Fig. 2. Stereoscopic view of the packing in the cell viewed perpendicular to the *ab* plane.

however, there is one intermolecular hydrogen-bonding interaction involving a hydroxyl H and a pyridine N (O$\cdots$N, 2·746 Å) (Fig. 2). The presence of the hydroxyl group has an effect on the conformation of the three rings about C(13). The *p*-chlorophenyl rings of parinol are rotated with respect to the plane of C(1)–C(13)–C(7), making dihedral angles with the least-squares planes of the rings C(1) to C(6) and C(7) to C(12) of 94·6 and 42·0° respectively. The ring-to-ring angle is 80·7°.

Torsion angles down the C(13)–C(7) and C(13)–C(1) vectors corresponding to the angles τ_1 [C(8–7–13–14)] and τ_2 [C(2–1–13–14)] as defined by Hovmöller, Norrestam & Palm (1976) are +87·0 (3) and −38·7 (3)° respectively. These compare with the corresponding angles in: *p,p'*-DDT (+87, −14°); dicofol, molecule *A* (hydrogen bonding *via* OH) (+78, −1°); dicofol, molecule *B* (no hydrogen bonding) (+90, −39°); and 1,1,1,2-tetrachloro-2,2-bis(*p*-chlorophenyl)ethane (+85, −25°).

Bond distances and angles are similar to those found for other analogous DDT compounds except that no significant distortion of the *exo* C(1) and C(7) angles is present. This is a feature of a number of members of this series.

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Structure of the 1:2 Complex of 2,5-Piperazinedione and Formic Acid

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Abstract. $C_4H_6N_2O_2 \cdot 2CH_2O_2$, triclinic, $P\bar{1}$, $a = 6\cdot852$ (2), $b = 7\cdot078$ (2), $c = 6\cdot253$ (2) Å, $\alpha = 109\cdot76$ (2), $\beta = 122\cdot12$ (2), $\gamma = 95\cdot28$ (3)°, $Z = 1$. The structure was solved by direct methods and refined by the block-diagonal least-squares method to an *R* of 0·057 from a set of 902 observed reflections. The piperazinedione ring is almost exactly planar.

Introduction. 2,5-Piperazinedione (DKP) is present in a number of molecules with important biological activity.

Some cyclic dipeptides also exhibit strong denaturing action on globular proteins, weakening the secondary structure by competing with their interchain hydrogen bonds (Cresenzi, Cesaro & Russo, 1973). In view of the fact that DKP can interact strongly with other molecules, we examined the crystal structures of some DKP complexes and here we report on the results for the DKP–formic acid complex.

DKP dissolves in formic acid and crystals of the complex grow very quickly. The crystal chosen for data

Table 1. *Final fractional atomic coordinates and isotropic thermal parameters*

B_{eq} is the isotropic equivalent of the anisotropic thermal parameters and is computed as $B_{eq} = 8\pi^2 (U_{11} U_{22} U_{33})^{1/3}$. The average standard deviation in B_{eq} for non-hydrogen atoms is 0·1 Å², and the average standard deviation for B for H atoms is 0·8 Å².

	x	y	z	B_{eq}/B (Å²)
O	0·2625 (2)	0·1300 (2)	−0·1542 (2)	4·0
O(1)	0·7100 (2)	0·4131 (2)	0·2282 (2)	4·2
O(2)	0·7218 (2)	0·3383 (2)	−0·1385 (2)	5·5
N	0·0918 (2)	0·0649 (2)	0·2803 (2)	3·3
C	0·1364 (2)	0·0663 (2)	−0·0876 (2)	3·3
Cᵃ	0·2511 (2)	0·1441 (2)	0·2212 (2)	3·5
C(1)	0·8129 (2)	0·4367 (2)	0·1095 (3)	4·0
H(1)[N]	0·154 (3)	0·101 (2)	0·472 (3)	4·4
H(2)[Cᵃ]	0·393 (3)	0·113 (3)	0·308 (4)	5·8
H(3)[Cᵃ]	0·315 (4)	0·302 (3)	0·312 (4)	4·0
H(4)[O(1)]	0·561 (4)	0·307 (3)	0·105 (4)	5·1
H(5)[C(1)]	0·971 (3)	0·541 (2)	0·247 (3)	6·3

collection was sealed in a capillary, as the crystals are efflorescent. Accurate cell dimensions were obtained by least-squares refinement of 2θ values of 35 high-angle reflections for which the $\alpha_1-\alpha_2$ peaks were resolved. The intensity data were collected manually (Cu $K\alpha$ radiation, $2\theta \leq 150°$) using the stationary-crystal technique (Furnas & Harker, 1955) with balanced Ni–Co Ross filters on a GE XRD-5 diffractometer. Of 987 reflections, 902 were considered observed from counting statistics $[I > 2\sigma(I)]$. Lorentz, polarization, and $\alpha_1-\alpha_2$ splitting corrections were made to obtain structure amplitudes from the observed intensities.

The structure was solved by direct methods using the program *MULTAN* (Germain, Main & Woolfson, 1971). A total of 102 reflections with $E > 1·5$ were used; out of eight phase sets, that with the second-highest figure of merit revealed all the non-hydrogen atoms. The structure was refined by the block-diagonal least-squares method, and H atoms were located from the difference electron density map. The structure was further refined with anisotropic temperature factors assigned to the non-hydrogen atoms; and the final $R = \sum ||F_o| - |F_c||/\sum |F_o| = 0·057$ for the 902 observed reflections and 0·063 for the complete set of 987 reflections.* The quantity minimized in the refinement was $w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F)$. The coordinates are listed in Table 1.

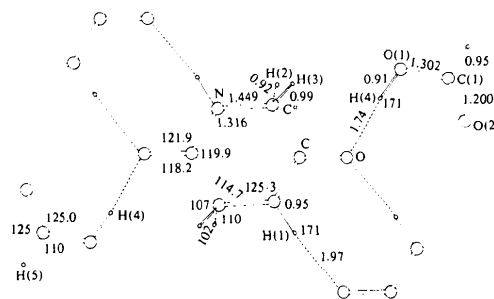**Discussion.** The bond lengths and angles are given in Fig. 1. The dimensions of the DKP ring are in good

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Fig. 1. Bond lengths (Å), bond angles (°) and hydrogen-bond parameters. The average standard deviations in bond lengths and angles involving non-hydrogen atoms are 0·002 Å and 0·2° respectively. For those involving H atoms they are 0·02 Å and 1° respectively.

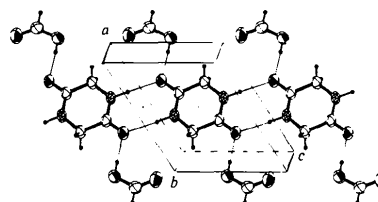Fig. 2. The crystal packing. The thermal ellipsoids for O, N and C atoms are drawn differently.

Table 2. *Torsion angles* (°)

Torsion angles are defined according to the IUPAC–IUB Commission on Biochemical Nomenclature (1970).

φ	C′–N–Cᵃ–C′	0·5 (2)°
ψ	N–Cᵃ–C′–N	0·4 (2)
ω	Cᵃ–C′–N–Cᵃ	0·5 (2)
	O–C′–Cᵃ–N	179·3 (2)
	O–C′–N–Cᵃ	179·2 (2)

agreement with the earlier observations for un-complexed DKP (Degeilh & Marsh, 1959).

In formic acid, the two C—O distances are significantly different: 1·200 and 1·302 Å, characteristic of carbonyl and hydroxyl O atoms. The H is *cis* to the carbonyl O as found in the low-temperature structure of formic acid (Nahringbauer, 1978).

The DKP ring is almost exactly planar, with all atoms, except the H atoms attached to the α-carbon atoms, lying in a plane. Table 2 gives the torsion angles of DKP. The magnitudes of the φ, ψ and ω angles are all less than 0·5°.

The complex is formed by strong O–H···O hydrogen bonds with a hydrogen-to-acceptor distance of 1·74 Å. The complexed molecules are held together by N–H···O hydrogen bonds. In each peptide, O is an acceptor and N is the donor of H atoms. This results in the formation of a hydrogen-bonded eight-membered ring around the center of symmetry and infinite ribbons parallel to the *c* axis (Fig. 2).

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.

Structure of diacetylmorphine (heroin): erratum. By DENNIS V. CANFIELD, *Forensic Science Coordinator, University of Southern Mississippi, Hattiesburg, MS 39401, USA*

Abstract

A typographical error in the *c* axis of diacetylmorphine [Canfield, Barrick & Giessen (1979), *Acta Cryst.* B35, 2806–2809] is corrected and the new torsion angles, bond lengths, and bond angles are reported. The correct value for the *c* axis is 16·092 (7) Å.

The *c* axis in diacetylmorphine was previously reported (Canfield, Barrick & Giessen, 1979) as 16·392 (7) Å, when in fact it should be 16·092 (7) Å. This typographical error
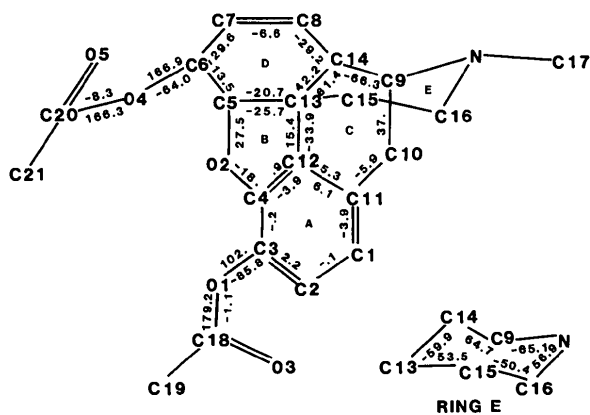

Fig. 1. Configurational drawing of diacetylmorphine with endocyclic torsion angles (°) for rings *A*, *B*, *C*, *D*, and *E*. The estimated average standard deviation in the torsion angles is 1·5°.
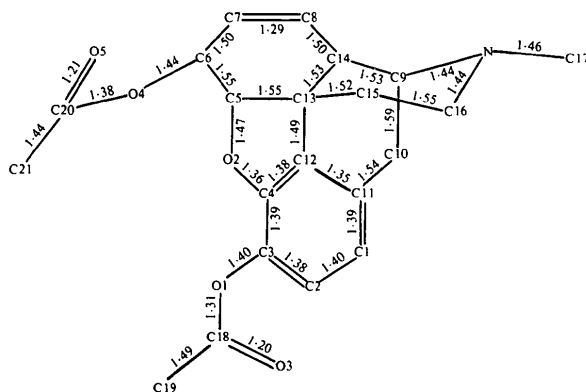

Fig. 2. Bond distances (Å). The estimated average standard deviation in the bond lengths is 0·014 Å.

occurred after the structure was determined. However, the incorrect value was used to calculate the torsion angles and bond lengths reported. The correct bond lengths and torsion angles are given in Figs. 1 and 2. The contact distances O(5)–C(19) and O(3)–C(21) reported in the original paper should be 3·222 (25) and 3·289 (20) Å respectively. Corrected bond angles have been deposited.*

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* A list of corrected bond angles has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36164 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.