distorted polyhedron at the transition metal and a tetrahedrally distorted polyhedron at the main-group metal atom. The Re(3) central atom coordinated with six ligands (four CO and two μ-I) fulfills the Rn closed-shell electron configuration and the In atom with four ligands [two univalent $Re(CO)_5$ and two μ-I] the Xe closed-shell electron configuration in accordance with the diamagnetism of the compound. The unsymmetrical four-membered ring is folded with a dihedral angle of 23·7° between the planes of $Re(3)I_2$ and InI_2, whereas such rings in compounds of the types $InI_4(\mu\text{-}I)_2$ (Forrester & Zalkin, 1964), $In_2[M'(CO)_5]_4(\mu\text{-}I)_2$ (M = Mn, Re) or $Re_2(CO)_8(\mu\text{-}I)_2$ (Darst, Lenhert, Lukehardt & Warfield, 1980) have planar or nearly planar shapes. It seems therefore possible that the change results from molecular packing requirements, because the shortest non-bonding O···O contact lengths are intermolecular [≤ 2·84 (2) Å]. In the sequence from $In_2[Re(CO)_5]_4(\mu\text{-}I)_2$ to the present compound it is recognizable that the subtended terminal bond angle at the In atom with the two univalent $Re(CO)_5$ groups is enlarged from 126·3 (1) to 133·5 (1)° and the ring bond angle subtended at the μ-I atom (or In atom) diminishes by about 2·4 (1)° [or 3·7 (1)°].

Intramolecular distances in both substances do not indicate interactions greater than van der Waals forces.

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5-η-Benzene-9-hydroxy-9,10-μ-triethylphosphinoaurio-5-ruthena-*nido*-decaborane(11) (Dichloromethane Solvate), $C_{12}H_{33}AuB_9OPRu$ (0·15CH_2Cl_2)

By Alistair J. McLennan and Alan J. Welch

Department of Chemistry, University of Edinburgh, Edinburgh EH9 3JJ, Scotland

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Abstract. (Crystallographic data assuming no solvate) M_r = 620·7, triclinic, $P\bar{1}$, a = 9·268 (3), b = 11·084 (5), c = 11·788 (3) Å, α = 87·87 (3), β = 86·803 (25), γ = 76·92 (3)°, V = 1177·3 (19) Å^3, Z = 2, D_x = 1·751 Mg m^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 6·91 mm^{-1}, $F(000)$ = 594, T = 291 K, R = 0·0578 for 3611 independent observed reflections. An r.m.s. misfit calculation shows that the cage of the title compound has a *nido* decaboranyl architecture. There is some evidence for a weak interaction between the 9,10-bridging Au atom and the 3c-2e Ru(5)—H(5,6)—B(6) unit.

Introduction. The reaction between $B_{10}H_{14}$ and $MeAuPR_3$ affords either the unique 'triple cluster' $[(B_{10}H_{12}Au)(AuPR_3)_4(AuB_{10}H_{12})]$ (1) [(1*a*) R = Et, (1*b*) R = Ph] (Wynd, Robins, Welch & Welch, 1985; Wynd, Welch & Parish, 1989), or the simple isolobally-substituted complex $[\mu\text{-}5,6\text{-}AuPR_3\text{-}B_{10}H_{13}]$ (2) [(2*a*) R = Cy, (2*b*) R = *o*-tol] (Wynd, McLennan, Reed & Welch, 1987). We are attempting to expand this chemistry to metal-containing analogues of $B_{10}H_{14}$. Several such compounds are known (Kennedy, 1986) including all four possible isomers of $[(\eta\text{-}C_6Me_6)RuB_9H_{13}]$ (Bown, Greenwood & Kennedy, 1986). Starting from a 5- or 6-metalla-B_9H_{13} species, reaction with $MeAuPR_3$ allows the possibility of the synthesis of clusters with heteronuclear metal—metal bonds. To minimize the possibility of steric control of the reaction we have synthesized $[5\text{-}(\eta\text{-}C_6H_6)\text{-}5\text{-}RuB_9H_{13}]$ (3) by an analogous route to that which afforded the appropriate C_6Me_6 species,

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and reacted the former with $MeAuPEt_3$. The results of crystallographic analysis of the product isolated from this reaction are presented here.

Experimental. [5-(η-C_6H_6)-5-RuB_9H_{13}] isolated (*via* preparative TLC, SiO_2 plates, CH_2Cl_2 eluant, R_f0·8) in *ca* 5% yield from a 1 mmol (based on Ru) reaction between [(η-C_6H_6)$RuCl_2$]$_2$ and freshly prepared [$HNEt_3$][$B_{10}H_{13}$] in CH_2Cl_2, as a yellow crystalline solid. NMR ($CDCl_3$, 291 K) $^{11}B(^1H)$ δ 31·17, 19·02, 9·10, 5·63 (2B, coincidental), 0·21, −8·28, −18·75 and −38·15. ^1H δ 5·98 (C_6H_6). Reaction with $MeAuPEt_3$ in CH_2Cl_2 over five days resulted in considerable darkening of the initially yellow solution. Red prismatic crystals were grown at 243 K. A single crystal, 0·03 × 0·03 × 0·02 cm, mounted on a glass fibre, was set on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$ X-radiation, graphite monochromator); cell parameters and orientation matrix from least-squares refinement ($13 < \theta \leq 14°$) of 25 centred reflections; data collection by ω–2θ scans in 96 steps with ω scan width ($0{\cdot}8 + 0{\cdot}34\tan\theta$)°; data ($h$:0 to 11, k: −13 to 13, l: −14 to 14) measured for $1 \leq \theta \leq 25°$ over 136 X-ray hours; slight crystal decay (possibly loss of solvate) for which correction made; corrections for Lorentz and polarization effects (Gould & Smith, 1986), and, following isotropic convergence, for absorption (Walker & Stuart, 1983); 5993 independent reflections measured, 3611 [$F \geq 2{\cdot}0\sigma(F)$] retained; structure solution *via* automatic direct methods (Sheldrick, 1986) (Au, Ru, P), and iterative full-matrix least-squares refinement (on F)/ΔF syntheses (Sheldrick, 1976) (all other atoms); weights assigned according to $w^{-1} = [\sigma^2(F) + 0{\cdot}003095F^2]$; PEt_3 ligand showed partial disorder, the best model of which involved disorder of two methylene C atoms [C(11) and C(31)] over two sites each, with optimized occupations 0·53 (3) [sites (A)] and 0·47 (3) [sites (B)]. In addition, significant electron density located *ca* 1·5 Å from (1,$\frac{1}{2}$,0), interpreted as the Cl atom of partial (0·3 molecules per unit cell, 0·15 molecules per molecule of auraruthenaborane based on peak heights in trial ΔF syntheses) CH_2Cl_2 of solvation; C_6H_6 ring idealized to regular, planar hexagon (C—C 1·395 Å), with coplanar H atoms bisecting the external C—C—C angle (C—H 1·08 Å); all atoms refined with individual anisotropic thermal parameters except ethyl C and solvate Cl (individual isotropic) and arene H [group isotropic, 0·103 (18) Å^2 at convergence]; ethyl-H, cage-H, hydroxy-H, solvate-C and solvate-H atoms not located; 193 variables, data:variable ratio > 18:1; max. shift/e.s.d. in final cycle < 0·07; $R = 0{\cdot}0578$, $wR = 0{\cdot}0810$, $S = 1{\cdot}124$; max. and min. residues in final ΔF synthesis 2·1 (near partial Cl) and −1·3 e Å^{-3}; scattering factors for C, H, B, P, O and Cl inlaid in *SHELX*76. Those for Ru and Au from *International Tables for X-ray Crystallography* (1974); figure drawn using *EASYORTEP* (Mallinson, 1982); all calculations on Amdahl 470 V/8 computer.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

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

	x	y	z	U_{eq}(Å^2)
Au	0·45305 (4)	0·04395 (4)	0·28345 (4)	0·0578 (3)
P	0·3155 (4)	−0·0786 (3)	0·2088 (3)	0·0677 (18)
C(1)	0·2121 (11)	0·4453 (6)	0·4728 (9)	0·086 (9)
C(2)	0·1235	0·4068	0·3960	0·090 (10)
C(3)	0·1146	0·2828	0·3956	0·100 (12)
C(4)	0·1944	0·1974	0·4719	0·095 (11)
C(5)	0·2831	0·2359	0·5486	0·088 (10)
C(6)	0·2919	0·3599	0·5490	0·085 (9)
B(1)	0·5877 (12)	0·2984 (11)	0·4071 (11)	0·056 (7)
B(2)	0·5010 (14)	0·4185 (11)	0·3015 (11)	0·060 (7)
B(3)	0·6917 (16)	0·3414 (15)	0·2836 (12)	0·075 (9)
B(4)	0·7465 (13)	0·1939 (15)	0·3421 (12)	0·073 (9)
Ru(5)	0·35462 (8)	0·29393 (7)	0·37528 (7)	0·0489 (5)
B(6)	0·3857 (18)	0·3694 (11)	0·1871 (16)	0·090 (10)
B(7)	0·5834 (18)	0·3713 (15)	0·1671 (11)	0·078 (10)
B(8)	0·7357 (16)	0·2188 (15)	0·1961 (14)	0·076 (9)
B(9)	0·6856 (14)	0·0786 (14)	0·2701 (13)	0·069 (8)
B(10)	0·5786 (12)	0·1433 (10)	0·3923 (10)	0·052 (6)
C(11A)	0·145 (3)	0·0135 (23)	0·1335 (22)	0·072 (8)
C(11B)	0·225 (5)	−0·042 (4)	0·079 (4)	0·112 (14)
C(12)	0·174 (3)	0·1065 (24)	0·0559 (22)	0·154 (8)
C(21)	0·200 (3)	−0·1451 (24)	0·3282 (22)	0·152 (8)
C(22)	0·110 (3)	−0·222 (3)	0·277 (3)	0·170 (9)
C(31A)	0·411 (3)	−0·177 (3)	0·096 (3)	0·091 (10)
C(31B)	0·402 (6)	−0·254 (5)	0·181 (5)	0·150 (20)
C(32)	0·5590 (25)	−0·2589 (21)	0·1334 (20)	0·133 (7)
Cl	0·9527 (20)	0·5708 (18)	0·1068 (16)	0·132 (5)
O	0·8066 (13)	−0·0446 (11)	0·2295 (11)	0·111 (3)

Discussion. Table 1* lists the coordinates of refined atoms and equivalent isotropic thermal parameters, whilst Table 2 details the internuclear distances and interbond angles determined. A perspective view of a single molecule is shown in Fig. 1.

The compound, which crystallizes with no important contacts between adjacent molecules, is shown by this study to be [5-(η-C_6H_6)-9-OH-μ-9,10-$AuPEt_3$-5-RuB_9H_{11}] (4), derived from (3) by isolobal replacement of μ-9,10-H by μ-9,10-$AuPEt_3$ and substitution of the H atom terminal to B(9) by OH. Although no H atoms were located, we are confident that, except for B(9), each B atom carries a terminal H atom. Bridging H atoms are probably located across the B(8)—B(9), Ru(5)—B(6) and B(6)—B(7) bonds. Evidence for the number and placement of

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* Lists of structure factors, phenyl H-atom positions and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52005 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Internuclear distances* (Å) *and interbond angles* (°) *in* [5-(η-C_6H_6)-9-OH-μ-9,10-$AuPEt_3$-5-RuB_9H_{11}]

Au—P	2·290 (3)	B(2)—Ru(5)	2·265 (13)
Au—Ru(5)	2·9411 (9)	B(2)—B(6)	1·932 (21)
Au—B(9)	2·270 (15)	B(2)—B(7)	1·765 (20)
Au—B(10)	2·251 (12)	B(3)—B(4)	1·728 (21)
P—C(11*A*)	1·92 (3)	B(3)—B(7)	1·725 (22)
P—C(11*B*)	1·78 (4)	B(3)—B(8)	1·698 (22)
P—C(21)	1·94 (3)	B(4)—B(8)	1·737 (21)
P—C(31*A*)	1·82 (3)	B(4)—B(9)	1·769 (21)
P—C(31*B*)	1·96 (6)	B(4)—B(10)	1·832 (19)
C(1)—Ru(5)	2·202 (9)	Ru(5)—B(6)	2·367 (16)
C(2)—Ru(5)	2·228 (9)	Ru(5)—B(10)	2·364 (12)
C(3)—Ru(5)	2·255 (9)	B(6)—B(7)	1·839 (23)
C(4)—Ru(5)	2·255 (9)	B(7)—B(8)	1·974 (22)
C(5)—Ru(5)	2·230 (9)	B(8)—B(9)	1·890 (21)
C(6)—Ru(5)	2·203 (9)	B(9)—B(10)	1·780 (19)
B(1)—B(2)	1·863 (17)	B(9)—O	1·628 (19)
B(1)—B(3)	1·809 (19)	C(11*A*)—C(12)	1·41 (4)
B(1)—B(4)	1·807 (19)	C(11*B*)—C(12)	1·62 (5)
B(1)—Ru(5)	2·225 (12)	C(21)—C(22)	1·48 (4)
B(1)—B(10)	1·756 (17)	C(31*A*)—C(32)	1·54 (4)
B(2)—B(3)	1·785 (20)	C(31*B*)—C(32)	1·50 (6)

P—Au—B(9)	140·4 (4)	C(3)—Ru(5)—C(4)	36·0 (3)
P—Au—B(10)	167·6 (3)	C(3)—Ru(5)—B(1)	164·2 (4)
B(9)—Au—B(10)	46·4 (5)	C(3)—Ru(5)—B(2)	141·3 (4)
Au—P—C(11*A*)	113·5 (8)	C(3)—Ru(5)—B(6)	105·3 (5)
Au—P—C(11*B*)	123·2 (14)	C(3)—Ru(5)—B(10)	132·4 (4)
Au—P—C(21)	110·7 (8)	C(4)—Ru(5)—C(5)	36·2 (3)
Au—P—C(31*A*)	114·5 (10)	C(4)—Ru(5)—B(1)	130·8 (4)
Au—P—C(31*B*)	120·6 (17)	C(4)—Ru(5)—B(2)	169·5 (4)
C(11*A*)—P—C(21)	94·4 (11)	C(4)—Ru(5)—B(6)	136·5 (5)
C(11*A*)—P—C(31*A*)	101·3 (12)	C(4)—Ru(5)—B(10)	100·5 (4)
C(11*B*)—P—C(21)	114·3 (16)	C(5)—Ru(5)—C(6)	36·7 (3)
C(11*B*)—P—C(31*B*)	97·7 (22)	C(5)—Ru(5)—B(1)	99·2 (4)
C(21)—P—C(31*A*)	120·1 (12)	C(5)—Ru(5)—B(2)	136·4 (4)
C(21)—P—C(31*B*)	83·2 (18)	C(5)—Ru(5)—B(6)	170·0 (5)
B(2)—B(1)—B(3)	58·1 (7)	C(5)—Ru(5)—B(10)	88·3 (4)
B(2)—B(1)—Ru(5)	66·6 (5)	C(6)—Ru(5)—B(1)	88·3 (4)
B(3)—B(1)—B(4)	57·1 (8)	C(6)—Ru(5)—B(2)	104·5 (4)
B(4)—B(1)—B(10)	61·9 (7)	C(6)—Ru(5)—B(6)	141·0 (5)
Ru(5)—B(1)—B(10)	71·8 (6)	C(6)—Ru(5)—B(10)	104·9 (4)
B(1)—B(2)—B(3)	59·4 (7)	B(1)—Ru(5)—B(2)	49·0 (4)
B(1)—B(2)—Ru(5)	64·4 (5)	B(1)—Ru(5)—B(10)	44·9 (4)
B(3)—B(2)—B(7)	58·1 (8)	B(2)—Ru(5)—B(6)	49·3 (5)
Ru(5)—B(2)—B(6)	68·1 (6)	B(2)—B(6)—Ru(5)	62·6 (6)
B(6)—B(2)—B(7)	59·4 (8)	B(2)—B(6)—B(7)	55·7 (8)
B(1)—B(3)—B(2)	62·4 (7)	B(2)—B(7)—B(3)	61·5 (8)
B(1)—B(3)—B(4)	61·4 (8)	B(2)—B(7)—B(6)	64·8 (8)
B(2)—B(3)—B(7)	60·3 (8)	B(3)—B(7)—B(8)	54·1 (8)
B(4)—B(3)—B(8)	60·9 (9)	B(3)—B(8)—B(4)	60·4 (9)
B(7)—B(3)—B(8)	70·4 (9)	B(3)—B(8)—B(7)	55·4 (8)
B(1)—B(4)—B(3)	61·5 (8)	B(4)—B(8)—B(9)	58·2 (8)
B(1)—B(4)—B(10)	57·7 (7)	Au—B(9)—B(10)	66·3 (6)
B(3)—B(4)—B(8)	58·7 (9)	B(4)—B(9)—B(8)	56·6 (8)
B(8)—B(4)—B(9)	65·2 (9)	B(4)—B(9)—B(10)	62·1 (8)
B(9)—B(4)—B(10)	59·2 (8)	B(4)—B(9)—O	119·0 (11)
C(1)—Ru(5)—C(2)	36·7 (3)	B(8)—B(9)—O	108·6 (10)
C(1)—Ru(5)—C(6)	36·9 (3)	B(10)—B(9)—O	141·4 (11)
C(1)—Ru(5)—B(1)	106·6 (4)	Au—B(10)—B(9)	67·4 (6)
C(1)—Ru(5)—B(2)	92·5 (4)	B(1)—B(10)—B(4)	60·5 (7)
C(1)—Ru(5)—B(6)	107·7 (5)	B(1)—B(10)—Ru(5)	63·4 (5)
C(1)—Ru(5)—B(10)	139·6 (4)	B(4)—B(10)—B(9)	58·6 (7)
C(2)—Ru(5)—C(3)	36·3 (3)	P—C(11*A*)—C(12)	114·6 (18)
C(2)—Ru(5)—B(1)	141·6 (4)	P—C(11*B*)—C(12)	111·8 (26)
C(2)—Ru(5)—B(2)	108·3 (4)	P—C(21)—C(22)	109·6 (18)
C(2)—Ru(5)—B(6)	92·9 (5)	P—C(31A)—C(32)	111·9 (19)
C(2)—Ru(5)—B(10)	165·1 (4)	P—C(31B)—C(32)	105·4 (31)

these μ-H atoms derives from the results of an 'r.m.s. misfit' (Gould & Taylor, 1986; Wynd *et al.*, 1987, 1989) calculation between the RuB_9 framework of (4) and that of [5-(η-C_6Me_6)-6-OMe-5-RuB_9H_{12}] in which the μ-H atoms were directly located (Bown, Fontaine, Greenwood, Kennedy & Thornton-Pett, 1988). In this calculation the r.m.s. misfit converged at 0·088 Å, indicating that the basic cage structures of the two complexes are the same, *i.e.* 5-ruthenadecaboranyl. Individually, the poorest misfit (0·198 Å) is for B(6), which in the title compound is ill-defined (note the unusual orientation of its thermal ellipsoid in Fig. 1).

The Au—Ru distance in (4) is 2·9411 (9) Å. Although this is probably too long to indicate a substantial metal—metal bond, there are structural indications of a weak link between the two metal atoms. Thus the Σ(angles at Au) in (4) is only 354·4°, *cf.* 359·7° in (2*a*) (Wynd *et al.*, 1987) and 359·4° in (2*b*) (Wynd *et al.*, 1989), and the difference in P—Au—B angles, P—Au—B(9) 140·4 (4), P—Au—B(10) 167·6 (3)°, clearly shows that Au is displaced out of the PB(9)B(10) plane towards B(6)/Ru(5). These data imply the existence of a weak bonding interaction between the 3c-2e Ru(5)—H(5,6)—B(6) bridge and a vacant 6*p* orbital on Au, similar to that previously identified in (2*a*) (Wynd *et al.*, 1989).

Two mechanistic implications arise from this structural study. Firstly, substitution of the [BH(5)] unit of $B_{10}H_{14}$ by [Ru(C_6H_6)] renders the μ-H atoms sufficiently distinct so that one (μ-9,10-H), presumably the most acidic, is preferentially substituted by an [AuPR_3] fragment. Secondly, the reaction of [5-(η-C_6H_6)-5-RuB_9H_{13}] with $MeAuPEt_3$ stops at the single μ-$AuPEt_3$ for μ-H product, unlike the equiv-

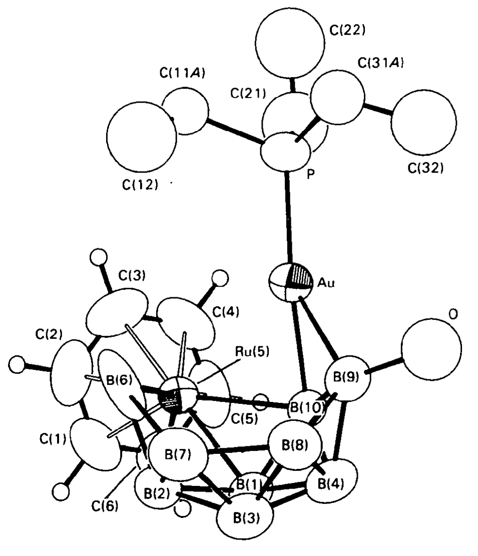

Fig. 1. Perspective view of [5-(η-C_6H_6)-9-OH-μ-9,10-$AuPEt_3$-5-RuB_9H_{11}] (50% thermal ellipsoids, except for H atoms which have an artificial radius of 0·1 Å for clarity). Only the major components of the disordered methylene C atoms are shown.

alent reaction between $B_{10}H_{14}$ and $MeAuPEt_3$, which affords the triethylphosphine triple cluster as the only isolable species, presumably *via* a multistage reaction. These points must remain conjectural, however, because of the (unexpected) finding of the OH function terminal to B(9). Since we cannot say whether hydrolysis occurred during reaction or work-up we clearly cannot be totally confident of the precise nature of the reacting species. Nevertheless, we are prompted by the findings reported herein to repeat and extend these synthetic and isolation procedures under strictly anhydrous conditions, and to study the reactions of a wider range of metalla-substituted *nido* and *arachno* boranes with $MeAuPR_3$. The results of these studies will be reported in future publications.

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Neutron Diffraction Study of Sodium Oxalate Monoperhydrate at 123 K*

BY BERIT FJÆRTOFT PEDERSEN†

Institute of Pharmacy, University of Oslo, PO Box 1068-*Blindern*, 0316 *Oslo* 3, *Norway*

AND ÅKE KVICK

Chemistry Department, Brookhaven National Laboratory, Upton, New York 11973, *USA*

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Abstract. $Na_2C_2O_4.H_2O_2$, $M_r = 168{\cdot}02$, monoclinic, $P2_1/c$, $a = 3{\cdot}521\,(2)$, $b = 8{\cdot}206\,(3)$, $c = 8{\cdot}950\,(3)$ Å, $\beta = 96{\cdot}63\,(3)°$, $V = 257{\cdot}0\,(5)$ Å^3, $Z = 2$, $D_x = 2{\cdot}17$, $D_m = 2{\cdot}09$ Mg m^{-3}, $\lambda = 1{\cdot}0559\,(2)$ Å, $\mu = 0{\cdot}0413$ mm^{-1}, $F(000) = 168$, $T = 123{\cdot}0\,(2)$ K. $R = 0{\cdot}0351$ for 2873 observations representing two sets of equivalent reflections. The hydrogen peroxide molecule is planar, *i.e.* the H—O—O—H dihedral angle is 180·0°; O—O = 1·467 (1), O—H = 1·0094 (9) Å, and O—O—H = 99·95 (8)°. The oxalate ion is centrosymmetric and planar, the two C—O distances are 1·2671 (6) and 1·2445 (7) Å, differing by 0·0226 Å, the longer one being the acceptor of the hydrogen bond. The C—C distance is especially long, 1·564 (1) Å.

Introduction. The structure of the title compound, $Na_2C_2O_4.H_2O_2$, was determined from X-ray film data by Pedersen & Pedersen (1964). The conformation of the H_2O_2 molecule was also studied by NMR spectroscopy. Both space-group symmetry and NMR spectra led to the conclusion that the dihedral angle of the H_2O_2 molecule was 180·0°, *i.e.* a planar conformation of the molecule was observed for the first time.

Several compounds containing H_2O_2 molecules have been studied (Pedersen, 1969, 1972; Adams &

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† Also affiliated to Department of Chemistry, University of Oslo.

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