Crystal Structure of a Dinuclear Dinitrogen Complex: Tetrachloro-{chlorotetrakis[dimethyl(phenyl)phosphine]rhenium(I)}-μ-dinitrogen-methoxymolybdenum(V)–Methanol–Hydrochloric Acid

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The crystal structure of the title complex has been determined from three-dimensional *X*-ray data. The N–N bond length is 1·18, with Mo–N 1·90, and Re–N 1·82 Å. These distances are consistent with a linear Re–N–N–Mo delocalised bonding scheme.

The rhenium atom is octahedrally co-ordinated to four phosphorus atoms (Re–P 2·48 Å), and a chlorine atom, *trans* to nitrogen (Re–Cl 2·42 Å). The molybdenum atom is octahedrally co-ordinated to four chlorine atoms (mean Mo–Cl 2·39 Å) and a methoxy-group *trans* to N (Mo–O 2·15 Å). The planes of phosphorus and chlorine atoms are parallel, and the atoms are in eclipsed positions.

Crystals are monoclinic, space group $P2_1/c$, with $a = 14{\cdot}918(1)$, $b = 11{\cdot}176(2)$, $c = 28{\cdot}162(3)$ Å, $\beta = 92{\cdot}6(1)°$, $Z = 4$. The structure was solved by the heavy-atom method by use of 3589 independent reflections, measured on a diffractometer, and refined by block-diagonal least-squares to R 0·059.

PREVIOUSLY reported crystal structures[1-5] containing the dinitrogen molecule have all had the N–N bond length within 0·03 Å of that in molecular nitrogen (1·1 Å).[6] Some asymmetrically bridged dinitrogen compounds, thought to contain the linear grouping M · · · N · · · N · · · Re (M = Nb, Mo, or Cr)[7,8] have exceptionally low $\nu(N_2)$,[9] down to 1630 cm^{-1}. A determination of the crystal structure of $[(PMe_2Ph)_4ClRe\text{-}N_2MoCl_4(OMe)]$ has confirmed the existence of an almost linear bridge system Re–N–N–Mo. The dimensions of this bridge are of interest in the understanding of the bonding of molecular nitrogen in transition-metal complexes. A preliminary account of this work has been published.[10]

EXPERIMENTAL

The compound was obtained according to reaction (1).

$$[ReCl(N_2)(PMe_2Ph)_4]\ (0{\cdot}22\ g) + [MoCl_4(thf)_2]\ (0{\cdot}21\ g) \xrightarrow[MeOH]{CH_2Cl_2} [(PMe_2Ph)_4ClReN_2MoCl_4(OMe)] \quad (1)$$

Dry light petroleum (b.p. 60—80 °C) was then added, and the solution filtered after 24 h to remove a dark oily precipitate. After setting aside the solution for 12 days, dark purple crystals were deposited (Found: C, 36·05; H, 4·7; Mo, 9·2; N, 2·45. Calc. for $C_{33}H_{47}Cl_5MoN_2OP_4Re$: C, 36·95; H, 4·2; Mo, 8·95; N, 2·60%).

Crystal Data.—$C_{33}H_{47}Cl_5MoN_2OP_4Re,CH_3OH,HCl$, M = 1140·9, Monoclinic, $a = 14{\cdot}918(1)$, $b = 11{\cdot}176(2)$, $c = 28{\cdot}162(3)$ Å, $\beta = 92{\cdot}6(1)°$, $U = 4690{\cdot}43$ Å^3, $Z = 4$, $D_c = 1{\cdot}614$. Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å; $\mu(\text{Mo-}K_\alpha) = 33{\cdot}5$ Space group $P2_1/c$ (C_{2h}^5).

Measurement of Intensities.—Preliminary unit-cell data were obtained from Weissenberg and precession photographs, and the space group uniquely determined. Lattice constants were determined from a least-squares refinement of the setting angles of 12 reflections which had been automatically centred on a Hilger and Watts four-circle diffractometer (Mo-K_α radiation). Data were collected for a crystal of dimensions 0·30 × 0·03 × 0·02 mm. Corrections were made for Lorentz and polarisation effects. No correction for absorption was made.

Intensity measurements (zirconium-filtered molybdenum radiation) were based on an ω—2θ scan with background measurements being taken at the end of each scan. Each background count was taken over 20 s and each peak-count was accumulated over 70 steps of 0·01° with 1 s counts at each step.

Measurements were made for all the independent reflections from $2° \leqslant \theta \leqslant 20°$, and resulted in 3589 reflections with $I \geqslant 3\sigma(I)$. No systematic variations in the intensities of three standard reflections, (300), (008), (334), measured after every 100 reflections, were observed.

Solution and Refinement of the Structure.—The structure was solved by Patterson and heavy-atom Fourier methods. The Patterson map enabled location of rhenium and molybdenum atoms. The first Fourier map revealed four of the five chlorine and three phosphorus atom positions. Two subsequent difference maps revealed all the non-hydrogen atom positions. Refinement by block-diagonal least-squares methods with unit weights and scattering factors taken from ref. 11 for non-hydrogen atoms gave R 0·08. All hydrogen atom positions, except those of the methoxy-group, were apparent on a difference map. They were included in the final refinement, with scattering factors from ref. 12, but not refined. Anisotropic vibration parameters were given to all non-carbon atoms, and isotropic vibration parameters to carbon and hydrogen. An analysis of $\Delta = |(|F_o| - |F_c|)|$ *vs.* F_o and $\sin\theta/\lambda$ caused the final weighting scheme to be adjusted as follows:

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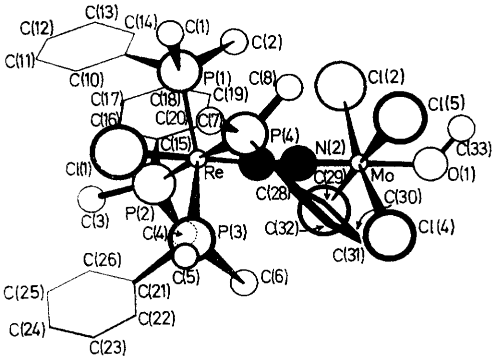

FIGURE 1 A perspective view of one molecule

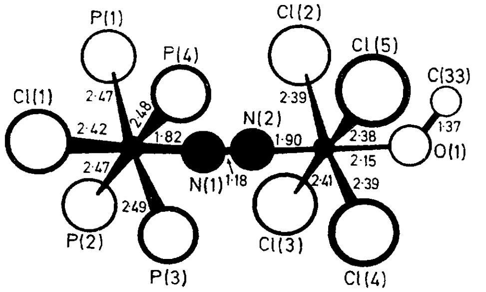

FIGURE 2 The bond lengths of the metal octahedra; carbon atoms have been removed for clarity

TABLE 1

Atomic co-ordinates ($\times 10^4$) and isotropic thermal parameters ($\times 10^4$)

	X	Y	Z	B/Å^2
Re	2116(1)	0985(1)	1861(0·5)	
Mo	4424(1)	3300(2)	1089(1)	*
Cl(1)	1070(4)	−0196(5)	2295(2)	*
Cl(2)	4966(4)	1577(7)	0688(3)	*
Cl(3)	3406(4)	3748(6)	0421(2)	*
Cl(4)	4066(4)	5187(6)	1430(3)	*
Cl(5)	5526(4)	2985(6)	1716(2)	*
P(1)	2825(3)	−0968(6)	1707(2)	*
P(2)	1282(3)	0860(5)	1084(2)	*
P(3)	1136(4)	2647(6)	2111(2)	*
P(4)	3064(4)	1198(5)	2601(2)	*
O(1)	5419(10)	4267(16)	0715(6)	*
N(1)	2964(10)	1863(16)	1578(6)	*
N(2)	3516(10)	2427(13)	1391(6)	*
C(1)	3069(16)	−1955(22)	2236(8)	3·8(6)
C(2)	3925(15)	−0820(22)	1439(8)	3·7(6)
C(3)	0120(16)	0258(23)	1090(9)	4·0(7)
C(4)	1140(16)	2349(23)	0794(9)	3·9(6)
C(5)	0993(17)	2774(25)	2740(9)	4·8(7)
C(6)	1487(15)	4147(23)	1947(8)	3·7(6)
C(7)	2745(14)	0303(20)	3096(8)	3·0(7)
C(8)	4257(15)	0781(22)	2530(8)	3·6(5)
C(9)	2194(15)	−2048(20)	1332(8)	3·2(6)
C(10)	1338(17)	−2387(24)	1468(9)	4·4(7)
C(11)	0892(17)	−3271(24)	1198(9)	4·5(7)
C(12)	1274(20)	−3823(28)	0830(10)	6·0(8)
C(13)	2087(22)	−3536(31)	0686(12)	7·4(10)
C(14)	2593(18)	−2606(26)	0950(10)	5·2(8)
C(15)	1770(14)	0073(20)	0591(7)	2·6(5)
C(16)	1289(15)	−0755(22)	0300(8)	3·8(6)
C(17)	1689(17)	−1236(25)	−0112(9)	5·0(8)
C(18)	2527(19)	−0853(28)	−0201(10)	5·7(8)
C(19)	3057(17)	−0027(24)	0056(9)	4·3(7)
C(20)	2631(17)	0446(24)	0464(9)	4·5(7)
C(21)	−0075(15)	2571(21)	1902(8)	3·3(6)
C(22)	−0420(15)	3369(22)	1558(8)	3·7(6)
C(23)	−1319(18)	3200(26)	1380(10)	5·3(8)
C(24)	−1821(18)	2325(26)	1591(10)	5·2(7)
C(25)	−1499(18)	1626(26)	1944(10)	5·1(7)
C(26)	−0596(15)	1758(22)	2126(8)	3·5(6)
C(27)	3243(14)	2684(19)	2851(8)	2·7(5)
C(28)	3177(17)	2899(24)	3342(9)	4·5(7)
C(29)	3411(20)	4094(31)	3519(11)	6·4(9)
C(30)	3627(16)	4962(23)	3213(9)	4·0(6)
C(31)	3717(17)	4755(24)	2754(9)	4·4(7)
C(32)	3539(14)	3608(20)	2558(8)	3·0(5)
C(33)	6291(17)	3985(27)	0628(9)	5·0(7)
Cl(6)	1389(10)	2931(16)	4255(8)	*
Cl(7)	1237(12)	0623(16)	4621(6)	*
O(2)	4807(24)	1463(34)	4643(13)	15·3(12)
C(34)	4790(29)	2500(43)	4415(16)	10·8(13)
H(1A)	2566	−2192	2350	5·5
H(1B)	3435	−2603	2158	
H(1C)	3387	−1470	2481	
H(2A)	4021	0000	1562	
H(2B)	4323	−1432	1567	
H(2C)	3880	−0931	1101	
H(3A)	−0094	0009	0739	
H(3B)	−0220	0584	0827	
H(3C)	0125	−0552	1102	
H(4A)	0804	2147	0710	
H(4B)	1876	2505	0710	
H(4C)	1169	2988	1030	
H(5A)	0804	2147	2840	
H(5B)	0663	3334	2821	
H(5C)	1211	3110	2831	
H(6A)	1624	4074	1725	
H(6B)	1918	4150	2138	
H(6C)	0814	4619	2074	
H(7A)	3155	0592	3267	
H(7B)	2749	−0565	3037	
H(7C)	2152	0481	3213	
H(8A)	4482	0867	2799	
H(8B)	4452	1330	2307	
H(8C)	4290	0357	2414	
H(10)	1088	−2053	1726	
H(11)	0283	−3476	1283	
H(12)	0936	−4659	0686	
H(13)	2440	−3831	0462	
H(14)	3189	−2439	0844	
H(16)	0681	−1046	0375	
H(17)	1539	−1874	−0305	
H(18)	2772	−1123	−0524	
H(19)	3629	0023	−0025	
H(20)	2878	1248	0624	
H(22)	−0026	3921	1391	
H(23)	−1544	3618	1100	
H(24)	−2521	2311	1476	
H(25)	−1976	1079	2069	
H(26)	−0322	1293	2403	
H(28)	2953	2273	3545	
H(29)	3217	3937	3692	
H(30)	3726	5705	3401	
H(31)	3880	5409	2553	
H(32)	3692	3386	2221	

* Anisotropic thermal parameters ($\times 10^5$) defined in the expression: $\exp -(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{23}kl + 2\beta_{31}lh + 2\beta_{12}hk)$

Re	192(5)	501(10)	132(2)	5(8)	11(24)	20(50)
Mo	222(10)	709(23)	134(3)	−52(13)	26(5)	52(83)
Cl(1)	218(32)	594(6)	143(11)	−56(38)	53(15)	69(23)
Cl(2)	380(40)	1059(89)	174(14)	26(50)	59(19)	−20(30)
Cl(3)	353(36)	1126(96)	129(96)	64(48)	−1(16)	53(28)
Cl(4)	443(43)	627(77)	194(14)	74(47)	−26(19)	47(28)
Cl(5)	266(35)	753(76)	168(13)	28(42)	−35(17)	−2(26)
Cl(6)	382(95)	1357(237)	412(57)	−18(119)	−208(59)	233(95)
Cl(7)	829(13)	1401(24)	245(39)	361(142)	−160(57)	185(78)
P(1)	200(30)	408(61)	110(19)	142(41)	0(14)	12(24)
P(2)	172(3)	480(6)	92(10)	13(40)	−5(14)	0(24)
P(3)	169(32)	566(7)	120(11)	81(39)	29(15)	−68(24)
P(4)	195(32)	478(69)	96(10)	6(38)	−1(14)	23(22)
O(1)	238(92)	1365(310)	287(148)	−128(13)	206(54)	213(103)
N(1)	156(10)	671(25)	68(3)	90(13)	11(4)	138(7)
N(2)	240(9)	94(15)	85(3)	94(9)	15(4)	84(5)

for $|F_o| < 64{\cdot}0$, $\sqrt{w} = 1{\cdot}0$, and for $|F_o| > 64{\cdot}0$, $\sqrt{w} = 128{\cdot}0/|F_o|$. Refinement ended at $R = 0{\cdot}059$, $R' = 0{\cdot}060$.

A ΔF synthesis calculated on the final parameters showed no peaks $>0{\cdot}6$ eÅ^{-3}, except about the rhenium atom.

Computation was carried out on an IBM 1130 computer, with programmes of *X*-RAY ARC system.[13] Observed and calculated structure factors are listed in Supplementary Publication No. SUP 21015 (17 pp. 1 microfiche).*

Atomic positional and thermal parameters are listed in Table 1, and important intramolecular dimensions in Table 2. Figure 1 shows a perspective view giving the labelling of the atoms other than hydrogen, and Figure 2 is a view of the co-ordination about the two metal atoms.

TABLE 2

Values of selected bond lengths and angles

(*a*) Distances (Å)

Re–Cl(1)	2·418(6)	Mo–O(1)	2·15(2)
Re–P(1)	2·471(6)	Mo–Cl(2)	2·39(1)
Re–P(2)	2·471(6)	Mo–Cl(3)	2·41(1)
Re–P(3)	2·486(6)	Mo–Cl(4)	2·39(1)
Re–P(4)	2·477(6)	Mo–Cl(5)	2·38(1)
Re–N(1)	1·815(15)	Mo–N(2)	1·90(1)
P(1)–C(1)	1·88(2)	P(3)–C(5)	1·80(3)
P(1)–C(2)	1·85(2)	P(3)–C(6)	1·82(3)
P(1)–C(9)	1·84(2)	P(3)–C(21)	1·88(2)
P(2)–C(3)	1·86(2)	P(4)–C(7)	1·80(2)
P(2)–C(4)	1·86(3)	P(4)–C(8)	1·86(2)
P(2)–C(15)	1·82(2)	P(4)–C(27)	1·82(2)
O(1)–C(33)	1·37(3)	N(1)–N(2)	1·18(3)

(*b*) Angles (°)

P(1)–Re–P(2)	89·9(2)	Cl(2)–Mo–Cl(3)	90·8(2)
P(2)–Re–P(3)	90·8(2)	Cl(3)–Mo–Cl(4)	89·2(2)
P(3)–Re–P(4)	90·6(2)	Cl(4)–Mo–Cl(5)	89·5(2)
P(4)–Re–P(1)	90·0(2)	Cl(5)–Mo–Cl(2)	89·6(2)
N(1)–Re–P(1)	95·0(5)	N(2)–Mo–Cl(2)	92·9(3)
N(1)–Re–P(2)	85·5(5)	N(2)–Mo–Cl(3)	91·1(5)
N(1)–Re–P(3)	98·7(5)	N(2)–Mo–Cl(4)	95·5(5)
N(1)–Re–P(4)	86·3(5)	N(2)–Mo–Cl(5)	94·2(5)
N(1)–Re–Cl(1)	175·3(5)	N(2)–Mo–O(1)	177·3(7)
P(1)–Re–P(3)	166·3(2)	Cl(2)–Mo–Cl(4)	171·0(3)
P(2)–Re–P(4)	174·8(2)	Cl(3)–Mo–Cl(5)	174·7(2)
Re–N(1)–N(2)	179·6(14)	C(1)–P(1)–C(2)	103·5(11)
Mo–N(2)–N(1)	178·7(9)	C(2)–P(1)–C(9)	105·4(11)
Mo–O(1)–C(33)	130·8(10)	C(1)–P(1)–C(9)	98·4(10)
		C(1)–P(1)–Re	116·7(8)
C(3)–P(2)–C(4)	103·9(11)	C(2)–P(1)–Re	112·8(7)
C(3)–P(2)–C(15)	103·8(11)	C(9)–P(1)–Re	117·9(7)
C(4)–P(2)–C(3)	97·9(10)		
Re–P(2)–C(3)	116·3(8)	C(5)–P(3)–C(6)	103·0(12)
Re–P(2)–C(4)	112·4(8)	C(5)–P(3)–C(21)	99·0(11)
Re–P(2)–C(15)	119·9(7)	C(6)–P(3)–C(21)	104·2(11)
		Re–P(3)–C(5)	115·8(9)
C(7)–P(4)–C(8)	103·5(10)	Re–P(3)–C(6)	115·9(8)
C(8)–P(4)–C(27)	98·4(10)	Re–P(3)–C(21)	116·6(7)
C(7)–P(4)–C(27)	104·3(10)		
Re–P(4)–C(7)	115·9(7)		
Re–P(4)–C(8)	113·5(8)		
Re–P(4)–C(27)	118·7(8)		

DISCUSSION

Description of the Molecular Structure.—The most significant feature of this analysis lies in the Re–N–N–Mo bridge. The nitrogen group bonds end-on to both metals, and the system is effectively linear. The lengthening of the N–N bond is accompanied by a significant shortening of both the Re–N and the Mo–N bond distances. The Re–N bond length [1·82(2) Å] in this compound may be compared directly with that in $[\mathrm{ReCl(N_2)(PMe_2Ph)_4}]$[4] (1·97 Å), and with the formal Re=N double bond in $[\mathrm{ReCl_3(NMe)(PEt_2Ph)_2}]$[14] (1·685 Å).

Reported[15,16] values of molybdenum(V)–nitrogen single-bond distances are 2·23 and 2·42 Å, but in the first compound the ligands are chelated and in the second the longer bond length is affected by the *trans*-effect from the Mo=O double bond. The Mo–$\mathrm{N_2}$ bond length (1·90 Å) found in the present compound indicates a considerable reduction from the single-bond distance.

The MO bonding scheme for the analogous $[\mathrm{(PMe_2Ph)_4ClReN_2CrCl_3(thf)_3}]$ has been described,[8] and this compound has essentially the same delocalised π-system. Although the N–N bond distance is formally that of a double bond, the lengthening of the bond arises, in this case, because the two 3*e* orbitals, which are bonding on nitrogen, are vacant.

Both metals are in an approximately octahedral environment. The Re–P and Re–Cl bond distances are as expected by comparison with those in the crystal structure of $\mathrm{ReCl(N_2)(PMe_2Ph)_4}$. Table 3 presents mean planes through various groups of atoms. The phosphorus atoms are out-of-plane by an alternate

TABLE 3

Planes through various groups of atoms. Equations are in the form $lX + mY + nZ = P$ where X is along the *a* axis, Y in the *ab* plane, and Z along the c^* axis. Deviations of the atoms ($\times 10^3$ Å) are given in square brackets

Plane (i): P(1)—(4)

$$0{\cdot}6907X + 0{\cdot}5668Y - 0{\cdot}4489Z = 0{\cdot}1956$$

[Re 93(1), N(1) 1906(16), Cl(1) −2321(6), P(1) −206(6), P(2) 204(6), P(3) −202(6), P(4) 204(6)]

Plane (ii)

Cl(2)—(5) $\quad 0{\cdot}7214X + 0{\cdot}5032Y - 0{\cdot}4756Z = 5{\cdot}208$

[Cl(2) 38(7), Cl(3) −38(6), Cl(4) 38(7), Cl(5) −38(6), Mo −149(2), O(1) 1999(17), N(2) −2050(5)]

Planes of benzene rings:

Plane (iii)

C(9)—(14) $\quad -0{\cdot}3734X + 0{\cdot}6927Y - 0{\cdot}6169Z = -5{\cdot}062$

Plane (iv)

C(15)—(20) $\quad -0{\cdot}3717X + 0{\cdot}7446Y - 0{\cdot}5543Z = -1{\cdot}801$

Plane (v)

C(21)—(26) $\quad 0{\cdot}3162X - 0{\cdot}6650Y - 0{\cdot}6765Z = -5{\cdot}693$

Plane (vi)

C(27)—(32) $\quad -0{\cdot}9491X + 0{\cdot}2667Y - 0{\cdot}1670Z = -4{\cdot}801$

Angles (°) between planes:

(iii)–(iv)	4·6	(i)–(ii)	4·3
(iii)–(v)	99·3	(iv)–(v)	103·8
(iii)–(vi)	50·0	(iv)–(vi)	49·9
(v)–(vi)	111·4		

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue.

[13] *X*-RAY ARC, IBM 1130 Program System for Crystallography, programmes by B. L. Vickery, D. Bright, P. R. Mallinson, A. Zalkin, M. E. Pippy, and R. F. Ahmed.

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step arrangement, with the rhenium atom approximately in the middle. This deviation from planarity is presumably the result of packing requirements of the phosphorus substituent groups. The best plane through phosphorus atoms lies almost parallel with the plane of the chlorine atoms (angle between normals, 4·3°); the phosphorus and chlorine atoms are in the eclipsed position with respect to each other; the appropriate angles to allow P(2), P(3), and P(4) to eclipse Cl(2), Cl(3), and Cl(4), lie in the range 85—95°.

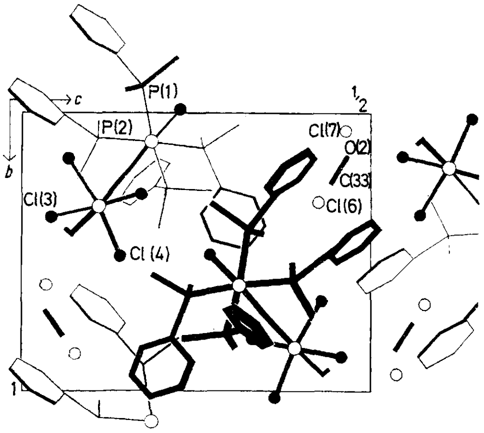

FIGURE 3 The structure projected on to the *bc* plane. The disordered chloride ions are represented by open circles and the methanol molecules by filled rectangles

The molybdenum atom is slightly out of the chlorine atom plane, displaced towards the oxygen atom. The Mo^V–Cl and Mo–O bond lengths are of the same order as those reported previously (2·28—2·46 Å)[17,18] for bridging atoms.

The crystal packing shows discrete molecules in an unusually open arrangement (Figure 3). This partially reflects the substantial steric requirements of the dimethyl(phenyl)phosphine ligand, but in addition, there is a clear channel through the centre of the structure. The difference map indicated this channel to be occupied by two separate atoms of electron density *ca.* 8, and two closely bonded atoms, whose high vibration parameters, combined with local peaks on the difference map, indicate some degree of disorder. The magnetic moment (μ_{eff} 1·85 B.M.) confirms the valency state of Mo^V. The situation was interpreted from a consideration of the chemical preparation, as being due to the presence of one CH_3OH per molecule, at least partially disordered, and one chlorine ion, from HCl, with an occupation 0·5 in each position. In view of the fact that the final difference map revealed hydrogen atom positions clearly, it seems unlikely that there are unlocated disordered solvent molecules in the apparently empty volume.

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[3/2344 *Received, 15th November,* 1973]

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