particularly significant if we compare these distances with those of carbonyl ligands occupying coordination sites of comparable symmetry, namely Ru(2)—C(5) [1.955 (5) Å] and Ru(3)—C(7) [1.937 (5) Å] that are *trans* to the opposite bridgehead atom P(1).

Amongst the growing family of edge double-bridged trinuclear complexes of the iron triad (Albers, Robinson & Coville, 1986), the present species is the first involving a phosphorus and an iodide as bridgehead atoms.

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Acta Cryst. (1992). C**48**, 1002–1004

Structure of Di(5-β-methylallyl-2-phenyl-1,3-oxazole)zinc(II) Dichloride

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(*Received* 1 *July* 1991; *accepted* 8 *November* 1991)

Abstract. Dichlorobis[5-(2-methylallyl)-2-phenyl-1,3-oxazole-*N*]zinc(II), $[ZnCl_2(C_{13}H_{13}NO)_2]$, $M_r = 534.79$, orthorhombic, $Fdd2$, $a = 26.374$ (3), $b = 12.220$ (2), $c = 16.278$ (3) Å, $V = 5246$ (2) Å^3, $Z = 8$, $D_x = 1.350$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 11.48$ cm^{-1}, $F(000) = 2208$, $T = 300$ K, final $R = 0.037$, $wR = 0.034$ for 1658 unique reflections with $F > 2\sigma(F)$. The compound consists of two substituted oxazole molecules complexed with one zinc dichloride molecule. The coordination around the Zn atom is a distorted tetrahedron. The oxazole ring is planar and is twisted 26° out of the phenyl-ring plane. The β-methylallyl is disordered.

Introduction. The reaction of *N*-cyanomethylbenzimidates with β-methylallylzinc chloride leads to the

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0108-2701/92/061002-03$06.00

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Zn(1)	0.0	0.0	0.0	0.042 (1)
Cl(1)	0.9968	0.1513 (1)	−0.9198 (1)	0.057 (1)
C(1)	0.9186 (2)	0.1735 (5)	0.8786 (3)	0.042 (3)
C(2)	0.9671 (2)	0.2000 (6)	0.8581 (4)	0.058 (3)
C(3)	0.9785 (2)	0.3046 (6)	0.8304 (4)	0.073 (4)
C(4)	0.9425 (3)	0.3834 (6)	0.8242 (5)	0.075 (4)
C(5)	0.8928 (3)	0.3591 (6)	0.8442 (4)	0.064 (4)
C(6)	0.8818 (2)	0.2526 (5)	0.8718 (4)	0.056 (3)
C(7)	0.9054 (2)	0.0623 (5)	0.9008 (3)	0.039 (3)
N(8)	0.9355 (1)	−0.0142 (4)	0.9326 (3)	0.039 (2)
C(9)	0.9077 (2)	−0.1102 (5)	0.9366 (3)	0.046 (3)
C(10)	0.8602 (2)	−0.0908 (5)	0.9099 (4)	0.045 (3)
O(11)	0.8591 (2)	0.0175 (4)	0.8867 (3)	0.072 (3)
C(12)	0.8136 (2)	−0.1640 (6)	0.9032 (5)	0.066 (4)
C(13)	0.826 (1)	−0.282 (1)	0.891 (1)	0.059 (3)
C(13*A*)	0.813 (1)	−0.237 (2)	0.832 (1)	0.064 (5)
C(14)	0.823 (1)	−0.349 (1)	0.950 (1)	0.083 (4)
C(14*A*)	0.797 (1)	−0.206 (2)	0.759 (2)	0.112 (8)
C(15)	0.836 (1)	−0.323 (3)	0.808 (2)	0.169 (11)
C(15*A*)	0.853 (1)	−0.330 (3)	0.821 (2)	0.075 (8)

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Zn(1)—Cl(1)	2.264 (1)	C(1)—C(2)	1.361 (7)
Zn(1)—N(8)	2.031 (4)	C(2)—C(3)	1.389 (9)
N(8)—C(7)	1.332 (7)	C(3)—C(4)	1.358 (9)
N(8)—C(9)	1.386 (7)	C(4)—C(5)	1.381 (9)
C(7)—O(11)	1.360 (6)	C(5)—C(6)	1.407 (9)
C(7)—C(1)	1.448 (7)	C(6)—C(1)	1.374 (8)
C(9)—C(10)	1.347 (8)	C(10)—C(12)	1.524 (7)
C(10)—O(11)	1.376 (7)		
C(12)—C(13)*	1.49 (1)	C(12)—C(13*A*)†	1.46 (2)
C(13)—C(14)*	1.28 (1)	C(13*A*)—C(14*A*)†	1.35 (3)
C(13)—C(15)*	1.46 (3)	C(13*A*)—C(15*A*)†	1.49 (4)
N(8)—Zn(1)—Cl(1)	110.5 (1)	N(8)—Zn(1)—N(8*B*)	114.6 (2)
N(8)—Zn(1)—Cl(1*B*)	105.8 (2)	C(6)—C(1)—C(7)	120.8 (5)
Cl(1)—Zn(1)—Cl(1*B*)	109.6 (1)	C(6)—C(1)—C(2)	118.5 (6)
C(7)—N(8)—Zn(1)	130.4 (4)	C(1)—C(2)—C(3)	120.2 (6)
C(9)—N(8)—Zn(1)	119.4 (4)	C(2)—C(3)—C(4)	121.7 (4)
C(9)—N(8)—C(7)	107.3 (4)	C(3)—C(4)—C(5)	119.6 (7)
N(8)—C(7)—C(1)	127.9 (5)	C(4)—C(5)—C(6)	118.0 (7)
C(1)—C(7)—O(11)	123.5 (5)	C(1)—C(6)—C(5)	122.1 (6)
N(8)—C(7)—O(11)	108.6 (5)	C(9)—C(10)—C(12)	132.0 (6)
C(9)—C(10)—O(11)	106.2 (5)	C(12)—C(10)—O(11)	121.9 (6)
C(7)—O(11)—C(10)	108.8 (5)		
C(10)—C(12)—C(13)*	113.5 (6)	C(10)—C(12)—C(13*A*)†	111.6 (9)
C(12)—C(13)—C(14)*	120.6 (10)	C(12)—C(13*A*)—C(14*A*)†	120.4 (20)
C(12)—C(13)—C(15)*	119.6 (15)	C(12)—C(13*A*)—C(15*A*)†	126.5 (18)
C(14)—C(13)—C(15)*	119.3 (16)	C(14*A*)—C(13*A*)—C(15*A*)†	110.5 (21)

Symmetry code: (*B*) − x, − y, z (twofold axis).

* Site occupation factor of 65%.
† Site occupation factor of 35%.

formation of an oxazole derivative. IR and ^{1}H NMR spectra confirm this hypothesis (Hajjem & Baccar, 1991). Elemental analysis indicates the presence of a residue. Atomic absorption spectra showed that the compound contains zinc. Single-crystal X-ray diffraction was carried out in order to determine the crystal and molecular structure.

Experimental. Bipyramidal colourless crystals were obtained from recrystallization in THF and chloroform. A crystal with dimensions 0.2 × 0.2 × 0.4 mm was used. A computer-controlled Stoe Stadi-4 single-crystal diffractometer with monochromated Mo $K\alpha$ radiation was used for data collection. The cell dimensions and the orientation matrix were determined by least-squares fit based on the setting of 58 reflections with 2θ ranging from 35.7 to 44.3°. The intensities were collected at 300 K in the range $3 < 2\theta < 45°$ corresponding to $(\sin\theta/\lambda)_{\max} = 0.54$ Å^{-1} using $2\theta/\Omega = 1/1$ learnt profile scanning mode. A total of 2095 reflections in the range h $0 \rightarrow 28$, k $-13 \rightarrow 13$, l $-18 \rightarrow 18$ was measured. 1720 unique reflections were obtained with $R_{\rm int} = 0.010$. 1658 reflections having $F > 2\sigma(F)$ were used for structure determination and refinement. Three standard reflections (022, 40$\bar{4}$, 02$\bar{2}$), measured every 120 min, remained stable. The intensities were corrected for Lorentz–polarization and, using face indices, an analytical absorption correction with $T_{\max} = 0.851$ and $T_{\min} = 0.807$ was performed. At the end of the refinement an isotropic extinction correction with a

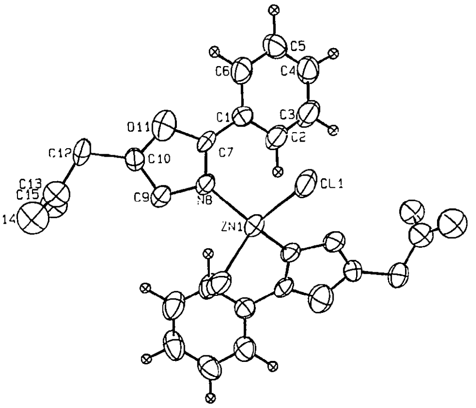

Fig. 1. General view of the molecule. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii. For the β-methylallyl radical, only the atoms having an occupancy of 0.65 are represented.

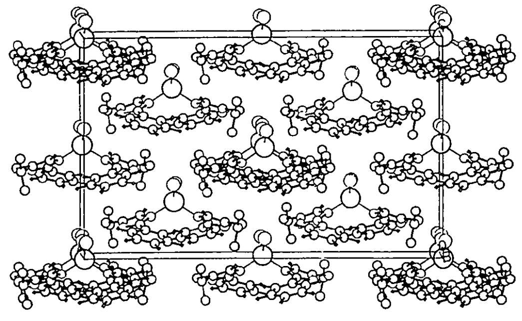

Fig. 2. Packing of the molecules in the cell.

coefficient $g = 2.7 \times 10^{-4}$ was also made. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and refined with *SHELX76* (Sheldrick, 1976). Final $R = 0.037$, $wR = 0.034$ and $S = 2.04$. The weighting scheme was $2.03/\sigma^2(F)$ and at the end of the refinement $(\Delta/\sigma)_{max}$ was 0.234. The H atoms of the phenyl and oxazole rings were located by geometrical calculations (C—H = 1.08 Å) with fixed isotropic thermal parameters (0.063 Å^2). The C atoms C13, C14 and C15 were refined with a site occupation factor of 65% (*A*) and 35% (*B*) for C13*A*, C14*A* and C15*A*. The thermal parameters of the atoms of this radical which are rather large were refined isotropically. In the final difference Fourier map $(\Delta\rho)_{min}$, $(\Delta\rho)_{max}$ were between −0.61 and 0.64 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on a VAX 8530 and an IBM 3090 computer.

Discussion. The atomic coordinates are listed in Table 1* and bond lengths and angles in Table 2. A projection of the molecule and of the packing of the molecules in the cell are shown in Figs. 1 and 2, respectively.

The coordination around the Zn atom is a distorted tetrahedron. The Zn—Cl and Zn—N(8) bond lengths, respectively 2.264 (1) and 2.031 (4) Å, are close to those reported for $ZnCl_2(imidazole)_2$ (Lundberg, 1966) and $ZnCl_2(pyridine)_2$ (Steffen & Palenik, 1976). The oxazole ring is planar as shown by the torsion angles N(8)—C(7)—O(11)—C(10) = −0.4 (6) and O(11)—C(7)—N(8)—C(9) = 1.5 (6)°. In this heterocycle the N(8)—C(7) bond of 1.332 (7) Å is longer than a double bond and the C(9)—N(8) bond of 1.386 (7) Å is shorter than a single bond. On the other hand, the C—O bonds of average length 1.36 Å have double-bond character. These values could be related to the aromaticity of the oxazole ring. The bond lengths in the phenyl ring are in the range 1.358 (9)–1.407 (9) Å. The torsion angles C(2)—C(1)—C(7)—N(8) = 26.1 (9) and C(6)—C(1)—C(7)—O(11) = 25.5 (8)° show that the oxazole ring is twisted about 25.8 (9)° out of the phenyl-ring plane. The low value of this twist angle and the short distance C(7)—C(1) = 1.448 Å could explain the conjugation between the two rings. The β-methylallyl is disordered.

One of the authors (AK) thanks the Deutscher Akademischer Austauschdienst for financial support and H. Fuess for helpful discussions. Support of the Fonds der Chemischen Industrie is gratefully acknowledged.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a diagram of the disordered β-methylallyl group have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54840 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0556]

Acta Cryst. (1992). C**48**, 1004–1007

The Synthesis and Structure of the Pyridine-Substituted Derivative of Tricarbonyl(η^5-methylcyclopentadienyl)manganese(I)

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(*Received* 13 *September* 1991; *accepted* 4 *November* 1991)

Abstract. Dicarbonyl(η^5-methylcyclopentadienyl)(pyridine)manganese(I), $C_{13}H_{12}MnNO_2$, $M_r = 269.18$, monoclinic, $P2_1/c$, $a = 7.761$ (5), $b = 20.578$ (10), $c = 15.634$ (11) Å, $\beta = 103.10$ (6)°, $V = 2432$ (3) Å^3, $Z = 8$ (two independent and structurally distinct molecules per asymmetric unit), $D_m =$

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0108-2701/92/061004-04$06.00