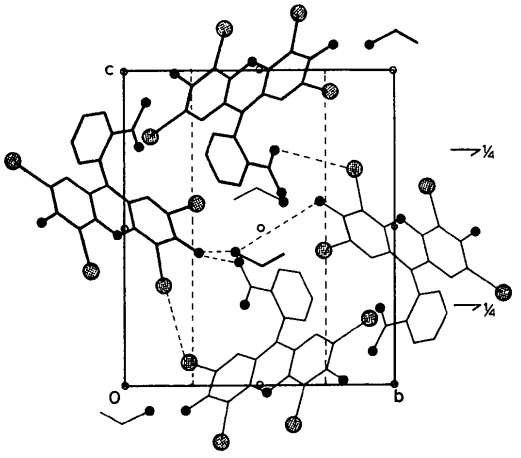

Fig. 2. Crystal packing of erythrosine B ethanolate projected along the *a* axis. The filled circles are oxygen and the stippled circles are iodine. The hydrogen bonding and short intermolecular I···I and I···O contacts are shown.

observed in the crystal structure of fluorescein perchlorate (Dubost, Leger, Colleter, Levillain & Fompeydie, 1981) and its lactoid form (Osborn & Rogers, 1975). The geometry of the xanthene ring system is consistent with a tautomer form in which the dye is a free acid with a keto oxygen at O(2) and a hydroxyl oxygen at O(8). The xanthene ring is folded 6° along the O(10)–C(5) bond and is more puckered than the perchlorate structure (2°). The carboxylic acid group is nearly coplanar (8°) with the benzene ring. This conformation is the same as that observed in the structure of fluorescein (Dubost *et al.*, 1981) and as found in the protein structure of the lactate dehydrogenase–erythrosine B binary complex (Wassarman & Lentz, 1971).

There are hydrogen bonds between O(8) of the dye and the hydroxyl oxygen of ethanol and between the carboxylic oxygen O(171) and O(172). As is frequently observed in thyroid hormone structures (Cody, 1980), there are short I···I [3·825 (1)] and I···O [3·196 (1) Å] van der Waals contacts in this structure (Fig. 2).

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Structures of Two Inactive Nifedipine Analog Calcium Channel Antagonists: (I) $C_{17}H_{17}N_3O_4$ and (II) $C_{19}H_{20}N_2O_6$

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Abstract. (I): Ethyl 5-cyano-2,6-dimethyl-4-(2-nitrophenyl)-1,4-dihydropyridine-3-carboxylate, $C_{17}H_{17}N_3O_4$, $M_r = 327{\cdot}34$, monoclinic, $P2_1/c$, $a = 7{\cdot}983$ (2), $b = 15{\cdot}614$ (4), $c = 13{\cdot}405$ (4) Å, $\beta = 100{\cdot}54$ (3)°, $V = 1643$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}32$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 0{\cdot}90$ cm^{-1}, $F(000) = 688$, $T = 295$ K, $R = 0{\cdot}051$ for 2319 observed reflections. (II): Dimethyl 2,6-dimethyl-4-[*trans*-(2-nitrophenyl)-

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ethylene]-1,4-dihydropyridine-3,5-dicarboxylate, $C_{19}H_{20}N_2O_6$, $M_r = 372{\cdot}38$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}429$ (1), $b = 29{\cdot}105$ (3), $c = 7{\cdot}534$ (1) Å, $V = 1848{\cdot}2$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}34$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 8{\cdot}01$ cm^{-1}, $F(000) = 784$, $T = 295$ K, $R = 0{\cdot}057$ for 2130 observed reflections. The 1,4-dihydropyridine (1,4-DHP) rings of nifedipine analogs (I) and (II) are both found in the boat conformation with N(1) and C(4) defining the bow and stern positions. The ester groups of both compounds are within ten degrees of coplanarity with their adjacent bonds of attachment to the DHP ring. Although (I) and (II) are inactive as calcium channel agents, these conformational features are similar to those exhibited by the two diametrically opposed agonist and antagonist classes of nifedipine analog drugs.

Introduction. Nifedipine and related 2,6-dimethyl-3,5-dicarboxyalkyl-4-(*ortho*/*meta*-substituted-phenyl)-1,4-dihydropyridine compounds have been shown to possess potent pharmacological activities toward relaxing cardiovascular and smooth muscle tissue (Fleckenstein, 1977; Janis & Triggle, 1983; Lee & Tsien, 1983). These compounds inhibit the normal excitation–contraction coupling in muscle tissue by blocking the flow of calcium ions through plasma membrane channels into the muscle cell. Radioligand binding studies (Bellemann, Ferry, Lubbecke & Glossmann, 1981; Gould, Murphy & Synder, 1982; Ehlert, Itoga, Roeske & Yamamura, 1982; Glossmann, Ferry, Lubbecke, Mewes & Hofmann, 1982; Bolger, Genco, Klockowski, Luchowski, Siegel, Janis, Triggle & Triggle, 1983; Bellemann, Schade & Towart, 1983) have characterized a plasma membrane receptor for these nifedipine analog antagonists which is pharmacologically relevant to the gating of the calcium channel and contractile inhibition. Recent reports have described a new class of nifedipine analog calcium channel agonists in which an ester group has either been replaced by a nitro group (Schramm, Thomas, Towart & Franckowiak, 1983) or linked to the 2-position of the DHP ring to form an adjacent lactone ring (Troug, 1984) as typified by the compounds BAY K 8644 and CGP 28 392 respectively. These agonists have been shown to bind to the same 1,4-DHP calcium channel receptor (Su, Janis & Triggle, 1984; Su, Swamy & Triggle, 1984; Janis, DeRampe, Sarmiento & Triggle, 1984). Direct measurements of calcium channel currents in single excitable cells (Brown, Kunze & Yatani, 1984; Hess, Lansman & Tsien, 1984) indicate that agonists bind to prolong a natural transient open state of the DHP receptor–channel complex while antagonists prefer to bind to the closed state. Structure activity studies of these two structurally similar classes of nifedipine analog compounds are of interest to identify those molecular features which are responsible for evoking these diametrically opposed responses. Two inactive nifedipine analogs, (I) ethyl 5-cyano-2,6-dimethyl-4-(2-nitrophenyl)-1,4-dihydropyridine-3-carboxylate and (II) dimethyl 2,6-dimethyl-4-[*trans*-(2-nitrophenyl)ethylene]-1,4-dihydropyridine-3,5-dicarboxylate were examined to explore to what extent these compounds might adopt molecular features thought to be important for either receptor binding or eliciting an agonist or antagonist response. Chemical formulae for nifedipine, BAY K 8644, CGP 28 392 and compounds (I) and (II) are illustrated in Fig. 1.

Experimental. Nifedipine analogs (I) and (II) were synthesized by standard Hantzsch procedures as reported by Loev, Goodman, Snader, Tedeschi & Macko (1974), both recrystallized from methanol as yellow rectangular plates. Crystal (I), 0·95 × 0·25 × 0·25 mm, Syntex/Nicolet *P*3 automated four-circle diffractometer, Nb-filtered Mo $K\alpha$ radiation, θ–2θ scan ($2 < \theta < 30°$). 4820 unique reflections recorded ($0 < h < 19$, $-1 < k < 22$, $-12 < l < 12$), 2319 with $I > 2\sigma(I)$. Crystal (II), 0·24 × 0·48 × 0·62 mm, Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan ($2 < \theta < 77°$). 2239 unique reflections measured ($0 < h < 10$, $-1 < k < 36$, $0 < l < 9$), 2130 with $I > 2\sigma(I)$. Precise cell constants were determined for each structure by a least-squares fit of 25 carefully centered Bragg angles in the $\sin\theta/\lambda$ range of 0·30 to 0·37 Å^{-1}. The two crystal structures were determined with the use of the *MULTAN* program (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined by full-matrix least squares on F, with weights of $4F_o^2/\sigma^2(I)$, $\sigma(I) = [\sigma(I)^2 + (pF_o)^2]^{1/2}$ and $p = 0{\cdot}03$. H-atom positions determined from electron density difference syntheses were refined isotropically and non-H-atom parameters anisotropically in the final refinement cycles. The C(21) methyl group of analog (I) was found to exhibit a twofold staggered rotational

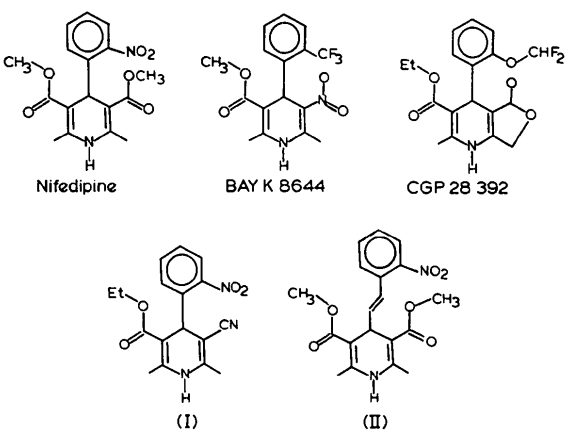

Fig. 1. Chemical structures of calcium channel drugs discussed in text.

disorder with regard to the C(2)–C(21) bond; the occupancy of each rotational conformer was fixed at 0·5 in the refinement. $R = 0{\cdot}051$, $wR = 0{\cdot}062$, $S = 2{\cdot}49$ for 297 variables and 2319 observed data for structure (I) and $R = 0{\cdot}057$, $wR = 0{\cdot}072$, $S = 3{\cdot}81$ for 324 variables and 2130 observed data for structure (II). $(\Delta/\sigma)_{max}$ for the final least-squares cycles were 0·32 and 0·48, and the minimum and maximum final difference electron densities were −0·26 to +0·36 and −0·33 to +0·35 e Å^{-3} respectively. No corrections for absorption or secondary extinction; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Refined fractional atomic coordinates and estimated standard deviations for the two compounds are given in Table 1,* non-hydrogen bond distances and angles in Table 2, and torsion angles for the 1,4-DHP rings are given in Table 3. Orthogonal perspectives of molecules (I) and (II) are shown in Figs. 2 and 3 which in addition indicate the atomic labeling scheme employed in this study.

Discussion. Structure–activity studies of the nifedipine analog calcium channel drugs have indicated the importance of *ortho* and *meta* aryl substitution in

* Lists of structure factors, anisotropic thermal parameters and hydrogen-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43512 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Analog (I)				
N(1)	0·6703 (2)	0·1698 (1)	0·0457 (1)	3·9 (1)
C(2)	0·6463 (3)	0·1910 (1)	0·1412 (2)	3·5 (1)
C(3)	0·6714 (2)	0·1311 (1)	0·2146 (1)	3·1 (1)
C(4)	0·7310 (2)	0·0408 (1)	0·1986 (1)	3·0 (1)
C(5)	0·7248 (2)	0·0242 (1)	0·0859 (1)	3·3 (1)
C(6)	0·6998 (3)	0·0876 (1)	0·0165 (2)	3·4 (1)
C(7)	0·9115 (2)	0·0277 (1)	0·2593 (1)	3·2 (1)
C(8)	1·0445 (3)	0·0727 (2)	0·2294 (2)	4·2 (1)
C(9)	1·2102 (3)	0·0654 (2)	0·2810 (2)	5·4 (1)
C(10)	1·2476 (4)	0·0115 (2)	0·3628 (2)	6·4 (1)
C(11)	1·1212 (4)	−0·0359 (2)	0·3930 (2)	5·9 (1)
C(12)	0·9552 (3)	−0·0276 (2)	0·3410 (2)	4·4 (1)
C(21)	0·5925 (5)	0·2809 (2)	0·1542 (3)	5·2 (1)
C(31)	0·6491 (3)	0·1556 (1)	0·3143 (2)	3·5 (1)
C(51)	0·7461 (3)	−0·0647 (1)	0·0548 (2)	4·0 (1)
C(54)	0·7728 (4)	−0·2094 (2)	0·1140 (3)	5·6 (1)
C(55)	0·9535 (4)	−0·2321 (3)	0·1207 (4)	7·6 (1)
C(61)	0·6982 (4)	0·0785 (2)	−0·0949 (2)	4·8 (1)
N(12)	0·8256 (4)	−0·0805 (2)	0·3764 (2)	6·2 (1)
N(32)	0·6333 (3)	0·1763 (1)	0·3936 (2)	5·0 (1)
O(1)	0·6884 (3)	−0·0483 (2)	0·3833 (2)	7·0 (1)
O(2)	0·8635 (4)	−0·1540 (2)	0·3986 (3)	11·7 (1)
O(52)	0·7559 (3)	−0·0902 (1)	−0·0285 (1)	6·7 (1)
O(53)	0·7504 (2)	−0·1191 (1)	0·1333 (1)	4·8 (1)
Analog (II)				
N(1)	0·6224 (3)	0·0946 (1)	0·4344 (3)	3·1 (1)
C(2)	0·5204 (3)	0·1046 (1)	0·5716 (3)	2·7 (1)
C(3)	0·5735 (2)	0·0998 (1)	0·7417 (3)	2·5 (1)
C(4)	0·7493 (3)	0·0921 (1)	0·7727 (3)	2·6 (1)
C(5)	0·8212 (3)	0·0648 (1)	0·6219 (3)	2·7 (1)
C(6)	0·7619 (3)	0·0698 (1)	0·4555 (3)	2·9 (1)
C(7)	1·0025 (3)	0·2031 (1)	0·7094 (3)	3·3 (1)
C(8)	1·0059 (5)	0·2242 (1)	0·8763 (5)	4·5 (1)
C(9)	1·0665 (6)	0·2682 (1)	0·9011 (6)	5·9 (1)
C(10)	1·1312 (5)	0·2920 (1)	0·7603 (6)	5·6 (1)
C(11)	1·1321 (5)	0·2725 (1)	0·5952 (6)	5·2 (1)
C(12)	1·0690 (4)	0·2286 (1)	0·5699 (4)	4·0 (1)
C(13)	0·8268 (3)	0·1388 (1)	0·7951 (3)	3·0 (1)
C(14)	0·9351 (3)	0·1566 (1)	0·6898 (3)	3·2 (1)
C(21)	0·3598 (4)	0·1205 (1)	0·5110 (4)	4·0 (1)
C(31)	0·4778 (3)	0·1058 (1)	0·9014 (3)	2·8 (1)
C(34)	0·2272 (4)	0·1183 (2)	1·0311 (4)	5·3 (1)
C(51)	0·9638 (3)	0·0371 (1)	0·6549 (4)	3·3 (1)
C(54)	1·1296 (5)	0·0055 (2)	0·8760 (7)	5·9 (1)
C(61)	0·8285 (4)	0·0512 (1)	0·2858 (4)	4·2 (1)
N(12)	1·0800 (4)	0·2093 (1)	0·3880 (4)	5·4 (1)
O(1)	1·0007 (5)	0·1793 (1)	0·3400 (3)	6·7 (1)
O(2)	1·1969 (6)	0·2209 (2)	0·2967 (6)	9·7 (1)
O(32)	0·5350 (2)	0·1062 (1)	1·0496 (2)	3·7 (1)
O(33)	0·3224 (2)	0·1107 (1)	0·8747 (2)	4·3 (1)
O(52)	1·0489 (4)	0·0200 (1)	0·5452 (4)	6·9 (1)
O(53)	0·9918 (3)	0·0327 (1)	0·8927 (3)	4·5 (1)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

	Analog (I)	Analog (II)		Analog (I)	Analog (II)
N(1)–C(2)	1·370 (3)	1·376 (3)	C(8)–C(9)	1·380 (3)	1·391 (5)
N(1)–C(6)	1·374 (3)	1·389 (3)	C(9)–C(10)	1·372 (5)	1·380 (6)
C(2)–C(3)	1·346 (3)	1·365 (3)	C(10)–C(11)	1·371 (5)	1·368 (6)
C(2)–C(21)	1·486 (4)	1·501 (4)	C(11)–C(12)	1·386 (4)	1·396 (5)
C(3)–C(4)	1·516 (3)	1·517 (3)	C(31)–N(32)	1·141 (3)	
C(3)–C(31)	1·432 (3)	1·459 (3)	C(31)–O(32)		1·216 (5)
C(4)–C(5)	1·525 (3)	1·513 (3)	C(31)–O(33)		1·333 (3)
C(4)–C(7)	1·533 (3)		O(33)–C(34)		1·443 (4)
C(4)–C(13)		1·517 (3)	C(51)–O(52)	1·202 (3)	1·202 (4)
C(13)–C(14)		1·315 (3)	C(51)–O(53)	1·349 (3)	1·344 (4)
C(14)–C(7)		1·477 (3)	C(12)–N(12)	1·469 (4)	1·484 (4)
C(5)–C(6)	1·347 (3)	1·357 (3)	N(12)–O(1)	1·224 (4)	1·157 (5)
C(5)–C(51)	1·469 (3)	1·469 (3)	N(12)–O(2)	1·210 (4)	1·248 (6)
C(6)–C(61)	1·498 (3)	1·497 (4)	C(54)–O(53)	1·451 (3)	1·448 (5)
C(7)–C(8)	1·392 (3)	1·399 (4)	C(54)–C(55)	1·472 (5)	
C(7)–C(12)	1·388 (3)	1·403 (4)			
N(1)–C(2)–C(3)	119·2 (2)	118·6 (2)	C(8)–C(7)–C(12)	116·5 (2)	115·7 (2)
N(1)–C(2)–C(21)	115·5 (2)	113·6 (2)	C(8)–C(7)–C(14)		120·0 (2)
N(1)–C(6)–C(5)	120·1 (2)	118·2 (2)	C(12)–C(7)–C(14)		124·3 (2)
N(1)–C(6)–C(61)	113·7 (2)	114·1 (2)	C(4)–C(13)–C(14)		125·7 (2)
C(2)–N(1)–C(6)	123·5 (2)	123·6 (2)	C(13)–C(14)–C(7)		124·5 (2)
C(2)–C(3)–C(4)	123·5 (2)	118·7 (2)	C(5)–C(6)–C(61)	126·3 (2)	127·8 (2)
C(2)–C(3)–C(31)	118·0 (2)	125·5 (2)	C(5)–C(51)–O(52)	127·4 (2)	126·8 (2)
C(4)–C(3)–C(31)	118·4 (2)	115·5 (2)	C(5)–C(51)–O(53)	111·2 (2)	111·3 (2)
C(3)–C(2)–C(21)	125·3 (2)	127·8 (2)	C(7)–C(8)–C(9)	121·8 (2)	122·1 (2)
C(3)–C(4)–C(5)	110·1 (2)	110·7 (2)	C(7)–C(12) C(11)	122·3 (2)	122·2 (2)
C(3)–C(4)–C(7)	109·8 (2)		C(7)–C(12)–N(12)	120·9 (2)	121·1 (2)
C(5)–C(4)–C(7)	111·1 (2)		C(8)–C(9)–C(10)	120·0 (2)	120·3 (3)
C(3)–C(4)–C(13)		107·8 (2)	C(9)–C(10)–C(11)	120·2 (3)	119·5 (3)
C(5)–C(4)–C(13)		112·4 (2)	C(10)–C(11)–C(12)	119·2 (3)	120·1 (3)
C(3)–C(31)–N(32)	178·7 (2)		C(11)–C(12)–N(12)	116·7 (2)	116·6 (2)
C(3)–C(31)–O(32)		122·6 (2)	C(31)–O(33)–C(34)		116·1 (2)
C(3)–C(31)–O(33)		115·6 (2)	O(32)–C(31)–O(33)		121·8 (2)
C(4)–C(5)–C(6)	122·2 (2)	119·4 (2)	C(51)–O(53)–C(54)	117·3 (2)	115·5 (2)
C(4)–C(5)–C(51)	117·2 (2)	119·2 (2)	O(52)–C(51)–O(53)	121·4 (2)	121·9 (2)
C(6)–C(5)–C(51)	120·6 (2)	121·1 (2)	O(53)–C(54)–C(55)	112·0 (2)	
C(4)–C(7)–C(8)	118·2 (2)		C(12)–N(12)–O(1)	119·1 (2)	122·5 (3)
C(4)–C(7)–C(12)	125·3 (2)		C(12)–N(12)–O(2)	117·0 (2)	117·1 (3)
			O(1)–N(12)–O(2)	123·9 (2)	119·2 (3)

Table 3. *Dihydropyridine ring torsion angles* (°)

	Analog (I)	Analog (II)
C(6)–N(1)–C(2)–C(3)	7·9 (3)	15·9 (3)
N(1)–C(2)–C(3)–C(4)	2·0 (3)	11·0 (3)
C(2)–C(3)–C(4)–C(5)	−10·6 (3)	−32·8 (3)
C(3)–C(4)–C(5)–C(6)	11·1 (3)	32·1 (3)
C(4)–C(5)–C(6)–N(1)	−3·2 (3)	−9·3 (3)
C(5)–C(6)–N(1)–C(2)	−7·2 (3)	−16·8 (3)

constraining the orientation of the phenyl ring to lie close to the N(1)–C(4) vertical symmetry plane of the 1,4-DHP ring (Loev *et al.*, 1974; Rodenkirchen, Bayer, Steiner, Bossert, Meyer & Moller, 1979). Diffraction studies (Triggle, Shefter & Triggle, 1980; Fossheim, Swarteng, Mostad, Romming, Shefter & Triggle, 1982) confirmed this intramolecular constraint and furthermore revealed that antagonist potency correlated more strongly with the degree of DHP ring flatness than with the degree of phenyl ring rotational coplanarity with the N(1)–C(4) DHP vertical symmetry plane. In such structures the DHP ring has a shallow boat conformation with N(1) and C(4) defining the stern and bow positions, the aryl group is situated above the bow of the boat in an axial or flagpole orientation with the *ortho* or *meta* substituent almost invariably positioned on the forward or bowsprit side of the phenyl ring. The nifedipine analog agonists BAY K 8644 and CGP 28 392 have also been shown to possess DHP conformations and phenyl ring orientations which are sufficiently similar to the antagonist class of drugs (Langs & Triggle, 1985) to suggest that differentiation between agonists and antagonists might be linked to a small preference in the *cis–trans* conformational equilibrium of the ester group or small differences in the hydrogen-bonding strengths of the DHP amine group. Compounds (I) and (II) were examined primarily to determine whether the inactivity of these compounds might be due to different DHP ring conformations or atypical orientations of the phenyl group which might preclude binding to the 1,4-DHP receptor channel complex.

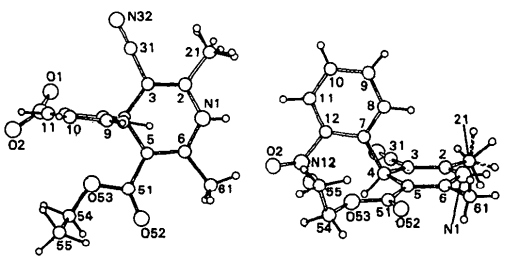

Fig. 2. Orthogonal perspective views of molecule (I) showing the numbering of the atoms. The bare numbers are for C atoms.

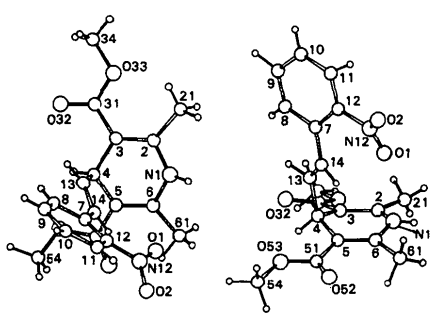

Fig. 3. Orthogonal perpectives and atomic labels for molecule (II).

The molecular features observed for the 3-cyano analog (I) include an extremely flat DHP boat conformation, with C(4) and N(1) defining the bow and stern positions; the sum of the absolute magnitudes of the six DHP ring torsion angles is only 42°. Values of this flatness index compiled for more than 30 crystallographically measured molecules (Langs & Triggle, unpublished observations) range from a minimum of 39 and 56° respectively for the most active nifedipine analog agonist and antagonist to a maximum of 87 and 121° respectively for those which exhibit only marginal potency. The phenyl ring of the 3-cyano analog is within 6° of the vertical bisecting symmetry plane of the DHP ring, compared with 30° shown by the potent antagonist nifedipine (Fossheim *et al.*, 1982). The plane of the ester group is inclined 6·0° from the plane defined by carbonyl carbon and the C(2), C(3) and C(4) atoms of the DHP ring, which compares favorably with the average value of 8·4 ±4·8° determined from 53 observations of ester functions obtained from the crystal structures of nifedipine derivatives (Langs & Triggle, unpublished observations). All these characteristics suggest that the inactivity of (I) may result either from an ability to promote a balanced population of both open and closed states of the calcium channel or an inability to stabilize either of these states by failing to bind to the 1,4-DHP receptor.

In contrast, the *trans*-ethylene analog (II) was found to have an extremely puckered DHP boat conformation with a torsion angle magnitude sum of 118°. This value is exceeded by only one other compound, 2,6-dimethyl-3,5-dicarboxyethyl-4-phenyl-1,4-dihydropyridine, a weak antagonist with a corresponding torsion sum of 121° (Hempel & Gupta, 1978). The *trans*-ethylene group was found to be twisted back over the DHP ring rather than extending outward away from the ring to the outer bowsprit side of the molecule. This orientation avoids a close van der Waals contact between H(13) and atoms O(32) and O(53) which would result were the ethylene group turned to the bowsprit side of the molecule, and allows the DHP ring to pucker more than normal. Large 4-(*ortho*-phenyl) substituents appear to flatten the DHP ring as a result of such contacts, and thereby increase the receptor binding and activity of these calcium channel drugs. Although one may have anticipated that the ethylene linkage of analog (II) would position the phenyl ring too far from the DHP ring and cause a steric obstruction in binding to the 1,4-DHP receptor, the extreme puckering of the DHP ring would be an additional factor affecting such binding.

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tert-Butylammonium Chloride at 115 K

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Abstract. $C_4H_{12}N^+.Cl^-$, $M_r = 109{\cdot}6$, orthorhombic, *Pbca*, $a = 17{\cdot}770\ (8)$, $b = 8{\cdot}877\ (4)$, $c = 8{\cdot}647\ (3)$ Å, $V = 1364{\cdot}0$ Å^3, $Z = 8$, $D_x = 1{\cdot}067$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 1{\cdot}98$ cm^{-1}, $F(000) = 480$, $T = 115$ K, $R = 0{\cdot}043$ for 1073 unique reflections with $I > 2\sigma(I)$. The *tert*-butylammonium ion in this ionic crystal is in its fully staggered conformation (all H atoms were found and their positions refined), and is hydrogen bonded to three neighboring Cl^- ions. The analysis was performed at 298 K as well as 115 K; the structure does not change, but the torsional motion of the *tert*-butyl group about the C–NH_3^+ axis increases appreciably, as observed in other structures, from an r.m.s. amplitude of about 5° to nearly 12°.

Introduction. The *tert*-butylammonium ion is frequently used as a perching guest in host–guest chemistry (Cram & Trueblood, 1981). The present analysis was carried out to provide information on the conformation of this simple ion and on its internal torsional motion (Trueblood & Dunitz, 1983). The current Cambridge Structural Database (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979) reports only ten structures that contain this ion; the only ones that give H-atom parameters are from this laboratory.

Experimental. Crystals were obtained unexpectedly from chloroform–benzene during an attempt at preparation of a *tert*-butylammonium salt of a *para*-cyclophane crown (Helgeson, 1978) and were initially believed to be such a salt. Colorless single crystal, 0·23 × 0·22 × 0·44 mm; Syntex $P\bar{1}$ diffractometer

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