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# The Crystal and Molecular Structure of Benzoyl-DL-leucylglycine Ethyl Ester

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The crystal structure of the modified dipeptide benzoyl-DL-leucylglycine ethyl ester has been determined by direct methods and refined by full-matrix least-squares calculations to $R=0.0388$ for 2496 observed reflexions. The crystals are monoclinic, $P2_1/n$, $a=15.104$ (8), $b=16.631$ (9), $c=14.700$ (8) Å, $\beta=91.28$ (2)° with two molecules per asymmetric unit. These two molecules have slightly different conformations and pack in the crystal in a manner similar to a twisted parallel pleated sheet.

## Introduction

The structure of benzoyl-DL-leucylglycine ethyl ester (Fig. 1) has been determined and compared with similar structures of heavy-atom derivatives (Rao, 1969; Chandrasekaran & Subramanian, 1969). The presence of two molecules in the asymmetric unit allows an internal check of molecular parameters and also a comparison with related structures.

## Experimental

Transparent lath-shaped crystals were obtained from ethanol/benzene. Preliminary precession photographs indicated space group $P2_1/n$ and cell dimensions obtained by least-squares refinement of the $\theta$ values of 20 reflexions on a four-circle diffractometer were $a=15.104$ (8), $b=16.631$ (9), $c=14.700$ (8) Å, $\beta=91.28$ (2)°. The crystal density by flotation in aqueous KI solution was 1·151 g cm$^{-3}$ indicating an asymmetric unit molecular weight of 640, *i.e.*, two molecules of $C_{17}H_{24}N_2O_4$ per asymmetric unit. The absorption

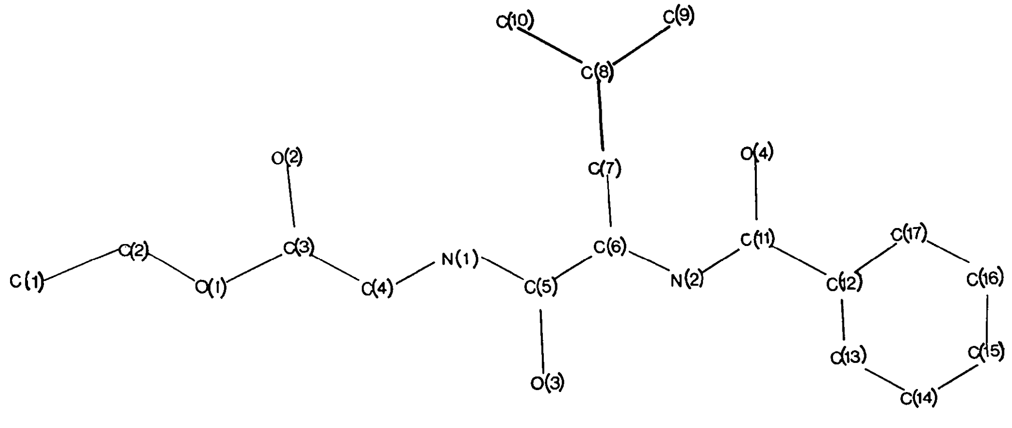

Fig. 1. Benzoylleucylglycine ethyl ester numbering scheme for non-H atoms.

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Table 1. *Refined atomic coordinates for non-*H *atoms* (×10⁴) *with e.s.d.'s in parentheses*

| | Molecule 1 | | | Molecule 2 | | |
|---|---|---|---|---|---|---|
| | *x* | *y* | *z* | *x* | *y* | *z* |
| C(1) | 7633 (10) | 2084 (14) | 1636 (10) | 1392 (9) | 396 (7) | −2309 (6) |
| C(2) | 6965 (4) | 2367 (8) | 1288 (5) | 1102 (8) | 456 (6) | −1486 (5) |
| O(1) | 6230 (2) | 2404 (3) | 1926 (2) | 1787 (3) | 103 (3) | −819 (2) |
| C(3) | 5429 (3) | 2476 (3) | 1579 (3) | 1601 (3) | 229 (2) | 34 (2) |
| O(2) | 5271 (2) | 2492 (3) | 800 (3) | 940 (2) | 554 (2) | 267 (2) |
| C(4) | 4772 (2) | 2525 (2) | 2324 (2) | 2308 (2) | −101 (2) | 646 (2) |
| N(1) | 3885 (2) | 2635 (2) | 1968 (2) | 2136 (2) | 73 (1) | 1582 (2) |
| C(5) | 3483 (2) | 3347 (2) | 1965 (2) | 2363 (2) | 773 (2) | 1955 (2) |
| O(3) | 3809 (1) | 3953 (1) | 2312 (2) | 2791 (2) | 1277 (1) | 1552 (2) |
| C(6) | 2613 (2) | 3359 (2) | 1428 (2) | 1992 (2) | 921 (2) | 2891 (2) |
| C(7) | 2798 (2) | 3676 (2) | 473 (2) | 1067 (2) | 1284 (2) | 2787 (2) |
| C(8) | 2046 (3) | 3530 (3) | −214 (3) | 607 (2) | 1483 (2) | 3670 (3) |
| C(9) | 2188 (4) | 4098 (5) | −1025 (3) | −294 (3) | 1871 (3) | 3451 (4) |
| C(10) | 1989 (4) | 2663 (4) | −500 (4) | 521 (4) | 763 (3) | 4282 (3) |
| N(2) | 1945 (2) | 3861 (1) | 1853 (2) | 2571 (2) | 1441 (1) | 3433 (2) |
| C(11) | 1633 (2) | 3660 (2) | 2668 (2) | 3188 (2) | 1158 (2) | 4006 (2) |
| O(4) | 1966 (1) | 3097 (1) | 3108 (2) | 3316 (2) | 424 (1) | 4090 (2) |
| C(12) | 854 (2) | 4103 (2) | 3017 (2) | 3734 (2) | 1741 (2) | 4539 (2) |
| C(13) | 425 (3) | 4709 (2) | 2535 (3) | 3772 (2) | 2554 (2) | 4343 (3) |
| C(14) | −330 (3) | 5057 (3) | 2899 (4) | 4325 (3) | 3057 (3) | 4841 (3) |
| C(15) | −649 (4) | 4797 (4) | 3713 (4) | 4823 (3) | 2761 (3) | 5549 (3) |
| C(16) | −229 (4) | 4207 (3) | 4188 (3) | 4778 (2) | 1962 (2) | 5763 (2) |
| C(17) | 531 (3) | 3864 (2) | 3853 (3) | 4249 (3) | 1450 (2) | 5252 (2) |

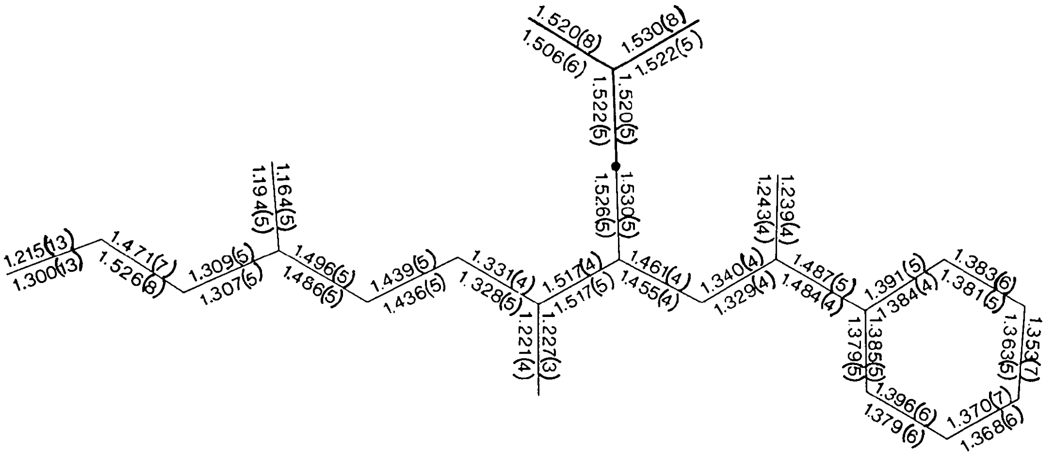

Fig. 2. Benzoylleucylglycine ethyl ester bond lengths with e.s.d.'s in parentheses.

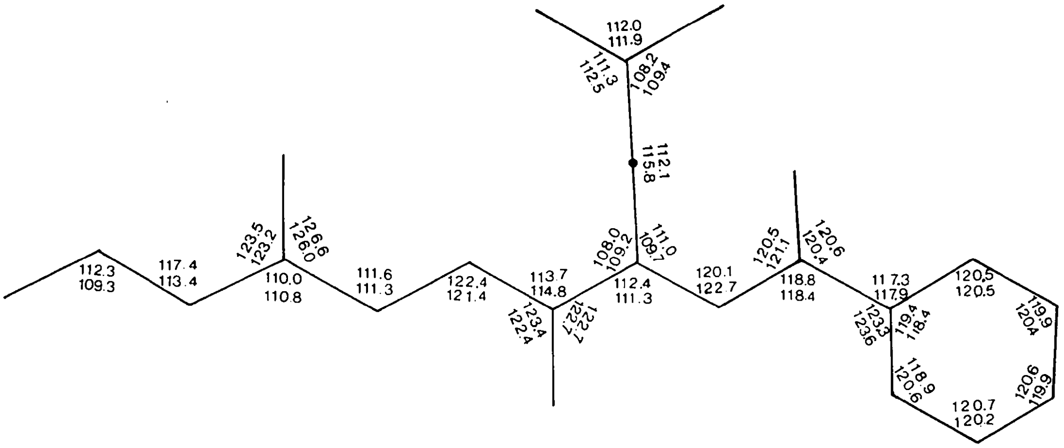

Fig. 3. Benzoylleucylglycine ethyl ester bond angles. E.s.d.'s are from 0·2° → 0·5° except for angles involving C(1), C(2), O(1), O(2), where they are 1° → 3°.

coefficient for Cu $K\alpha$ is 6·83 cm$^{-1}$. Intensity data were collected on a Hilger & Watts Y290 four-circle diffractometer using an $\omega$–$2\theta$ scan with counting steps of 0·01° and a scan range of 0·80°. 3804 reflexions were collected of which 2496 were significant ($I \geq 3\sigma$).

The structure was solved by direct methods with the program *MULTAN* (Germain, Main & Woolfson, 1971). Phases were calculated for the 248 $E$ values greater than 1·9 using the reflexions $6,13,\bar{1}$, 434, $1,15,\bar{2}$ as origin-defining reflexions and 743, 131 and $10\bar{1}$ as the starting set. Eight sets of phases were calculated and that using all phase angles in the starting set as 360° gave the highest figure of merit (1·153). An $E$ map calculated with the 248 $E$ values gave 40 atomic peaks in stereochemically feasible positions. Successive Fourier syntheses revealed the remaining six atoms. The conventional $R$ value at this stage was 0·38.

The structure was refined by full-matrix least-squares calculations using a modified version of the *SFLS* program of D. W. J. Cruickshank and J. F. Smith. The function minimized was $\sum w(|F_o|-G|F_c|)^2$ where $|F_o|$, $|F_c|$ and $G$ are the observed and calculated structure amplitudes and scale factor respectively, and $w$ is a weight for each reflexion given by: $w=$ if $|F_o|>F_{\min}$ then $\{1-\exp[-a(\sin\theta/\lambda)^2]\}/(b+|F_o|+c|F_o|^2+d|F_o|^3)$ else $e$. The final values used for the constants

Table 2. *Anisotropic thermal parameters of non-*H *atoms* (× 10$^4$) *with e.s.d.'s in parentheses*

$U^2=U_{11}l_1^2+U_{22}l_2^2+U_{33}l_3^2+2U_{32}l_3l_2+2U_{31}l_3l_1+2U_{21}l_2l_1$ (Cruickshank, 1964).

Molecule 1

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $2U_{32}$ | $2U_{31}$ | $2U_{21}$ |
|---|---|---|---|---|---|---|
| C(1) | 2074 (112) | 6088 (316) | 2673 (131) | −4437 (351) | 2997 (213) | −3978 (320) |
| C(2) | 657 (32) | 4243 (153) | 1258 (48) | −635 (135) | 528 (66) | −1022 (122) |
| O(1) | 569 (19) | 2603 (48) | 924 (20) | −287 (49) | 269 (31) | −651 (45) |
| C(3) | 657 (26) | 1063 (31) | 650 (26) | −234 (42) | 157 (40) | −229 (41) |
| O(2) | 981 (23) | 2620 (53) | 719 (22) | −442 (50) | 242 (34) | −312 (54) |
| C(4) | 567 (20) | 768 (23) | 722 (21) | −26 (35) | 62 (34) | −350 (34) |
| N(1) | 511 (16) | 456 (16) | 745 (17) | 97 (25) | −57 (26) | −118 (26) |
| C(5) | 522 (18) | 437 (19) | 563 (18) | 29 (30) | 57 (29) | 42 (32) |
| O(3) | 602 (13) | 505 (13) | 791 (14) | 228 (23) | −131 (22) | 52 (21) |
| C(6) | 511 (18) | 447 (16) | 568 (18) | 53 (28) | −32 (29) | 8 (27) |
| C(7) | 661 (20) | 674 (20) | 560 (19) | −26 (32) | 34 (32) | −27 (33) |
| C(8) | 832 (27) | 1355 (38) | 565 (22) | 96 (48) | −219 (39) | −230 (50) |
| C(9) | 1496 (49) | 1952 (61) | 724 (30) | −471 (69) | −288 (59) | −873 (91) |
| C(10) | 1466 (46) | 1499 (48) | 888 (31) | 896 (65) | −449 (60) | 710 (67) |
| N(2) | 475 (14) | 420 (13) | 507 (15) | −44 (23) | 22 (23) | −44 (22) |
| C(11) | 491 (18) | 382 (17) | 590 (20) | 55 (31) | −161 (31) | 22 (28) |
| O(4) | 673 (14) | 510 (12) | 676 (13) | −214 (23) | 39 (21) | −204 (22) |
| C(12) | 517 (18) | 469 (17) | 653 (21) | 116 (31) | −123 (31) | −52 (30) |
| C(13) | 694 (23) | 754 (24) | 882 (24) | −107 (41) | 95 (39) | −522 (41) |
| C(14) | 884 (31) | 968 (31) | 1286 (39) | −143 (58) | 52 (57) | −789 (52) |
| C(15) | 1001 (34) | 1322 (42) | 1098 (36) | 62 (68) | 702 (60) | −677 (65) |
| C(16) | 1076 (35) | 1165 (36) | 910 (29) | −4 (57) | 606 (54) | −530 (61) |
| C(17) | 819 (25) | 793 (24) | 661 (23) | 54 (38) | 248 (39) | −244 (41) |

Molecule 2

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $2U_{32}$ | $2U_{31}$ | $2U_{21}$ |
|---|---|---|---|---|---|---|
| C(1) | 3419 (148) | 2558 (110) | 1150 (60) | −282 (129) | −1521 (148) | −1209 (207) |
| C(2) | 3089 (117) | 2505 (96) | 797 (41) | 461 (96) | −1141 (107) | −2568 (176) |
| O(1) | 1498 (29) | 1712 (33) | 521 (17) | 86 (35) | −254 (33) | −693 (49) |
| C(3) | 918 (30) | 681 (23) | 606 (25) | 49 (36) | −116 (41) | −181 (44) |
| O(2) | 1060 (23) | 1278 (25) | 928 (20) | 113 (36) | −516 (35) | −811 (42) |
| C(4) | 808 (22) | 496 (18) | 557 (19) | 157 (30) | −185 (33) | −147 (32) |
| N(1) | 689 (17) | 406 (15) | 512 (15) | 42 (25) | −119 (24) | 119 (25) |
| C(5) | 588 (19) | 400 (18) | 551 (18) | −59 (32) | −284 (30) | 7 (30) |
| O(3) | 973 (17) | 535 (13) | 677 (14) | 35 (23) | 197 (25) | 392 (26) |
| C(6) | 621 (19) | 431 (16) | 506 (17) | −59 (27) | −232 (29) | 109 (29) |
| C(7) | 554 (19) | 645 (19) | 598 (19) | 56 (32) | −139 (30) | 128 (32) |
| C(8) | 685 (23) | 671 (22) | 756 (23) | 147 (36) | 143 (35) | 194 (34) |
| C(9) | 672 (26) | 1225 (35) | 1179 (33) | 432 (58) | 268 (45) | 188 (49) |
| C(10) | 1572 (46) | 907 (31) | 995 (31) | −376 (52) | 1022 (62) | −4 (60) |
| N(2) | 598 (16) | 360 (13) | 525 (14) | −3 (23) | −297 (26) | 66 (23) |
| C(11) | 592 (19) | 414 (20) | 481 (17) | −39 (28) | −46 (30) | −91 (29) |
| O(4) | 914 (17) | 422 (14) | 798 (15) | 133 (22) | −522 (25) | −191 (23) |
| C(12) | 517 (17) | 475 (19) | 447 (16) | 77 (28) | −111 (28) | −47 (28) |
| C(13) | 579 (18) | 527 (21) | 583 (18) | 94 (31) | −180 (30) | 45 (30) |
| C(14) | 684 (22) | 599 (20) | 734 (22) | 311 (36) | −149 (38) | 79 (35) |
| C(15) | 682 (23) | 834 (29) | 824 (25) | 302 (43) | −449 (40) | 185 (40) |
| C(16) | 900 (28) | 925 (32) | 891 (27) | −48 (47) | −1033 (46) | −18 (47) |
| C(18) | 843 (26) | 646 (22) | 758 (23) | 38 (38) | −580 (42) | −133 (38) |

Table 3. *Refined hydrogen atom coordinates* ($\times 10^4$) *and isotropic temperature factors* ($\times 10^3$)

$U=\overline{U^2}$ = mean square amplitude of vibration (Cruickshank, 1964).

| | Molecule 1 | | | | Molecule 2 | | | |
|---|---|---|---|---|---|---|---|---|
| | *x* | *y* | *z* | *U* | *x* | *y* | *z* | *U* |
| H′(C1) | 8082 (109) | 1942 (99) | 1076 (108) | 311 (67) | 1079 (41) | 510 (37) | −2703 (42) | 119 (19) |
| H″(C1) | 7575 (81) | 1565 (69) | 2176 (79) | 232 (43) | 1327 (99) | −214 (91) | −1919 (102) | 247 (69) |
| H‴(C1) | 7552 (26) | 2775 (24) | 1764 (27) | 61 (11) | 2107 (54) | 766 (50) | −2078 (55) | 165 (28) |
| H′(C2) | 6833 (54) | 1872 (52) | 830 (53) | 160 (27) | 1063 (17) | −108 (16) | −1627 (17) | 2 (6) |
| H″(C2) | 6773 (55) | 2744 (52) | 875 (55) | 162 (27) | 635 (91) | 923 (82) | −1281 (90) | 265 (55) |
| H′(C4) | 4947 (28) | 3001 (26) | 2762 (28) | 68 (12) | 2908 (27) | 195 (25) | 485 (27) | 66 (12) |
| H″(C4) | 4832 (24) | 1992 (22) | 2687 (24) | 51 (10) | 2299 (24) | −673 (23) | 601 (25) | 54 (10) |
| H(N1) | 3592 (24) | 2220 (22) | 1771 (25) | 50 (10) | 1850 (19) | −232 (17) | 1871 (19) | 27 (7) |
| H(C6) | 2362 (17) | 2790 (16) | 1384 (18) | 17 (7) | 1958 (16) | 425 (15) | 3218 (16) | 10 (6) |
| H′(C7) | 3320 (22) | 3375 (20) | 251 (22) | 42 (9) | 711 (23) | 927 (21) | 2424 (23) | 46 (9) |
| H″(C7) | 2887 (19) | 4253 (18) | 528 (19) | 28 (8) | 1118 (23) | 1791 (21) | 2381 (23) | 47 (9) |
| H(C8) | 1443 (36) | 3776 (33) | 54 (37) | 98 (16) | 966 (20) | 1864 (18) | 4007 (20) | 28 (8) |
| H′(C9) | 2831 (38) | 3937 (34) | −1369 (39) | 109 (18) | −659 (29) | 1514 (27) | 3124 (30) | 75 (13) |
| H″(C9) | 1760 (39) | 3981 (35) | −1477 (39) | 110 (18) | −613 (31) | 2010 (29) | 4033 (32) | 82 (14) |
| H‴(C9) | 2252 (43) | 4770 (39) | −803 (49) | 127 (21) | −182 (33) | 2362 (30) | 2946 (34) | 89 (15) |
| H′(C10) | 1548 (38) | 2560 (34) | −945 (39) | 107 (17) | 167 (39) | 298 (35) | 3901 (40) | 113 (18) |
| H″(C10) | 2612 (47) | 2473 (42) | −819 (46) | 137 (23) | 202 (37) | 949 (33) | 4810 (38) | 104 (17) |
| H‴(C10) | 1899 (39) | 2306 (36) | 61 (40) | 113 (18) | 1119 (24) | 567 (22) | 4531 (25) | 52 (10) |
| H(N2) | 1789 (21) | 4307 (19) | 1537 (22) | 37 (9) | 2479 (17) | 1964 (15) | 3397 (17) | 13 (6) |
| H(C13) | 641 (25) | 4854 (23) | 1938 (25) | 57 (10) | 3385 (21) | 2791 (19) | 3854 (22) | 38 (9) |
| H(C14) | −601 (28) | 5454 (26) | 2541 (29) | 70 (12) | 4371 (25) | 3656 (23) | 4729 (25) | 55 (10) |
| H(C15) | −1218 (36) | 5059 (34) | 3944 (36) | 104 (17) | 5231 (25) | 3097 (23) | 5874 (25) | 54 (10) |
| H(C16) | −423 (33) | 3987 (30) | 4833 (35) | 92 (15) | 5131 (24) | 1737 (22) | 6223 (24) | 49 (10) |
| H(C17) | 850 (27) | 3386 (24) | 4174 (27) | 65 (12) | 4260 (25) | 889 (23) | 5396 (25) | 55 (10) |

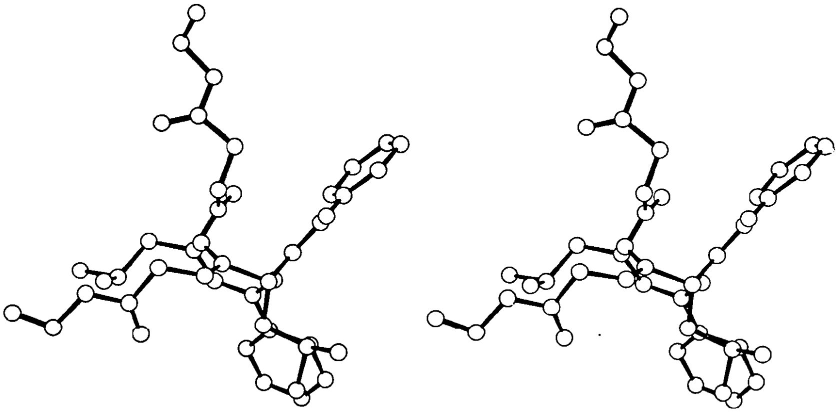

Fig. 4. Stereo view of the asymmetric unit in L configuration with hydrogen atoms omitted. Viewing direction parallel to **b**.

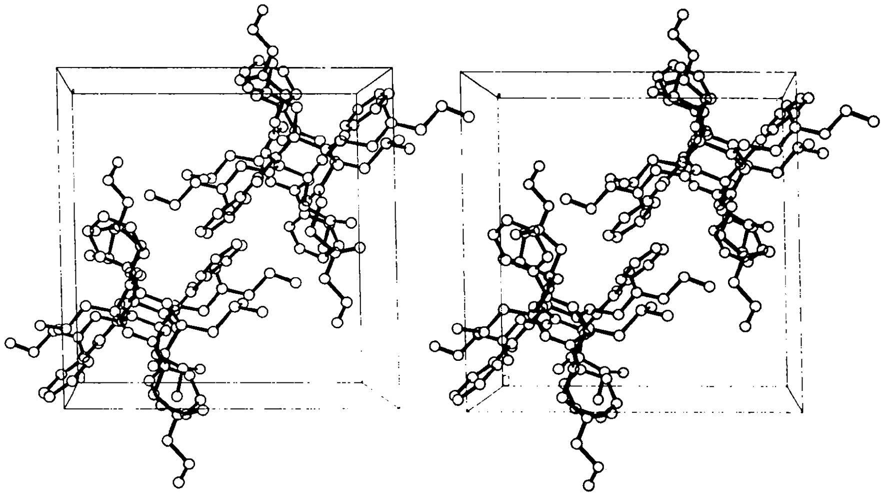

Fig. 5. Packing of molecules in the unit cell. Stereo view parallel to **b**.

were $a=3{\cdot}00$, $b=60{\cdot}0$, $c=4{\cdot}0\times10^{-3}$, $d=0{\cdot}00$, $e=0{\cdot}00$ and $F_{\min}=0{\cdot}00$. Refinement of positional and anisotropic thermal parameters reduced $R$ to 0·085 at which stage a difference map revealed all hydrogen atoms except those attached to C(1) and C(2). The ethyl ester groups, from their thermal parameters, were clearly disordered and for this reason their hydrogen atom positions were calculated from stereochemical considerations. The positional and anisotropic thermal parameters of the non-hydrogen atoms of the two independent molecules and all 48 hydrogen atom positions and isotropic thermal parameters were refined separately in alternate cycles owing to the limitations of computer store. This refinement was terminated when all parameter shifts were less than 0·1 e.s.d. at which stage $R=0{\cdot}0388$ for observed reflexions, 0·0640 for all reflexions and $R$(weighted) = 0·0557.

Scattering factors used were O, N and C (Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal, 1955) and H (Stewart, Davidson & Simpson, 1965).*

All calculations other than the refinement were carried out with programs from the Birkbeck College Crystallography Program Library run on the University of London CD6600 or the University College London IBM360 computers.

## Discussion

The fractional coordinates of all non-hydrogen atoms are given in Table 1 and their anisotropic thermal parameters in Table 2. All bond lengths (Fig. 2) and angles (Fig. 3) are reasonable except in the case of dimensions involving atoms C(1), C(2), O(1) and O(2) which are highly disordered. There are no significant differences between the crystallographically independent molecules. A correction for thermal motion does not significantly improve the dimensions within the disordered groups. The fractional coordinates of the hydrogen atoms and their isotropic thermal parameters are given in Table 3. The abnormally long bond lengths found in the leucyl side chain of *N*-methyl-DL-leucylglycine hydrobromide (Chandrasekaran & Subramanian, 1969) and L-leucylglycine hydrobromide (Rao, 1969) are not found in the present structure. Values are in good agreement with the structure of di-L-leucine hydrochloride (Golič & Hamilton, 1972). The conformations of the molecules may be described in terms of the principal torsion angles (Table 4) from which it is seen that the two molecules in the asymmetric unit differ chiefly in a rotation of 30° about the bond C(6)–N(2). Fig. 4 is a stereo view of the asymmetric unit with hydrogen atoms omitted for clarity.

Table 4. *Torsion angles*

Values correspond to L configuration at $C^{\alpha}$. Convention is that of the IUPAC-IUB Commission on Biochemical Nomenclature (1970).

| Angle | Molecule 1 | Molecule 2 |
|---|---|---|
| O(2)–C(3)—C(4)—N(1) | −178·2 | −177·1 |
| C(3)–C(4)—N(1)—C(5) | 101·8 | 83·8 |
| C(4)–N(1)—C(5)—C(6) | 174·5 | 173·9 |
| N(1)–C(5)—C(6)—N(2) | −142·6 | −151·4 |
| C(5)–C(6)—N(2)—C(11) | 64·8 | 94·2 |
| C(6)–N(2)—C(11)–C(12) | 170·1 | 178·9 |
| N(2)–C(11)–C(12)–C(13) | 179·5 | −167·6 |
| C(5)–C(6)—C(7)—C(8) | 166·8 | −178·5 |
| C(6)–C(7)—C(8)—C(9) | 74·1 | 57·4 |
| C(6)–C(7)—C(8)—C(10) | −162·3 | −178·1 |

Intermolecular hydrogen bonds are formed through atoms N(1), N(2), O(3) and O(4) of both molecules but the oxygen atoms of the ethyl ester group are found in a predominantly hydrophobic environment sandwiched between benzene rings and the leucyl side chains. The molecules pack in the crystal as separate columns of D and L molecules (Fig. 5); there are no hydrogen bonds between a D and an L molecule. This packing is consistent with the disorder of the ethyl ester groups and high thermal parameters of the benzene and leucyl side chain. The hydrogen bonding is similar to that of a parallel pleated sheet but with adjacent chains rotated by about 90° as shown in Fig. 6. This twisted $\beta$-structure is being increasingly found as an important secondary structure in globular proteins (Liljas *et al.*, 1972; Wright, Alden & Kraut, 1969) and

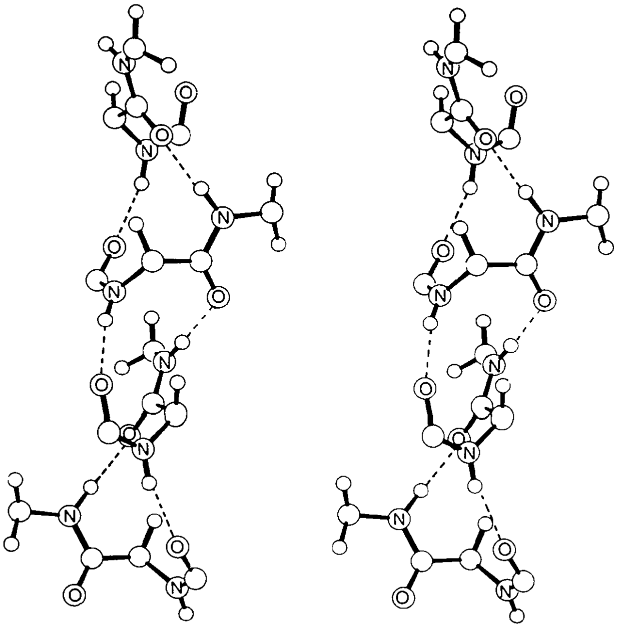

Fig. 6. Hydrogen-bonding system showing parallel pleated sheet structure. Atoms of the ester, leucyl and phenyl group are omitted as these do not involve hydrogen bonds. Large unlabelled atoms are carbon and small atoms hydrogen. The L configuration is shown.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31009 (12 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

thus its occurrence in a small crystal structure may be of considerable interest. Hydrogen-bond distances and angles are given in Table 5.

Table 5. *Hydrogen-bond lengths and angles*

| X | H | Y | X···Y | ∠X···H···Y |
|---|---|---|---|---|
| N(1)$_2^{(i)}$ | H(N1)$_2^{(i)}$ | O(3)$_1^{(ii)}$ | 2·873 (5) Å | 175·5 (2·7)° |
| O(3)$_2^{(i)}$ | H(N1)$_1^{(ii)}$ | N(1)$_1^{(i)}$ | 2·856 (5) | 168·6 (2·9) |
| N(2)$_2^{(i)}$ | H(N2)$_2^{(i)}$ | O(4)$_1^{(i)}$ | 2·937 (4) | 164·6 (2·4) |
| O(4)$_2^{(i)}$ | H(N2)$_1^{(ii)}$ | N(2)$_1^{(ii)}$ | 2·968 (5) | 168·1 (2·8) |

Subscript 1 or 2 refers to molecule 1 or 2 within the asymmetric unit. Superscripts: (i) $x,y,z$. (ii) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

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# The Crystal Structure of 7-Amino-2*H*,4*H*-*vic*-triazolo[4,5-*c*]-1,2,6-thiadiazine-1,1-dioxide Monohydrate (ATTM)

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The crystal structure of 7-amino-2*H*,4*H*-*vic*-triazolo[4,5-*c*]-1,2,6-thiadiazine-1,1-dioxide monohydrate has been determined by single-crystal X-ray diffraction techniques. The compound crystallizes in the monoclinic system, space group $P2_1/c$ with $a=10{\cdot}552$ (2), $b=8{\cdot}509$ (2), $c=8{\cdot}835$ (2) Å, $\beta=90{\cdot}34$ (1)° and $Z=4$. Intensity data were collected with a four-circle diffractometer using Mo $K\alpha$ radiation. The crystal structure was solved by a three-dimensional Patterson synthesis and refined by the least-squares method to a final $R$ of 0·055 for 1708 observed reflexions. The packing in the crystal is determined by hydrogen bonds in which all available groups participate. The water molecule has a trigonal coordination.

## Introduction

This compound (hereinafter ATTM) was known to be similar to the previously reported anhydrous analogue (hereinafter ATT) (Foces-Foces, Cano & García-Blanco, 1974), and the present work was undertaken in order to determine the main characteristics of the N–SO$_2$–N group and compare them with those of ATT. Initially, the chemical formula of ATTM was not exactly known. The structure analysis confirmed the formulation $C_3H_4N_6O_2S.H_2O$.

## Experimental

A summary of the crystal data is given in Table 1.

Table 1. *Crystal data*

Standard deviations, given in parentheses refer to the least significant digit.

$C_3H_4N_6O_2S.H_2O$
Space group: $P2_1/c$
$\lambda$(Mo $K\bar{\alpha}$) = 0·7107 Å
$a=10{\cdot}553$ (2) Å
$b=8{\cdot}509$ (2) Å
$c=8{\cdot}835$ (2) Å
$\beta=90{\cdot}34$ (1)°

M.W. 206·06
$Z=4$
$V=791{\cdot}6$ Å$^3$
$D_{obs}$(flotation) = 1·69 g cm$^{-3}$
$D_{cal}$ = 1·72 g cm$^{-3}$
$F_{000}=424$
$\mu=3{\cdot}946$ cm$^{-1}$

A crystal of approximate dimensions 0·50 × 0·20 × 0·08 mm was mounted on a Philips four-circle single-crystal diffractometer. Intensities were recorded in the $\omega$–$2\theta$ scan mode with $\theta$ between 2° and 30°. Graphite monochromatized Mo $K\alpha$ radiation was used. 2302 reflexions were measured, of which 1708 had intensities