parameters refined, $R = 0.069$, $wR = 0.070$, unit weights. Max. Δ/σ 0·01, max. difference map peak 0·22, min. −0·24 e Å^{-3}. In view of the high temperature factors for atoms C91 and O9 [largest values O9: U_{33} 0·247 Å^2, U_{22} 0·203 Å^2; C91: U_{33} 0·146 Å^2] the electron density in the vicinity was examined closely for indications of statistical disorder of these atoms. There were no such indications. Other programs used were *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Scattering factors from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction.

Discussion. Atomic numbering is shown in Fig. 1. Atomic coordinates of the non-hydrogen atoms are given in Table 1,* with bond lengths in Table 2. There are two hydrogen bonds in the structure both involving O2 and O4. O4⋯O2(2−x, −y, $\frac{1}{2}$+z) 2·81 (2) Å and O2⋯O4(x, y, −1+z) 2·74 (2)Å. O9 is not involved in any close contacts. The cyclohexane ring has a chair conformation. Fig. 2 is a view of the unit cell showing hydrogen bonding.

The authors wish to thank Dr J. Meinwald for supplying the compound and for his continuing interest.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43504 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
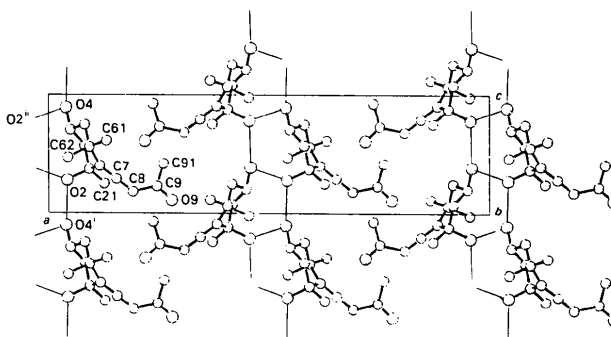

Fig. 2. View of unit cell along b showing hydrogen bonding as thin lines. Symmetry codes: (i) $x, y, -1+z$; (ii) $2-x, -y, \frac{1}{2}+z$.

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Molecular Structure Analysis of Benzamide Neuroleptics and Analogs. XI.
exo-2,3-Dihydro-N-(8-benzyl-8-azabicyclo[3.2.1]oct-3-yl)-1,3-benzodioxole-5-carboxamide and exo-2,3-Dihydro-N-(8-benzyl-8-azabicyclo[3.2.1]oct-3-yl)-1,4-benzodioxin-4-carboxamide

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Abstract. (I) $C_{22}H_{24}N_2O_3$. $M_r = 364·4$, monoclinic, $P2_1/n$, $a = 12·833$ (5), $b = 12·012$ (6), $c = 12·515$ (5) Å, $\beta = 101·12$ (3)°, $V = 1893·0$ Å^3, $Z = 4$, $D_x = 1·28$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0·71069$ Å, $\mu = 0·49$ cm^{-1}, $F(000) = 776$, $R = 0·04$ for 1720 reflections. (II) $C_{22}H_{25}N_2O_3^+.Cl^-.H_2O$. $M_r = 418·9$, mono-clinic, $P2_1/c$, $a = 15·580$ (1), $b = 6·865$ (1), $c = 19·928$ Å, $\beta = 104·85$ (1)°, $V = 2060·2$ Å^3, $Z = 4$, $D_x = 1·35$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1·54178$ Å, $\mu = 17·94$ cm^{-1}, $F(000) = 888$, $R = 0·04$ for 1929 reflectons. (III) $C_{23}H_{26}N_2O_3$. $M_r = 378·5$, monoclinic, $P2_1/n$, $a = 12·994$ (3), $b = 12·435$ (3), $c = 12·417$ (4) Å, β

$= 102 \cdot 36 \, (2)^\circ$, $V = 1959 \cdot 8 \, \text{Å}^3$, $Z = 4$, $D_x = 1 \cdot 28 \, \text{g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069 \, \text{Å}$, $\mu = 0 \cdot 49 \, \text{cm}^{-1}$, $F(000) = 808$, $R = 0 \cdot 04$ for 1523 reflections. (IV) $C_{23}H_{26}N_2O_3 \cdot C_6H_6O_6$. $M_r = 552 \cdot 6$, triclinic, $P\bar{1}$, $a = 11 \cdot 031 \, (1)$, $b = 14 \cdot 283 \, (2)$, $c = 9 \cdot 839 \, (1) \, \text{Å}$, $\alpha = 98 \cdot 96 \, (1)$, $\beta = 115 \cdot 69 \, (1)$, $\gamma = 90 \cdot 70 \, (1)^\circ$, $V = 1374 \cdot 3 \, \text{Å}^3$, $Z = 2$, $D_x = 1 \cdot 33 \, \text{g cm}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1 \cdot 54178 \, \text{Å}$, $\mu = 7 \cdot 39 \, \text{cm}^{-1}$, $F(000) = 584$, $R = 0 \cdot 04$ for 3045 reflections. All measurements at room temperature. Of these four inactive title compounds only (II) does not exhibit the intrabenzamidic hydrogen bond similar to that occurring in the active benzamides. Bond distances and angles are normal.

Introduction. This work is part of a more general study undertaken on a recent class of neuroleptics: the 3-benzamido-*N*-benzyl nortropanes. These compounds contain two of the main structural features observed in several families of neuroleptics, namely the piperidine ring and the benzamide group.

Unexpectedly, the title compounds exhibit a lack of antipsychotic activity though the bridge between the two methoxy groups of the benzamide moiety is the only difference from tropapride (Durant, de Beys, Collin & Evrard, 1986; Jalfre, Bucher, Dorme, Mocquet & Porsolt, 1983), the well known active nortropane benzamide analogue. Therefore, it seemed of interest to analyse the X-ray crystal structure of these molecules in both protonated and base forms.
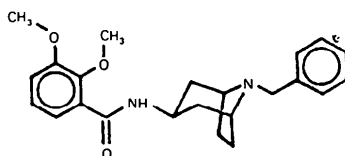
Tropapride
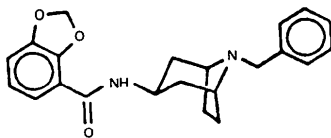
(I) Base

(II) Hydrochloride (+H₂O)
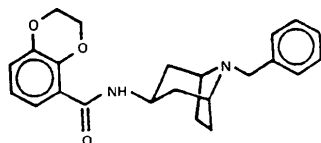
(III) Base

(IV) Fumarate

Experimental. Compound (I) crystallized from an acetone/ethanol mixture at room temperature. Colourless prismatic crystal $0 \cdot 28 \times 0 \cdot 29 \times 0 \cdot 42$ mm for all X-ray measurements. Enraf–Nonius CAD-4 diffractometer. Lattice parameters from least-squares refinement of 25 medium-angle reflections. No absorption correction. No intensity variation of standard reflection. $4 \le 2\theta \le 52^\circ$. 3715 independent reflections measured $(-15 \le h \le 15, \ 0 \le k \le 14, \ 0 \le l \le 15)$, 1720 observed $[I \ge 2 \cdot 5\sigma(I)]$. Direct methods (*MULTAN*80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All non-H atoms found in the best FOM *E* map. Full-matrix least-squares refinement on *F* with *SHELX*76 (Sheldrick, 1976). All H atoms located on difference Fourier map and not refined. Anisotropic temperature factors (U_{ij}) for all non-H atoms and isotropic ones for H atoms (corresponding to the isotropic temperature factors of the carrier atoms incremented by $0 \cdot 02$). $R = 0 \cdot 04$, $wR = 0 \cdot 04$, $w = 1 \cdot 0/[\sigma^2(F) + 0 \cdot 001F^2]$, $(\Delta/\sigma)_{max} = -0 \cdot 329$ $[U_{33}$ of C(27)$]$, $S = 0 \cdot 05$; max. and min. heights in final difference Fourier synthesis $0 \cdot 15$ and $-0 \cdot 01 \, \text{e Å}^{-3}$. Scattering factors from *SHELX*76. The three other compounds were solved with the same X-ray analysis procedure.

Compound (II) crystallized from an acetone solution at room temperature. Colourless prismatic crystal $0 \cdot 33 \times 0 \cdot 10 \times 0 \cdot 03$ mm. $4 \le 2\theta \le 148^\circ$. 4191 independent reflections measured $(0 \le h \le 19, \ -8 \le k \le 0, \ -24 \le l \le 24)$, 1929 observed $[I \ge 2 \cdot 5\sigma(I)]$. All non-H atoms found in the best FOM *E* map (*MULTAN*80). Four H atoms located on difference Fourier map; the others calculated and fixed. $R = 0 \cdot 04$, $wR = 0 \cdot 05$, $w = 1 \cdot 0/[\sigma^2(F) + 0 \cdot 001F^2]$. $(\Delta/\sigma)_{max} = -0 \cdot 477$ $[U_{23}$ of C(4)$]$, $S = 0 \cdot 36$; max. and min. heights in final difference Fourier synthesis $0 \cdot 42$ and $-0 \cdot 30 \, \text{e Å}^{-3}$.

Crystals of compound (III) were obtained from an acetone solution at room temperature. Colourless prismatic crystal $0 \cdot 26 \times 0 \cdot 21 \times 0 \cdot 22$ mm. $4 \le 2\theta \le 52^\circ$. 3843 independent reflections measured $(-16 \le h \le 16, \ 0 \le k \le 15, \ 0 \le l \le 15)$, 1523 observed $[I \ge 2 \cdot 5\sigma(I)]$. All non-H atoms found in the best FOM *E* map (*SHELX*76). H(16) calculated; all the others located on difference Fourier map and fixed. $R = 0 \cdot 04$, $wR = 0 \cdot 05$, $w = 1 \cdot 0/[\sigma^2(F) + 0 \cdot 0005F^2]$. $(\Delta/\sigma)_{max} = -0 \cdot 344$ $[U_{23}$ of C(26)$]$, $S = 0 \cdot 37$; max. and min. heights in final difference Fourier synthesis $0 \cdot 23$ and $-0 \cdot 32 \, \text{e Å}^{-3}$.

Compound (IV) crystallized from an acetone solution at room temperature. Colourless prismatic crystal $0 \cdot 28 \times 0 \cdot 13 \times 0 \cdot 40$ mm. $4 \le 2\theta \le 148^\circ$. 5564 independent reflections measured $(-13 \le h \le 13, \ -17 \le k \le 17, \ 0 \le l \le 12)$, 3045 observed $[I \ge 2 \cdot 5\sigma(I)]$. Direct methods (*DIRDIF*, Beurskens, 1981). All H atoms located on difference Fourier map and refined. $R = 0 \cdot 04$, $wR = 0 \cdot 04$, $w = 1 \cdot 0/[\sigma^2(F) + $

$0.01F^2$]. $(\Delta/\sigma)_{max} = -0.694$ [x of C(13)], $S = 0.42$; max. and min. heights in final difference Fourier synthesis 0.20 and -0.25 e Å^{-3}.
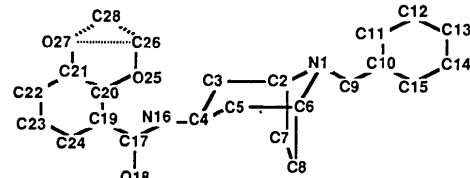
For the four compounds, XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) was used for the molecular geometry analysis.

Table 1. *Final atomic coordinates* ($\times 10^4$) *and* B_{eq} *values with e.s.d.'s in parentheses*

$$B_{eq} = 8\pi^2 U_{eq}(\text{Å}^2); \quad U_{eq} = \tfrac{1}{3}\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}		x	y	z	B_{eq}
Compound (I)					Compound (II)				
N(1)	3038 (1)	9541 (2)	1727 (2)	3.77 (1)	N(1)	1616 (2)	3279 (4)	2277 (1)	3.41 (0)
C(2)	3356 (2)	10309 (2)	2657 (2)	5.09 (1)	C(6)	1616 (2)	5276 (5)	2599 (2)	3.77 (1)
C(3)	4565 (2)	10258 (2)	2969 (2)	5.57 (1)	C(5)	1981 (2)	5036 (5)	3384 (2)	3.86 (1)
C(4)	4935 (2)	9095 (2)	3345 (2)	4.22 (1)	C(4)	2947 (2)	4326 (5)	3554 (2)	3.41 (1)
C(5)	4351 (2)	8210 (2)	2573 (2)	4.44 (1)	C(3)	3060 (2)	2572 (5)	3122 (2)	3.41 (0)
C(6)	3164 (2)	8462 (2)	2282 (2)	4.23 (1)	C(2)	2589 (2)	2958 (5)	2337 (2)	3.40 (1)
C(7)	2811 (2)	9839 (3)	3564 (2)	7.27 (1)	C(8)	2247 (3)	6441 (6)	2267 (2)	4.63 (1)
C(8)	2651 (2)	8597 (3)	3281 (2)	6.08 (1)	C(7)	2873 (2)	4905 (6)	2083 (2)	4.43 (1)
C(9)	1934 (2)	9737 (2)	1160 (2)	4.48 (1)	C(9)	1039 (2)	3250 (6)	1537 (2)	4.46 (1)
C(10)	1850 (2)	10649 (2)	321 (2)	3.86 (1)	C(10)	1005 (2)	1330 (6)	1170 (2)	4.08 (1)
C(11)	886 (2)	11198 (2)	−6 (2)	4.42 (1)	C(11)	430 (2)	−102 (6)	1265 (2)	4.81 (1)
C(12)	765 (2)	11995 (2)	−819 (2)	5.40 (1)	C(12)	327 (3)	−1826 (6)	881 (2)	5.81 (1)
C(13)	1608 (2)	12274 (2)	−1308 (2)	5.82 (1)	C(13)	827 (3)	−2106 (6)	405 (2)	6.25 (1)
C(14)	2573 (2)	11740 (2)	−979 (2)	5.68 (1)	C(14)	1412 (3)	−704 (8)	314 (2)	6.21 (1)
C(15)	2693 (2)	10935 (2)	−177 (2)	4.85 (1)	C(15)	1505 (3)	1044 (6)	694 (2)	5.34 (1)
N(16)	6095 (1)	9027 (2)	3442 (2)	4.71 (1)	N(16)	3275 (2)	3844 (4)	4296 (1)	3.78 (0)
C(17)	6674 (2)	8226 (2)	4031 (2)	4.93 (1)	C(17)	3545 (2)	5263 (5)	4763 (2)	3.74 (1)
O(18)	6274 (1)	7515 (2)	4513 (2)	8.30 (1)	O(18)	3548 (2)	7012 (4)	4599 (1)	5.46 (1)
C(19)	7849 (2)	8241 (2)	4074 (2)	4.98 (1)	C(19)	3855 (2)	4693 (5)	5508 (2)	3.59 (1)
C(20)	8403 (2)	9080 (3)	3687 (2)	5.21 (1)	C(20)	4287 (2)	2983 (5)	5741 (2)	3.44 (1)
C(21)	9489 (2)	9025 (3)	3751 (3)	6.85 (1)	C(21)	4615 (2)	2584 (5)	6443 (2)	3.85 (1)
C(22)	10082 (2)	8125 (4)	4203 (3)	8.66 (1)	C(22)	4510 (2)	3846 (6)	6947 (2)	4.35 (1)
C(23)	9529 (2)	7264 (4)	4579 (3)	8.57 (1)	C(23)	4056 (3)	5577 (6)	6721 (2)	4.68 (1)
C(24)	8446 (2)	7311 (3)	4512 (2)	6.67 (1)	C(24)	3748 (2)	6000 (5)	6030 (2)	4.34 (1)
O(25)	8025 (2)	10059 (2)	3198 (2)	7.03 (1)	O(25)	4471 (2)	1439 (4)	5363 (1)	4.48 (0)
C(26)	8916 (3)	10552 (3)	2811 (3)	8.68 (1)	C(26)	5074 (3)	201 (6)	5851 (2)	4.42 (1)
O(27)	9839 (2)	9960 (2)	3310 (2)	9.44 (1)	O(27)	5006 (2)	769 (4)	6530 (1)	5.43 (1)
					Cl(28)	1009 (1)	68 (2)	3169 (1)	5.29 (0)
					O(29)	2473 (2)	296 (5)	4627 (2)	6.96 (1)
Compound (III)					Compound (IV)				
N(1)	3007 (2)	440 (2)	1766 (2)	3.33 (1)	N(1)	1872 (2)	1769 (1)	3145 (2)	2.86 (1)
C(2)	3106 (3)	1492 (3)	2315 (3)	3.93 (1)	C(2)	2854 (2)	2444 (2)	3013 (3)	2.94 (1)
C(3)	4265 (3)	1796 (3)	2588 (3)	4.19 (1)	C(3)	3044 (3)	3360 (2)	4134 (3)	3.14 (1)
C(4)	4912 (3)	976 (3)	3375 (3)	3.93 (1)	C(4)	3640 (3)	3187 (2)	5796 (3)	3.25 (1)
C(5)	4595 (3)	−173 (3)	3018 (3)	4.72 (1)	C(5)	2898 (3)	2329 (2)	5930 (3)	3.40 (1)
C(6)	3389 (3)	−278 (3)	2711 (3)	4.35 (1)	C(6)	2726 (3)	1484 (2)	4677 (3)	3.53 (1)
C(7)	2648 (3)	1333 (4)	3343 (4)	6.16 (1)	C(7)	4119 (3)	1898 (2)	3445 (3)	3.84 (1)
C(8)	2877 (3)	138 (5)	3643 (3)	6.68 (1)	C(8)	4053 (3)	1281 (2)	4572 (3)	4.31 (1)
C(9)	1922 (3)	207 (3)	1197 (3)	4.08 (1)	C(9)	1304 (3)	899 (2)	1889 (3)	3.79 (1)
C(10)	1843 (3)	−696 (3)	368 (3)	3.44 (1)	C(10)	769 (3)	1142 (2)	306 (3)	3.49 (1)
C(11)	2681 (3)	−977 (3)	−103 (3)	4.25 (1)	C(11)	−413 (4)	1582 (3)	−300 (4)	5.81 (1)
C(12)	2575 (3)	−1772 (3)	−898 (3)	4.67 (1)	C(12)	−911 (5)	1758 (3)	−1776 (5)	8.32 (2)
C(13)	1611 (3)	−2302 (3)	−1252 (3)	4.73 (1)	C(13)	−223 (6)	1500 (3)	−2663 (5)	7.84 (2)
C(14)	777 (3)	−2028 (3)	−778 (3)	4.67 (1)	C(14)	946 (5)	1063 (3)	−2063 (4)	6.48 (1)
C(15)	883 (3)	−1228 (3)	27 (3)	3.81 (1)	C(15)	1437 (3)	881 (2)	−587 (4)	4.60 (1)
N(16)	6049 (2)	1090 (3)	3448 (2)	4.18 (1)	N(16)	3594 (2)	4033 (2)	6825 (3)	3.65 (1)
C(17)	6613 (3)	1890 (4)	4011 (3)	4.54 (1)	C(17)	4525 (2)	4776 (2)	7382 (3)	3.01 (1)
O(18)	6203 (2)	2578 (3)	4486 (3)	9.12 (1)	O(18)	5503 (2)	4795 (1)	7084 (2)	4.51 (1)
C(19)	7780 (3)	1948 (3)	4037 (3)	4.00 (1)	C(19)	4349 (2)	5626 (2)	8375 (3)	2.82 (1)
C(20)	8379 (3)	1123 (3)	3709 (3)	3.58 (1)	C(20)	3343 (2)	5674 (2)	8880 (3)	2.99 (1)
C(21)	9449 (3)	1278 (3)	3762 (3)	4.24 (1)	C(21)	3261 (3)	6524 (2)	9755 (3)	3.50 (1)
C(22)	9923 (3)	2246 (4)	4129 (3)	4.95 (1)	C(22)	4180 (3)	7296 (2)	10161 (3)	3.93 (1)
C(23)	9339 (3)	3066 (4)	4456 (3)	5.13 (1)	C(23)	5200 (3)	7236 (2)	9685 (3)	4.14 (1)
C(24)	8279 (3)	2913 (3)	4413 (3)	4.82 (1)	C(24)	5283 (3)	6418 (2)	8813 (3)	3.68 (1)
O(25)	7895 (2)	176 (2)	3324 (2)	4.71 (1)	O(25)	2434 (2)	4903 (1)	8489 (2)	3.96 (1)
C(26)	8563 (4)	−643 (5)	3095 (6)	12.68 (3)	C(26)	1667 (3)	4942 (2)	9366 (4)	4.21 (1)
O(27)	10074 (2)	502 (2)	3447 (3)	6.43 (1)	O(27)	2279 (2)	6621 (1)	10252 (2)	4.89 (1)
C(28)	9522 (4)	−379 (4)	2898 (6)	11.89 (3)	C(28)	1150 (3)	5908 (2)	9447 (4)	4.78 (1)
					C(29)	4477 (2)	−217 (1)	9367 (2)	3.90 (1)
					C(30)	4279 (2)	−42 (1)	7831 (2)	4.41 (1)
					O(31)	5107 (2)	443 (1)	7673 (2)	6.37 (1)
					O(32)	3170 (2)	−445 (1)	6652 (2)	5.98 (1)
					O(33)	8474 (2)	4704 (1)	7090 (2)	5.92 (1)
					O(34)	6598 (1)	3798 (1)	5490 (2)	5.91 (1)
					C(35)	7887 (2)	4085 (1)	6003 (2)	3.82 (1)
					C(36)	8549 (2)	3575 (2)	5102 (3)	4.22 (1)
					C(37)	8042 (2)	2800 (2)	4081 (2)	4.03 (1)
					C(38)	8832 (2)	2258 (2)	3351 (2)	3.91 (1)
					O(39)	9835 (1)	2696 (1)	3384 (2)	5.17 (1)
					O(40)	8461 (2)	1395 (1)	2798 (2)	5.76 (1)

Table 2. *Atom numbering, bond lengths* (Å) *and valence angles* (°)

	(I)	(II)	(III)	(IV)
N(1)–C(2)	1·479 (3)	1·506 (4)	1·468 (4)	1·501 (4)
C(2)–C(3)	1·526 (4)	1·571 (5)	1·519 (5)	1·522 (4)
C(3)–C(4)	1·520 (3)	1·516 (5)	1·533 (5)	1·538 (4)
C(4)–C(5)	1·532 (3)	1·535 (4)	1·526 (5)	1·519 (5)
C(5)–C(6)	1·527 (4)	1·530 (5)	1·537 (5)	1·529 (4)
C(6)–N(1)	1·465 (3)	1·514 (4)	1·473 (4)	1·515 (3)
C(2)–C(7)	1·550 (4)	1·534 (5)	1·533 (7)	1·533 (4)
C(7)–C(8)	1·538 (5)	1·543 (6)	1·545 (8)	1·544 (5)
C(6)–C(8)	1·530 (4)	1·542 (6)	1·542 (6)	1·540 (3)
N(1)–C(9)	1·476 (3)	1·518 (4)	1·464 (4)	1·516 (4)
C(9)–C(10)	1·507 (3)	1·502 (6)	1·512 (5)	1·507 (4)
C(10)–C(11)	1·392 (3)	1·377 (6)	1·386 (6)	1·384 (5)
C(11)–C(12)	1·384 (3)	1·397 (6)	1·383 (5)	1·379 (6)
C(12)–C(13)	1·383 (4)	1·386 (7)	1·401 (5)	1·399 (9)
C(13)–C(14)	1·385 (3)	1·369 (7)	1·382 (6)	1·369 (7)
C(14)–C(15)	1·381 (3)	1·406 (7)	1·396 (5)	1·382 (5)
C(10)–C(15)	1·392 (4)	1·386 (6)	1·395 (5)	1·385 (6)
C(4)–N(16)	1·472 (3)	1·473 (4)	1·469 (5)	1·467 (4)
N(16)–C(17)	1·346 (3)	1·338 (4)	1·340 (5)	1·336 (3)
C(17)–O(18)	1·216 (3)	1·245 (4)	1·224 (6)	1·236 (4)
C(17)–C(19)	1·497 (4)	1·491 (5)	1·511 (6)	1·506 (4)
C(19)–C(20)	1·374 (4)	1·374 (5)	1·400 (6)	1·397 (4)
C(20)–C(21)	1·382 (4)	1·386 (5)	1·391 (6)	1·403 (4)
C(21)–C(22)	1·379 (5)	1·369 (6)	1·384 (6)	1·374 (4)
C(22)–C(23)	1·387 (6)	1·397 (6)	1·383 (7)	1·393 (5)
C(23)–C(24)	1·377 (4)	1·368 (5)	1·380 (6)	1·367 (4)
C(19)–C(24)	1·405 (4)	1·415 (5)	1·396 (5)	1·401 (4)
C(20)–O(25)	1·370 (4)	1·374 (5)	1·372 (4)	1·368 (3)
O(25)–C(26)	1·450 (5)	1·443 (5)	1·406 (7)	1·443 (5)
C(21)–O(27)	1·364 (4)	1·378 (4)	1·372 (5)	1·369 (4)
O(27)–C(26)	1·419 (4)	1·438 (5)	–	–
O(27)–C(28)	–	–	1·404 (6)	1·442 (3)
C(26)–C(28)	–	–	1·360 (9)	1·503 (4)
C(2)–N(1)–C(6)	101·0 (2)	102·0 (2)	100·9 (2)	101·7 (2)
C(2)–N(1)–C(9)	111·8 (2)	114·1 (3)	112·3 (3)	115·7 (2)
C(6)–N(1)–C(9)	112·1 (2)	111·1 (2)	112·9 (3)	110·6 (2)
N(1)–C(2)–C(3)	106·8 (2)	107·9 (3)	108·0 (3)	107·3 (2)
N(1)–C(2)–C(7)	104·7 (2)	102·5 (2)	105·1 (3)	102·5 (2)
C(3)–C(2)–C(7)	112·2 (2)	112·0 (3)	112·9 (3)	113·7 (2)
C(2)–C(3)–C(4)	110·9 (2)	109·8 (3)	111·0 (3)	111·4 (2)
C(3)–C(4)–N(16)	109·1 (2)	109·3 (3)	112·5 (3)	110·6 (2)
C(3)–C(4)–C(5)	110·9 (2)	112·1 (2)	111·1 (3)	111·3 (2)
N(16)–C(4)–C(5)	112·2 (2)	111·2 (3)	108·3 (3)	110·1 (3)
C(4)–C(5)–C(6)	110·9 (2)	110·2 (3)	110·4 (3)	110·6 (3)
N(1)–C(6)–C(5)	107·6 (2)	107·2 (2)	107·3 (3)	107·6 (2)
N(1)–C(6)–C(8)	105·5 (2)	102·6 (3)	105·6 (3)	102·4 (2)
C(5)–C(6)–C(8)	113·1 (2)	112·6 (3)	111·5 (3)	113·0 (2)
C(2)–C(7)–C(8)	104·1 (2)	105·3 (3)	103·6 (3)	104·8 (3)
C(6)–C(8)–C(7)	103·4 (2)	105·0 (3)	103·5 (3)	105·1 (2)
N(1)–C(9)–C(10)	112·6 (2)	115·0 (3)	113·2 (3)	112·7 (2)
C(9)–C(10)–C(11)	119·3 (2)	120·1 (4)	122·0 (3)	121·1 (3)
C(9)–C(10)–C(15)	122·3 (2)	120·2 (4)	119·0 (4)	119·5 (3)
C(11)–C(10)–C(15)	118·3 (2)	119·6 (4)	119·0 (3)	119·3 (3)
C(10)–C(11)–C(12)	120·7 (2)	121·0 (4)	121·0 (3)	119·9 (4)
C(11)–C(12)–C(13)	120·4 (2)	119·2 (4)	120·3 (4)	120·3 (4)
C(12)–C(13)–C(14)	119·1 (2)	120·2 (4)	118·7 (3)	119·6 (4)
C(13)–C(14)–C(15)	120·6 (2)	120·6 (4)	121·0 (3)	119·8 (5)
C(14)–C(15)–C(10)	120·7 (2)	119·4 (4)	120·0 (4)	120·9 (3)
C(4)–N(16)–C(17)	121·9 (2)	120·0 (3)	122·3 (3)	122·8 (3)
N(16)–C(17)–O(18)	122·1 (2)	122·8 (3)	121·7 (3)	122·3 (3)
N(16)–C(17)–C(19)	117·2 (2)	117·7 (3)	118·7 (4)	118·5 (2)
O(18)–C(17)–C(19)	120·6 (2)	119·5 (3)	119·6 (4)	119·2 (2)
C(17)–C(19)–C(20)	125·5 (2)	124·6 (3)	125·2 (3)	125·0 (2)
C(17)–C(19)–C(24)	118·3 (2)	119·8 (3)	116·1 (4)	116·1 (3)
C(20)–C(19)–C(24)	116·1 (2)	115·6 (3)	118·7 (3)	118·8 (3)
C(19)–C(20)–C(21)	121·9 (3)	122·2 (3)	119·8 (3)	119·3 (2)
C(19)–C(20)–O(25)	128·7 (2)	128·7 (3)	119·2 (3)	119·9 (2)
C(21)–C(20)–O(25)	109·4 (3)	109·1 (3)	120·9 (3)	120·8 (3)
C(20)–C(21)–C(22)	122·1 (3)	122·2 (3)	120·4 (4)	120·9 (3)
C(20)–C(21)–O(27)	110·1 (3)	110·0 (3)	122·4 (3)	122·0 (2)

Table 2 (*cont.*)

	(I)	(II)	(III)	(IV)
C(22)–C(21)–O(27)	127·7 (3)	127·7 (3)	117·1 (3)	117·1 (2)
C(21)–C(22)–C(23)	116·5 (3)	116·5 (3)	120·3 (4)	119·5 (3)
C(22)–C(23)–C(24)	121·7 (4)	121·6 (4)	119·5 (4)	120·3 (3)
C(23)–C(24)–C(19)	121·6 (3)	121·9 (3)	121·3 (4)	121·1 (3)
C(20)–O(25)–C(26)	105·5 (2)	106·0 (2)	115·8 (3)	114·8 (2)
O(25)–C(26)–O(27) C(28)	107·0 (3)	106·4 (3)	119·4 (5)	108·8 (3)
C(21)–O(27)–C(26) C(28)	106·2 (2)	105·3 (3)	114·5 (3)	113·7 (2)
O(27)–C(28)–C(26)	–	–	119·6 (6)	109·3 (2)

Discussion. Atomic coordinates and thermal parameters are given in Table 1.* Table 2 shows the atom numbering, bond lengths and valence angles.

Three of the four compounds here reported show an intramolecular hydrogen bond between N(16) and O(25), leading to the formation of a virtual six-membered ring as observed in the active nortropane benzamides (Collin, Durant & Evrard, 1986; Collin, Evrard & Durant, 1986). [(I): N(16)···O(25) 2·839 (3), H(16)···O(25) 2·009 (3) Å, N(16)–H(16)···O(25) 134·9 (1)°; (III): N(16)···O(25) 2·687 (4), H(16)··· O(25) 1·773 (3) Å, N(16)–H(16)···O(25) 138·5 (2)°; (IV): N(16)···O(25) 2·662 (4), H(16)···O(25) 1·970 (40) Å, N(16)–H(16)···O(25) 133·9 (26)°.]

In compound (II), the amidic nitrogen atom N(16) is engaged in a hydrogen bond with the water molecule [N(16)···O(29) 2·890 (5), H(16)···O(29) 2·053 (4) Å, N(16)–H(16)···O(29) 144·2 (2)°]. This water molecule is bonded to Cl(28) [O(29)···Cl(28) 3·205 (4), H(291)···Cl(28) 2·459 (2) Å, O(29)– H(291)···Cl(28) 153·1 (3)°]. The resulting geometry prevents the formation of an intramolecular hydrogen bond between the amide and the methoxy oxygen atom [N(16)–H(16)···O(25)].

The network of intermolecular hydrogen bonds is given in Table 3 and Fig. 1. For the non-protonated forms, the crystal packing results only in van der Waals interactions. For compound (II), molecules are linked together by H bonds between N(1) and N(16) *via* hydrochloric acid and water molecules; for compound (IV), by means of 1·5 fumaric acid molecules present in the asymmetric unit.

No particular comments need be made about bond distances and valence angles.

The molecules can be described by means of four planes. In Table 4 the dihedral angles between these planes and the characteristic distances are compared with those of tropapride. These data show that a similar disposition of the fragments is observed for both ethylenedioxy compounds [(III) and (IV)], except

Table 3. *Intermolecular bridges*

D—H⋯A	D—H⋯A (°)	D⋯A (Å)	H⋯A (Å)
Compound (II)			
N(1)—H(1)⋯Cl(28)	177·1 (2)	3·127 (3)	2·096 (2)
N(16)—H(16)⋯O(29)	144·2 (2)	2·890 (5)	2·053 (4)
O(29)—H(291)⋯Cl(28)	153·1 (3)	3·205 (4)	2·459 (2)
Compound (IV)			
N(1)—H(1)⋯O(39^i)	176·7 (39)	2·700 (3)	1·862 (39)
O(32)—H(321^{ii})⋯O(40^{ii})	172·2 (29)	2·526 (3)	1·560 (37)
O(34)—H(341)⋯O(18)	176·0 (25)	2·633 (3)	1·658 (35)

Symmetry codes: (i) −1 + x, y, z; (ii) 1 − x, −y, 1 − z.

Table 4. *Distances (Å) between main features and dihedral angles (°) between mean planes*

	(I)	(II)	(III)	(IV)	Tropapride
Distances					
a	3·75	3·83	3·76	3·77	3·86
b	7·80	7·90	7·81	7·84	7·83
c	5·50	5·63	5·54	5·48	5·45
Dihedral angles					
A—D	67·1	71·2	64·3	82·0	87·3
B—D	45·8	31·5	43·6	57·7	49·0
B—C	10·2	31·9	13·5	5·6	14·5

Fig. 2. Stereoscopic view of a rigid fitting between (III) (full line) and (IV) (dotted line) obtained by superposition of the N(1), C(2), C(3), C(4), C(5) and C(6) atoms (Lejeune, Michel & Vercauteren, 1984).

that the benzamide and benzyl groups can lie either side of the piperidine bisector plane (Fig. 2). On the other hand, the two methylenedioxy compounds [(I) and (II)] are differently oriented because of the absence of the intrabenzamidic hydrogen bond in the protonated form (II) [dihedral angle B—C: 10·2° for (I) and 31·9° for (II)]. Finally we observe the similarity between the two bases (I) and (III).

To conclude, it appears that both methylenedioxy and ethylenedioxy analogues can, like 'tropapride', form the characteristic intrabenzamidic H bond. Hence the lack of *in vivo* activity of these analogues cannot be explained in this way.
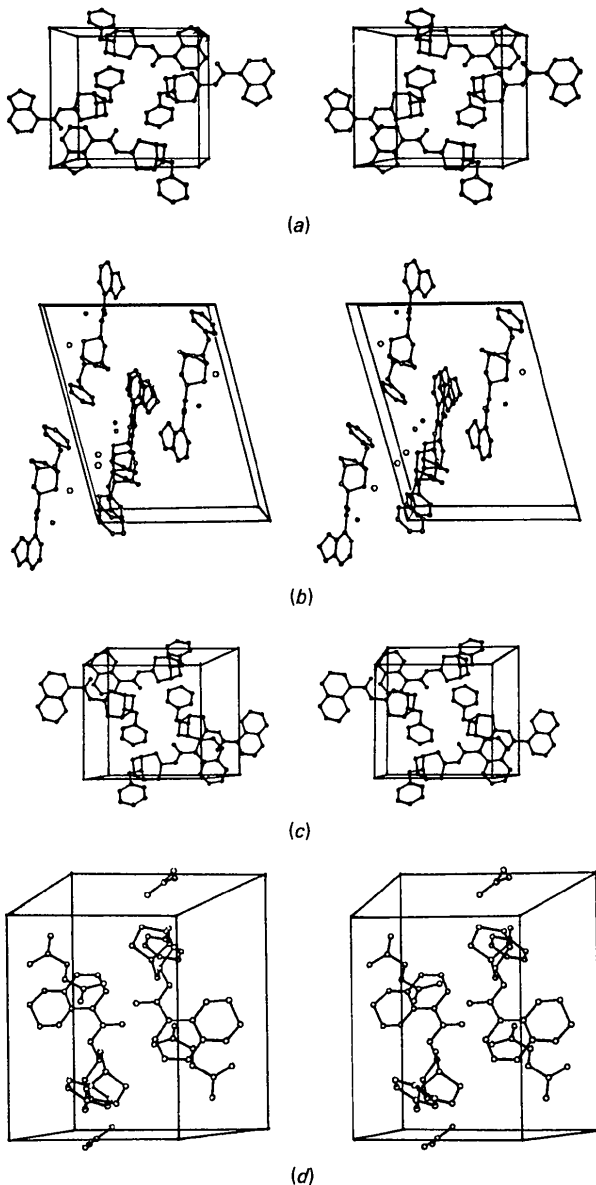
Fig. 1. Stereoviews of molecular conformations and crystal packings: (a) compound (I), (b) compound (II), (c) compound (III), (d) compound (IV).

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Structure of (1R)-1,8,8-Trimethyl-3-oxabicyclo[3.2.1]octane-2,4-dione (Camphoric Anhydride)

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Abstract. $C_{10}H_{14}O_3$, $M_r = 182 \cdot 22$, orthorhombic, $P2_12_12_1$, $a = 6 \cdot 486$ (1), $b = 11 \cdot 203$ (2), $c = 13 \cdot 027$ (2) Å, $V = 946 \cdot 6$ Å^3, $Z = 4$, $D_x = 1 \cdot 278$ (1) Mg m^{-3}, λ(Cu $K\alpha$) $= 1 \cdot 5418$ Å, μ(Cu $K\alpha$) $= 0 \cdot 778$ mm^{-1}, $F(000) = 392$, $T = 290$ (1) K, $R = 0 \cdot 034$ for 905 observed independent reflections. The six-membered ring shows a half-boat conformation with marked non-planarity of the anhydride moiety.

Introduction. Knowledge of the ring geometry is indispensable for interpretation of the NMR spectra of glutaric anhydrides (Koer, de Hoog & Altona, 1975). The existence of large differences among ring torsion angles points to the strong influence of the substituents on the conformation of these compounds (Hašek, 1985). Changes of the ring and chromophore geometry are responsible for the strong substituent and solvent dependence of circular dichroism (CD) spectra of cyclic anhydrides. Interesting examples of this are the camphoric acid derivatives with a bicyclic skeleton for which the CD curves are strongly solvent dependent (Połoński, 1983). The structure of the bicyclic title compound was required for the analysis of its CD spectrum.

Experimental. Crystals of the title compound were grown from toluene. Unit-cell parameters were determined from oscillation and Weissenberg photographs, which showed orthorhombic symmetry with systematic absences $h00$ for h odd, $0k0$ for k odd and $00l$ for l odd, indicating space group $P2_12_12_1$. A needle-like crystal of dimensions $0 \cdot 75 \times 0 \cdot 25 \times 0 \cdot 20$ mm was used for data collection on a Stoe automatic four-circle diffractometer. Accurate cell parameters were obtained from a least-squares fit of θ values for 43 high-order reflections. The following data collection regime was applied: ω–2θ scan, 55 steps of $0 \cdot 2°$, count time per step $0 \cdot 3$–$1 \cdot 8$ s, $\theta_{max} = 60°$ ($0 \le h \le 7$, $0 \le k \le 12$, $0 \le l \le 14$), two standard intensities ($00\bar{6}$ and 040) were checked every 90 min with no significant variation of their intensities. 926 unique reflections were measured of which 21 with $I < 2\sigma(I)$ were treated as unobserved in subsequent calculations. Lorentz and polarization but no absorption corrections were applied.

The structure was solved by direct methods and refined by full-matrix least squares using the *XTAL* package of programs (Stewart & Hall, 1983) on a VAX computer. The correct enantiomer was chosen on the basis of the known chirality of natural camphor, from which the compound was synthesized. H-atom positions were taken from a difference Fourier map and refined individually with isotropic temperature factors. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Minimized function $\sum w(F_o - F_c)^2$, with final values of $R = 0 \cdot 034$ and $wR = 0 \cdot 033$; $w = 1/\sigma^2(F)$. Ratio of maximum parameter shift to e.s.d. in the last cycle of refinement was $0 \cdot 02$ for non-hydrogen atoms and $0 \cdot 05$