SHORT COMMUNICATIONS

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Acta Cryst. (1978). B**34**, 2956

Bis(*N*-acetylglycinato)-1,10-phenanthrolinecopper(II): erratum.

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In the paper by Battaglia, Bonamartini Corradi, Marcotrigiano & Pellacani [*Acta Cryst.* (1977), B**33**, 3886–3888] the coordinates of the copper atom are given incorrectly. The correct values are $x = 0, y = 0{\cdot}1424, z = 0$.

All the relevant information is given in the Abstract.

Acta Cryst. (1978). B**34**, 2956–2957

Dibenzo-*p*-dioxin: a refinement.

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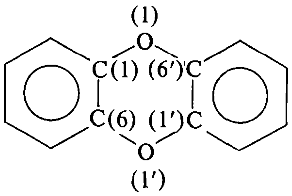
In agreement with Senma, Taira, Taga & Osaki [*Cryst. Struct. Commun.* (1973), **2**, 311–314] we find that the space group of dibenzo-*p*-dioxin is *C*2/*c*, and not *Cc* as reported by Cordes & Fair [*Acta Cryst.* (1974), B**30**, 1621–1623].

Two reports on the structure of dibenzo-*p*-dioxin (DPDO) (I) have appeared



(I)

In the first (Senma, Taira, Taga & Osaki, 1973), the structure was refined in the centrosymmetric space group *C*2/*c* using 948 visually-estimated photographic data, while in the second (Cordes & Fair, 1974; C&F hereinafter) the refinement was carried out in the noncentrosymmetric space group *Cc*, using 233 Mo *Kα* diffractometer data. The latter authors state that the space group *Cc* proved to be correct, presumably because they were unable to refine the structure in the centrosymmetric space group *C*2/*c*. They attribute the large discrepancies in the derived structural parameters (*e.g.* a difference of 7° for two chemically equivalent C—C—O angles) and the large magnitudes of their e.s.d.'s (2° for bond angles and up to 0·04 Å for bond distances) to the presence of a small percentage of intense reflections in their data set.

Structural confirmation of the basic dibenzo-*p*-dioxin nucleus is important since certain halogenated dioxin derivatives are now known to possess extreme toxicity (Schwetz, Norris, Sparchu, Rowe, Gehring, Emerson & Gerbig, 1973; McConnell & Moore, 1976) and biological potency (Poland & Glover, 1973).

We have successfully refined the parameters of C&F using their intensity data in the centrosymmetric space group *C*2/*c*. The resulting internal consistency in bond distances and bond angles and excellent agreement with the values reported in the literature for similar compounds (see below) clearly indicate the space group to be *C*2/*c*, and not *Cc* as assigned by C&F.

In order to refine the structure of DPDO in space group *C*2/*c* the coordinates reported by C&F had to be transformed so as to place the molecular center of inversion at the crystallographic center of inversion at ($\frac{1}{4}$,$\frac{1}{4}$,0), and not at (0,0,0). This was done by adding (0·25,0,–0·25) to the coordinates of the centrosymmetric half of the molecule (non-hydrogen atoms only). The refinement was carried out on *F* by the full-matrix least-squares procedure. The weights were assigned according to the formula $w = 1/\{1 + [(|F_o| -$

Table 1. *Atomic fractional coordinates and thermal parameters with associated e.s.d.'s*

	x	y	z	B (Å^2)
O(1)	0·3714 (4)	0·2510 (10)	0·0023 (3)	5·1 (1)
C(1)	0·2018 (6)	0·4176 (15)	0·0530 (5)	4·2 (2)
C(2)	0·1525 (6)	0·5875 (15)	0·1063 (5)	4·6 (2)
C(3)	0·2232 (6)	0·7602 (16)	0·1616 (5)	5·0 (2)
C(4)	0·3419 (6)	0·7616 (16)	0·1640 (5)	5·0 (2)
C(5)	0·3898 (6)	0·5888 (15)	0·1101 (4)	4·7 (2)
C(6)	0·3195 (6)	0·4182 (16)	0·0556 (4)	4·2 (2)
HC(2)	0·064	0·577	0·103	6·0
HC(3)	0·191	0·915	0·199	6·0
HC(4)	0·394	0·932	0·201	6·0
HC(5)	0·477	0·582	0·107	6·0

Table 2. *Selected bond distances* (Å), *and bond angles* (°), *and their e.s.d.'s for DPDO and tetrachlorodibenzo-p-dioxin* (*TCDD*)

	DPDO			TCDD
	This work	Cordes & Fair (1974)	Senma *et al.* (1973)	Boer *et al.* (1972)
C—O	1·383 (8)	1·37 (3), 1·39 (3)	1·378 (3)	1·377 (3), 1·379 (3)
	1·383 (7)	1·38 (3), 1·39 (4)	1·387 (3)	1·378 (3), 1·378 (3)
C—O—C	116·4 (5)	112 (2), 118 (2)	116·3 (2)	115·6 (2), 115·8 (2)
C—C—O	121·9 (6)	118 (2), 119 (2)	122·0 (2)	122·2 (2), 122·2 (2)
(internal)	121·7 (6)	123 (2), 125 (2)	121·7 (2)	122·2 (2), 122·0 (2)
C—C—O	117·7 (6)	121*, 121*	117·9 (2)	117·5 (2), 117·8 (2)
(external)	117·6 (6)	115*, 115*	117·6 (2)	117·5 (2), 117·7 (2)

* Calculated from the coordinates given by C&F.

$25)/15]^2\}^{1/2}$. The atomic scattering factors for C and N were taken from *International Tables for X-ray Crystallography* (1962), and those for H from Stewart, Davidson & Simpson (1965). The H atoms were located from a difference Fourier map. Their positions were kept fixed. The positional and isotropic temperature parameters for C and O atoms were refined together with a scale factor.

The final residuals, R_1 [$=\sum(||F_o|-|F_c||)/\sum|F_o|$] and R_2 {$=[\sum w(|F_o|-|F_c|)^2/\sum(|F_o|)^2]^{1/2}$} for the refinement in the centrosymmetric space group *C*2/*c*, were 0·057 and 0·062, respectively, with 222 observations and 29 variables.* The structure was also refined in the noncentrosymmetric space group *Cc* using the same weighting scheme as that used for the centrosymmetric refinement to facilitate statistical comparison; R_1 and R_2 in this case were 0·045 and 0·048, respectively, with 55 variables. These latter results are essentially the same as those obtained by C&F. The improvement in the weighted residual R_2 for refinement in the noncentrosymmetric space group *Cc* over that in the centrosymmetric *C*2/*c* (0·048 *vs* 0·062) was found to be statistically significant, the significance level for accepting *C*2/*c* being less than 0·005 (Hamilton, 1965). However, the latter is definitely a better choice (Donohue, 1971) since the resulting molecular geometry is chemically more reasonable and is in much better accord with those of other related compounds (see below).

The final positional and thermal parameters and their e.s.d.'s derived from the last cycle of refinement are given in Table 1.

The C—C bond distances in the benzene ring range from 1·366 (9) to 1·384 (9) Å with a mean of 1·377 Å, while the bond angles range from 119·1 (6) to 120·7 (7)° with a mean of 120·0°. The C—H bond distances range from 1·03 to 1·15 Å. The benzene ring was refined as a rigid group by C&F. The bond distances and bond angles involving the O atoms in the dioxin ring found by us are compared in Table 2 with those given by C&F, and also by Senma *et al.* (1973) and Boer, van Remoortere, North & Neuman (1972). Examination of Table 2 shows that our values are in excellent agreement with those reported by these other workers, whereas those of C&F have extremely large errors and inconsistencies. A least-squares plane through the 12 carbon atoms shows that atoms C(1) through C(6) of one benzene ring deviate from this plane by −0·004, −0·004, 0·004, 0·004, −0·001 and −0·010 Å, respectively, while their centrosymmetric counterparts in the other benzene ring deviate (necessarily) by exactly the same amount in the opposite direction. The two O atoms deviate from this plane by ±0·001 Å only. This, again, is a much better description of the planarity of the DPDO molecule than that obtained by C&F, where deviations in C atoms range from 0·01 to 0·07 Å, and the two O atoms deviate by 0·16 and 0·17 Å respectively.

The computer programs used in the present analysis were modified versions of the Busing, Martin & Levy least-squares refinement and function and error programs, Zalkin's Fourier program, and Doedens's least-squares-plane program.

We are grateful to Professor D. J. Hodgson for providing the laboratory facilities.

Note added in proof: The cell dimensions used were those of C & F: a = 11·69 (1), b = 5·09 (1), c = 15·11 (1) Å, β = 100·29 (1)°.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33598 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.