unfavourable because of the shorter non-bonding C(2)···O and H(21)···O distances and because of the preference for a staggered rather than an eclipsed disposition of bonds, if one regards a double bond as being decomposed into two bent bonds (Dunitz & Strickler, 1968). In α,β-unsaturated acids, where the *syn* form implies eclipsing of double bonds, some acids have either the *anti* form or show orientational *anti–syn* disordering, indicating a delicate balance between the two factors mentioned above. In monomethyl *trans,trans*-muconate (Rabinovich & Schmidt, 1967) the orientation of the methoxycarbonyl group is synplanar, while the carboxyl group shows the antiplanar conformation and exhibits no disorder, in contrast to monomethyl fumarate (Leiserowitz & Tang, 1977) where the conformations of ester and acid groups are identical to that in monomethyl *trans,trans*-muconate, though the acid group shows some orientational disorder. The difference in the C=O and C–O(H) distances, agreeing well with the mean reported for carboxyl groups without orientational disorder in 16 acids (Leiserowitz, 1976), and the well-defined position of the O(H) H atom indicate absence of orientational disorder in the carboxyl group of monomethyl succinate. The dihedral angle C(1)–C(2)–O(1)–O(2)/C(3)–C(4)–O(3)–O(4) is 82·2°. The distance of C(5) from the best plane through C(3), C(4), O(3) and O(4) is 0·11 Å. The H atoms of the methyl group are in the *anti* position, while in dimethyl *meso*-tartrate an approximately equal distribution of the *syn* and *anti* forms is present.

The molecules are hydrogen-bonded across centres of symmetry, thus forming cyclic centrosymmetric dimers. The O–H···O angle is close to linear (172·5°) with an O···O distance of 2·66 Å. The deviation of H(1) from the plane of C(1), O(1) and O(2) is 0·11 Å. The carboxyl groups forming the dimer are not coplanar; the displacement between them is 0·17 Å, which agrees well with the average value reported for a number of centrosymmetric dimers (Jeffrey & Sax, 1963). The stacking of the dimers is determined by van der Waals interactions; no short distances except the hydrogen bond are present. The structure consists of two stacks of hydrogen-bonded pairs of molecules, centred at $(0,0,\frac{1}{2})$ and $(\frac{1}{2},0,\frac{1}{2})$, which are related by the *a* glide plane. The molecular packing is shown in Fig. 2.

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1-Benzotriazoleacetic Acid

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Abstract. $C_6H_4N_3CH_2COOH$, M_r = 177·2, monoclinic, $P2_1/c$, a = 13·26 (4), b = 4·53 (1), c = 15·18 (4) Å, β = 113·1 (2)°, Z = 4, V = 838 Å^3, D_c = 1·41 g cm^{-3}, $F(000)$ = 368. The compound crystallized from ethanol solution. The radiation used was Mo $K\alpha$. The structure was solved by direct methods. Least-squares refinement with 846 observed reflexions converged at R = 0·044 for 146 parameters. The benzotriazole residue is planar. Strong O–H···N hydrogen bonds, 2·69 Å in length and arranged across a glide plane, link the molecules together to form infinite chains running parallel to the *c* axis.

Introduction. A prismatic crystal (0·16 × 0·18 × 0·5 mm) elongated along **b** was selected for data collection on a Siemens AED automatic diffractometer by the θ–2θ scan procedure. About 1000 independent reflexions were measured out to $\sin\theta/\lambda = 0{\cdot}65$ Å^{-1}, 846 of which were taken as observed $[I > 2{\cdot}5\sigma(I)]$.

The structure factors were derived without a correction for absorption $[\mu(\text{Mo } K\alpha) = 1{\cdot}13 \text{ cm}^{-1}]$.

The structure was solved by a straightforward application of the computer program *MULTAN* (Germain, Main & Woolfson, 1970).

The E map calculated from the best set of phases showed all the non-hydrogen atoms. Their coordinates were refined by the block-diagonal least-squares method with individual temperature factors, initially isotropic and then anisotropic.

All H atoms were unambiguously located from a difference electron density map. Further least-squares refinement included the H atoms, allowing their positional and isotropic thermal parameters to shift. The final R index for the observed reflexions was 0·044.* The weighting scheme used during the refinement procedure was $w = [5 + 1{\cdot}67|F_o| + 0{\cdot}05|F_o|^2]^{-1}$ for the observed reflexions. The final atomic parameters are listed in Tables 1 and 2. Atomic scattering factors for the non-hydrogen atoms were taken from Cromer & Mann (1968), and for the H atoms from Moore (1963).

Table 1. *Atomic coordinates* ($\times 10^4$) *of the non-hydrogen atoms with estimated standard deviations in parentheses*

	x	y	z
N(1)	1573 (3)	5056 (8)	1177 (2)
N(2)	1115 (3)	3097 (9)	457 (2)
N(3)	1738 (3)	2967 (9)	−32 (2)
C(4)	3492 (4)	5543 (12)	115 (3)
C(5)	4242 (4)	7523 (13)	687 (4)
C(6)	4146 (4)	8878 (12)	1497 (4)
C(7)	3281 (4)	8255 (11)	1754 (3)
C(8)	2516 (3)	6223 (10)	1163 (2)
C(9)	2611 (3)	4877 (11)	372 (3)
C(10)	1012 (3)	5799 (11)	1799 (3)
C(11)	1556 (3)	4432 (11)	2785 (3)
O(12)	1074 (2)	5282 (8)	3354 (2)
O(13)	2325 (3)	2779 (10)	3008 (2)

Table 2. *Positional* ($\times 10^3$) *and thermal* ($\times 10$) *parameters of the hydrogen atoms with standard deviations in parentheses*

	x	y	z	B (Å^2)
H(C4)	354 (3)	467 (11)	−49 (3)	28 (11)
H(C5)	482 (4)	821 (13)	51 (3)	36 (12)
H(C6)	468 (4)	1025 (14)	186 (4)	55 (5)
H(C7)	315 (4)	922 (11)	231 (3)	29 (11)
H(C10)	98 (4)	803 (11)	188 (3)	30 (11)
H'(C10)	29 (4)	487 (11)	153 (3)	30 (11)
H(O12)	141 (4)	412 (15)	397 (4)	54 (15)

Discussion. The X-ray analysis of the title compound was carried out as part of a comparative structural study on benzotriazole systems *N*-substituted in positions 1 and 2. Many of these derivatives display antimetabolic activity.

Intramolecular bond lengths and angles with the numbering scheme are shown in Fig. 1. Their values are uncorrected for the thermal motion.

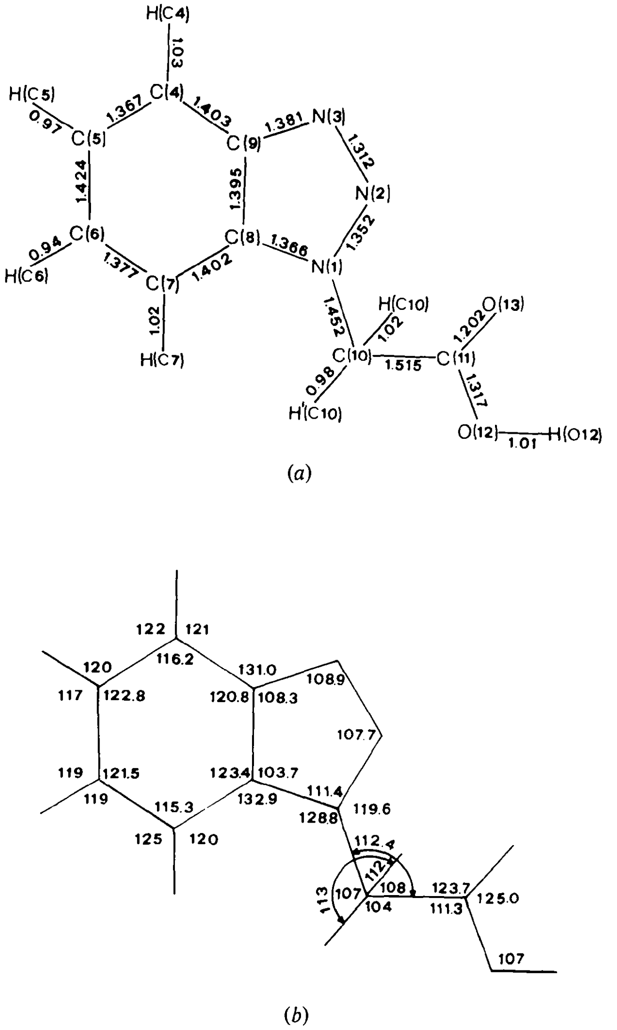

Fig. 1. (*a*) Bond lengths (Å) and numbering scheme. (*b*) Bond angles (°). Taking into account also the e.s.d.'s of the cell parameters, the ranges for the e.s.d.'s are: 0·005–0·009 Å for bonds involving non-hydrogen atoms, 0·05–0·07 Å for bonds involving H atoms, 0·7–1·3° for angles involving non-hydrogen atoms, 4–9° for angles involving H atoms.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32377 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

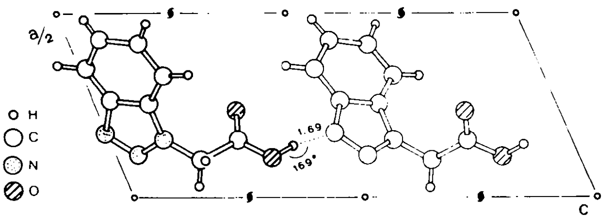

Fig. 2. Perspective view of the crystal structure along the *b* axis showing the molecular conformation [the torsion angles C(8)–N(1)–C(10)–C(11) and N(1)–C(10)–C(11)–O(13) are −78·1 and −4·2° respectively]. The dotted line indicates the hydrogen bond.

The molecular arrangement of the benzotriazolyl group is identical within the experimental error with that found in the crystal structures of benzotriazole (Escande, Galigné & Lapasset, 1974), *cis*-1-(6-acetoxymethyltetrahydro-2-pyranyl)-5,6-dichlorobenzotriazole (*cis*-ATD) (Fayos & Garcia-Blanco, 1972) and 2-(4-*O*-acetyl-2,3-dideoxy-β-L-*glycero*-pent-2-enopyranosyl)-5,6-dichlorobenzotriazole (*trans*-CLBA) (Lopez de Lerma, Martínez-Carrera & García-Blanco, 1973).

Bond distances indicate that substantial delocalization occurs in the heterocyclic nine-membered system. This is also strictly planar, atomic displacements from the least-squares plane being no larger than 0·01 Å.

The crystal packing is shown in Fig. 2. The molecules are linked in infinite chains parallel to the *c* axis by a strong O–H···N hydrogen bond, which is nearly linear. It involves the carboxylic O(12)–H and the $\mathrm{N(3^{i})}$ atom of the molecule related by the glide plane [O···N 2·69, H···N 1·69 Å, O–H···N angle 169°].

This result confirms the ability of the N(3) atom to behave as an excellent hydrogen-bond acceptor, as previously recognized in the crystal structures of benzotriazole and *cis*-ATD.

All the other intermolecular distances appear to be in the normal range but there are two contacts shorter than the sum of the van der Waals radii: those formed by O(13) with $\mathrm{H(C4^{i})}$ at $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, 2·47 Å, on the one side, and with $\mathrm{H(C7^{ii})}$ at $(x, y - 1, z)$, 2·42 Å, on the other side. Moreover, the arrangement of C(11), $\mathrm{H(C4^{i})}$ and $\mathrm{H(C7^{ii})}$ around the O atom is approximately planar trigonal. This situation may be indicative of some donor character in hydrogen bonding from the C atoms in positions 4 and 7, as suggested by Fayos & García-Blanco (1972).

We thank Professor F. Sparatore for providing us with the crystalline sample.

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2-*exo*-Hydroxy-7-methylbicyclo[2.2.1]heptane-7-*syn*-carboxylic Acid*†

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Abstract. $C_9H_{14}O_3$, monoclinic, $P2_1/c$, $a = 11\cdot58\,(2)$, $b = 6\cdot874\,(10)$, $c = 12\cdot02\,(2)$ Å, $\beta = 115\cdot21\,(5)^\circ$, $Z = 4$, $D_x = 1\cdot306\,(7)$, $D_m = 1\cdot32\,(1)$ g cm^{-3}, at 22°C. For 798 reflections with $I > \sigma$, $R = 0\cdot056$ and $R_w = 0\cdot032$. The norbornane skeleton shows little distortion by the substituent groups, and the O(hydroxyl)–C(carboxyl) distance (2·87 Å) is less crowded than in a salt of a related 2,6,2-methylhydroxy acid which lactonizes more rapidly.

Introduction. This compound was studied as part of a program to establish the dimensions and their variation for a variety of substituted norbornane molecules (Chapuis, Zalkin & Templeton, 1973, 1977). There has

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* Structures of Bicyclo[2.2.1] Systems. III.

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