

while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1 while a view showing the nearly D_{3h} symmetry of the complex is shown in Fig. 2.

Related literature. A review the chemical and physical properties of related triangular bridged structures has been reported (Cannon & White, 1988).

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The 1/2 Complex of Mercury(II) Trifluoroacetate and Pyridine *N*-Oxide

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Abstract. Bis(pyridine *N*-oxide)bis(trifluoroacetate)mercury(II), $[\text{Hg}(\text{C}_2\text{F}_3\text{O}_2)_2(\text{C}_5\text{H}_5\text{NO})_2]$, $M_r = 616.8$, monoclinic, $C2/c$, $a = 12.52$ (1), $b = 21.33$ (2), $c = 7.62$ (1) Å, $\beta = 107.9$ (1)°, $V = 1936.4$ Å³, $Z = 4$, $D_x = 2.12$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 77.06$ cm⁻¹, $F(000) = 1160.0$, $T = 290$ K, $R = 0.022$ for 887 unique observed reflexions. Distorted octahedral coordination around the Hg atom on a twofold axis with Hg—O 2.167 (6) Å to trifluoroacetate, Hg—O 2.447 (5) Å to pyridine oxide and Hg—O 2.548 (5) Å to a further centrosymmetrically related pyridine oxide. Centrosymmetrically related Hg atoms are thus bridged along *c* by pyridine oxide. The CF₃ group shows disorder, occupying two alternative orientations.

Experimental. Crystals grown from an equimolar solution of mercury(II) trifluoroacetate and pyridine *N*-oxide in dichloromethane were acicular along *c*. Cell dimensions, initially from Weissenberg photographs [$\lambda(\text{Cu } K\alpha) = 1.542$ Å], refined from setting angles of 20 reflexions in the range $10 < 2\theta < 40^\circ$ on STADI-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$). This was also used for measurement of 952 unique intensities from a crystal

$0.2 \times 0.09 \times 0.05$ mm for the layers *hk0* to *hk7*, standard measured every 20 reflexions, < 5% intensity variation, $h - 11$ to 10, $k 0$ to 20, max. $(\sin\theta)/\lambda = 0.55$ Å⁻¹; variable ω scan, $2\theta'$ fixed, stationary background count. Lp correction and absorption correction (point by point integration, max. and min. transmission factors 0.501, 0.271) using the programs of *SHELX76* (Sheldrick, 1976). The same programs were also used in the structure determination involving 887 reflexions for which $I > 3\sigma(I)$. Hg-atom position and confirmation of space group from Patterson maps. Positions of non-H atoms were from ΔF map which indicated disorder of the CF₃ group with occupation of two alternative orientations. During initial least-squares refinement, based on *F*, two sets of F-atom positions with complementary site occupations were refined alternately, the minor set [F(4), F(5), F(6)] with site occupation 0.25 were subsequently fixed in positions with refineable isotropic vibrations. Refinement of: Hg, position and U_{ij} ; C, N, O and F(1), F(2), F(3), positions and U_{iso} ; H, common U_{iso} with calculated positions (C—H = 1.08 Å) gave $R = 0.039$. Refinement of anisotropic U_{ij} for C, N, O and F atoms reduced *R* to 0.022 and significantly improved the e.s.d.'s, the results quoted refer to this refinement. Interlayer scale factors were refined at an intermediate stage. Final $\Delta/\sigma < 0.04$, variations in final *F* map +0.43 to -0.46 e Å⁻³. *R*

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Table 1. Fractional atomic coordinates ($\times 10^4$; Hg $\times 10^5$) and U_{eq} ($\text{\AA}^2 \times 10^3$; Hg $\text{\AA}^2 \times 10^4$)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	U_{eq}
Hg	00000*	2642 (2)	25000*	381 (5)
C(1)	2275 (7)	799 (5)	3191 (12)	48 (9)
C(2)	3528 (10)	760 (6)	3419 (19)	91 (16)
C(3)	294 (9)	1745 (4)	5320 (14)	65 (11)
C(4)	930 (11)	2299 (5)	5908 (17)	83 (15)
C(5)	1949 (13)	2282 (5)	7153 (18)	92 (16)
C(6)	2358 (11)	1720 (6)	7906 (17)	94 (15)
C(7)	1733 (10)	1171 (5)	7381 (15)	70 (13)
N	715 (7)	1204 (3)	6084 (10)	48 (9)
O(1)	1852 (5)	1306 (3)	2934 (9)	67 (7)
O(2)	1818 (5)	274 (3)	3302 (9)	58 (6)
O(3)	101 (5)	687 (2)	5532 (7)	49 (5)
F(1)	3758 (8)	1076 (6)	2096 (18)	160 (19)†
F(2)	4112 (7)	996 (5)	5069 (17)	143 (15)†
F(3)	3911 (7)	188 (4)	3394 (19)	113 (15)†
F(4)	4140*	1240*	3640*	116 (13)‡
F(5)	4010*	300*	4700*	190 (25)‡
F(6)	3710*	420*	2080*	163 (18)‡

* Fixed position.

† Site occupation 0.75.

‡ Site occupation 0.25.

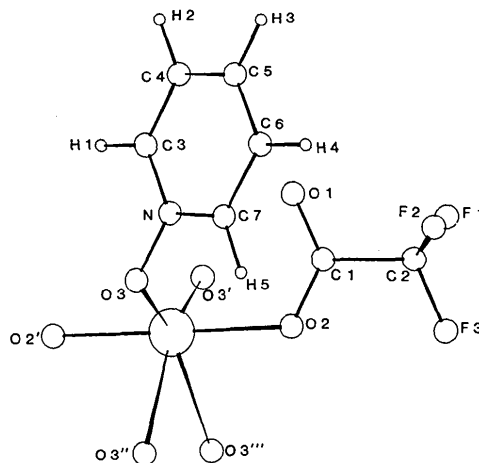


Fig. 1. Unit of structure with atom labelling, coordination around the Hg atom is also shown (symmetry code as in Table 2).

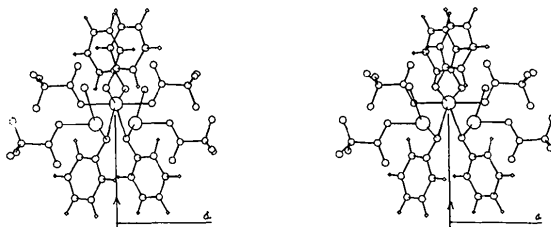


Fig. 2. Projection of part of the structure onto (001). * Twofold axis at (0, y, 0.25) is shown.

Table 2. Distances (\AA) and angles ($^\circ$) around the Hg atom

Hg—O(2)	2.167 (6)		
Hg—O(3)	2.447 (5)		
Hg—O(3')	2.548 (5)		
O(2)—Hg—O(2)'	178.9 (3)	O(3)—Hg—O(3')	136.8 (3)
O(2)—Hg—O(3)	89.2 (3)	O(3)—Hg—O(3'')	74.6 (3)
O(2)—Hg—O(3')	90.4 (3)	O(3)—Hg—O(3''')	148.4 (3)
O(2)—Hg—O(3'')	94.6 (3)	O(3'')—Hg—O(3''')	74.5 (3)
O(2)—Hg—O(3''')	86.3 (3)		

Symmetry codes: (') $-x, y, \frac{1}{2}-z$; (') $-x, -y, 1-z$; (''') $x, -y, z-\frac{1}{2}$.

$= 0.022$, $wR = 0.029$, $S = 1.79$, 143 parameters refined, $Q = 6.20$, $w = 2.322/[\sigma^2(F) + 0.000158F^2]$.

Atomic scattering factors and mass absorption coefficient for Hg atoms from Cromer & Mann (1968) and Cromer & Liberman (1970), other atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters in Table 1,* bond distances and angles around the Hg atom in Table 2. The unit of structure with atomic labelling and the Hg-atom coordination is shown in Fig. 1, a projection of part of the structure is shown in Fig. 2.

Related literature. Three structures of Hg^{II} complexes with pyridine oxide have been reported. Tris(mercury(II) thiocyanate) bis(pyridine N-oxide) (Grdenic, Kamenar, Sikirica & Vernic, 1976), (pyridine N-

* Lists of structure factors, H-atom coordinates, bond lengths and angles not involving the Hg atom and anisotropic vibrational factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53644 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

oxide)dichloromercury (Sawitzki & von Schnering, 1974), hexakis(pyridine 1-oxide)mercury(II) perchlorate (Kepert, Taylor & White, 1973). Two complexes of mercury(II) trifluoroacetate with pyridine have been reported (Halfpenny, Small & Thorpe, 1978; Halfpenny & Small, 1978). One other complex of mercury(II) trifluoroacetate and an O donor ligand, 1,4-dioxane, has been described (Small, 1982). A preliminary account of disorder involving the trifluoroacetate group has been given (Small, 1987).

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Bis(acetato)(4-benzyl-1,7-diphenyl-2,4,6-triazahepta-2,5-diene- $\kappa^2 N^2, N^6$)copper(II)

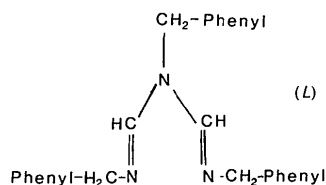
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Abstract. $[\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{23}\text{H}_{23}\text{N}_3)]$, $M_r = 523.09$, monoclinic, $P2_1/c$, $a = 11.03$ (1), $b = 10.29$ (1), $c = 25.03$ (2) Å, $\beta = 116.8$ (1)°, $V = 2535.7$ Å³, $Z = 4$, $D_x = 1.370$ g cm⁻³, $F(000) = 1092$, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 8.94$ cm⁻¹, $T = 290$ K, $R = 0.048$ for 1305 unique observed reflexions. Coordination around Cu is approximately square planar. Cu is in the plane of the bidentate amidine group with Cu—N 1.991 (7) and 1.991 (9) Å; O atoms of each acetate group are respectively 0.512 (9) and 0.925 (8) Å above and below this plane, with Cu—O 1.972 (7) and 1.960 (6) Å. Cu—O contacts to the other O atoms of the acetate groups are 2.531 (8) and 2.483 (7) Å.

Experimental. The ligand (*L*) 4-benzyl-1,7-diphenyl-2,4,6-triazahepta-2,5-diene was prepared by the reaction of ethyl orthoformate and benzylamine on *N,N'*-dibenzylformamidine.* By reacting the ligand (*L*) with copper(II) acetate in methanol, the title



compound was obtained as bright-blue needles elongated parallel to **b**. Cell dimensions, initially from Weissenberg photographs [$\lambda(\text{Cu } K\alpha) = 1.542$ Å], refined from setting angles of 20 reflexions in the range $10 < 2\theta < 40^\circ$ on STADI-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$). This was also used for measurement of 3297 intensities (2926 unique, $R_{\text{int}} = 0.022$) from a crystal $0.06 \times 0.08 \times 0.30$ mm for the layers $h0l$ to $h10l$, standard measured every 20 reflexions (overall decrease $< 10\%$, allowed for by linear interpolation), $h - 10$ to 9, $k 0$ to 10, $l 0$ to 24, max. $(\sin\theta)/\lambda = 0.50$ Å⁻¹; variable ω

* Full details of the preparation of the ligand (*L*) and lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53614 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

scan, $2\theta'$ fixed, stationary background count. Lp but not absorption correction made. 1306 reflexions for which $I > 3\sigma(I)$ were used in the structure determination. Cu-atom position obtained from Patterson maps, other non-H positions from subsequent ΔF maps. Refinement, using F , of non-H-atom positions (the C atoms in the phenyl groups being constrained as rigid groups with C—C = 1.395 Å) and U_{ij} . H atoms in calculated positions (C—H = 1.08 Å) with common isotropic U refined in CH, CH₂, CH₃ and phenyl groups respectively. At an intermediate stage interlayer scale factors were refined. 299 parameters refined in two blocks alternately until $\Delta/\sigma < 0.05$, and largest features on ΔF map were $+0.29$ to -0.29 e Å⁻³, $R = 0.048$, $wR = 0.043$, $S = 1.318$, $Q = 4.36$ and $w = 1.273/[\sigma^2(F) + 0.000131F^2]$. SHELX76 (Sheldrick, 1976) programs were used throughout.

Table 1. Fractional atomic coordinates ($\times 10^4$; Cu $\times 10^5$) and U_{eq} (Å² $\times 10^3$; Cu $\times 10^4$) values

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Cu	15910 (11)	24418 (14)	55527 (5)	379 (10)
C(1)	1382 (9)	4461 (87)	4658 (4)	46 (10)
N(1)	623 (8)	3427 (7)	4791 (3)	37 (9)
C(2)	-635 (10)	3397 (10)	4444 (4)	42 (12)
N(2)	-1531 (8)	2553 (10)	4492 (3)	47 (8)
C(3)	-1248 (10)	1661 (10)	4939 (4)	43 (12)
N(3)	-119 (8)	1503 (7)	5391 (3)	40 (9)
C(4)	-73 (9)	443 (8)	5810 (4)	46 (10)
C(5)	-2993 (8)	2739 (11)	4053 (3)	50 (10)
C(25)	5133 (9)	1673 (11)	5511 (5)	86 (14)
C(24)	3725 (12)	1709 (13)	5492 (5)	56 (14)
O(1)	3403 (6)	2836 (6)	5619 (3)	47 (7)
O(2)	3032 (7)	731 (7)	5384 (3)	68 (9)
C(27)	2576 (9)	3288 (9)	7272 (3)	54 (10)
C(26)	2181 (10)	3203 (11)	6614 (4)	42 (12)
O(3)	2352 (7)	2096 (6)	6413 (3)	41 (7)
O(4)	1687 (7)	4135 (6)	6278 (3)	63 (8)
C(7)	3237 (7)	4733 (6)	4376 (3)	52 (11)
C(8)	3973 (7)	4373 (6)	4069 (3)	66 (15)
C(9)	3630 (7)	3244 (6)	3724 (3)	78 (15)
C(10)	2551 (7)	2476 (6)	3684 (3)	79 (13)
C(11)	1815 (7)	2836 (6)	3991 (3)	58 (13)
C(6)	2158 (7)	3965 (6)	4337 (3)	39 (10)
C(13)	-3584 (7)	3178 (5)	2988 (3)	59 (12)
C(14)	-3941 (7)	2775 (5)	2403 (3)	71 (15)
C(15)	-4024 (7)	1453 (5)	2269 (3)	67 (14)
C(16)	-3750 (7)	533 (5)	2718 (3)	68 (14)
C(17)	-3393 (7)	936 (5)	3302 (3)	56 (12)
C(12)	-3310 (7)	2259 (5)	3437 (3)	43 (11)
C(19)	-1097 (7)	2086 (7)	6265 (3)	56 (13)
C(20)	-1350 (7)	2467 (7)	6740 (3)	69 (12)
C(21)	-825 (7)	1742 (7)	7268 (3)	79 (16)
C(22)	-47 (7)	637 (7)	7321 (3)	73 (16)
C(23)	207 (7)	257 (7)	6845 (3)	60 (12)
C(18)	-318 (7)	981 (7)	6318 (3)	40 (10)