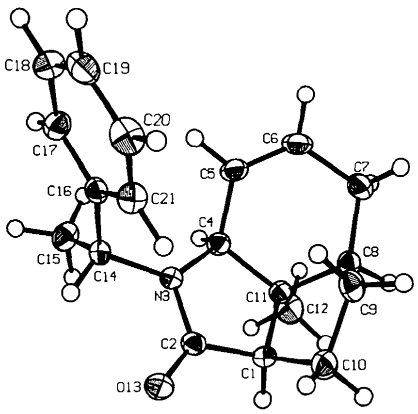

Fig. 1. View showing atom-labelling scheme. Thermal ellipsoids are scaled to the 50% probability level.

thermal parameters for non-H atoms are listed in Table 1, while bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1. The least-squares-planes program was supplied by Cordes (1983); other computer programs from reference 11 of Gadol & Davis (1982).

Related literature. (1) is a synthetic precursor to the naturally occurring alkaloid dendrobine. The tricyclic ring system of (1) with various functional groups has been used as an intermediate in the total synthesis of dendrobine (Inubushi, Kikuchi, Ibuka, Tanaka, Saji & Tokane, 1972; Yamada, Suzuki, Hayakawa, Aoki, Nakamura, Nagase & Hirata, 1972; Kende, Bentley, Mader & Ridge, 1974; Roush, 1980).

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2-(1,2-Dibromoethyl)-1,4-benzodioxan

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Abstract. $C_{10}H_{10}Br_2O_2$, $M_r = 322\cdot00$, monoclinic, $P2_1/c$, $a = 9\cdot4088\ (9)$, $b = 19\cdot616\ (2)$, $c = 5\cdot958\ (1)$ Å, $\beta = 94\cdot61\ (2)°$, $V = 1095\cdot99$ Å^3, $Z = 4$, $D_x = 1\cdot95$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 7\cdot21$ mm^{-1}, $F(000) = 624$, $T = 293$ K, $R = 0\cdot036$ for 1357 reflexions. The crystals examined proved to be the racemate of the *R,S* isomer. The dioxan ring adopts a half-chair conformation with the ethyl group *trans* to the dioxan C—C bond. The Br atoms are *trans* to each other, with the torsion angle Br—C—C—Br = 170·2 (4)°.

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Experimental. White needles, m.p. 378–379 K, available from previous studies. (Schroth, Reinhardt, Kranke & Streichenbach, 1963; Schroth & Werner, 1967). Enraf–Nonius CAD-4F diffractometer, monochromated Mo *Kα* radiation. $2\theta_{\rm max}$ 50°, 2113 reflexions, $+h$ $+k$ $\pm l$. Two check reflexions, no intensity change. Crystal size 0·4 × 0·3 × 0·2 mm, ψ-scan absorption correction, min. and max. correction factors 0·849, 1·223. 1918 unique reflexions ($R_{\rm int}$ = 0·019), 1357 with $F > 3\sigma(F)$ used in refinement. Index range $|h| \leq 10$, $|k| \leq 22$, $|l| \leq 7$. Cell constants refined from 25 reflexions with $\theta \simeq 14°$.

Structure solution by combination of Patterson and direct methods which together located the Br—C—C—Br side chain. Remainder of molecule found during conventional refinement. H atoms located and refined with individual isotropic thermal parameters. All non-H atoms anisotropic.

Final refinement minimizing $\sum w(|F_o|-|F_c|)^2$, 170 refined parameters, R = 0·036, wR = 0·041, w = 1·3645/[$\sigma^2(F)$ + 0·000529F^2], mean shift/e.s.d. = 0·130, max. = 0·463, max., min. $\Delta\rho$ = 0·65 (near Br), −0·39 e Å^{-3}.

Table 1. *Relative atomic coordinates* (× 10⁴) *for non-hydrogen atoms with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$(Å^2 × 10^3)
O1	1927 (4)	4742 (2)	−2779 (6)	46 (1)
C2	2908 (6)	4385 (3)	−1268 (8)	43 (1)
C3	3024 (7)	4736 (3)	1018 (9)	58 (2)
O4	3590 (5)	5405 (2)	796 (6)	60 (1)
C5	3200 (6)	6455 (3)	8873 (11)	55 (2)
C6	2605 (6)	6830 (3)	−2873 (11)	60 (2)
C7	1783 (6)	6507 (3)	−4580 (10)	60 (2)
C8	1565 (6)	5817 (3)	−4543 (9)	49 (1)
C9	2988 (6)	5760 (3)	8971 (9)	45 (1)
C10	2160 (5)	5439 (3)	−2770 (8)	43 (1)
C11	2465 (5)	3648 (3)	−1197 (9)	44 (1)
Br12	654 (1)	3543 (1)	196 (1)	61 (1)
C13	2253 (6)	3305 (3)	−3438 (9)	46 (1)
Br14	4067 (1)	3228 (1)	−4820 (1)	54 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°) *and selected torsion angles* (°)

C2—O1	1·420 (6)	C10—O1—C2	112·9 (4)
C10—O1	1·385 (6)	C3—C2—O1	109·9 (4)
C3—C2	1·522 (7)	C11—C2—O1	108·9 (4)
C11—C2	1·505 (7)	C11—C2—C3	114·1 (4)
O4—C3	1·426 (7)	O4—C3—C2	109·5 (4)
C9—O4	1·375 (6)	C9—O4—C3	114·0 (4)
C6—C5	1·358 (9)	C9—C5—C6	121·1 (5)
C9—C5	1·379 (8)	C7—C6—C5	119·2 (6)
C7—C6	1·381 (9)	C8—C7—C6	121·1 (6)
C8—C7	1·369 (9)	C10—C8—C7	119·6 (5)
C10—C8	1·374 (7)	C5—C9—O4	118·9 (5)
C10—C9	1·396 (7)	C10—C9—O4	121·9 (5)
Br12—C11	1·965 (5)	C10—C9—C5	119·2 (5)
C13—C11	1·494 (7)	C8—C10—O1	118·5 (4)
Br14—C13	1·959 (6)	C9—C10—O1	121·8 (4)
		C9—C10—C8	119·7 (5)
		Br12—C11—C2	111·4 (4)
		C13—C11—C2	115·1 (4)
		C13—C11—Br12	105·8 (3)
		Br14—C13—C11	110·6 (4)
O1—C2—C3—O4	−63·4 (4)	C3—C2—C11—Br12	55·9 (3)
C3—C2—C11—C13	176·3 (3)	Br12—C11C13—Br14	−170·2 (4)

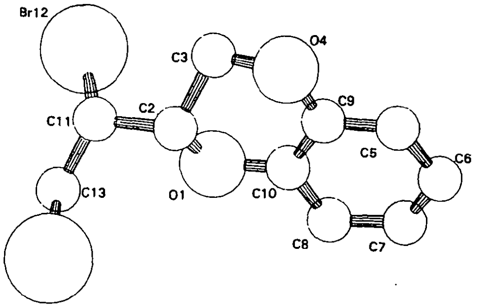

Fig. 1. View of a molecule of the title compound with atom numbering.

Programs used *SHELX76* (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *SHELX76*. Final atomic coordinates are given in Table 1, bond lengths and angles in Table 2.* The molecular structure is illustrated in Fig. 1.

Related literature. Previous work showed that the preparative route used gave a mixture of the racemic *R,S* and *R,R* diastereoisomers (Schroth *et al.*, 1963). The present determination has identified the isomer melting at 378–379 K as *R,S* and hence that melting at 336–337 K is the *R,R* isomer. The dimensions of the benzodioxan moiety are very close to those found recently in bis(1,4-benzodioxan)silver(I) perchlorate (Barnes & Blyth, 1985).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44362 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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