monomethyl iodide are in similar environments to the ether oxygen and quaternary ammonium nitrogen atoms in acetylcholine. It has been suggested (Hey, 1952; Albert, 1979) that these may be 'pharmacodynamic' groups associated with nicotine-like activity, although the stereospecificity of nicotine indicates that there are more than two groups involved in producing the response.

We thank the SERC for support.

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The Structures of Two Chiral Bicyclic Phosphonamides

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(*Received* 8 *November* 1985; *accepted* 3 *February* 1986)

Abstract. The crystal structures of two enantiomerically related phosphonamides have been determined by X-ray diffraction. Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}54178$ Å, room temperature. Compound (3), 2-*sec*-butyl-2,3,3a,-4,5,6,7,7a-octahydro-1,3-dimethyl-1*H*-1,3,2-benzodiazaphosphole 2-oxide, $C_{12}H_{25}N_2OP$, m.p. = 369 K, $M_r = 244{\cdot}32$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}210\,(5)$, $b = 9{\cdot}310\,(3)$, $c = 18{\cdot}384\,(10)$ Å, $V = 1405$ Å^3, $Z = 4$, $D_m = 1{\cdot}14$, $D_x = 1{\cdot}155$ Mg m^{-3}, $\mu = 1{\cdot}60$ mm^{-1}, $F(000) = 536$. Compound (4), 2,3,3a,4,5,6,7,7a-octahydro-1,3-dimethyl-2-(1-methyl-3-butenyl)-1*H*-1,3,2-benzodiazaphosphole 2-oxide, $C_{13}H_{25}N_2OP$, m.p. = 360–362 K, $M_r = 256{\cdot}33$ orthorhombic, $P2_12_12_1$, $a = 8{\cdot}292\,(3)$, $b = 10{\cdot}047\,(1)$, $c = 17{\cdot}876\,(2)$ Å, $V = 1489$ Å^3, $Z = 4$, $D_m = 1{\cdot}13$, $D_x = 1{\cdot}143$ Mg m^{-3}, $\mu = 1{\cdot}53$ mm^{-1}, $F(000) = 560$. $R = 0{\cdot}042$ with 1239 observed reflections for (3) and $R = 0{\cdot}039$ with 1621 observed reflections for (4). Except for a difference in the substituents attached to P, the two molecules are mirror images of one another, thus clearly confirming the chirality-controlling reactions. Each molecule is constituted of a five-membered ring of the envelope type including a tetrahedrally coordinated phosphorus atom. To this five-membered ring is fused a cyclohexane ring in the chair conformation.

Introduction. In previous work from these laboratories we reported on the design, synthesis and reactivity of the enantiomeric chiral and topologically unique bicyclic phosphonamides (1) and (2) (Hanessian, Delorme, Beaudoin & Leblanc, 1984, 1985). Inherent in the design of these compounds were elements of chirality, stereoelectronic effects and symmetry, associated with the general structure in question. Based on these features, it was anticipated that the anion generated from the (*R,R*) isomer, for example, would have a *pro-S* bias in reactions with carbonyl compounds and electrophiles. Indeed, when the potassium salt was allowed to react with 4-butylcyclohexanone, the corresponding ethylidene derivative was obtained with better than 90% enantiomeric excess (Hanessian *et al.*, 1984, 1985). In order to assess the facial selectivity of the corresponding phosphonamide anion, reaction was affected with various alkyl halides. In this instance, the orientation of the newly introduced alkyl group would reflect the diastereofacial bias of the phosphonamide anion.

Thus, when the lithium or potassium anions of (1) and (2) were treated respectively with ethyl iodide and allyl bromide, crystalline products (3) and (4) were formed in high yield in each case. X-ray crystal-

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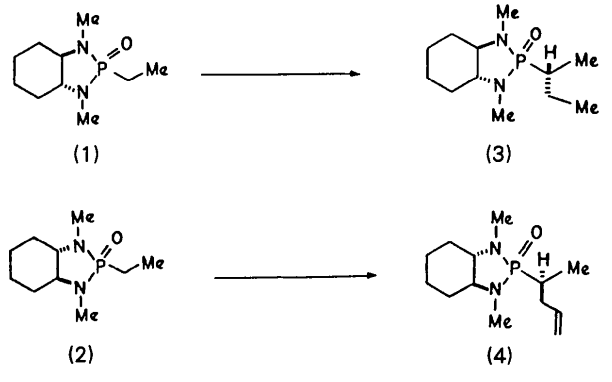
lographic analysis confirmed their structures, thus proving the originally anticipated stereochemical bias of the phosphonamide anions.



Experimental. Crystals of (3) and (4) recrystallized from ether/petroleum ether mixtures. Crystals mounted in a random orientation on Nonius CAD-4 diffractometer. Unit-cell dimensions and crystal orientations by least-squares fit to angular settings of 25 well-centered reflections in ranges $8 \leq \theta \leq 46°$ and $34 \leq \theta \leq 80°$ for (3) and (4) respectively. For both (3) and (4), absent spectra of type $h00$, $h \neq 2n$, $0k0$, $k \neq 2n$ and $00l$, $l \neq 2n$ uniquely determine space group $P2_12_12_1$. Density by flotation in aqueous $ZnCl_2$ solutions. Intensity data collected, for both compounds, in *hkl* octant under conditions listed in Table 1, with graphite-monochromatized Cu $K\alpha$ radiation. Data reduction to structure factors in usual manner.* No absorption correction. Crystal size: 0·20 × 0·28 × 0·40 mm (3); 0·12 × 0·45 × 0·55 mm (4). Structures solved by direct methods (*MULTAN*78). Block-diagonal least-squares refinement, all atoms anisotropic and H atoms isotropic. In (4), three H atoms on C(12) and C(13) clearly visible on difference Fourier synthesis. All attempts to refine coordinates of these atoms led to meaningless C—H distances and very high temperature factors. Consequently these three H atoms were kept at fixed positions. Function minimized: $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$. Scattering curves for P, O, N, C from Cromer & Mann (1968) and from Stewart, Davidson & Simpson (1965) for H atoms. Real and imaginary parts of anomalous dispersion of P from Cromer & Liberman (1970).

Discussion. The absolute configuration for (3) and (4) was established using the anomalous dispersion of P. Structure-factor calculations were performed for each configuration by changing x, y, z into $\bar{x}, \bar{y}, \bar{z}$. The final atomic coordinates,* given in Table 2, correspond to the unambiguously established absolute configurations (see Table 1). In order to discuss the X-ray results, the

Table 1. *Data collection and refinement summary*

	(3)	(4)
$2\theta_{max}(°)$	140·0	150·0
hkl range	$0 \leq h \leq 10$	$0 \leq h \leq 10$
	$0 \leq k \leq 11$	$0 \leq k \leq 12$
	$0 \leq l \leq 22$	$0 \leq l \leq 22$
Largest fluctuation of intensity reference (%)	1·1	2·0
Acceptance level, $I/\sigma(I) \geq$	3·00	1·96
Number of measured reflections	1546	1783
Number of observed reflections	1239	1621
Av. Δ/σ	0·09	0·08
Max. Δ/σ	0·45	0·25
R	0·042	0·039
wR	0·051	0·050
S	1·83	1·87
Residual electron density (e Å^{-3})	−0·39, 0·26	−0·26, 0·35
R for the other configuration	0·048	0·044
wR for the other configuration	0·059	0·056
S for the other configuration	2·12	2·12

Table 2. *Final atomic coordinates for* $C_{12}H_{25}N_2OP$ (3) *and* $C_{13}H_{25}N_2OP$ (4) ($\times 10^5$ *for* P; $\times 10^4$ *for* O,N,C) *and* U_{eq} (Å^2 $\times 10^4$ *for* P,O,N,C)

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

	x	y	z	U_{eq}
(3)				
P(1)	83371 (11)	22003 (9)	7340 (5)	442
O(1)	8079 (3)	2948 (3)	41 (1)	555
N(1)	6736 (3)	2090 (3)	1299 (1)	449
N(2)	8594 (3)	437 (3)	679 (2)	477
C(1)	7436 (5)	−326 (3)	1145 (2)	476
C(2)	6872 (5)	−1798 (4)	891 (2)	632
C(3)	5644 (6)	−2370 (4)	1434 (2)	743
C(4)	4227 (6)	−1338 (5)	1569 (3)	737
C(5)	4816 (5)	168 (4)	1770 (2)	598
C(6)	5980 (5)	681 (4)	1193 (2)	460
C(7)	5605 (5)	3302 (4)	1311 (2)	591
C(8)	10161 (5)	−250 (4)	544 (3)	696
C(9)	10027 (5)	2993 (4)	1215 (2)	560
C(10)	10268 (6)	2380 (6)	1963 (2)	823
C(11)	9888 (6)	4662 (4)	1202 (2)	706
C(12)	11520 (7)	5391 (5)	1335 (3)	893
(4)				
P	44962 (7)	36612 (6)	45827 (3)	412
O	4184 (2)	2734 (2)	5208 (1)	542
N(1)	2987 (3)	4692 (2)	4344 (1)	455
N(2)	4661 (3)	2981 (2)	3738 (1)	470
C(1)	3536 (3)	3583 (3)	3207 (1)	449
C(2)	2871 (4)	2677 (3)	2602 (1)	596
C(3)	1695 (4)	3494 (4)	2124 (2)	741
C(4)	389 (4)	4167 (4)	2592 (2)	840
C(5)	1095 (4)	4997 (3)	3231 (2)	677
C(6)	2162 (3)	4109 (3)	3689 (1)	470
C(7)	1901 (4)	5143 (3)	4937 (2)	624
C(8)	6136 (4)	2400 (3)	3449 (2)	661
C(9)	6279 (3)	4628 (3)	4786 (1)	454
C(10)	6652 (4)	5686 (3)	4208 (2)	692
C(11)	6220 (4)	5165 (3)	5592 (2)	644
C(12)	7862 (4)	5413 (4)	5901 (2)	725
C(13)	8432 (6)	6436 (4)	6172 (3)	1091

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* The programs used here are modified versions of *NRC*-2, data reduction, *NRC*-10, bond distances and angles, and *NRC*-22, mean planes (Ahmed, Hall, Pippy & Huber, 1973); *FORDAP*, Fourier and Patterson maps (A. Zalkin); *MULTAN*78, multisolution program (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); *NUCLS*, least-squares refinement (Doedens & Ibers, 1967) and *ORTEP*, stereodrawings (Johnson, 1965).

* Lists of structure factors, anisotropic temperature factors, H-atom parameters and least-squares-planes data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42809 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

atomic numbering adopted for (3) and (4) is shown in Fig. 1 while stereoviews of the molecules are represented in Fig. 2. The bond distances and bond angles of (3) and (4) are compared in Table 3.

The two molecules are of opposite configurations but, in both cases, the methyl substituents at N(1) and N(2) are in equatorial positions and *trans* to one another.

The five-membered rings are both of the envelope type with the fold along N(1)–C(1) and N(2)–C(6) for (3) and (4), respectively. The atoms N(1), N(2), C(1), P in (3) or (4) are within 5σ of their respective least-squares planes. The above groups of atoms make practically identical dihedral angles of 140° with plane N(1)–C(1)–C(6) in (3) and (4) respectively

The coordination around the phosphorus atom is tetrahedral, since the average of the six angles involving P is 109·3° for each of (3) and (4). There are, however, some distortions since P is part of a five-membered ring so the angular values are in the range 117·4 (2)° for N(1)–P(1)–O(1) to 94·4 (1)° for N(1)–P(1)–N(2) for (3) and from 116·8 (1) to 94·8 (1)° for (4). The tetrahedral coordination around P is also established by noting that the phosphorus atom in (3) is 0·536 (1) Å

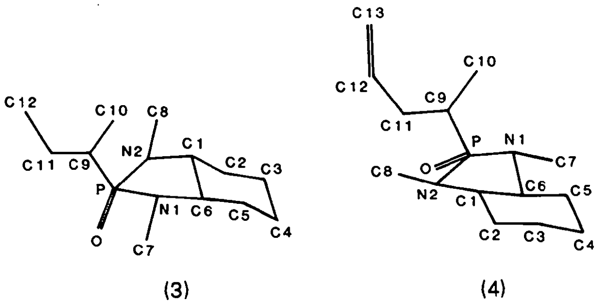

Fig. 1. Schematic representation of the molecules and atomic numbering.

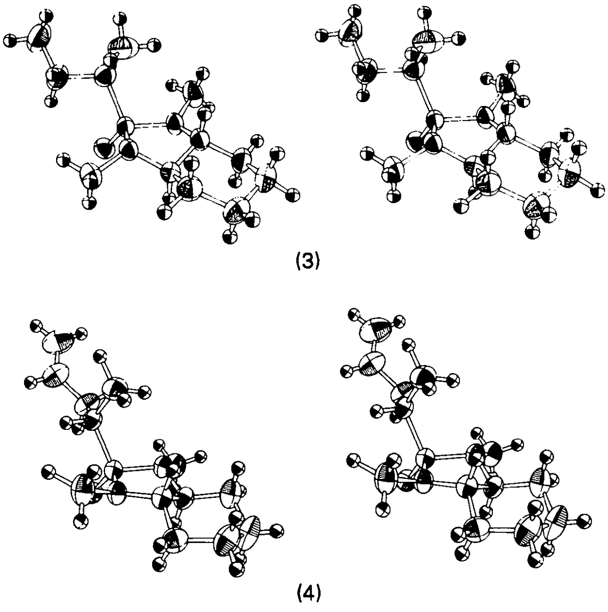

Fig. 2. Stereopairs showing the structures of $C_{12}H_{25}N_2OP$, (3), and $C_{13}H_{25}N_2OP$, (4).

Table 3. *Distances* (Å) *and angles* (°) *for* $C_{12}H_{25}N_2OP$, (3), *and* $C_{13}H_{25}N_2OP$, (4)

	(3)	(4)		(3)	(4)
C(1)–C(2)	1·520 (5)	1·517 (4)	N(2)–C(1)	1·464 (5)	1·462 (3)
C(2)–C(3)	1·515 (6)	1·535 (5)	N(1)–C(7)	1·461 (5)	1·463 (4)
C(3)–C(4)	1·529 (7)	1·526 (5)	N(2)–C(8)	1·458 (5)	1·450 (4)
C(4)–C(5)	1·529 (6)	1·531 (5)	P(1)–O(1)	1·467 (3)	1·478 (2)
C(5)–C(6)	1·505 (6)	1·500 (4)	P(1)–C(9)	1·803 (4)	1·806 (3)
C(6)–C(1)	1·522 (5)	1·523 (4)	C(9)–C(10)	1·502 (6)	1·514 (4)
C(6)–N(1)	1·464 (4)	1·477 (3)	C(9)–C(11)	1·558 (6)	1·539 (4)
N(1)–P(1)	1·678 (3)	1·679 (2)	C(11)–C(12)	1·522 (7)	1·490 (5)
P(1)–N(2)	1·658 (3)	1·663 (2)	C(12)–C(13)	—	1·231 (6)
C(1)–C(2)–C(3)	108·5 (3)	107·9 (2)	C(7)–N(1)–P(1)	117·4 (2)	117·8 (2)
C(2)–C(3)–C(4)	113·1 (4)	112·5 (3)	N(1)–P(1)–O(1)	117·0 (6)	116·8 (1)
C(3)–C(4)–C(5)	112·0 (4)	112·3 (3)	N(2)–P(1)–O(1)	115·8 (1)	116·2 (1)
C(4)–C(5)–C(6)	108·8 (3)	108·0 (3)	O(1)–P(1)–C(9)	110·0 (2)	109·3 (1)
C(5)–C(6)–C(1)	110·1 (3)	109·8 (2)	C(9)–P(1)–N(1)	108·9 (1)	109·2 (1)
C(6)–C(1)–C(2)	109·5 (3)	109·9 (2)	C(9)–P(1)–N(2)	109·7 (2)	109·7 (1)
C(6)–C(1)–N(2)	104·2 (3)	104·7 (2)	P(1)–N(2)–C(8)	123·8 (2)	123·9 (2)
C(1)–N(2)–P(1)	111·2 (2)	111·5 (2)	C(8)–N(2)–C(1)	117·4 (3)	118·3 (2)
N(2)–P(1)–N(1)	94·4 (1)	94·8 (1)	P(1)–C(9)–C(10)	113·3 (3)	114·1 (2)
P(1)–N(1)–C(6)	107·7 (2)	107·6 (2)	P(1)–C(9)–C(11)	110·1 (3)	110·5 (2)
N(1)–C(6)–C(1)	103·1 (3)	103·9 (2)	C(10)–C(9)–C(11)	113·7 (3)	113·5 (2)
C(5)–C(6)–N(1)	117·4 (3)	118·0 (2)	C(9)–C(11)–C(12)	112·2 (4)	112·1 (3)
C(2)–C(1)–N(2)	117·1 (3)	116·5 (2)	C(11)–C(12)–C(13)	—	129·5 (4)
C(6)–N(1)–C(7)	115·1 (3)	114·3 (2)			

	(3)	(4)
Six-membered ring		
C(1)–C(2)–C(3)–C(4)	54·3 (5)	−54·1 (3)
C(2)–C(3)–C(4)–C(5)	−52·4 (5)	53·2 (4)
C(3)–C(4)–C(5)–C(6)	53·8 (5)	−55·4 (4)
C(4)–C(5)–C(6)–C(1)	−60·2 (4)	61·3 (3)
C(5)–C(6)–C(1)–C(2)	64·7 (4)	−65·9 (3)
C(6)–C(1)–C(2)–C(3)	−59·8 (4)	59·8 (3)
Five-membered ring		
C(1)–N(2)–P(1)–N(1)	−2·6 (2)	1·9 (2)
N(2)–P(1)–N(1)–C(6)	−24·5 (2)	23·7 (2)
P(1)–N(1)–C(6)–C(1)	42·6 (3)	−40·9 (2)
N(1)–C(6)–C(1)–N(2)	−43·2 (3)	41·2 (2)
C(6)–C(1)–N(2)–P(1)	27·4 (3)	−25·7 (2)
Others		
C(7)–N(1)–C(6)–C(5)	−63·1 (4)	64·6 (3)
C(7)–N(1)–P(1)–O(1)	−34·4 (3)	32·0 (2)
C(7)–N(1)–P(1)–C(9)	91·1 (3)	−92·6 (2)
C(7)–N(1)–P(1)–N(2)	−156·3 (3)	154·6 (2)
C(8)–N(2)–C(1)–C(2)	−60·6 (4)	59·5 (3)
C(8)–N(2)–P(1)–C(9)	−39·5 (3)	41·0 (3)
C(8)–N(2)–P(1)–O(1)	85·8 (3)	−83·6 (2)
C(8)–N(2)–P(1)–N(1)	−151·1 (3)	153·4 (2)
C(8)–N(2)–C(1)–C(6)	178·4 (3)	−179·0 (2)
C(2)–C(1)–C(6)–N(1)	−169·3 (3)	167·0 (2)
C(2)–C(1)–N(2)–P(1)	148·5 (3)	−147·2 (2)
C(3)–C(2)–C(1)–C(6)	−59·8 (4)	59·8 (3)
C(3)–C(2)–C(1)–N(2)	−178·0 (3)	178·6 (2)
C(4)–C(5)–C(6)–C(1)	−60·2 (4)	61·2 (3)
C(4)–C(5)–C(6)–N(1)	−177·7 (3)	180·0 (2)
C(5)–C(6)–C(1)–N(2)	−169·2 (3)	168·3 (2)
C(5)–C(6)–N(1)–P(1)	163·8 (3)	−162·7 (2)
C(9)–P(1)–N(2)–C(1)	109·2 (2)	−110·5 (2)
C(9)–P(1)–N(1)–C(6)	−137·0 (2)	136·5 (2)
O(1)–P(1)–N(1)–C(6)	97·5 (2)	−99·0 (2)
O(1)–P(1)–N(2)–C(1)	−125·5 (2)	124·9 (2)
C(10)–C(9)–P(1)–N(2)	−57·1 (3)	55·7 (2)
C(10)–C(9)–P(1)–N(1)	45·0 (3)	−47·0 (2)
C(10)–C(9)–P(1)–O(1)	174·4 (3)	−175·8 (2)
C(11)–C(9)–P(1)–N(1)	−83·6 (3)	82·4 (2)
C(11)–C(9)–P(1)–O(1)	45·8 (3)	−46·5 (2)
C(11)–C(9)–P(1)–N(2)	174·3 (3)	−175·0 (2)
C(12)–C(11)–C(9)–C(10)	73·2 (5)	−76·3 (3)
C(12)–C(11)–C(9)–P(1)	−158·5 (3)	154·1 (2)
C(13)–C(12)–C(11)–C(9)	—	125·0 (4)

above the plane formed by N(1), N(2) and O(1) and 0·528 (1) Å above the corresponding plane in (4). These distances in the 75 molecular fragments $\genfrac{}{}{0pt}{}{N}{N}\!\!>\!P{=}O$ found in the Cambridge Data File average 0·52 Å.

Reported P—O distances fall in the range 1·40 to 1·62 Å. Our values of 1·467 (3) Å in (3) and 1·478 (2) Å in (4) agree reasonably well with the average of 1·495 Å calculated for 75 P—O distances in the $\genfrac{}{}{0pt}{}{N}{N}\!\!>\!P{=}O$ molecular fragments in the Cambridge Data File. It should be noted that the distribution of these P—O distances presents two maxima. The predominant one is centered around 1·478 Å, the secondary maximum is centered at 1·585 Å. The P—N distances are 1·678 (3), 1·658 (3) Å in (3) and 1·679 (2) and 1·663 (2) Å in (4). Their average of 1·669 Å is slightly larger than the average value of 1·650 Å computed for the 75 above fragments found in the Cambridge Data File. The values of the torsion angles in the five-membered rings are listed in Table 3. Although they are of opposite signs they compare very well, to within 2°, in (3) and (4). None of the groups of four atoms, consisting of a nitrogen and its three neighbors, are coplanar. This is usually measured by the sum, $\sum N$, of the angles around each nitrogen. One finds in (3) $\sum N(1) = 340{\cdot}2\ (7)$ and $\sum N(2) = 352{\cdot}4\ (7)°$ and in (4) $\sum N(1) = 339{\cdot}7\ (6)$ and $\sum N(2) = 353{\cdot}7\ (6)°$ instead of $\sum N = 360°$ for a planar group. Consequently, N(1) and N(2) are above the planes formed by their three neighbors by as much as 0·390 (3) and 0·238 (3) Å in (3) and 0·406 (2) and 0·221 (2) Å in (4). These two sets of measurements also reveal that in both compounds the pyramid with N(2) at its apex is clearly more flattened than that with N(1).

The six-membered rings

Both six-membered rings in (3) and (4) are in the chair conformation. The torsion angles of interest, listed in Table 3, show a similar pattern. In both compounds the largest deviations [64·7 (4)° in (3), −65·9 (3)° in (4)] from the 'standard' torsion angle, $\theta = 55{\cdot}8°$, involve the C(1)—C(6) bond (Bucourt & Hainault, 1965). This is the bond which is also part of the five-membered ring. The bond angles [average 110·3 (3)° in (3), 110·1 (3)° in (4)] are normal and follow the same pattern when individual values in (3) and (4) are compared. The bond distances have averages of 1·520 (6) Å in (3) and 1·522 (5) Å in (4). The C(5)—C(6) bond distance is significantly shorter in (3) [1·505 (6) Å] and (4) [1·500 (4) Å] when compared to the averages [1·523 (6) and 1·526 (5) Å] of the other five distances in the ring.

The substituents on P

The substituent attached to P is a *sec*-butyl group in (3) and a 1-methyl-3-butenyl group in (4). All bond distances and angles in these groups are normal except for the C(12)=C(13) double bond in (4) which is unusually short, with a value of 1·231 (6) Å, for the $C(sp^2){=}C(sp^2)$ distance. A C=C distance of 1·314 (6) Å has been reported for ethylene at 85 K (van Nes & Vos, 1977) while values of 1·333 (2) and 1·339 (3) Å have been reported at room temperature by Raman spectroscopy (Dowling & Stoicheff, 1958) and electron diffraction in the gas phase (Bartell & Bonham, 1959) respectively. The C(12)=C(13) distance observed here is thus significantly shorter than in ethylene or some of its derivatives. However, this is not the only situation of this type. Of the 122 allyl fragments found in the Cambridge Data File, 14 have a C=C bond distance in the range 1·207 to 1·250 Å. These short distances are usually accompanied by relatively high thermal parameters and sometimes the associated H atoms could not be located in the difference Fourier syntheses. See for example: Wilson, Rekers, Packard & Elder (1980), Watson & Taira (1977) or Tomita, Rosenstein & Almeida-Santos (1978). In only one case was the relatively close contact (3·5–3·6 Å) between allyl groups of two different molecules suggested as the reason for the short C=C bond (Marsh & Waser, 1970). In our situation, the molecules are arranged in such a way that there are no two allyl groups near one another.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support and Mr M. Olivier for the measurements on the Nonius diffractometer.

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Structure of 2-[2-(Thymin-1-yl)ethylthio]acetic Acid

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(*Received* 21 *January* 1986; *accepted* 10 *February* 1986)

Abstract. $C_9H_{12}N_2O_4S$, $M_r = 244·26$, monoclinic, $P2_1$, $a = 8·610$ (2), $b = 12·427$ (2), $c = 5·070$ (1) Å, $\beta = 92·69$ (2)°, $V = 541·9$ (2) Å^3, $Z = 2$, $D_m = 1·48$, $D_x = 1·497$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 29·9$ cm^{-1}, $F(000) = 256$, $T = 293$ K, $R = 0·064$ for 461 observed reflexions. The molecule takes a folded conformation. Bond distances and angles are normal. The carboxyl group is doubly hydrogen-bonded to O(4) and N(3) of the thymine moiety of the adjacent molecule related by 2_1 with an O···O distance of 2·61 (1) Å and an O···N distance of 2·84 (1) Å.

Introduction. As part of studies on elementary patterns in protein–nucleic acid interactions, we have found hydrogen-bonding schemes between carboxyl groups and nucleic acid bases (Ohki, Takenaka, Shimanouchi & Sasada, 1975, 1976, 1977; Takenaka, Ohki & Sasada, 1980; Takenaka & Sasada, 1982*a*; Fujita, Takenaka & Sasada, 1982, 1984), using model crystals that contain both components. The present paper deals with hydrogen bonds between a carboxyl group and thymine.

Experimental. 1-(2-Chloroethyl)thymine, derived from its 2-hydroxyethyl derivative (Shibata, Takenaka, Sasada & Ohki, 1985), was reacted with mercaptoacetic acid ethyl ester. After de-esterification, plate crystals of the title compound were obtained from an aqueous solution. D_m by flotation in a mixture of bromoform and cyclohexane. Rigaku four-circle diffractometer; graphite-monochromated Mo $K\alpha$ radiation; crystal size 0·5 × 0·2 × 0·05 mm; unit-cell dimensions determined with 38 reflexions ($11 < 2\theta < 25°$); 589 independent reflexions measured in the range $2 < 2\theta < 42°$, h −8~8, k 0~12, l 0~5; 461 reflexions with $F_o > 3\sigma$ considered observed; ω-scan mode, scan rate 2° min^{-1}, scan width 1·75° in ω. Five reference reflexions monitored every 50 reflexions showed no significant intensity deterioration. Corrections for Lorentz and polarization factors, but not for absorption. Standard deviations $\sigma^2(F_o) = \sigma_P^2(F_o) + qF_o^2$, where σ_P was evaluated by counting statistics and q was estimated to be $4·67 \times 10^{-4}$ from measurement of monitored reflexions (McCandlish & Stout, 1975).

Structure solved by the heavy-atom method and refined by the full-matrix least-squares method; geometrically assigned H atoms included in structure-factor calculations isotropically; $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/\sigma^2(F_o)$; final R value 0·064 for 461 reflexions with $F_o > 3\sigma$ ($wR = 0·056$, $S = 1·48$); maximum shift of parameters 0·08σ, $\Delta\rho$ peak 0·27 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *TPAT* (Takenaka & Sasada, 1971), *LSAP*80 (Takenaka & Sasada, 1980), *DCMS*82 (Takenaka & Sasada, 1982*b*) and *LISTUP* (Takenaka & Sasada, 1983). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows the molecular structure with the atom numbering. The molecule as a whole takes a folded conformation; the torsion angles are given in the figure. The distance between the centres of the pyrimidine ring and the carboxyl group is 5·16 Å. Bond distances and angles of the thymine moiety are in agreement with those of thymine derivatives, *e.g.*

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* Lists of structure factors, anisotropic thermal parameters of non-H atoms, H-atom coordinates and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42822 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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