# Crystal and Molecular Structure of Carbonylnitrosyl(p-tolyl isocyanide)-bis(triphenylphosphine)osmium(o) Perchlorate

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The crystal and molecular structure of the title compound has been determined from three-dimensional $X$-ray data collected by counter methods. Crystals are monoclinic, space group $P2_1/n$, with $Z = 4$ in a unit cell of dimensions $a = 13.257(4)$, $b = 21.48(2)$, $c = 14.820(6)$ Å, and $\beta = 99.33(2)°$. Least-squares refinement has led to a final $R$ of 0.072 for 2 433 observed reflections. The co-ordination geometry about osmium is that of a trigonal bipyramid with carbonyl, nitrosyl, and isocyanide groups in the trigonal plane; the triphenylphosphine groups occupy apical positions. All three ligands of the trigonal plane are bound linearly to the osmium.

STRUCTURAL data available on complexes containing the isocyanide ligand [1-7] suggest that its normal mode of co-ordination is linear or very nearly linear and that the metal–carbon bond has a bond order greater than one. The results of an i.r. spectral investigation [8] of the π-bonding ability of the isocyanide group suggest that it can allow a considerable amount of π back donation, but that when both carbonyl and isocyanide are present the former will receive a greater share of the π electrons. Similar evidence suggests that isocyanide is a stronger π-bonding ligand than substituted phosphines.[9] For the cation [Os(CO)(CNR)(NO)(PPh₃)₂]⁺, the i.r. band at 1 700 cm⁻¹ associated with the nitrosyl stretching allows no prediction of the manner of binding of this group to the metal and its effect on the co-ordination geometry.[10] Accordingly we have undertaken a single-crystal $X$-ray analysis of [Os(CO)(CNR)(NO)(PPh₃)₂][ClO₄] (R = p-tolyl).

## EXPERIMENTAL

The crystals were orange-brown plates. Unit-cell constants were determined from a least-squares refinement [11] of the setting angles of 12 reflections centred on a Hilger and Watts automatic four-circle diffractometer.

*Crystal Data.*—[C₄₅H₃₇N₂O₂OsP₂][ClO₄], $M = 989.4$, Monoclinic, $a = 13.257(4)$, $b = 21.48(2)$, $c = 14.820(6)$ Å, $\beta = 99.33(2)°$, $U = 4 165$ Å³, $D_m = 1.57$, $Z = 4$, $F(000) = 1 848$, $D_c = 1.58$. Space group $P2_1/n$. Mo-$K_\alpha$ radiation, $\lambda = 0.710 7$ Å; $\mu(\text{Mo-}K_\alpha) = 34.6$ cm⁻¹.

Intensity data were collected by a symmetric $2\theta$—$\omega$ scan of 1.40° at a scan rate of 0.02° s⁻¹, with background counted for 10 s at each end of the scan. Of 4 530 reflections examined with a Bragg angle of less than 21° [sin(θ/λ) ⩽ 0.51], 2 423 independent reflections had $I > 3\sigma(I)$. Data were processed according to the procedure of Ibers [12] with $p$ initially given a value of 0.04. In the later stages of the refinement this parameter was increased to 0.08 so as to further downweight the large reflections. Absorption corrections were applied,[13] the scaling factors ranging from 1.25 to 2.10.

[1] F. A. Cotton, T. G. Dunne, and J. S. Wood, *Inorg. Chem.,* 1965, **4**, 318.
[2] J. K. Stalick and J. A. Ibers, *J. Amer. Chem. Soc.,* 1970, **92**, 5333.
[3] R. S. Dickson and J. A. Ibers, *J. Organometallic Chem.,* 1972, **36**, 191.
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[9] W. D. Horrocks and R. C. Taylor, *Inorg. Chem.,* 1963, **2**, 723.
[10] G. R. Clark, K. R. Grundy, W. R. Roper, J. M. Waters, and K. R. Whittle, *J.C.S. Chem. Comm.,* 1972, 119.
[11] W. R. Busing and H. A. Levy, *Acta Cryst.,* 1967, **22**, 457.
[12] P. W. R. Corfield, R. J. Doedens, and J. A. Ibers, *Inorg. Chem.,* 1967, **6**, 197.
[13] W. R. Busing and H. A. Levy, *Acta Cryst.,* 1957, **10**, 180.

A ' sharpened ' Patterson synthesis revealed the atomic site of osmium ($R$ 0.28) and from the subsequent electron-density map the two phosphorus atoms were located ($R$ 0.23). From a second electron-density map the remaining 54 non-hydrogen atoms were located ($R$ 0.17). Atomic scattering factors for osmium were taken from ref. 14 and for all the other atoms from ref. 15. No attempt was made to distinguish the nitrosyl and carbonyl groups and initially both were treated as carbonyl. Four cycles of block-diagonal least-squares refinement assuming isotropic thermal motion lowered $R$ to 0.117 and $R'$ to 0.111 {$R' = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o^2]^{\frac{1}{2}}$, where the weight, $w$, is given by $4F_o^2/\sigma^2(F_o^2)$}. The assumption of anisotropic thermal motion for osmium,
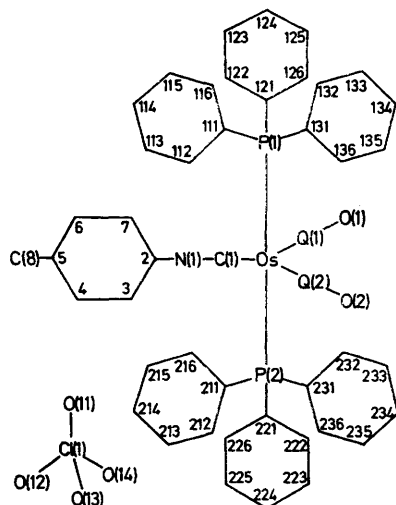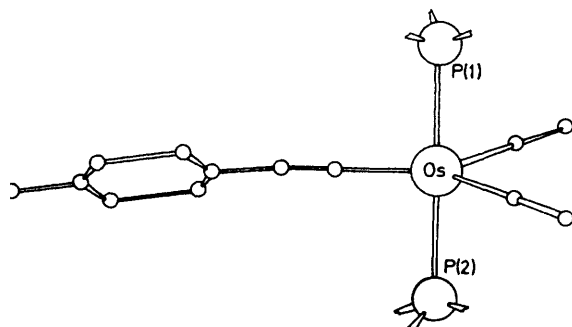


FIGURE 1   Numbering system



FIGURE 2   Co-ordination geometry

phosphorus, and chlorine resulted in a further lowering of $R$ to 0.083 and $R'$ to 0.093. Hydrogen positions associated with the $p$-tolyl isocyanide group were estimated and included in the calculation with $B$ 5.0 Å.$^2$ Anisotropic thermal motion was then assumed for all non-hydrogen atoms except those of the triphenylphosphine phenyl rings, which were treated as rigid groups (hydrogen atoms were not included). The effects of anomalous dispersion were also included in the calculation of $F_c$, values of $\Delta f'$ and $\Delta f''$ for osmium, phosphorus, and chlorine being taken from ref. 16. Full-matrix least-squares refinement converged with $R$ 0.078 and $R'$ 0.088. Inspection of $\langle w(\Delta F)^2\rangle$

14 D. T. Cromer and J. T. Waber, *Acta Cryst.*, 1965, **18**, 104.
15 ' International Tables for *X*-Ray Crystallography,' vol. III, 1962, pp. 202—203.

showed it to be dependent on $|F_o|$, the greatest divergence from an approximately constant value occurring where $|F_o|$ was large. Hence $p$ was increased to 0.08. No clear distinction between the nitrogen and carbon atoms of the nitrosyl and carbonyl ligands was possible and as a better

TABLE 1

Atomic co-ordinates and isotropic temperature parameters with estimated standard deviations in parentheses

| Atom | $x/a$ | $y/b$ | $z/c$ | $B/\text{Å}^2$ |
|---|---|---|---|---|
| Os | 0.108 16(7) | 0.061 16(5) | 0.285 47(7) | |
| Cl(1) | 0.119 0(7) | 0.342 7(5) | 0.406 1(7) | |
| P(1) | −0.042 9(5) | 0.098 1(3) | 0.189 0(5) | |
| P(2) | 0.270 0(5) | 0.033 1(3) | 0.374 5(5) | |
| O(1) | 0.012 5(17) | 0.063 9(13) | 0.446 7(14) | |
| O(2) | 0.114 2(16) | −0.050 1(12) | 0.164 7(16) | |
| O(11) | 0.060 4(40) | 0.344 8(30) | 0.326 7(40) | |
| O(12) | 0.200 7(23) | 0.377 5(14) | 0.393 5(24) | |
| O(13) | 0.064 0(34) | 0.364 0(21) | 0.461 6(31) | |
| O(14) | 0.131 7(27) | 0.281 7(14) | 0.416 8(34) | |
| N(1) | 0.235 8(20) | 0.169 9(13) | 0.215 6(16) | |
| Q(1) * | 0.046 6(19) | 0.062 2(14) | 0.374 9(17) | 6.2(6) |
| Q(2) * | 0.115 8(23) | −0.006 6(15) | 0.211 9(20) | 7.2(8) |
| C(1) | 0.188 4(20) | 0.132 0(15) | 0.242 4(23) | |
| C(2) | 0.308 8(22) | 0.207 4(13) | 0.191 1(20) | |
| C(3) | 0.373 5(23) | 0.197 2(16) | 0.131 5(22) | |
| C(4) | 0.444 8(22) | 0.237 6(20) | 0.114 8(21) | |
| C(5) | 0.453 1(23) | 0.293 4(16) | 0.160 8(24) | |
| C(6) | 0.389 2(34) | 0.304 3(21) | 0.217 8(31) | |
| C(7) | 0.313 8(24) | 0.265 5(16) | 0.232 9(20) | |
| C(8) | 0.529 0(30) | 0.339 3(19) | 0.139 0(31) | |
| C(111) | −0.116 1(12) | 0.154 7(7) | 0.238 8(12) | 3.8(6) |
| C(112) | −0.066 2(9) | 0.195 9(10) | 0.304 0(12) | 4.3(6) |
| C(113) | −0.121 5(13) | 0.242 2(9) | 0.340 4(12) | 5.1(7) |
| C(114) | −0.226 7(13) | 0.247 3(7) | 0.311 6(13) | 4.9(7) |
| C(115) | −0.276 6(9) | 0.206 1(10) | 0.246 4(13) | 4.6(6) |
| C(116) | −0.221 3(12) | 0.159 8(9) | 0.210 0(11) | 3.9(6) |
| C(121) | −0.137 5(14) | 0.035 2(9) | 0.156 5(14) | 5.0(7) |
| C(122) | −0.158 3(15) | −0.006 4(10) | 0.223 5(10) | 4.9(6) |
| C(123) | −0.235 4(17) | −0.050 7(9) | 0.202 6(13) | 7.0(8) |
| C(124) | −0.291 9(14) | −0.053 4(10) | 0.114 6(16) | 7.6(8) |
| C(125) | −0.271 1(16) | −0.011 9(11) | 0.047 6(11) | 6.8(8) |
| C(126) | −0.194 0(17) | 0.032 4(10) | 0.068 6(12) | 6.2(8) |
| C(131) | −0.016 2(15) | 0.132 3(9) | 0.082 5(12) | 3.3(5) |
| C(132) | −0.052 0(24) | 0.192 1(13) | 0.058 8(15) | 6.1(8) |
| C(133) | −0.025 6(31) | 0.221 3(10) | −0.018 2(17) | 7.3(9) |
| C(134) | 0.036 6(17) | 0.190 6(11) | −0.071 5(13) | 6.2(8) |
| C(135) | 0.072 4(25) | 0.130 8(14) | −0.047 7(15) | 6.9(9) |
| C(136) | 0.046 0(30) | 0.101 6(10) | 0.029 2(17) | 7.7(10) |
| C(211) | 0.377 2(10) | 0.046 3(8) | 0.313 7(10) | 2.7(5) |
| C(212) | 0.377 9(13) | 0.014 7(11) | 0.231 6(12) | 5.1(7) |
| C(213) | 0.461 0(15) | 0.020 8(9) | 0.185 4(10) | 5.1(7) |
| C(214) | 0.543 4(11) | 0.058 6(10) | 0.221 3(11) | 4.5(5) |
| C(215) | 0.542 7(13) | 0.090 2(11) | 0.303 4(12) | 5.4(7) |
| C(216) | 0.459 6(15) | 0.084 1(8) | 0.349 6(10) | 3.9(6) |
| C(221) | 0.300 6(13) | 0.081 4(8) | 0.478 3(10) | 3.8(6) |
| C(222) | 0.364 9(23) | 0.058 0(8) | 0.554 6(12) | 5.5(6) |
| C(223) | 0.391 6(18) | 0.095 1(11) | 0.632 0(10) | 5.1(7) |
| C(224) | 0.354 0(16) | 0.155 7(9) | 0.633 0(11) | 6.0(8) |
| C(225) | 0.289 6(24) | 0.179 2(8) | 0.556 7(14) | 6.2(8) |
| C(226) | 0.262 9(18) | 0.142 0(11) | 0.479 4(11) | 4.8(7) |
| C(231) | 0.276 9(13) | −0.045 2(6) | 0.414 2(11) | 3.3(5) |
| C(232) | 0.347 1(11) | −0.087 9(9) | 0.389 9(11) | 4.6(6) |
| C(233) | 0.352 3(14) | −0.147 8(8) | 0.426 6(13) | 5.8(7) |
| C(234) | 0.287 4(15) | −0.165 0(7) | 0.487 6(13) | 4.6(6) |
| C(235) | 0.217 2(12) | −0.122 3(10) | 0.511 9(11) | 5.1(7) |
| C(236) | 0.212 0(13) | −0.062 3(9) | 0.475 2(12) | 4.9(6) |

\* Atom Q has a scattering factor of $\frac{1}{2}(f_C + f_N)$.

approximation a form factor of $\frac{1}{2}(f_C + f_N)$ was applied to both. [These atoms have been designated as Q(1) and Q(2).] A similar procedure $^{17}$ was used in the structural analysis of [Mn(CO)$_2$(NO)(PPh$_3$)$_2$] where no distinction

16 D. T. Cromer, *Acta Cryst.*, 1965, **18**, 17.
17 J. H. Enemark and J. A. Ibers, *Inorg. Chem.*, 1967, **6**, 1575.

between nitrosyl and carbonyl groups was possible. Further refinement gave final $R$ factors of $R$ 0.072, and $R'$ 0.087. The final difference map showed no peaks indicating the presence of a solvent molecule nor were alternative positions for the perchlorate oxygen atoms apparent.

The numbering system of the molecule is given in Figure 1, the co-ordination geometry in Figure 2. Final atomic parameters are listed with their standard deviations in Tables 1, bond distances and angles in Tables 2 and 3.

### TABLE 2

Bond lengths (Å), with estimated standard deviations in parentheses

| | | | |
|---|---|---|---|
| Os–P(1) | 2.400(7) | N(1)–C(1) | 1.14(4) |
| Os–P(2) | 2.406(7) | N(1)–C(2) | 1.35(4) |
| Os–Q(1) | 1.67(2) | C(2)–C(3) | 1.35(4) |
| Os–Q(2) | 1.84(3) | C(2)–C(7) | 1.39(4) |
| Os–C(1) | 2.02(3) | C(3)–C(4) | 1.34(5) |
| P(1)–C(111) | 1.79(2) | C(4)–C(5) | 1.37(5) |
| P(1)–C(121) | 1.85(2) | C(5)–C(6) | 1.31(6) |
| P(1)–C(131) | 1.83(2) | C(5)–C(8) | 1.48(5) |
| P(2)–C(211) | 1.82(2) | C(6)–C(7) | 1.35(6) |
| P(2)–C(221) | 1.85(2) | Cl(1)–O(11) | 1.30(6) |
| P(2)–C(231) | 1.78(2) | Cl(1)–O(12) | 1.35(3) |
| O(1)–Q(1) | 1.22(3) | Cl(1)–O(13) | 1.27(5) |
| O(2)–Q(2) | 1.15(4) | Cl(1)–O(14) | 1.33(3) |

### TABLE 3

Bond angles (°) with estimated standard deviations in parentheses

| | | | |
|---|---|---|---|
| P(1)–Os–P(2) | 173.5(2) | P(1)–C(121)–C(122) | 119(2) |
| P(1)–Os–Q(1) | 90.9(9) | P(1)–C(121)–C(126) | 121(2) |
| P(1)–Os–Q(2) | 91.7(1.0) | P(1)–C(131)–C(132) | 119(2) |
| P(1)–Os–C(1) | 89.6(9) | P(1)–C(131)–C(136) | 121(2) |
| P(2)–Os–Q(1) | 94.4(9) | P(2)–C(211)–C(212) | 118(2) |
| P(2)–Os–Q(2) | 89.8(1.0) | P(2)–C(211)–C(216) | 122(2) |
| P(2)–Os–C(1) | 83.9(9) | P(2)–C(221)–C(222) | 120(2) |
| Q(1)–Os–Q(2) | 124.5(1.3) | P(2)–C(221)–C(226) | 120(2) |
| Q(1)–Os–C(1) | 126.4(1.3) | P(2)–C(231)–C(232) | 122(2) |
| Q(2)–Os–C(1) | 109.1(1.3) | P(2)–C(231)–C(236) | 117(2) |
| Os–Q(1)–O(1) | 172(2) | C(1)–N(1)–C(2) | 168(3) |
| Os–Q(2)–O(2) | 176(3) | N(1)–C(2)–C(3) | 130(3) |
| Os–C(1)–N(1) | 177(3) | N(1)–C(2)–C(7) | 114(3) |
| Os–P(1)–C(111) | 116(1) | C(3)–C(2)–C(7) | 117(3) |
| Os–P(1)–C(121) | 112(1) | C(2)–C(3)–C(4) | 124(3) |
| Os–P(1)–C(131) | 113(1) | C(3)–C(4)–C(5) | 118(3) |
| Os–P(2)–C(211) | 113(1) | C(4)–C(5)–C(6) | 118(3) |
| Os–P(2)–C(221) | 112(1) | C(4)–C(5)–C(8) | 118(3) |
| Os–P(2)–C(231) | 114(1) | C(6)–C(5)–C(8) | 124(4) |
| C(111)–P(1)–C(121) | 102(1) | C(5)–C(6)–C(7) | 125(4) |
| C(111)–P(1)–C(131) | 106(1) | C(2)–C(7)–C(6) | 118(3) |
| C(121)–P(1)–C(131) | 107(1) | O(11)–Cl(1)–O(12) | 103(3) |
| C(211)–P(2)–C(221) | 104(1) | O(11)–Cl(1)–O(13) | 105(3) |
| C(211)–P(2)–C(231) | 108(1) | O(11)–Cl(1)–O(14) | 101(3) |
| C(221)–P(2)–C(231) | 105(1) | O(12)–Cl(1)–O(13) | 117(3) |
| P(1)–C(111)–C(112) | 119(2) | O(12)–Cl(1)–O(14) | 118(3) |
| P(1)–C(111)–C(116) | 121(2) | O(13)–Cl(1)–O(14) | 111(3) |

Final observed and calculated structure factors are listed in Supplementary Publication No. SUP 21780 (30 pp., 1 microfiche),* together with thermal parameters, rigid-group parameters, hydrogen-atom positions, intra- and inter-molecular distances, details of planes, and root-mean-square amplitudes of vibration.

### DESCRIPTION AND DISCUSSION

The crystal structure consists of monomeric ions. The configuration about osmium is that of a slightly distorted trigonal bidyramid with the triphenylphos-

* For details see Notices to Authors No. 7 in *J.C.S. Dalton,* **1975**, Index issue.

phine groups occupying the apical positions and the carbonyl, nitrosyl, and *p*-tolyl isocyanide groups occupying the trigonal plane. These last three groups all bond in a linear manner and the least-squares plane through C(1), Q(1), and Q(2) shows that the osmium atom lies in the trigonal plane. The angles of the trigonal plane, C(1)–Os–Q(1), C(1)–Os–Q(2), and Q(1)–Os–Q(2) [126.4(1.3), 109.1(1.3), and 124.5(1.3)°], show some deviations from the ideal value (120°). The P(1)–Os–P(2) angle [173.5(2)°] also shows a slight distortion from the ideal geometry.

In the *p*-tolyl isocyanide ligand Os–C(1), C(1)–N(1), and N(1)–C(2) bond lengths are 2.02(3), 1.14(4), and 1.35(4) Å respectively. The covalent radius for a single Os–C bond is 2.02 Å,[18] hence it can be assumed that in this complex little or no π bonding occurs, an effect resulting from the presence of linear nitrosyl and carbonyl groups which compete more successfully for the π electrons. The lack of π bonding between the osmium atom and the isocyanide group is also seen by comparing the Os–C(isocyanide) bond length with those involving the carbonyl ligand where distances in the range 1.84—1.94 Å are frequently observed (see later). The C(1)–N(1) bond is as expected for a carbon–nitrogen triple bond; the N(1)–C(2) distance [1.35(4) Å] is in agreement with values for comparable bonds [1.39(8) and 1.38(9) Å] in [MnBr(CO)$_3$(CNPh)$_2$].[19] The angles Os–C(1)–N(1) and C(1)–N(1)–C(2) are 176.6(2.8) and 168.0(3.1)° respectively, the latter showing a small deviation from linearity. Other deviations from ideal values are found for the angles N(1)–C(2)–C(3) and N(1)–C(2)–C(7) which are expected to be equivalent but for which significantly different values [129.7(2.8) and 113.6(2.7)°] are observed. In its present position the isocyanide group is sandwiched between two phenyl rings, one from a phosphine group in the same cation [C(211)–(216)] and another from an adjacent phosphine in a second cation [C(231)–(236)]. The closest approach made by atoms C(2)–(8) to the atoms of these two phenyl rings is 3.50 Å. If, however, the angles C(2)–N(1)–C(1), N(1)–C(2)–C(3), and N(1)–C(2)–C(7) are assumed to have the predicted values of 180, 120, and 120°, then from a model it can be seen that atoms C(2)–(8) approach closely to one of the phenyl rings [C(231)–(236)]. If atomic sites are estimated for this position of the isocyanide group and inter-molecular contacts calculated several are <3.2 Å [*viz.* C(4)···C(234) 3.1, C(5)···C(234) 3.0, C(5)···C(235) 2.8, C(6)···C(234) 3.2, and C(8)···C(235) 2.8 Å]. The observed distortions thus probably arise from these steric interactions. The mean C–C distance associated with the isocyanide phenyl ring is 1.35 Å, a standard deviation of 0.03 Å being calculated from a statistical spread of values. The angles do not differ significantly from 120°. Similarly, the bond lengths and angles of the methyl group are as expected. The phenyl ring of

[18] L. Pauling, 'The Nature of the Chemical Bond,' 3rd edn., Cornell University Press, Ithaca, New York, 1960.
[19] D. Bright and O. S. Mills, *J.C.S. Dalton,* **1974,** 219.

the *p*-tolyl isocyanide ligand is itself planar but does not lie in the equatorial plane of the trigonal bipyramid. Some rotation about the C(2)–N(1) bond is found so that the two planes are mutually inclined at 15.7°. This effect is at least partly caused by inter- and intra-molecular repulsions.

The carbonyl and nitrosyl groups, which were not distinguished, are both linear and have bond angles of 172.4(2.5) and 175.8(2.7)°. The Os–Q(1) and Os–Q(2) bond lengths [1.67(2) and 1.84(3) Å] are significantly different and suggest that Q(1) is probably nitrogen and Q(2) carbon. This distinction follows from the fact that metal–nitrogen bond lengths associated with a linear nitrosyl group are generally shorter than are metal–carbonyl bond distances; *e.g.* in [Os(NO)$_2$(OH)-(PPh$_3$)$_2$]$^+$,[20] which also contains a linear nitrosyl group, Os–N is 1.63(1) Å, whereas in [Os(CO)$_3$(PPh$_3$)$_2$] Os–C is 1.90 and 1.93(5) Å.[21]

The Os–P bond lengths [2.400(7) and 2.406(7) Å] do not differ from those found in the trigonal-bipyramidal osmium(0) complex [Os(CO)$_3$(PPh$_3$)$_2$] [2.32(4), 2.35(4),

and 2.42(4) Å].[21] The mean P–C bond length and Os–P–C, C–P–C, and P–C–C bond angles [1.82(3) Å; 113(1), 105(2), and 120(2)°] are as expected and compare well with such values for [Os(CO)$_3$(PPh$_3$)$_2$].[21]

The large anisotropic temperature parameters of the perchlorate anion together with bond lengths ranging from 1.27(5) to 1.35(3) Å and angles ranging from 101(3) to 118(3)° suggest that this group is disordered. For this reason the atomic sites of the perchlorate anion will only be approximate. Corrections for thermal motion [22] have been calculated and these lie in the range 0.06—0.08 Å. The corrected values are as expected for a normal perchlorate group.[23]

Non-bonded intra- and inter-molecular distances have been calculated and are deposited with the Supplementary data. None of these approaches is thought to indicate hydrogen bonding.[24]

[20] G. R. Clark, J. M. Waters, and K. R. Whittle, *J.C.S. Dalton*, 1975, 463.
[21] J. K. Stalick and J. A. Ibers, *Inorg. Chem.*, 1969, **8**, 419.
[22] D. W. J. Cruickshank, *Acta Cryst.*, 1956, **9**, 757.
[23] R. B. Gillespie, R. A. Sparks, and K. N. Trueblood, *Acta Cryst.*, 1959, **12**, 867.
[24] W. C. Hamilton and J. A. Ibers, 'Hydrogen Bonding in Solids,' Benjamin, New York, 1968.