2937 reflections
199 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0650P)^2 + 0.2606P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.70131 (6)	0.30295 (6)	0.83579 (6)	0.0533 (2)
O11	0.6243 (2)	0.1883 (2)	0.9845 (2)	0.0736 (5)
O12	0.8506 (2)	0.3819 (2)	0.8028 (2)	0.0766 (5)
C3	0.6044 (2)	0.5542 (2)	0.6399 (2)	0.0520 (5)
C2	0.4418 (2)	0.6274 (2)	0.6451 (2)	0.0510 (5)
C21	0.4235 (3)	0.7428 (3)	0.6968 (3)	0.0678 (6)
O1	0.3113 (2)	0.51281 (15)	0.7520 (2)	0.0601 (4)
C5	0.3758 (2)	0.3874 (2)	0.8366 (2)	0.0470 (4)
C4	0.5551 (2)	0.4349 (2)	0.8014 (2)	0.0459 (4)
O6	0.37838 (14)	0.28125 (14)	0.78863 (14)	0.0475 (3)
C7	0.2149 (2)	0.2337 (2)	0.8058 (2)	0.0510 (5)
C71	0.2448 (3)	0.1265 (3)	0.7414 (3)	0.0661 (6)
C8	0.1040 (2)	0.1696 (3)	0.9711 (2)	0.0594 (5)
C9	0.0887 (3)	0.2756 (3)	1.0360 (3)	0.0711 (7)
C10	0.2573 (2)	0.3379 (2)	1.0018 (2)	0.0557 (5)
O10	0.2993 (2)	0.3512 (2)	1.0930 (2)	0.0741 (5)
C11	0.7522 (2)	0.2317 (2)	0.7038 (2)	0.0508 (5)
C12	0.6806 (3)	0.0986 (2)	0.7477 (3)	0.0601 (5)
C13	0.7329 (4)	0.0394 (3)	0.6460 (3)	0.0769 (7)
C14	0.8517 (3)	0.1131 (3)	0.5040 (3)	0.0769 (7)
C15	0.9215 (3)	0.2453 (3)	0.4611 (3)	0.0771 (7)
C16	0.8732 (3)	0.3046 (3)	0.5611 (3)	0.0638 (6)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

S1—O11	1.435 (2)	C5—O6	1.406 (2)
S1—O12	1.439 (2)	C5—C4	1.527 (2)
S1—C11	1.765 (2)	C5—C10	1.536 (3)
S1—C4	1.785 (2)	O6—C7	1.458 (2)
C3—C4	1.524 (3)	C7—C8	1.510 (3)
C3—C2	1.528 (3)	C7—C71	1.511 (3)
C2—O1	1.449 (2)	C8—C9	1.516 (3)
C2—C21	1.506 (3)	C9—C10	1.499 (3)
O1—C5	1.417 (2)	C10—O10	1.207 (3)
O11—S1—O12	119.08 (12)	C4—C5—C10	115.8 (2)
O11—S1—C11	108.05 (10)	C3—C4—C5	102.70 (15)
O12—S1—C11	106.49 (10)	C3—C4—S1	115.85 (13)
O11—S1—C4	108.28 (10)	C5—C4—S1	119.20 (14)
O12—S1—C4	104.53 (10)	C5—O6—C7	114.59 (14)
C11—S1—C4	110.24 (9)	O6—C7—C8	109.4 (2)
C4—C3—C2	101.3 (2)	O6—C7—C71	106.5 (2)
O1—C2—C21	109.5 (2)	C8—C7—C71	112.5 (2)
O1—C2—C3	104.5 (2)	C7—C8—C9	111.2 (2)
C21—C2—C3	114.2 (2)	C10—C9—C8	111.2 (2)
C5—O1—C2	111.61 (14)	O10—C10—C9	124.4 (2)
O6—C5—O1	111.0 (2)	O10—C10—C5	121.5 (2)
O6—C5—C4	108.99 (14)	C9—C10—C5	114.1 (2)
O1—C5—C4	104.85 (15)	C12—C11—S1	119.5 (2)
O6—C5—C10	111.0 (2)	C16—C11—S1	119.7 (2)
O1—C5—C10	105.0 (2)		

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992*b*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9-(Carboxymethyl)-2,6-dichloropurine Ethyl Ester. An Intermediate for Peptidic Nucleic Acid Synthesis

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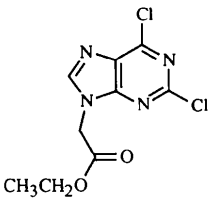
Abstract

The title compound (ethyl 2,6-dichloropurine-9-acetate, $C_9H_8Cl_2N_4O_2$) is an intermediate for the synthesis

of peptidic nucleic acids (PNA's) containing purine nucleobases. The endocyclic bond angle at each halogen substitution site is expanded by at least 2° compared to the corresponding angles in unsubstituted purine. The extended side chain emerges almost orthogonally from the heterocycle.

Comment

The use of PNA's (Egholm, Nielsen, Buchardt & Berg, 1992; Egholm *et al.*, 1993) offers the possibility of inhibiting gene expression in a controlled manner through triplex formation (Thuong & Hélène, 1993) with duplex DNA using Hoogsteen hydrogen bonds (Cheng & Pettitt, 1992). Our interest in the development of PNA's containing purine bases capable of forming stable triplexes in a pH-independent manner has prompted the synthesis of the versatile synthetic intermediate (1). Regioselective substitution of the Cl atom at the more reactive C6 position can be achieved using amine and alkoxide nucleophiles with concomitant amide formation or transesterification of the carboxyethyl group. Higher temperature and longer reaction time result in nucleophilic substitution of the C2 position.



(1)

The C—Cl bond distances (Table 2) do not support a simplistic correlation of ground-state bond length and strength with reactivity towards nucleophiles; the less reactive C2—Cl2 bond of length 1.730 (3) Å is marginally longer than the more reactive C6—Cl6 bond of length 1.721 (2) Å. Relative stability of the intermediates is more likely to determine the order of reactivity, which remains the same whether the organic substituent is a carboxyalkyl or a sugar and attached at N9 or N7 (Worthington, Schwalbe & Fraser, 1995). Cl substitution on the heterocycle significantly expands the endocyclic bond angle at the attached atom. Whereas the average N1—C2—N3 and N1—C6—C5 angles are 127.1 and 119.2°, respectively, in three accurately determined purine structures in the 9*H* tautomeric form (Takeda, Ohashi & Sasada, 1974; Itai, Yamada, Okamoto & Iitaka, 1977; Valle, Piazzogna & Ettore, 1985), and 127.3 and 122.8°, respectively, in three derivatives of 6-chloropurine (Sternglanz & Bugg, 1975; Mishnev, Bleidelis, Liepin'sh, Ramzaeva & Goncharova, 1979; Mishnev, Bleidelis, Goncharova & Ramzaeva, 1982), corresponding angles are 129.8 (2) and 121.5 (2)° in the present structure. Thus, Cl substitution at a position on the six-membered ring of purine appears to open the ring angle by at least 2°. A corresponding increment of 1.4° was found for chlorobenzenes (Domenicano, Vaciago & Coulson, 1975).

As in some other purine rings (Takeda, Ohashi & Sasada, 1974; Sternglanz & Bugg, 1975), the atoms of the heterocycle are coplanar within ±0.02 Å, but slightly buckled at the ring junction, with atoms C4 and C5 relatively 'down' and C2 and C8 'up'. The side chain minimizes steric hindrance by emerging almost orthogonally from the heterocycle; the torsion angle C4—N9—C10—C11 is 81.0 (3)°. Subsequent bonds are antiperiplanar giving an extended chain, which imposes an eclipsed conformation [−12.8 (4)°] on N9—C10—C11—O11.

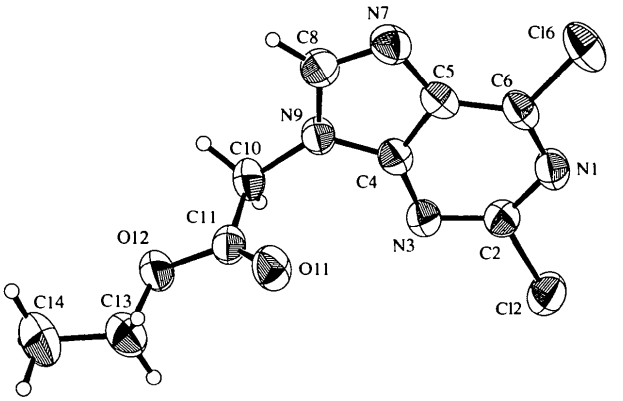


Fig. 1. *ORTEP*II view (Johnson, 1976) of the title molecule. Displacement ellipsoids are shown at the 50% probability level.

Experimental

To a solution of 2,6-dichloropurine (5.36 g, 28.3 mmol) in dry CH_3CN (40 ml) was added K_2CO_3 (4.71 g, 34.1 mmol) and ethyl bromoacetate (5.57 g, 33.0 mmol). After stirring at room temperature under argon for 48 h, the product solution was filtered and the solvent evaporated under vacuum. The residue was subjected to flash column chromatography on silica and compound (1) was eluted with EtOAc. (1) was recrystallized from methanol (4.00 g, 52%, m.p. 394–396 K). TLC (EtOAc): R_f 0.55. IR (KBr disc): ν_{max} 3109, 3001, 1731, 1586, 1554, 1401, 1344, 1236, 1152, 1018 cm^{-1}. ^1H NMR [250.1 MHz, $(CD_3)_2SO$]: δ 1.22 (*t*, 3H, J = 7.13 Hz, CH_3), 4.20 (*q*, 2H, J = 7.13 Hz, CH_2), 5.25 (*s*, 2H, CH_2), 8.71 p.p.m. (*s*, 1H, H8). ^{13}C NMR [62.9 MHz, $(CD_3)_2SO$]: δ 40.0 (CH_3), 44.5 (CH_2), 62.8 (CH_2), 130.2 (C5), 146.2 (C8), 151.8 (C2), 153.1 (C4, C6), 166.1 p.p.m. (CO). MS (electrospray): *m/z* (I_r) 277 (M^+H, 30%), 275 (M^+H, 100%), 243 (4%), 241 (12%). Elemental analyses were satisfactory.

Crystal data

$C_9H_8Cl_2N_4O_2$
M_r = 275.09

Mo $K\alpha$ radiation
λ = 0.71069 Å

Monoclinic
$C2/c$
$a = 25.5850\ (14)$ Å
$b = 11.841\ (2)$ Å
$c = 8.012\ (3)$ Å
$\beta = 104.03\ (2)°$
$V = 2354.8\ (10)$ Å^3
$Z = 8$
$D_x = 1.552$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 9.7–14.2°$
$\mu = 0.546$ mm^{-1}
$T = 293\ (2)$ K
Tabular
$0.5 \times 0.5 \times 0.25$ mm
Very pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2524 measured reflections
2055 independent reflections
1462 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.0271$
$\theta_{max} = 24.95°$
$h = -30 \rightarrow 30$
$k = 0 \rightarrow 14$
$l = -1 \rightarrow 9$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R(F) = 0.0353$
$wR(F^2) = 0.1064$
$S = 1.015$
2054 reflections
187 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.5523P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.002$

$\Delta\rho_{max} = 0.267$ e Å^{-3}
$\Delta\rho_{min} = -0.207$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0024 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0.09837 (8)	0.3243 (2)	0.5420 (3)	0.0461 (5)
C2	0.13531 (10)	0.2584 (2)	0.6414 (3)	0.0420 (6)
Cl2	0.11702 (3)	0.11776 (6)	0.64249 (10)	0.0571 (2)
N3	0.18326 (8)	0.2842 (2)	0.7380 (3)	0.0402 (5)
C4	0.19291 (9)	0.3944 (2)	0.7313 (3)	0.0377 (5)
C5	0.15866 (10)	0.4748 (2)	0.6351 (3)	0.0417 (6)
C6	0.11059 (10)	0.4330 (2)	0.5391 (3)	0.0443 (6)
Cl6	0.06350 (3)	0.51922 (8)	0.40991 (10)	0.0688 (3)
N7	0.18100 (9)	0.5814 (2)	0.6631 (3)	0.0505 (6)
C8	0.22708 (11)	0.5640 (2)	0.7722 (4)	0.0475 (6)
N9	0.23691 (8)	0.4528 (2)	0.8188 (3)	0.0399 (5)
C10	0.28615 (10)	0.4061 (2)	0.9266 (3)	0.0420 (6)
C11	0.32372 (10)	0.3645 (2)	0.8221 (3)	0.0401 (6)
O11	0.31164 (7)	0.3516 (2)	0.6690 (2)	0.0543 (5)
O12	0.37172 (7)	0.3472 (2)	0.9250 (2)	0.0483 (5)
C13	0.41401 (12)	0.3045 (4)	0.8490 (5)	0.0610 (8)
C14	0.46559 (14)	0.3184 (5)	0.9798 (8)	0.097 (2)

Table 2. *Selected geometric parameters* (Å, °)

N1—C6	1.326 (4)	C6—Cl6	1.721 (2)
N1—C2	1.331 (3)	N7—C8	1.303 (3)
C2—N3	1.318 (3)	C8—N9	1.376 (3)
C2—Cl2	1.730 (3)	N9—C10	1.453 (3)
N3—C4	1.331 (3)	C10—C11	1.502 (4)
C4—N9	1.361 (3)	C11—O11	1.200 (3)
C4—C5	1.393 (3)	C11—O12	1.319 (3)
C5—C6	1.375 (4)	O12—C13	1.455 (4)
C5—N7	1.381 (3)	C13—C14	1.482 (5)
C6—N1—C2	116.3 (2)	C6—C5—C4	114.9 (2)
N3—C2—N1	129.8 (2)	N7—C5—C4	110.8 (2)
N3—C2—Cl2	115.8 (2)	N1—C6—C5	121.5 (2)
N1—C2—Cl2	114.4 (2)	N1—C6—Cl6	116.9 (2)
C2—N3—C4	111.0 (2)	C5—C6—Cl6	121.6 (2)
N3—C4—N9	127.9 (2)	C8—N7—C5	103.6 (2)
N3—C4—C5	126.5 (2)	N7—C8—N9	114.2 (2)
N9—C4—C5	105.6 (2)	C4—N9—C8	105.8 (2)
C6—C5—N7	134.3 (2)		
C4—N9—C10—C11	81.0 (3)	C8—N9—C10—C11	−92.9 (3)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DATREDXL* (Brookhaven National Laboratory & Univ. of Birmingham, 1986). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1024). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tropinyl 2-Isopropylbenzo[*b*]thiophene-3-carboxylate, $C_{20}H_{25}NO_2S$

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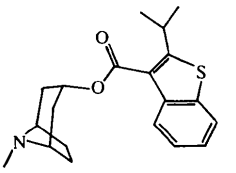
Abstract

The title tropinyl ester, 8-methyl-8-azabicyclo[3.2.1]oct-3-yl 2-isopropylbenzo[*b*]thiophene-3-carboxylate, is an analogue of the parasympathomimetic neurotransmitter acetyl choline. The molecule is stabilized in the observed conformation by an intramolecular C···O interaction.

Comment

Certain benzofused heterocycles, *viz.* benzo[*b*]thiophenes (Campaigne, Knapp, Neiss & Bosin, 1970; Bosin & Campaigne, 1977), substituted at various positions, have been found to selectively inhibit the action of thromboxane synthase without significantly inhibiting the action of prostacyclin synthase or cyclooxygenase. They are, therefore, useful as therapeutic agents for the treatment of thrombosis, ischaemic heart disease, stroke and migrane. We synthesized the title compound, (I), an analogue of the parasympathomimetic neurotransmitter acetyl choline, with the expectation that it may have a wide range of pharmacological effects.



(I)

The structure of (I) was confirmed by IR, ^{1}H NMR and mass spectroscopy, and elemental analyses. The three-dimensional structure of the tropine ester has now been determined by X-ray diffraction methods. An *ORTEP*II (Johnson, 1976) diagram with the atomic numbering scheme is shown in Fig. 1.

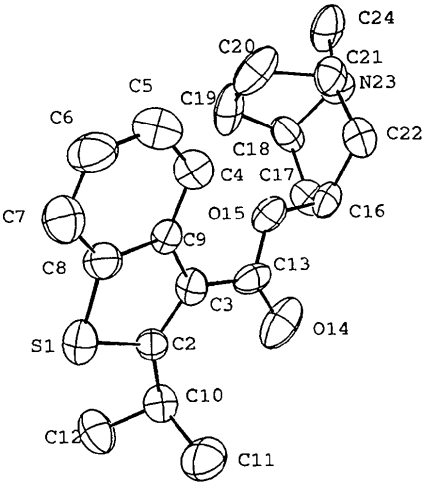


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecule with displacement ellipsoids at the 50% probability level.

Beers & Reich (1970) have reported several partly and fully rigid molecules that are active as agonists or antagonists of acetyl choline. Conformational analysis of several of these agonists and antagonists can be used as a basis for the definition of structural parameters necessary for the range of activity of this class of compounds. A distance of about 5.9 Å between the receptor anionic site and the positively charged centre of the molecule plays a crucial role in their biological functions. The intramolecular N23···O15 distance (5.9 Å) in the present structure, which is similar to that observed in strychnine (Robertson & Beevers, 1951), indicates the suitability of the compound as a potential drug. The observed conformation of the molecule (Fig. 1) appears to be stabilized in part by a C—H···O interaction [C10···O14 = 2.96 (9) Å] and the restrictions introduced by the steric interactions between C12 and C11 on the one hand and O14 on the other. In any case, the N23···O15 distance is unaffected by the conformation of this part of the molecule.