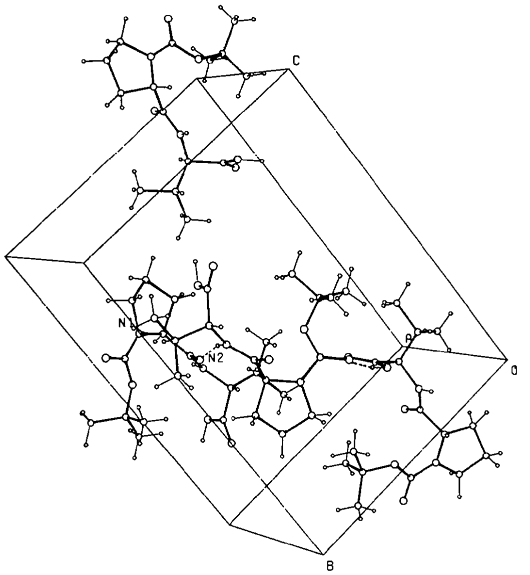

Fig. 2. Perspective view of the crystal packing of Boc-L-Pro-D-Val-OH. Hydrogen bonds are indicated by dashed lines.

The molecular arrangement in the crystal (Fig. 2) is influenced by two intermolecular hydrogen bonds: N(2)···O(3′) = 2·880 (4) Å (symmetry code: 0·5 + *x*, 1·5 − *y*, 1 − *z*) and O(4)···O(2′) = 2·659 (4) Å (symmetry code: 1·5 − *x*, 2 − *y*, −0·5 + *z*) linking the molecules along $\overline{ac}$. As observed in the crystal structure of related oligopeptides this favours an intermolecular packing of polymeric hydrogen-bonded sheets of peptide molecules which have only van der Waals contacts between each other.

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Tris(3,5-dimethyl-1-pyrazolyl)methane, $C_{16}H_{22}N_6$

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Abstract. $M_r = 298\cdot39$, monoclinic, $P2_1/n$, $a = 17\cdot573\,(11)$, $b = 23\cdot000\,(10)$, $c = 18\cdot105\,(8)$ Å, $\beta = 113\cdot89\,(4)°$, $V = 6691\,(6)$ Å^3, $Z = 16$, $D_x = 1\cdot19$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot820$ cm^{-1}, $F(000) = 2560$, $T = 291$ K. Final $R = 0\cdot108$ for 4137 observed reflexions. None of the four independent molecules shows threefold symmetry. In spite of the absence of crystallographic symmetry between these four molecules, their conformations are very similar. There is an approximate inversion center. The helical conformation is in good agreement with published results for comparable molecules.

Introduction. This seems to be the first structure determination of a tris(azolyl)methane derivative. Compared to triphenylmethane, the particularity of this molecule is the asymmetry of the five-membered rings. Even in the case of a regular helical conformation, in addition to the two enantiomers corresponding to the left-handed and the right-handed helix, this asymmetry

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters*

For H(C1) and C(17) to C(22) isotropic thermal parameters are given.

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} or B(Å^2)	x	y	z	B_{eq} or B(Å^2)
		Molecule *A*				Molecule *B*		
C(1)	3650 (5)	8629 (4)	2443 (5)	2·89	8179 (5)	9390 (4)	2135 (5)	2·63
N(2)	3531 (5)	8689 (3)	3188 (5)	3·33	8299 (5)	9547 (4)	2942 (5)	3·76
N(3)	2963 (5)	9068 (4)	3246 (5)	3·78	8796 (5)	10016 (4)	3322 (5)	3·86
C(4)	3073 (8)	9028 (5)	4026 (7)	4·63	8704 (7)	10048 (5)	4040 (7)	4·86
C(5)	3706 (8)	8647 (5)	4461 (7)	5·25	8174 (7)	9621 (6)	4109 (6)	5·12
C(6)	4003 (6)	8439 (5)	3918 (7)	3·91	7917 (6)	9312 (5)	3405 (6)	3·77
N(7)	2882 (4)	8743 (3)	1760 (4)	2·91	8937 (5)	9432 (3)	2022 (4)	2·96
N(8)	2420 (5)	8268 (3)	1365 (5)	3·61	9399 (5)	8940 (3)	2191 (5)	3·27
C(9)	1756 (6)	8501 (5)	782 (6)	3·74	10108 (6)	9086 (5)	2139 (7)	4·23
C(10)	1782 (6)	9105 (4)	786 (6)	3·58	10104 (7)	9670 (5)	1936 (7)	4·50
C(11)	2508 (6)	9249 (4)	1424 (6)	2·98	9347 (7)	9888 (5)	1859 (6)	3·90
N(12)	4307 (5)	9004 (3)	2442 (5)	2·97	7521 (5)	9732 (3)	1547 (5)	3·22
N(13)	4741 (5)	9359 (3)	3079 (5)	3·60	7046 (5)	10098 (3)	1778 (5)	3·38
C(14)	5296 (6)	9614 (4)	2852 (7)	4·24	6451 (6)	10275 (4)	1090 (6)	3·70
C(15)	5233 (7)	9438 (5)	2083 (7)	4·28	6523 (7)	10021 (4)	411 (7)	4·03
C(16)	4595 (7)	9037 (5)	1847 (7)	4·40	7200 (6)	9675 (4)	723 (6)	3·62
H(C1)	3837 (5)	8187 (4)	2400 (5)	2·19	7989 (5)	8939 (4)	2040 (5)	2·19
C(17)	2521 (8)	9386 (6)	4296 (8)	6·86	9181 (8)	10527 (5)	4627 (8)	6·22
C(18)	4692 (8)	8010 (5)	4013 (7)	5·65	7334 (7)	8802 (5)	3108 (6)	4·76
C(19)	1099 (7)	8101 (5)	212 (7)	5·26	10801 (8)	8658 (5)	2289 (8)	6·19
C(20)	2832 (7)	9851 (5)	1730 (6)	4·50	8978 (7)	10501 (5)	1669 (7)	5·28
C(21)	5889 (8)	10057 (5)	3417 (7)	5·89	5797 (7)	10692 (5)	1111 (7)	5·20
C(22)	4200 (7)	8694 (5)	1068 (6)	4·84	7609 (7)	9281 (5)	296 (7)	5·37
		Molecule *C*				Molecule *D*		
C(1)	6340 (6)	2599 (4)	2699 (5)	3·22	1817 (6)	1841 (4)	2976 (6)	2·97
N(2)	6418 (5)	2485 (3)	1949 (4)	2·96	1695 (5)	1677 (3)	2182 (5)	3·26
N(3)	7003 (5)	2092 (3)	1953 (5)	3·71	1153 (5)	1233 (4)	1799 (5)	3·74
C(4)	6886 (7)	2052 (5)	1170 (7)	4·11	1191 (6)	1194 (5)	1078 (6)	4·32
C(5)	6221 (7)	2414 (5)	668 (6)	4·28	1756 (7)	1595 (5)	998 (6)	3·98
C(6)	5951 (6)	2688 (4)	1191 (6)	3·92	2078 (6)	1900 (4)	1714 (6)	3·74
N(7)	7114 (5)	2517 (3)	3375 (4)	3·02	1068 (5)	1819 (3)	3095 (5)	3·33
N(8)	7598 (5)	3004 (3)	3691 (5)	3·60	589 (5)	2317 (4)	2883 (5)	4·12
C(9)	8269 (6)	2792 (5)	4291 (7)	4·47	−116 (7)	2173 (5)	2931 (7)	4·97
C(10)	8218 (7)	2197 (5)	4359 (7)	4·77	−112 (7)	1600 (5)	3181 (7)	5·06
C(11)	7485 (6)	2028 (4)	3755 (6)	3·98	637 (8)	1381 (5)	3251 (6)	4·89
N(12)	5694 (5)	2250 (3)	2766 (4)	2·94	2466 (5)	1491 (3)	3577 (5)	3·36
N(13)	5262 (4)	1847 (3)	2193 (4)	2·97	2915 (5)	1099 (3)	3363 (5)	3·66
C(14)	4750 (6)	1610 (4)	2490 (6)	3·44	3482 (6)	904 (4)	4053 (7)	3·95
C(15)	4838 (6)	1847 (4)	3234 (6)	3·59	3407 (7)	1169 (4)	4710 (6)	3·76
C(16)	5451 (6)	2255 (4)	3393 (6)	3·53	2770 (7)	1545 (4)	4401 (6)	4·01
H(C1)	6168 (6)	3049 (4)	2699 (5)	2·19	2021 (6)	2289 (4)	3054 (6)	2·19
C(17)	7451 (7)	1653 (5)	944 (7)	5·29	665 (7)	747 (5)	470 (7)	5·44
C(18)	5267 (7)	3137 (5)	1054 (7)	5·23	2707 (7)	2381 (5)	1987 (6)	4·57
C(19)	8950 (8)	3214 (6)	4780 (8)	6·65	−816 (9)	2628 (6)	2729 (9)	7·59
C(20)	7097 (7)	1411 (5)	3533 (7)	4·90	982 (8)	767 (5)	3475 (7)	5·64
C(21)	4138 (7)	1141 (5)	2014 (6)	4·62	4109 (7)	445 (3)	4052 (7)	5·65
C(22)	5855 (7)	2669 (5)	4114 (6)	4·47	2378 (7)	1959 (5)	4814 (7)	5·32

will multiply the number of possible conformers. The structure determination will in fact show a completely asymmetric conformation of the molecule.

Experimental. Compound synthesized by Avila, Elguero, Julia & del Mazo (1984). Crystals obtained by slow evaporation from *n*-heptane. Parallelepiped crystal with dimensions 0·12 × 0·18 × 0·28 mm. D_m not measured. Lattice parameters refined using 15 reflexions in the range $5 < 2\theta < 15°$. Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$ radiation. 9717 independent reflexions with $\sin\theta/\lambda \leq 0{\cdot}561$ Å^{-1}, range of *hkl*: h $0\rightarrow19$, k $0\rightarrow25$, l $-20\rightarrow18$, 4137 with $I \geq 2{\cdot}5\sigma(I)$. The small proportion of observed reflexions is due to the poor quality of the crystal which also explains the rather high R value. Standard reflexion (172) checked every 50 reflexions: no significant deviation. Structure solution: a misplaced 17-atom fragment obtained by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The shift vector (0·352, 0·0, 0·0) found by the application of the *TRADIR* procedure of *DIRDIF*81 (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981). The shifted fragment was expanded to four complete molecules by *DIRDIF*81. Anisotropic least-squares refinement (*SHELX*76, Sheldrick, 1976) using F; methyl isotropic. H of central C atoms in computed positons. $w = 1/(\sigma^2 + 0{\cdot}00362F^2)$, $R = 0{\cdot}108$, $R_w = 0{\cdot}114$, $S = 4{\cdot}1$ for 4137 observed reflexions. Final max. shift to error = 0·31. Max. and min. heights in final difference Fourier synthesis: 0·6 and −0·3 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Final atomic parameters are given in Table 1.*

There are four independent molecules in the asymmetric unit. As can be seen from Table 4,* the molecules can be divided into two groups (*A*,*C* and *B*,*D*). Inside each of the groups, the two molecules are approximately related by an inversion center at (0·5, 0·56, 0·25). Their conformations are nearly perfectly the same, the mean absolute difference between the torsional angles being only 2°. Comparing the two groups, the difference becomes slightly larger (6°), but still shows that there is a similarity between the molecules of different groups.

Fig. 1 is a stereoscopic view of molecule *A* giving the numbering of the atoms (*PLUTO*, Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2. A mean value is also given for each of the chemically equivalent bonds.

The helical conformation can be defined by the angle between the mean plane through the pyrazole ring and that defined by C(1), H(C1) and the first N atom of the ring. The values for the three rings averaged over the four molecules are 29 (3), 23 (2) and 62 (1)° [ranges: 24·5 (7)–35·5 (8); 19·8 (7)–27·1 (9); 59·9 (7)–64·4 (7)°]. The molecules do not have threefold symmetry, in contrast with trimesitylmethane (Blount & Mislow, 1975) and tris(1-pyrazolyl)phosphine (Cobbledick & Einstein, 1975) for which the angles are respectively 38 and 36°. They are comparable to the average of our 12 angles: 41 (6)°. Our results are also in good agreement with the values observed by Riche & Pascard-Billy (1974) in triphenylmethane (two independent molecules: 34,30,53 and 38,21,47°).

Defining a plane through the three N atoms bonded to the central C atom, one can assign the α conformation to the region lying on the same side of the plane as the C(1)—H bond and β to the other one. It can be seen that in the four independent molecules, the three other N atoms of the rings adopt a βαβ conformation. This asymmetry in the orientation of the three rings also affects the C(1)—N—C bond angles whose average values are respectively 128·0 (6), 131·9 (8) and 126·6 (4)°, the larger angles corresponding to the α conformation.

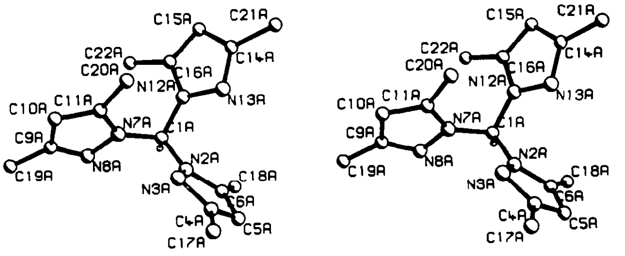

Fig. 1. Stereoscopic view of molecule *A* with atom numbering.

Table 2. *Molecular geometry*

(*a*) Bond distances (Å) and mean values of chemically equivalent bonds

	A	*B*	*C*	*D*	Weighted averages
C(1)—N(2)	1·45 (1)	1·44 (1)	1·44 (1)	1·42 (1)	1·440 (3)
C(1)—N(7)	1·44 (1)	1·43 (1)	1·43 (1)	1·42 (2)	
C(1)—N(12)	1·44 (1)	1·45 (1)	1·44 (1)	1·46 (1)	
N(2)—N(3)	1·36 (1)	1·38 (1)	1·37 (1)	1·38 (1)	1·369 (3)
N(7)—N(8)	1·38 (1)	1·35 (1)	1·38 (1)	1·38 (1)	
N(12)—N(13)	1·37 (1)	1·36 (1)	1·37 (1)	1·35 (1)	
N(2)—C(6)	1·37 (1)	1·38 (2)	1·37 (1)	1·38 (2)	1·365 (4)
N(7)—C(11)	1·36 (1)	1·37 (1)	1·34 (1)	1·36 (2)	
N(12)—C(16)	1·37 (2)	1·37 (1)	1·37 (2)	1·37 (1)	
N(3)—C(4)	1·35 (2)	1·38 (2)	1·35 (2)	1·34 (2)	1·331 (4)
N(8)—C(9)	1·33 (1)	1·33 (2)	1·33 (1)	1·32 (2)	
N(13)—C(14)	1·34 (2)	1·32 (1)	1·34 (2)	1·32 (1)	
C(4)—C(5)	1·38 (2)	1·39 (2)	1·42 (1)	1·40 (2)	1·399 (5)
C(9)—C(10)	1·39 (1)	1·39 (2)	1·38 (2)	1·39 (2)	
C(14)—C(15)	1·41 (2)	1·41 (2)	1·40 (2)	1·39 (2)	
C(5)—C(6)	1·37 (2)	1·37 (2)	1·37 (2)	1·38 (1)	1·365 (4)
C(10)—C(11)	1·37 (1)	1·38 (2)	1·37 (1)	1·37 (2)	
C(15)—C(16)	1·38 (2)	1·35 (1)	1·37 (1)	1·34 (1)	
C(4)—C(17)	1·50 (2)	1·52 (2)	1·52 (2)	1·52 (2)	1·522 (3)
C(9)—C(19)	1·51 (1)	1·50 (2)	1·52 (2)	1·54 (2)	
C(14)—C(21)	1·52 (2)	1·51 (2)	1·52 (1)	1·53 (2)	
C(6)—C(18)	1·52 (2)	1·51 (2)	1·53 (2)	1·50 (2)	
C(11)—C(20)	1·51 (1)	1·53 (2)	1·56 (2)	1·53 (2)	
C(16)—C(22)	1·52 (2)	1·55 (2)	1·54 (1)	1·54 (2)	

(*b*) Bond angles (°) (e.s.d.'s range from 0·7 to 1·2°)

	A	*B*	*C*	*D*
N(2)—C(1)—N(7)	110	112	111	112
N(2)—C(1)—N(12)	111	111	111	111
N(7)—C(1)—N(12)	111	112	112	111
C(1)—N(2)—N(3)	121	121	118	120
C(1)—N(2)—C(6)	127	128	130	128
N(3)—N(2)—C(6)	112	112	112	112
N(2)—N(3)—C(4)	104	103	104	104
N(3)—C(4)—C(5)	112	113	112	112
N(3)—C(4)—C(17)	118	117	119	120
C(5)—C(4)—C(17)	130	130	130	128
C(4)—C(5)—C(6)	105	105	105	106
N(2)—C(6)—C(5)	107	108	108	106
N(2)—C(6)—C(18)	122	121	121	123
C(5)—C(6)—C(18)	132	131	132	131
C(1)—N(7)—N(8)	117	115	118	115
C(1)—N(7)—C(11)	131	133	130	134
N(8)—N(7)—C(11)	112	112	112	110
N(7)—N(8)—C(9)	104	106	104	105
N(8)—C(9)—C(10)	113	111	112	112
N(8)—C(9)—C(19)	119	123	118	120
C(10)—C(9)—C(19)	129	127	130	128
C(9)—C(10)—C(11)	105	106	107	105
N(7)—C(11)—C(10)	107	106	106	108
N(7)—C(11)—C(20)	125	123	124	123
C(10)—C(11)—C(20)	128	132	130	129
C(1)—N(12)—N(13)	122	121	122	122
C(1)—N(12)—C(16)	126	127	126	127
N(13)—N(12)—C(16)	112	111	112	111
N(12)—N(13)—C(14)	103	105	103	105
N(13)—C(14)—C(15)	114	112	113	111
N(13)—C(14)—C(21)	118	119	120	120
C(15)—C(14)—C(21)	128	129	127	129
C(14)—C(15)—C(16)	103	105	104	106
N(12)—C(16)—C(15)	108	108	108	107
N(12)—C(16)—C(22)	122	122	121	122
C(15)—C(16)—C(22)	130	130	131	131
H(C1)—C(1)—N(2)	109	108	108	107
H(C1)—C(1)—N(7)	109	107	107	107
H(C1)—C(1)—N(12)	107	108	108	108

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* Lists of structure factors, anisotropic thermal parameters and torsion angles (Table 4) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39289 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2′,2′-Dimethylspiro[bicyclo[2.2.1]hept-5-ene-7,1′-cyclopropane]-2,3-dicarbonitrile, $C_{13}H_{14}N_2$

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Abstract. $M_r = 198\cdot13$, monoclinic, $P2_1/c$, $a = 10\cdot973\,(6)$, $b = 7\cdot611\,(3)$, $c = 13\cdot842\,(3)$ Å, $\beta = 91\cdot27\,(3)^\circ$, $U = 1155\cdot7\,(8)$ Å^3, $Z = 4$, $D_x = 1\cdot14$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 6\cdot4$ mm^{-1}, $F(000) = 424$, $T = 298$ K. Final $R = 0\cdot092$ for 1285 observed unique reflections. The structure of the title compound has been established and correlates well with the observed NMR data. The bond lengths and angles are normal.

Introduction. During the course of our photochemical studies, we obtained a crystalline 1:1 adduct from irradiation (254 nm) of 3,3-dimethyl-6-methylenecyclohexa-1,4-diene (0·06 mol dm^{-3}) in acetonitrile in the presence of fumaronitrile (0·14 mol dm^{-3}). The ^{1}H NMR spectrum and other data did not distinguish among possible isomers. This X-ray structural study was undertaken to establish the structure.

Experimental. Platy crystals, dimensions $0\cdot10 \times 0\cdot83 \times 0\cdot77$ mm. Syntex $P2_1$ four-circle diffractometer. $2\theta_{\max} = 50^\circ$, scan range $\pm\,1\cdot0^\circ$ (2θ) around $K\alpha_1$–$K\alpha_2$ angles, scan speed 2–29° min^{-1}, depending upon intensity of a 2 s pre-scan; backgrounds at each end of scan for 0·25 scan time. Three standard reflections monitored every 100 reflections showed irregularities during data collection (probably due to counter instability of electronic origin); data rescaled using a sliding point-to-point scale to correct for this. Unit-cell dimensions and standard deviations by least-squares fit to 15 high-angle reflections. 2299 reflections measured, 2023 unique, 738 unobserved $[I/\sigma(I) < 3\cdot0]$. Range of hkl: $h\ -12\rightarrow13$, $k\ 0\rightarrow8$, $l\ 0\rightarrow16$. No absorption correction. Systematic absences indicated space group $P2_1/c$. Structure solution readily achieved using direct-methods link of *SHELXTL* (Sheldrick, 1981). Refinement by cascaded least squares on F (unit weights) using anisotropic temperature factors for all non-H atoms. H atoms (fixed isotropic temperature factors of 0·07–0·08 Å^2) inserted at calculated positions, with methyl groups treated as rigid bodies. Final $R = 0\cdot092$, $S = 1\cdot6$. The relatively high R value is attributed to the counter instability already noted. As the structure solution proceeded satisfactorily and bond e.s.d.'s were not excessive, it was not felt that recollection would be worthwhile. $(\Delta/\sigma)_{\max} = 0\cdot18$. Max. and min. height in final ΔF map 0·3 and $-0\cdot4$ e Å^{-3}. Computing with *SHELXTL* on a Data General Nova 3. Scattering factors from *International Tables for X-ray Crystallography* (1974).

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