawn with *ORTEP* (Johnson, 1965).

Discussion. A view of the complex is shown in Fig. 1. Final fractional coordinates and thermal parameters and interatomic distances and angles are given in Tables 1 and 2.*

The mercury atom of the (β-methoxyethyl)mercury(II), $MeOCH_2CH_2Hg^{II}$, group is bound to N(7) of a deprotonated theophylline group, theo$^-$, with Hg–N(7) 2·08 (1) Å, a similar bond length to that found for Hg–N(7) or Hg–N(9) binding in $MeHg^{II}$ complexes of the related purines 9-methylguanine, 2·09 (2) Å (Canty, Tobias, Chaichit & Gatehouse, 1980), adenine, 2·07–2·10 Å (Hubert & Beauchamp, 1980; Prizant, Olivier, Rivest & Beauchamp, 1981, 1982), and inosine, 2·11 (2) Å (Bélanger-Gariéby & Beauchamp, 1982). The Hg atom is 0·548 (1) Å from the mean plane of the theophylline group.*

The C(1′)–Hg–N(7) moiety is slightly bent from linearity, 173·3 (6)°, with the bending occurring *opposite* two weak intermolecular Hg···N(9$^{\rm i}$) [2·91 (1) Å] and Hg···O(1$'^{\rm ii}$) [2·89 (1) Å] interactions. These interactions are 0·2–0·3 Å less than the sum of the van der Waals radii of O (1·4 Å) or N (1·5 Å) (Pauling, 1960), and Hg (*ca* 1·73 Å) (Canty & Deacon, 1980). The N(9$^{\rm i}$)–Hg–O(1$'^{\rm ii}$) angle, 95·9 (3)°, between the two weak donor atoms contrasts with the near-linear NHgN′ arrangement for the weakly bonded donors in bis[2-(dimethylaminomethyl)phenyl]mercury(II) (Atwood, Berry, Stobart & Zaworotko, 1983). The [Hg(CH_2CH_2OMe)(theo)] molecules are thus weakly linked into a three-dimensional network *via* these bridging interactions.

Two other distances, Hg···O(2$^{\rm i}$) [3·12 (1) Å] and Hg···O(6) [3·11 (1) Å] are approximately equal to the van der Waals radius sum. Although they are only 0·2–0·3 Å longer than contacts viewed as weakly bonding, the disposition of O(2$^{\rm i}$) and O(6) about Hg is highly irregular, whereas Hg···N(9$^{\rm i}$) and Hg···O(1$'^{\rm ii}$) are mutually perpendicular to C(1′)–Hg–N(7). Thus we prefer to regard the effective coordination number of mercury as four with use of *sp* hybrids to bond the two

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* Lists of structure factors and mean-plane data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39424 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

close donor atoms, and *p* orbitals (that are perpendicular to each other and the *sp*-hybrid axis) to bind $N(9^{i})$ and $O(1'^{ii})$ weakly.

This work was supported by a grant from the National Health and Medical Research Council.

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Acta Cryst. (1984). C**40**, 1536–1540

Structures of Three Aluminum Alkoxide Complexes having the Formula $[Al(OR)_2(acac)]_n$

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(*Received* 18 *October* 1983; *accepted* 25 *April* 1984)

Abstract. Compound (I): 1,1-bis(acetylacetonato)-bis(μ-trimethylsiloxy)-2,2-bis(trimethylsiloxy)-dialuminum(III), $[Al_2(C_5H_7O_2)_2(C_3H_9OSi)_4]$, $M_r = 608\cdot95$, trigonal, $P3_121$, $a = 11\cdot170\,(9)$, $c = 25\cdot87\,(3)$ Å, $V = 2795$ Å^3, $Z = 3$, $D_x = 1\cdot09$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 2\cdot4$ cm^{-1}, $F(000) = 983\cdot86$, room temperature, $R = 0\cdot0622$ for 1320 unique observed reflections. Compound (II): acetylacetonatobis(triphenylsiloxy)aluminum(III), $[Al(C_5H_7O_2)(C_{18}H_{15}OSi)_2]$, $M_r = 676\cdot9$, monoclinic, $P2_1/c$, $a = 16\cdot05\,(1)$, $b = 8\cdot961\,(5)$, $c = 26\cdot42\,(2)$ Å, $\beta = 104\cdot78\,(5)°$, $V = 3674\cdot4$ Å^3, $Z = 4$, $D_x = 1\cdot22$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot6$ cm^{-1}, $F(000) = 1423\cdot81$, room temperature, $R = 0\cdot0597$ for 2934 unique observed reflections. Compound (III): 1,1,2-tris(acetylacetonato)-1,2;1,2;2,3;2,3-tetra-μ-isopropoxy-3,3-diisopropoxytrialuminum(III), $[Al_3(C_3H_7O)_6(C_5H_7O_2)_3]$, $M_r = 731\cdot9$, monoclinic, $P2_1$, $a = 9\cdot77\,(2)$, $b = 24\cdot81\,(2)$, $c = 9\cdot98\,(1)$ Å, $\beta = 115\cdot7\,(1)°$, $V = 2179$ Å^3, $Z = 2$, $D_x = 1\cdot11$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot33$ cm^{-1}, $F(000) = 791\cdot89$, room temperature, $R = 0\cdot0740$ for 2335 unique observed reflections. (I) is a symmetric molecule containing tetrahedral and octahedral Al sites on a twofold axis. (II) is a distorted tetrahedral monomer. (III) is a linear trimer containing one tetrahedral and two octahedral Al sites. The complexes studied contain varying degrees of steric crowding about the Al atom, and this steric crowding results in distortions of the geometry at each of the Al sites.

Introduction. In previous years, considerable research has been devoted towards understanding the structures of aluminum alkoxide complexes (Bradley, Mehrotra & Gaur, 1978). A unifying theme of this work is the tendency of Al to maximize its coordination number, resulting in associated molecules containing tetrahedral and octahedral metal centers. Although five-coordinate complexes have been postulated (Mehrotra & Mehrotra, 1961), structural evidence to support their

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