Crystal and Molecular Structure of Dicarbonylnitrosylbis(triphenylphosphine)osmium(0) Perchlorate–Dichloromethane

By **George R. Clark, Joyce M. Waters,* and Kenneth R. Whittle,** Chemistry Department, University of Auckland, Auckland, New Zealand

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 $a = 17.031(2)$, $b = 13.951(1)$, $c = 17.270(1)$ Å, and $\beta = 104.33(1)°$. Block-diagonal least-squares refinement gave R 0.057 for 4 281 observed reflections. The co-ordination geometry about osmium is that of a trigonal bipyramid with the two triphenylphosphine groups apical. The trigonal plane is formed by the linear nitrosyl and two carbonyl ligands. As a result of statistical disordering in this basal plane it has not been possible to differentiate between the NO and CO groups.

Two modes of bonding have now been established for the nitrosyl ligand and in five-co-ordinate complexes these appear to be associated with different co-ordination geometries. The linear mode, in which the nitrosyl group is formulated as NO^+, is associated with the trigonal bipyramidal arrangement as in the d^8 complex, $[IrH(NO)(PPh_3)_3]$,[1] whereas the bent mode, formulated as NO^-, has been found in conjunction with the tetragonal pyramidal geometry as in the d^6 complex, $[IrI(NO)(CO)(PPh_3)_2]$.[2]

The complex, $[Os(CO)_2(NO)(PPh_3)_2]ClO_4$ shows a nitrosyl stretching band in the i.r. spectrum at 1 750 cm^{-1}, suggesting that this group may be linear [*i.e.* complex is formulated as $Os^0(NO^+)$, a d^8 species] and the co-ordination geometry that of a trigonal bipyramid.[3] This has been found for the electronically similar complex $[Os(CO)_3(PPh_3)_2]$.[4] However, the dicarbonyl complex may be prepared *via* $[OsCl(CO)(NO)(PPh_3)_2]$, the iridium analogue of which has been shown by *X*-ray analysis [5] to contain a bent nitrosyl and have tetragonal pyramidal geometry. Mingos and Ibers [1] emphasise the need for caution in predicting nitrosyl geometry on the basis of i.r. evidence, particularly when the nitrosyl band falls in or near the region 1 600–1 720 cm^{-1}. Thus, in view of the uncertainty concerning the nitrosyl bonding mode and complex geometry we have undertaken a single-crystal analysis of $[Os(CO)_2(NO)(PPh_3)_2]ClO_4$.

EXPERIMENTAL

Crystals were reddish brown and tabular. Unit-cell constants were determined from a least-squares refinement [6] of the setting angles of twelve reflections centred on a Hilger and Watts automatic four-circle diffractometer.

Crystal Data.—$[C_{38}H_{30}NO_3OsP_2]^+[ClO_4]^-,CH_2Cl_2$, M = 985.2, Monoclinic, $a = 17.031(2)$, $b = 13.951(1)$, $c = 17.270(1)$ Å, $\beta = 104.33(1)°$, $U = 3\,976$ Å^3, $D_m = 1.63$, $Z = 4$, $D_c = 1.65$. Space group $P2_1/n$. Cu-K_α radiation, $\lambda = 1.5418$ Å, μ(Cu-K_α) = 91.6 cm^{-1}. Crystal size 0.20 × 0.12 × 0.12 mm.

Intensity data were collected by a symmetric 2θ—ω scan of 1.0° at a scan rate of 0.02° s^{-1}, with background counted for 10 s at each end of the scan. Of 5 860 reflections examined within Bragg angle of 57° ($\sin\theta/\lambda = 0.54$) 4 281 independent reflections were recorded with intensities $> 3\sigma(I)$. Data were processed according to Ibers [7] with $p = 0.04$, and were corrected for Lorentz and polarisation factors and for absorption.[8]

A sharpened Patterson synthesis revealed the positions of the osmium and two phosphorus atoms and the subsequent structure-factor calculation gave R 0.27. From the corresponding electron-density map all non-hydrogen atoms, except those of the solvent molecule and one oxygen of the perchlorate group, were located. No attempt was made to distinguish between nitrosyl and carbonyl groups at this stage and all three were treated as carbonyl. These additional forty-three atoms reduced R to 0.20. The four remaining non-hydrogen atoms were located from a second electron-density map and R fell to 0.18. Atomic scattering factors used for the osmium atom were taken from ref. 9 and for all other atoms from ref. 10. Four cycles of block-diagonal least-squares refinement assuming isotropic thermal motion lowered R to 0.087. The function minimised was $\Sigma w(|F_o| - |F_c|)^2$, with the weight w given by $4F_o^2/\sigma^2(F_o^2)$. Anisotropic thermal motion was now assumed for all atoms other than those of the phenyl rings and the carbon of the dichloromethane molecule and three further cycles of refinement were computed. In these cycles the effects of anomalous dispersion were included in the calculation of F_c, the values of $\Delta f'$ and $\Delta f''$ for osmium, phosphorus, and chlorine being from ref. 11.

Attempts were then made to differentiate between the nitrosyl and carbonyl groups by considering bond distances and by analysis of the thermal parameters. No single model proved better than any other. {Similar difficulties have been reported previously, *e.g.* in the structure of $[Mn(NO)(CO)_2(PPh_3)_2]$.[12]} Accordingly a model was adopted in which statistical disorder between the three groups was assumed, and a scattering factor corresponding to $(1/3f_N + 2/3f_O)$ was chosen for each of the three atoms directly bonded to osmium. These atoms were relabelled NC(1), NC(2), NC(3). Refinement continued with the atoms of the disordered nitrosyl and carbonyl groups being restricted to isotropic thermal parameters, and convergence was reached with R 0.057 and R' 0.075 $\{R' = [\Sigma w(|F_o| - |F_c|)^2/\Sigma wF_o^2]^{\frac{1}{2}}\}$. Final atomic positional parameters, with their standard deviations, are listed in Table 1, the numbering system of the molecule is given in the Figure, bond distances and angles with their standard deviations are given in Tables

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[2] D. M. P. Mingos, W. T. Robinson, and J. A. Ibers, *Inorg. Chem.*, 1971, **10**, 1043.

[3] For a preliminary account of the preparation, reactions, and structure of $[Os(CO)_2(NO)(PPh_3)_2]ClO_4$ see G. R. Clark, K. R. Grundy, W. R. Roper, J. M. Waters, and K. R. Whittle, *J.C.S. Chem. Comm.*, 1972, 119.

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2 and **3**. Final observed and calculated structure factors are listed in Supplementary Publication No. SUP 21435 (20 pp., 1 microfiche),* together with anisotropic thermal parameters and details of root-mean-square amplitudes of vibration.

TABLE 1

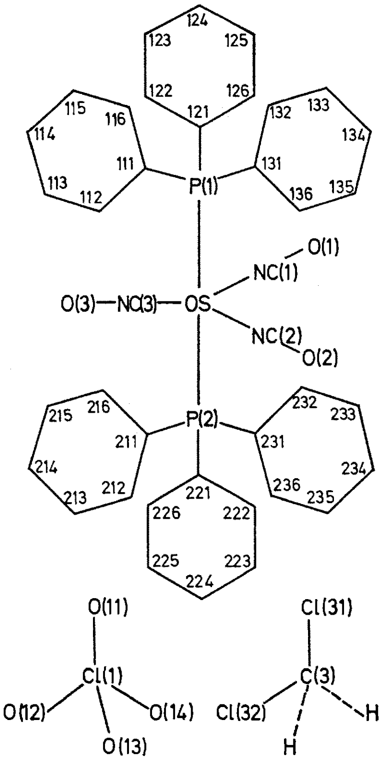
Atomic co-ordinates and isotropic temperature parameters

Atom	*x*/*a*	*y*/*b*	*z*/*c*	*B*/Å^2
Os	0.156 15(3)	0.149 82(3)	0.414 79(3)	
Cl(1)	0.0051(2)	0.2004(3)	0.0764(2)	
Cl(31)	0.2963(4)	0.3967(6)	0.6934(6)	
Cl(32)	0.3673(4)	0.5765(5)	0.6805(8)	
P(1)	0.2202(1)	0.0034(2)	0.3898(2)	
P(2)	0.0954(1)	0.2961(2)	0.4445(2)	
O(1)	0.1974(6)	0.2434(8)	0.2732(6)	7.5(2)
O(2)	0.2596(7)	0.1497(8)	0.5838(7)	7.8(2)
O(3)	−0.0057(7)	0.0514(8)	0.3803(6)	8.0(2)
O(11)	0.0471(10)	0.1789(18)	0.0284(9)	
O(12)	0.0306(14)	0.2922(13)	0.0794(13)	
O(13)	0.0243(7)	0.1707(7)	0.1564(6)	
O(14)	−0.0760(7)	0.2125(14)	0.0451(7)	
NC(1)	0.0565(7)	0.0905(9)	0.3936(7)	5.3(2)
NC(2)	0.1825(7)	0.2061(9)	0.3275(7)	4.6(2)
NC(3)	0.2203(7)	0.1503(8)	0.5173(7)	4.6(2)
C(3)	0.3771(10)	0.4566(14)	0.6744(10)	9.7(4)
C(111)	0.1627(5)	−0.0662(7)	0.3063(5)	3.5(2)
C(112)	0.1672(6)	−0.1663(7)	0.3086(6)	4.4(2)
C(113)	0.1264(6)	−0.2191(8)	0.2400(6)	5.0(2)
C(114)	0.0829(6)	−0.1731(8)	0.1733(6)	4.9(2)
C(115)	0.0782(6)	−0.0733(8)	0.1720(6)	4.9(2)
C(116)	0.1172(6)	−0.0205(7)	0.2382(6)	4.4(2)
C(121)	0.3191(5)	0.0218(6)	0.3705(5)	3.0(2)
C(122)	0.3373(6)	−0.0163(8)	0.3032(6)	4.7(2)
C(123)	0.4170(7)	−0.0081(9)	0.2935(7)	5.5(2)
C(124)	0.4758(6)	0.0420(8)	0.3515(6)	5.0(2)
C(125)	0.4560(6)	0.0820(7)	0.4160(6)	4.5(2)
C(126)	0.3773(6)	0.0740(7)	0.4262(6)	4.4(2)
C(131)	0.2406(5)	−0.0762(7)	0.4761(5)	3.7(2)
C(132)	0.3096(6)	−0.1317(8)	0.4941(6)	4.7(2)
C(133)	0.3245(7)	−0.1928(9)	0.5621(7)	5.4(2)
C(134)	0.2693(7)	−0.1979(10)	0.6085(7)	6.2(3)
C(135)	0.1978(8)	−0.1434(9)	0.5886(8)	6.2(3)
C(136)	0.1832(6)	−0.0833(8)	0.5230(7)	5.2(2)
C(211)	0.1548(5)	0.4039(7)	0.4383(5)	3.2(2)
C(212)	0.1173(6)	0.4922(8)	0.4214(6)	4.4(2)
C(213)	0.1638(8)	0.5748(11)	0.4198(8)	6.3(3)
C(214)	0.2471(7)	0.5678(9)	0.4343(7)	5.9(3)
C(215)	0.2854(7)	0.4801(8)	0.4521(7)	5.5(2)
C(216)	0.2399(6)	0.3966(8)	0.4547(6)	4.6(2)
C(221)	0.0793(5)	0.2943(6)	0.5449(5)	3.3(2)
C(222)	0.1018(6)	0.3712(8)	0.5968(6)	4.8(2)
C(223)	0.0847(7)	0.3681(9)	0.6719(7)	6.1(3)
C(224)	0.0499(7)	0.2895(9)	0.6968(7)	5.6(2)
C(225)	0.0251(7)	0.2149(9)	0.6448(7)	5.9(3)
C(226)	0.0402(7)	0.2160(8)	0.5678(7)	5.4(2)
C(231)	−0.0038(5)	0.3203(6)	0.3791(5)	3.3(2)
C(232)	−0.0656(6)	0.3584(7)	0.4104(6)	4.2(2)
C(233)	−0.1402(6)	0.3809(8)	0.3588(6)	5.1(2)
C(234)	−0.1520(7)	0.3661(8)	0.2775(7)	5.5(2)
C(235)	−0.0911(7)	0.3277(9)	0.2461(7)	5.7(2)
C(236)	−0.0163(6)	0.3045(8)	0.2967(6)	4.6(2)

RESULTS AND DISCUSSION

The crystal structure consists of monomeric ions. The configuration about osmium is that of a slightly distorted trigonal bipyramid with linear nitrosyl and carbonyl groups lying in the equatorial plane and the two triphenylphosphine groups in the apical positions. Since the nitrosyl co-ordinates as NO^+ the osmium has a formal oxidation state of zero.

The three metal–ligand distances within the trigonal plane [1.84, 1.85, and 1.84(1) Å] are all equivalent. The close agreement amongst these bond lengths and the consistency of the isotropic temperature factors of the atoms of the nitrosyl and carbonyl groups supports the



The numbering system of the molecule

TABLE 2

Bond lengths (Å) with estimated standard deviations in parentheses

Os–P(1)	2.405(2)	C(132)–C(133)	1.42(2)
Os–P(2)	2.401(3)	C(133)–C(134)	1.38(2)
Os–NC(1)	1.84(1)	C(134)–C(135)	1.40(2)
Os–NC(2)	1.85(1)	C(135)–C(136)	1.38(2)
Os–NC(3)	1.84(1)	C(211)–C(212)	1.38(1)
P(1)–C(111)	1.811(9)	C(211)–C(216)	1.41(1)
P(1)–C(121)	1.813(9)	C(212)–C(213)	1.40(2)
P(1)–C(131)	1.823(9)	C(213)–C(214)	1.38(2)
P(2)–C(211)	1.830(9)	C(214)–C(215)	1.38(2)
P(2)–C(221)	1.822(9)	C(215)–C(216)	1.41(2)
P(2)–C(231)	1.816(9)	C(221)–C(222)	1.39(1)
O(1)–NC(1)	1.16(1)	C(221)–C(226)	1.39(1)
O(2)–NC(2)	1.16(1)	C(222)–C(223)	1.40(2)
O(3)–NC(3)	1.18(1)	C(223)–C(224)	1.37(2)
C(111)–C(112)	1.40(1)	C(224)–C(225)	1.37(2)
C(111)–C(116)	1.39(1)	C(225)–C(226)	1.42(2)
C(112)–C(113)	1.42(2)	C(231)–C(232)	1.40(1)
C(113)–C(114)	1.37(2)	C(231)–C(236)	1.40(1)
C(114)–C(115)	1.39(2)	C(232)–C(233)	1.40(2)
C(115)–C(116)	1.38(1)	C(233)–C(234)	1.38(2)
C(121)–C(122)	1.38(1)	C(234)–C(235)	1.39(2)
C(121)–C(126)	1.40(1)	C(235)–C(236)	1.39(2)
C(122)–C(123)	1.41(2)	Cl(1)–O(11)	1.26(2) *
C(123)–C(124)	1.41(2)	Cl(1)–O(12)	1.35(2) *
C(124)–C(125)	1.36(2)	Cl(1)–O(13)	1.40(1) *
C(125)–C(126)	1.40(1)	Cl(1)–O(14)	1.36(1) *
C(131)–C(132)	1.38(1)	C(3)–Cl(31)	1.71(2) *
C(131)–C(136)	1.42(2)	C(3)–Cl(32)	1.69(2) *

* Corrections (Å) for these bond lengths are: Cl(1)–O(11) 0.07, Cl(1)–O(12) 0.07, Cl(1)–O(13) 0.04, Cl(1)–O(14) 0.06, C(3)–Cl(31) 0.04, and C(3)–Cl(32) 0.04.

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1974, Index issue.

view that the structure determined does indeed represent the average of three disordered arrangements. It is further noted that with such disorder the Os–NC bond distances should appear to be intermediate between the values expected for metal–carbonyl and metal–linear-nitrosyl bonds. This is observed, *e.g.* Os–NO (linear) is 1.63(1) in $[Os(OH)(NO)_2(PPh_3)_2]^+$,[13] and Os–CO 1.90, 1.93(5) Å in $[Os(CO)_3(PPh_3)_2]$.[4] The Os–P bond lengths [2.405(2) and 2.401(3) Å] are also as expected.[4] The mean P–C and C–C bond lengths of the triphenylphosphine groups are 1.819(7) and 1.39(2) Å, the standard deviations being calculated from a statistical spread of values. The mean Os–P–C, C–P–C, P–C–C, and C–C–C angles are 113(1), 105(1), 119.8(9), and 119.7(8)° and agree with the typical values found in $[Os(CO)_3(PPh_3)_2]$.[4]

TABLE 3

Bond angles (°), with estimated standard deviations in parentheses

P(1)–Os–P(2)	177.96(8)
P(1)–Os–NC(1)	91.1(3)
P(1)–Os–NC(2)	90.1(3)
P(1)–Os–NC(3)	89.3(3)
P(2)–Os–NC(1)	90.0(3)
P(2)–Os–NC(2)	91.0(3)
P(2)–Os–NC(3)	88.6(3)
NC(1)–Os–NC(2)	115.2(5)
NC(1)–Os–NC(3)	119.5(5)
NC(2)–Os–NC(3)	125.3(5)
Os–NC(1)–O(1)	178.7(1.0)
Os–NC(2)–O(2)	178.0(1.0)
Os–NC(3)–O(3)	178.2(1.0)
Os–P(1)–C(111)	114.7(3)
Os–P(1)–C(121)	113.3(3)
Os–P(1)–C(131)	111.9(3)
Os–P(2)–C(211)	114.4(3)
Os–P(2)–C(221)	111.5(3)
Os–P(2)–C(231)	114.2(3)
C(111)–P(1)–C(121)	105.9(4)
C(111)–P(1)–C(131)	106.3(4)
C(121)–P(1)–C(131)	103.9(4)
C(211)–P(2)–C(221)	106.3(4)
C(211)–P(2)–C(231)	104.7(4)
C(221)–P(2)–C(231)	105.0(4)
P(1)–C(111)–C(112)	119.8(7)
P(1)–C(111)–C(116)	120.3(7)
P(1)–C(121)–C(122)	120.8(7)
P(1)–C(121)–C(126)	118.6(7)
P(1)–C(131)–C(132)	120.3(8)
P(1)–C(131)–C(136)	119.4(8)
P(2)–C(211)–C(212)	120.7(7)
P(2)–C(211)–C(216)	118.9(7)
P(2)–C(221)–C(222)	121.4(7)
P(2)–C(221)–C(226)	118.5(7)
P(2)–C(231)–C(232)	120.2(7)
P(2)–C(231)–C(236)	119.0(7)
C(112)–C(111)–C(116)	119.8(9)
C(111)–C(112)–C(113)	118.8(9)
C(112)–C(113)–C(114)	120.7(1.0)
C(113)–C(114)–C(115)	119.9(1.0)
C(114)–C(115)–C(116)	120.3(1.0)
C(111)–C(116)–C(115)	120.5(9)
C(122)–C(121)–C(126)	120.6(9)
C(121)–C(122)–C(123)	119.4(1.0)
C(122)–C(123)–C(124)	119.2(1.0)
C(123)–C(124)–C(125)	120.5(1.0)
C(124)–C(125)–C(126)	120.6(1.0)
C(121)–C(126)–C(125)	119.5(9)
C(132)–C(131)–C(136)	120.3(9)
C(131)–C(132)–C(133)	119.3(1.0)
C(132)–C(133)–C(134)	120.2(1.2)
C(133)–C(134)–C(135)	120.3(1.2)
C(134)–C(135)–C(136)	119.9(1.2)
C(131)–C(136)–C(135)	120.0(1.1)
C(212)–C(211)–C(216)	120.3(9)
C(211)–C(212)–C(213)	120.2(1.0)
C(212)–C(213)–C(214)	119.8(1.2)
C(213)–C(214)–C(215)	120.6(1.2)
C(214)–C(215)–C(216)	120.4(1.1)
C(211)–C(216)–C(215)	118.7(1.0)
C(222)–C(221)–C(226)	120.0(9)
C(221)–C(222)–C(223)	119.1(1.0)
C(222)–C(223)–C(224)	121.6(1.1)
C(223)–C(224)–C(225)	119.4(1.1)
C(224)–C(225)–C(226)	120.6(1.1)
C(221)–C(226)–C(225)	119.2(1.0)
C(232)–C(231)–C(236)	120.7(9)
C(231)–C(232)–C(233)	119.4(9)
C(232)–C(233)–C(234)	119.7(1.0)
C(233)–C(234)–C(235)	121.2(1.1)
C(234)–C(235)–C(236)	119.9(1.1)
C(231)–C(236)–C(235)	119.1(1.0)
O(11)–Cl(1)–O(12)	92(2)
O(11)–Cl(1)–O(13)	123(1)
O(11)–Cl(1)–O(14)	117(1)
O(12)–Cl(1)–O(13)	104(1)
O(12)–Cl(1)–O(14)	100(1)
O(13)–Cl(1)–O(14)	113(1)
Cl(31)–C(3)–Cl(32)	112(1)

The three ligands in the trigonal plane are distorted from ideal geometry, subtending angles of 115.2, 119.5, and 125.3(5)° at the osmium atom. The deviation of these angles from the ideal value of 120° is probably caused by a close intermolecular approach of 3.10 Å between atoms O(1) and C(113) (at $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$) which results in a contraction of the C(1)–Os–NC(1) angle to minimise this interaction. The osmium atom lies in the trigonal plane. The P(1)–Os–P(2) angle deviates only slightly from linearity [177.96(8)°].

Within the perchlorate group considerable disorder is suggested by the variation in bond length and bond angles [1.26(2)—1.40(1) Å, and 92(2)—123(1)°] and by the large thermal parameters observed. In an attempt to improve the model for this group anisotropic thermal motion has been assumed, but the final model is only approximate. Corrections to the bond lengths calculated from the anisotropic thermal parameters of the oxygen atoms by the method of Cruickshank [14] are listed in Table 2.

The dichloromethane also has large thermal parameters with Cl–C bond lengths of 1.71(2) and 1.69(2) Å and Cl–C–Cl 112(1)°. In the free molecule [15] a rather longer C–Cl distance is observed (1.77 Å). However the values determined for the dicarbonyl complex compare favourably with those from the analysis [16] of $[IrI(O_2)(CO)(PPh_3)_2]\cdot CH_2Cl_2$ [1.63(4) and 1.70(4) Å], where the dichloromethane also has large anisotropic temperature parameters. In the dicarbonyl complex there is an intermolecular contact Cl(31) · · · C(222) 3.35 Å (van der Waals distance 3.4 Å),[17] but the atoms do not have the correct geometry for hydrogen bonding to occur. Since dichloromethane is undergoing large thermal motion, the position

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[17] W. C. Hamilton and J. A. Ibers, 'Hydrogen Bonding in Solids,' Benjamin, New York, 1968.

of the molecule is only approximate and these bond lengths have also been corrected for thermal motion.

Intermolecular distances $\leqslant$3.4 Å have been calculated and are listed in Table 4. The shortest contact distances involve oxygen atoms of the perchlorate anion and carbonyl or nitrosyl groups with carbon atoms of the phenyl rings. In no case are the atoms in a geometry which would allow hydrogen-bond formation.[17] The perchlorate anion and the dichloromethane molecule lie in channels which extend throughout the structure.

Table 4

Intermolecular distances $\leqslant$3.4 Å

	Symmetry position *	Translation	Distance
Cl(31) $\cdots$ C(222)	(x,y,z)	(0,0,0)	3.34
O(1) $\cdots$ O(13)	(x,y,z)	(0,0,0)	3.29
O(1) $\cdots$ C(112)	$(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$	(0,0,0)	3.24
O(1) $\cdots$ C(113)	$(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$	(0,0,0)	3.11
O(2) $\cdots$ C(234)	$(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$	(0,0,0)	3.32
O(2) $\cdots$ C(235)	$(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$	(0,0,0)	3.30
O(12) $\cdots$ C(125)	$(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$	(−1,0,−1)	3.30
O(13) $\cdots$ C(3)	$(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$	(−1,0,−1)	3.15
O(13) $\cdots$ C(116)	(x,y,z)	(0,0,0)	3.24
O(13) $\cdots$ C(236)	(x,y,z)	(0,0,0)	3.27

* Of second atom.

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