displacements of −0·013 (4) [C(12)] and 0·055 (2) Å [O(11)]. Although the oxime group also lies essentially within this plane, a small amount of twist about the C(7)–C(1) bond is evident from the calculated displacements of C(7) [0·015 (3) Å], N(8) [0·064 (3) Å] and O(9) [0·069 (3) Å]. The overall planarity of the molecule is also demonstrated in that the largest deviation of an atom from the mean plane calculated for all 12 non-hydrogen atoms is only 0·042 (2) Å [O(11)].

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1,3-Dioxole-2-spiro-4′-(3′,3′-diethylpyrrolidin-2′-one)

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Abstract. $C_9H_{17}NO_3$, orthorhombic, *Pbca*, $a = 14\cdot164$ (4), $b = 15\cdot128$ (4), $c = 10\cdot012$ (4) Å, $M_r = 187\cdot2$, $Z = 8$, $D_x = 1\cdot16$ g cm^{-3}. The molecules are linked into centrosymmetric dimers through N–H···O(=C) hydrogen bonds of length 2·91 Å. The pyrrolidine ring is in the envelope conformation with C(4) 0·467 Å above the plane of the other ring atoms.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings for 15 reflexions ($\pm hkl$) on a Syntex $P2_1$ diffractometer (Cu K, $\lambda = 1\cdot54178$ Å). Intensity measurements were carried out in the θ–2θ mode ($3\cdot5 \leq 2\theta \leq 135\cdot0°$) with graphite-monochromated Cu $K\alpha$ radiation, at scan speeds varying linearly between 2·93 (150 counts s^{-1} and below) and 29·30° min^{-1} (5000 counts s^{-1} and above). Scan and background times were equal. Lorentz and polarization corrections were made but no absorption corrections [μ(Cu K) = 6·3 cm^{-1}] were applied. After application of the acceptance criterion $F > 3\cdot0\sigma(F)$, 1589 unique reflexions were retained for the refinement. The structure was solved by direct methods (*SHELX* 76, G. M. Sheldrick) and refined by full-matrix least squares with anisotropic temperature factors for all the nonhydrogen atoms. The H atom positional parameters were refined with the geometrical constraints d(N–H) = 1·02 ± 0·02, d(C–H) = 1·08 ± 0·02 Å. Group isotropic temperature factors were introduced for the ethyl and 1,3-dioxole ring H atoms. The terminal value of R_G [$= (\Sigma w\Delta^2/\Sigma wF_o^2)^{1/2}$] was 0·057, with R_w ($\Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o|$) 0·056 and R 0·063.‡ The

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‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33191 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

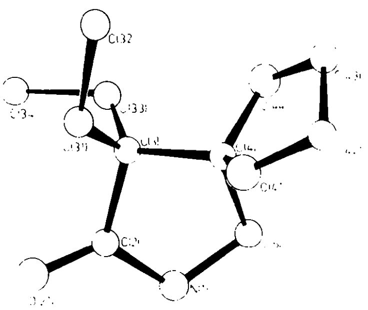


Fig. 1. The molecule in perspective with the numbering system.

Table 1. *Positional parameters* ($\times 10^4$) *for the non-hydrogen atoms*

	x	y	z
N(1)	435 (1)	5029 (1)	6770 (2)
C(5)	776 (2)	5237 (1)	8112 (2)
C(4)	1285 (1)	4381 (1)	8499 (2)
C(3)	775 (1)	3644 (1)	7708 (2)
C(2)	429 (1)	4169 (1)	6494 (2)
O(41)	2247 (1)	4418 (1)	8118 (1)
C(42)	2767 (2)	4713 (2)	9266 (3)
C(43)	2197 (2)	4400 (2)	10415 (3)
O(44)	1275 (1)	4251 (1)	9908 (1)
C(31)	1407 (1)	2878 (1)	7248 (2)
C(32)	1786 (2)	2294 (2)	8378 (3)
C(33)	9898 (2)	3339 (2)	8522 (3)
C(34)	9268 (2)	2671 (2)	7834 (4)
O(21)	169 (1)	3842 (1)	5425 (1)

Table 2. *Positional parameters* ($\times 10^3$) *and isotropic temperature factors* ($\times 10^3$) *for the hydrogen atoms*

	x	y	z	U(Å^2)
H(1)	19 (2)	548 (1)	611 (2)	59 (8)
H(51)	129 (2)	575 (2)	816 (3)	95 (11)
H(52)	21 (1)	538 (2)	877 (2)	47 (8)
H(421)	348 (1)	456 (3)	928 (5)	162 (9)
H(422)	270 (3)	542 (1)	938 (4)	162 (9)
H(431)	210 (3)	481 (2)	1128 (3)	162 (9)
H(432)	248 (3)	378 (2)	1079 (4)	162 (9)
H(311)	199 (1)	319 (2)	671 (2)	64 (4)
H(312)	103 (2)	250 (2)	656 (2)	64 (4)
H(321)	223 (2)	267 (2)	904 (3)	128 (6)
H(322)	124 (2)	196 (2)	893 (4)	128 (6)
H(323)	227 (2)	183 (2)	794 (4)	128 (6)
H(331)	20 (2)	304 (2)	942 (2)	64 (4)
H(332)	939 (2)	386 (1)	877 (3)	64 (4)
H(341)	896 (3)	291 (2)	691 (3)	128 (6)
H(342)	862 (2)	257 (3)	840 (4)	128 (6)
H(343)	967 (3)	211 (2)	760 (5)	128 (6)

weights adopted were those from the counting statistics. Complex neutral-atom scattering factors were employed (Cromer & Waber, 1965; Cromer & Liberman, 1970). The atom numbering is given in Fig. 1. Positional parameters and, for H, isotropic thermal parameters are in Tables 1 and 2.

Discussion. Pyrrolidine-2,4-diones have achieved significance on account of their biological activity and pharmacological applicability, and as partial structures of vitamin B_{12} analogues (Melenteva, Pekel & Berezovskii, 1969). For example, an analgesic and sedative action has been established for 1,5-diphenyl-3-butylpyrrolidine-2,4-dione (Habicht, 1962). Derivatives of the pyrrolidin-2-ones have also been shown to have an important effect on the central nervous system (Lunsford, Cale, Ward, Franko & Jenkins, 1964). As part of a systematic study of pyrrole and succinimide derivatives we have carried out an X-ray analysis of (I) (Engel, 1972).

Table 3. *Bond lengths* (Å)

C(5)–N(1)	1·461 (3)	C(2)–N(1)	1·330 (3)
C(4)–C(5)	1·532 (4)	C(3)–C(4)	1·546 (3)
O(41)–C(4)	1·414 (3)	O(44)–C(4)	1·425 (3)
C(2)–C(3)	1·534 (3)	C(31)–C(3)	1·533 (4)
C(33)–C(3)	1·554 (4)	O(21)–C(2)	1·235 (3)
C(42)–O(41)	1·436 (4)	C(43)–C(42)	1·483 (5)
O(44)–C(43)	1·417 (4)	C(32)–C(31)	1·533 (4)
C(34)–C(33)	1·514 (5)		

Table 4. *Bond angles* (°)

C(2)–N(1)–C(5)	113·8 (2)	C(4)–C(5)–N(1)	101·9 (2)
C(3)–C(4)–C(5)	105·1 (2)	O(41)–C(4)–C(5)	110·5 (2)
O(41)–C(4)–C(3)	109·8 (2)	O(44)–C(4)–C(5)	111·2 (2)
O(44)–C(4)–C(3)	113·8 (2)	O(44)–C(4)–O(41)	106·4 (2)
C(2)–C(3)–C(4)	100·4 (2)	C(31)–C(3)–C(4)	115·3 (2)
C(31)–C(3)–C(2)	109·8 (2)	C(33)–C(3)–C(4)	108·5 (2)
C(33)–C(3)–C(2)	108·3 (2)	C(33)–C(3)–C(31)	113·5 (2)
C(3)–C(2)–N(1)	109·9 (2)	O(21)–C(2)–N(1)	125·1 (2)
O(21)–C(2)–C(3)	125·0 (2)	C(42)–O(41)–C(4)	106·8 (2)
C(43)–C(42)–O(41)	104·1 (3)	O(44)–C(43)–C(42)	105·8 (3)
C(43)–O(44)–C(4)	108·9 (2)	C(32)–C(31)–C(3)	114·7 (2)
C(34)–C(33)–C(3)	115·5 (3)		

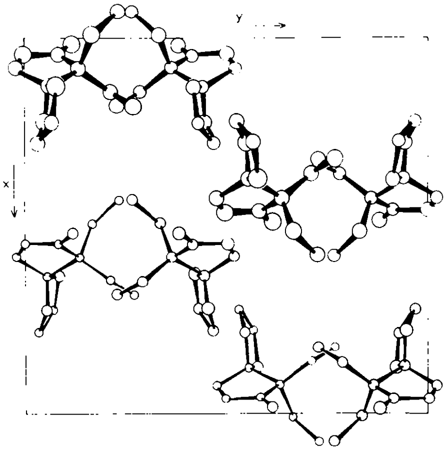
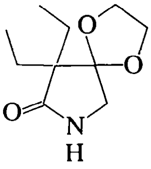


Fig. 2. Projection of the unit cell perpendicular to [001].



(I)

The molecules of (I) are linked into centrosymmetric dimers through two N–H···O(=C) hydrogen bonds of length 2·91 Å. The pyrrolidine ring is in the envelope conformation with C(4) 0·467 Å above the least-squares plane through the other ring atoms [unit weights, distances from the plane: N(1) −0·005, C(2) 0·005, C(3) −0·003, C(5) 0·003, O(21) −0·001 Å]. A similar conformation is adopted by the 1,3-dioxole ring [unit weights, distances from the plane: C(4) −0·018, O(41) −0·395, C(42) 0·017, C(43) −0·028, O(44) 0·029 Å]. Bond lengths and angles are normal (Tables 3 and 4). As a result of π interaction the N(1)—C(2) distance of 1·330 (3) Å is much shorter than the N(1)—C(5) distance of 1·461 (3) Å. A projection of the unit cell perpendicular to [001] is given in Fig. 2.

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5,5′-Dibromo-3,3′-diethyl-4,4′-dimethyl-2,2′-pyrromethene Hydrobromide

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Abstract. $C_{15}H_{19}N_2Br_2^+Br^-$, monoclinic, $C2/c$, a = 16·140 (8), b = 8·871 (6), c = 12·537 (8) Å, β = 106·57 (5)°, M_r = 467·0, D_x = 1·73 g cm^{-3}, Z = 4. The molecule contains a space-group C_2 axis with the Br$^-$ anion in a disordered site ±0·793 Å from the best least-squares plane of the pyrromethene system. The chromophore displays a slightly twisted *syn-Z* conformation (interplanar angle 13·0°) with a wide methine bridge angle of 133·8 (6)°. The disordered Br$^-$ anions are each involved in two non-linear N—H···Br hydrogen bonds of length 3·228 and 3·225 Å.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings for 15 reflexions (±hkl) on a Syntex $P2_1$ diffractometer (Cu K, λ = 1·54178 Å). Intensity measurements were carried out in the θ–2θ mode ($3·5 \leq 2\theta \leq 135·0°$) with graphite-monochromated Cu $K\alpha$ radiation, at scan speeds varying linearly between 2·93 (150 counts s^{-1} and below) and 29·30° min^{-1} (1500 counts s^{-1} and above). Scan and background times were equal. Lorentz, polarization and a numerical absorption correction [μ(Cu $K\alpha$) = 81·2 cm^{-1}; crystal size: 0·106 × 0·088 × 1·2 mm] were applied. After application of the rejection criterion $I \leq 2·0\sigma(I)$, 1420 unique reflexions were retained for the refinement. The structure was solved by Patterson and difference syntheses and refined by full-matrix least squares with anisotropic temperature factors for all the non-hydrogen atoms. The Br$^-$ anion [Br(1)] occupies a disordered site. As a result of the shortness of the distance between these C_2 symmetry-related sites [1·590 Å] Br(1) has unrealistically high values of U_{11},

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