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The Crystal and Molecular Structure of Hexacyclo[10,3,1,0^{2,10},0^{3,7},0^{6,15},0^{9,14}]hexadecane, an Ethano-Bridged Diamantane

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The structure of a hydrocarbon, m.p. $110-115^{\circ}$ C, obtained from two different cyclooctatetraene dimers by hydrogenation followed by aluminum halide rearrangement, has been determined to be an ethanodiamantane, hexacyclo[10,3,1,0^{2,10},0^{3,7},0^{6,15},0^{9,14}]hexadecane. The crystals are monoclinic with unitcell dimensions: $a = 9 \cdot 151 \pm 0 \cdot 001$, $b = 6 \cdot 510 \pm 0 \cdot 001$, $c = 19 \cdot 733 \pm 0 \cdot 003$ Å and $\beta = 98 \cdot 62 \pm 0 \cdot 02^{\circ}$. The space group is $P2_1/c$ with four molecules in the unit cell. The structure was solved by a straightforward application of the symbolic addition procedure and refined by full-matrix least-squares methods with anisotropic thermal parameters for the carbon atoms and isotropic thermal parameters for the hydrogen atoms. The final *R* value is 0.044 for 1615 observed reflections. The average standard deviation in the C-C bond distances is 0.002 Å and that in the C-H bond is 0.02 Å. The C-C bond distances lie in the range $1 \cdot 516 - 1 \cdot 552$ Å (mean $1 \cdot 534$ Å), and the bond angles range from 99.2 to $114 \cdot 5^{\circ}$ (mean $109 \cdot 1^{\circ}$). The molecule has seven six-membered rings; the mean torsion angle in all of them is greater than the value of 56° found in cyclohexane itself. The cyclopentane ring is in the envelope conformation with the puckered atom deviating 0.4 Å from the plane of the remaining atoms.

Introduction

The ethano-bridged diamantane molecules I and II are prepared by aluminum halide rearrangement of two different isomeric polycyclic hydrocarbons III and IV. Isomer I (about 15%) had a melting point of 110– 111.5° , while II (about 85%) melted between 206.5–208°. The lower melting isomer formed suitable crystals and



the X-ray structure analysis of this compound was initiated to determine which of the two possible structures (I or II) was actually correct. A preliminary communication on this investigation including details of the chemical reactions has already been published (Rao, Sundaralingam, Osawa, Wiskott & Schleyer, 1970).

The crystal structures of four other cage hydrocarbons, congressane (Karle & Karle, 1965), adamantane (Nordman & Schmitkons, 1965), diadamantane (Alden, Kraut & Taylor, 1968) and tetramantane (Schleyer, Osawa & Drew, 1968) have been determined to date. In the former two cases the molecules occupy a special position in the lattice and thus are constrained to the corresponding symmetry. In diadamantane the two halves of the molecule are related by an inversion center.

Experimental

Crystals of the compound were supplied by Professor P. von R. Schleyer of Princeton University. A crystal of dimensions $0.15 \times 0.2 \times 0.5$ mm was chosen for the analysis and was sealed inside a Lindemann capillary tube to minimize sublimation. Preliminary oscillation and Weissenberg photographs showed the crystal to be monoclinic with space group $P2_1/c$. The cell constants (Table 1) were determined using reflections with medium 2θ angle on a Picker four-circle diffractometer and Ni-filtered Cu radiation. Intensity data were collected by the θ -2 θ scan method at a scan speed of 2° min⁻¹ on the Picker four-circle diffractometer. The crystal was mounted with the b axis coincident with the φ axis of the goniostat. 2080 reflections with $2\theta \leq$ 134° were measured. Using the criterion that $I > 1.5\sigma$ (1), 1615 reflections were considered observed, and were corrected for the usual Lorentz and polarization factors.

Table 1. Crystal data

| Stoichiometry | $C_{16}H_{22}$ |
|-------------------------|----------------------------------|
| Cell dimensions | $a = 9.151 \pm 0.001$ Å |
| | $b = 6.510 \pm 0.001$ |
| | $c = 19.733 \pm 0.003$ |
| | $\beta = 98.62 \pm 0.02^{\circ}$ |
| Systematic absences | 0k0, k = 2n + 1; h0l, l = 2n + 1 |
| Space group | $P2_{1}/c$ |
| Ż | 4 |
| Observed density | 1.220 g.cm ⁻³ |
| Calculated density | 1.228 g.cm ⁻³ |
| μ for Cu K α | 9·2 cm ⁻¹ |

Structure analysis and refinement

The intensity distribution showed characteristics of hypercentering. The structure was solved by a straightforward application of the symbolic addition procedure (Karle & Karle, 1966). Two hundred sixty-eight reflections with E > 1.4 were used for the sign determination. The three origin-determining signs and three symbolic phases used initially are given in Table 2. The signs were generated by a program written by Long (1965). The most probable solution was found to be correct, and the E map calculated with these signs showed all the carbon atoms in the structure.

Table 2. Reflections used to assign the origin phases and initiate the process of sign determination

| h -4 3 3 | k 3 3 1 | <i>I</i> 10 10 13 | E 5·21 4·11 4·00 | Sign + + + origin |
|-------------------|------------------|----------------------------|---------------------------|-------------------------------|
| 0 | 3 | 6 | 3·84 | a |
| 0 | 1 | 2 | 3·44 | b |
| -4 | 1 | 6 | 3·26 | c |

Three cycles of isotropic full-matrix least-squares (Busing, Martin & Levy, 1962) reduced the R value to 0.09. A difference electron-density map calculated at this stage revealed the positions of all the hydrogen atoms. The refinement was continued, with anisotropic thermal parameters for the heavy atoms and isotropic thermal parameters for the hydrogen atoms, until the shifts in the parameters were less than 10% of the estimated standard deviations. The final R value was 0.044 for the observed reflections and 0.064 for all the reflections including the unobserved ones. The scattering factors of carbon were taken from International Tables for X-ray Crystallography (1962), while those of hydrogen were taken from Stewart, Davidson & Simpson (1965). The weighting scheme was based on the counting statistics with an electronic instability of 2% (Stout & Jensen, 1968).

The positional and thermal parameters of the heavy atoms are listed in Table 3; those of hydrogen atoms are listed in Table 4. An ORTEP plot (Johnson, 1965) of the molecule in stereo is presented in Fig. 1. The anisotropic thermal ellipsoids have essentially similar semi-axes. The thermal parameters of the hydrogen atoms are very reasonable, and those of the hydrogen atoms H(4') and H(5') in the ethano-bridge are the smallest, because they are less free to vibrate than the others. The observed and calculated structure factors are given in Table 5, where the low-angle reflections suspected of secondary extinction are marked with an 'E' and were given zero weight during refinement. The unobserved reflections are marked with an asterisk.

Table 4. Coordinates and isotropic thermal parameters of the hydrogen atoms

Coordinates are $\times 10^4$ and standard deviations refer to the least significant digits

| | x | У | Z | $B(Å^2)$ |
|--------|------------|------------|-----------|----------|
| H(1) | 6099 (16) | 1855 (22) | 2799 (7) | 1.1 |
| H(2) | 6643 (17) | 5364 (25) | 3099 (8) | 1.9 |
| H(3) | 5236 (17) | 5583 (24) | 4022 (8) | 2.3 |
| H(4) | 3154 (25) | 3712 (36) | 3623 (10) | 6.6 |
| H(4') | 4059 (18) | 3513 (26) | 2913 (8) | 2.8 |
| H(5) | 3487 (23) | 254 (33) | 3814 (10) | 6.1 |
| H(5') | 4362 (17) | - 56 (25) | 3125 (8) | 2.2 |
| H(6) | 5843 (20) | -471 (30) | 4388 (9) | 4.3 |
| H(7) | 4926 (19) | 2710 (26) | 4758 (8) | 3.5 |
| H(8) | 7428 (20) | 2039 (28) | 5332 (9) | 4.2 |
| H(8') | 7174 (17) | 4510 (25) | 5137 (8) | 2.2 |
| H(9) | 9494 (17) | 3333 (23) | 4808 (7) | 2.1 |
| H(10) | 8107 (19) | 6023 (27) | 4178 (9) | 3.5 |
| H(11) | 9425 (18) | 5770 (25) | 3187 (8) | 2.3 |
| H(11') | 10482 (21) | 5056 (31) | 3907 (10) | 4∙8 |
| H(12) | 10580 (21) | 2572 (28) | 2980 (9) | 4.3 |
| H(13) | 10797 (21) | 1237 (30) | 4159 (9) | 4.6 |
| H(13') | 9982 (19) | - 402 (27) | 3577 (9) | 3.5 |
| H(14) | 8670 (18) | -116 (25) | 4572 (8) | 2.8 |
| H(15) | 7207 (19) | - 797 (28) | 3478 (8) | 3.5 |
| H(16) | 8164 (17) | 3280 (28) | 2980 (9) | 4.3 |
| H(16') | 8390 (17) | 770 (25) | 2565 (8) | 2.2 |

Table 3. Coordinates and anisotropic thermal parameters of the carbon atoms

All numbers are multiplied by 10⁵ and standard deviations refer to the least significant digits.

| | x | y | z | β_{11} | β_{22} | β_{33} | β_{12} | β_{13} | β_{23} |
|-------------------------|------------|------------|-----------|--------------|--------------|--------------|--------------|--------------|--------------|
| C(1) | 69871 (16) | 22048 (25) | 31272 (7) | 878 (19) | 2015 (44) | 177 (4) | -70 (24) | 36 (7) | - 46 (11) |
| C(2) | 68089(17) | 42724 (24) | 34778 (7) | 1042 (21) | 1689 (41) | 203 (5) | 81 (24) | 57 (8) | 79 (11) |
| C(3) | 54690 (18) | 41977 (26) | 38682 (8) | 1044 (23) | 2375 (49) | 237 (5) | 323 (28) | 93 (9) | -6 (13) |
| $\tilde{C}(4)$ | 41050 (19) | 31780 (33) | 34450 (9) | 914 (23) | 3975 (69) | 304 (6) | 160 (32) | 67 (9) | 3 (17) |
| č | 43142 (20) | 8429 (33) | 35845 (9) | 1083 (26) | 3873 (69) | 325 (6) | 688 (35) | 118 (10) | -21 (17) |
| Č | 57782 (19) | 6709 (28) | 40780 (8) | 1269 (26) | 2425 (53) | 261 (5) | 363 (29) | 100 (10) | 128 (13) |
| $\tilde{C}(\tilde{7})$ | 58016 (18) | 27029 (29) | 44696 (8) | 1131 (23) | 3059 (58) | 201 (4) | -47 (30) | 154 (8) | -2 (13) |
| $\tilde{\mathbf{C}(8)}$ | 72375 (20) | 31225 (29) | 49418 (8) | 1466 (27) | 3119 (59) | 132 (4) | 17 (33) | 62 (9) | - 37 (13) |
| Č(9) | 84951 (17) | 30709 (26) | 45220 (8) | 1083 (22) | 2183 (47) | 198 (4) | -37 (26) | -42 (8) | -41 (12) |
| $\tilde{C}(10)$ | 82583 (18) | 47255 (24) | 39606 (8) | 1124 (23) | 1665 (43) | 229 (5) | -148 (24) | 54 (9) | -73 (11) |
| C(11) | 95799 (19) | 47326 (28) | 35659 (9) | 1151 (25) | 2660 (55) | 299 (6) | -470 (30) | 101 (10) | -16 (15) |
| $\tilde{C}(12)$ | 97254 (17) | 26211 (30) | 32469 (9) | 890 (21) | 3183 (59) | 277 (5) | - 99 (29) | 131 (9) | - 120 (15) |
| $\vec{C}(13)$ | 99124 (19) | 9471 (29) | 37925 (9) | 1089 (25) | 2728 (56) | 342 (6) | 378 (30) | 20 (10) | - 95 (15) |
| C(14) | 85850 (18) | 9655 (25) | 41818 (8) | 1113 (23) | 1872 (44) | 242 (5) | 175 (26) | - 30 (9) | 59 (12) |
| C(15) | 71276 (17) | 5698 (24) | 36935 (8) | 1126 (23) | 1572 (41) | 231 (4) | -98 (25) | 41 (8) | -29 (11) |
| C(16) | 83149 (18) | 21918 (28) | 27440 (8) | 1105 (23) | 2909 (55) | 221 (5) | - 56 (30) | 129 (8) | -138 (13) |

Discussion

The bond lengths and angles involving the carbon atoms are given in Fig. 2. The average standard deviation in the C-C distances is 0.002 Å (range 0.0015 to 0.0025 Å) and that in the C-C-C angle is 0.13° (range 0.11 to 0.15°). The molecule may be regarded as having

a mirror plane containing atoms 1, 16, 12, 9, 8, 7 and the midpoint of the 4-5 bond. The C-C distances listed in Table 6 have been classified into the different bond types. A bond type for a given bond is specified by two numbers representing the number of C-C bonds in which the terminal carbon atoms participate. Except for the bonds involved in the strained bridge, the bonds

| Table 5. Observed and calculated structure factors | (×10) | |
|--|-------|--|
|--|-------|--|

| -10.0 | 16 19 -15 18 52 66 20 8• 0 22 12 -11 | 17 50 3 18 13 -13 19 29 31 -6-1-1 | 10 54 10 11 45 26 12 56 -43 13 10+ -10 14 124 -8 | 5 166 180 6 91 96 7 38 18 8 19 16 7 66 -36 | 10 15 16 20 30 5 21 40 1 -4.2.L | 7 14 3 8 130 125 9 89 -84 10 133 133 11 14 15 | 5 25 23 6 16 18 7 96 -10 8 60 6 9 00 -3 | 1 310 311 2 172 -173 3 235 -253 4 481 408 | 1 52 54 2 22 21 3 104 -113 4 116 124 5 04 -3 | 1 18 -11 2 00 11 1 00 88 5 20 -24 | 7 16 -36 8 63 -63 9 166 -168 10 15 56 11 16 -11 | 7 % VB 8 116 120 9 30 33 10 47 -48 11 73 -73 | 11 12 11 12 40 11 | 0 45 -48 1 129 133 2 121 -123 3 51 -50 4 15 13 |
|---|--|---|---|---|--|--|---|---|--|--|---|--|--|--|
| 10 02 -30 12 20 18 -9.0.0 | 0 152 145 2 346 -343 4 728 -754 4 505 505 | 1 107 106 2 98 99 3 28 -28 6 66 -53 5 82 -78 | 15 7100 14 43 -70 17 24 23 18 14 14 19 152 150 20 128 -4 | 10 37 10 11 27 10 12 16 -9 13 26 -25 16 68 -50 15 51 -68 | 1 21 -17 2 39 -34 3 54 -40 4 93 -45 5 74 -14 | 13 108 -108 14 92 96 15 72 -68 16 74 -3 17 26 21 | -413-1 | 5 /60 -201 5 162 135 7 100 5 8 168 -155 9 55 -65 10 26 -20 | 7 22 23 8 94 6 9 19 -18 10 21 -19 11 29 5 | 4 80 76 7 95 -46 8 74 6 9 61 -68 | 13 15 13 14 41 30 15 14 -13 16 19 -39 | 13 3* 0 14 14 -13 15 6* 4 16 51 48 | c 11+ -11 1 40 -18 2 12 31 1 0+ 51 - 38 -38 | 6 52 53 7 9* 13 8 59 -56 9 8* 8 |
| 4 53 49 10 00 -0 12 45 -37 16 30 5 | 10 10 -11 12 90 -11 14 73 74 14 202 203 | | 1 4 4 1.1.4 | 8-1-L 0 160 165 | 14 -11 4 22 234 4 7 -44 10 12 -10 | 10 10 -36 10 10 -30 20 10 -21 21 -6 -0 | 1 3- 5 2 71 -69 1 08 5 78 75 | 11 57 -28 12 75 -73 13 93 104 14 85 -91 15 27 30 | | 11 40 -40 12 134 -18 13 57 -33 14 104 -4 | 3-4-4 0 23 24 1 51 -48 2 20 -18 | -7+5+L 1 124 120 | 1 12 -10 1 12 -10 1 10 - 3 1 10 - 3 | 12 11 |
| -8.0.1 2 224 225 1 158 155 | 18 30 -28 20 94 -13 4+0+L | 11 15 -13 12 21 20 13 5 3 14 24 25 15 12 -1 | 0 7795 1019 1 11 8 2 27 28 3 148 -151 4 258 249 | 2 32 -28 3 89 88 4 103 145 5 11 33 6 11 -25 | 11 108 -103 12 446 -447 13 126 -129 14 16 -18 15 130 133 | 3+2+1 3-17-13 1-19-9 2-6868 | 137 139 7 24 -24 8 94 -73 9 26 -28 10 39 -43 | 14 96 96 17 57 -56 18 61 60 19 116 9 20 18 1 | 7+3+L 2 29 29 1 115 -116 2 196 164 | 18 10 28 17 18 -11 18 77 -71 19 87 -86 | 1 14 -11 2 2 18 3 14 0 4 17 -34 7 40 -59 | 1 15 -11 1 4 -11 | 7.3.L | 1 22 18 2 51 56 3 65 - 56 |
| 10 24 -10 12 4 13 14 24 -30 | 2 176 -163 546 541 6 462 451 8 231 -211 | 16 60 -1 17 100 -6 18 35 33 19 40 -3 20 13 13 | 5 (67 160 6 87 85 7 66 -8 8 155 -151 9 139 -161 | 7 66 -66 6 39 -58 7 26 -30 10 17 15 | 10 10 11 17 105 101 18 110 -0 19 10 -15 20 100 -0 | 5 153 -150 5 108 109 5 50 51 7 100 95 | 11 18 -19 12 30 1 13 18 16 16 75 -69 15 62 61 | 21 30 3 0.3.c 1 101 -130 | 1 50 -15 1 50 -15 1 50 -156 | -2.4.1 1 18 -11 2 8 - 8 3 57 -58 | • 19 -78 9 14 -36 10 42 41 11 04 5 12 44 45 | 2 -11 9 43 -00 10 4 -70 11 7 -00 12 0 -1 | 2 46 45 1 10 -3 1 9 -36 7 34 -35 | • 0, 0, 5 15 - 35 • • 5 - • 6 7 5• - 14 8 30 - 31 |
| 18 37 40 -74046 2 347 339 | 12 43 44 14 30 -16 16 30 -33 18 40 -45 20 84 0 | -3+1+L | | ***** ***** | -34240 | 10 199 -196 11 279 -196 12 196 -193 | -7+3+L 1 17 -18 1 29 -11 | 1 229 -22V 1 229 -22V 1 49 149 3 111 -11V 6 409 -641 | 27 26 10 21 18 11 11 10 12 21 -71 | 5 76 78 5 76 78 6 8 11 7 20 18 6 97 -93 | 13 47 -44 14 13 -11 15 17 14 | 13 20 23 14 13 13 15 14 -4 14 11 -7 17 26 20 | 1 0+ 4 8+5+L 0 9: 41 | • 13 (3) 10 • • • 5 11 • 7 • • • 12 • • • • |
| • 302 -304 • 71 -71 • 52 54 10 25 -5 12 3• -16 | 5.0.1 0 190 190 2 43 -41 | • 58 •8 5 •9 ••6 • 111 •109 7 37 -31 • 17 •• | 16 20 26 17 74 11 18 96 96 19 39 -6 20 09 0 | | 3 4 -6 5 36 -31 5 67 69 6 75 -100 7 17 10 | 14 29 30 15 200 201 14 86 -61 17 85 48 | • 131 131 • 32 -29 • 351 -15• • 79 -15• | 121 -113 124 120 10150 141 1124 -25 | 8+3+L U 23 -28 I 55 -50 2 18 -15 | 10 25 -25 11 100 0 12 02 63 13 37 -60 14 76 78 | 0 21 2+ 1 124 125 2 127 -110 3 68 71 | -1+5+6 1 52 51 2 25 21 | -0+0+. | 0 20 -23 1 00 -01 2 01 -01 |
| -0-0-L | • 18 -14 • 29 -28 10 81 -76 12 107 -101 | 10 77 -70 11 28 -24 12 147 -139 11 91 -90 | 21 13 -15 22 25 24 24144 | 7 21 -20 6 16 16 9 17 -16 10+1+1 | 8 46 45 9 11 -6 10 282 -276 11 11* -13 12 44 -41 | ···· | 9 63 39 10 73 73 11 59 61 12 66 66 13 22 721 | 13 100 -5 14 50 48 15 33 -38 16 118 118 17 38 -18 | 3 31 31 6 6J -36 3 32 36 6 26 25 7 16 15 | 15 50 -80 16 80 -80 17 30 20 18 18 -14 19 35 36 | 5 83 -86 6 51 53 7 70 -1 8 16 14 9 26 28 | | 1 106 106 21 18 5 24 -18 6 82 -74 7 43 -41 | • 21 -20 • 14 15 • 19 -34 7 14 10 • 11 34 |
| 2 229 -211 - 108 -106 6 95 -71 8 46 73 10 16 39 | 10 0.0.L | 15 40 58 16 159 -159 17 70 69 18 68 -66 19 00 -4 | 1 102 -101 2 98 98 3 212 -206 57 55 | | 15 176 160 14 233 234 15 150 144 16 34 5 17 35 -34 | 0 52 53 1 12 -14 2 16 -11 5 11 10 4 49 -46 | · · · · · | 10 53 -50 10 47 -46 20 13 -8 21 23 -16 | • • • • • | -1.4.L | 10 40 10 11 50 -48 12 22 21 13 48 49 | • 28 21 10 11• -1 11 12 30 12 26 15 13 45 -50 | ■ 15 =35 =5+5+6 1 78 78 | 10 12 11 North |
| 12 76 -76 19 31 -31 16 20 -19 18 10* 8 20 67 -61 | 0 92 91 2 22 20 4 80 83 6 42 -43 8 50 -48 | 20 22 28 21 18 -21 -6-1-1 | 6 136 -168 7 66 39 8 111 106 9 90 10 136 -153 | 5 61 50 -10+2+L 1 40 0 | 20 74 -6 21 44 0 22 30 29 | 6 92 -73 7 54 -56 4 89 -84 9 107 -103 10 263 -255 | 1 88 84 2 31 29 3 17 -23 5 23 -21 | 0 40 48 1 84 0 2 45 -41 3 80 -80 | 0 24 24 1 39 39 2 61 61 3 22 25 4 22 -19 | 4 141 -136 5 102 -103 6 126 118 7 18 -19 | | 14 44 41 15 13 -11 16 44 5 17 18 26 | 1 40 41 40 41 40 -11 40 -100 40 -100 4 23 26 | |
| -3+0+1 2 19 -38 4 265 -170 - 151 -155 | 10 16 28 12 0* 0 16 48 -88 16 2* -1 | 2 79 86 3 19 -14 4 11 -4 3 40 39 | 12 3* 4 13 68 -64 14 24 18 15 73 70 16 132 -133 | 2 23 20 2 14 -14 4 10 -33 | -2-2-4 1 67 66 2 17 -10 3 100 -111 | 11 52 50 12 121 120 13 211 214 14 83 84 15 40 38 | 5 110 -10 6 100 -76 7 0 0 8 72 73 9 132 136 | • 163 -159 • 51 50 • 27 -19 7 152 163 • 165 131 | * * - 33 • 34 - 33 | 9 30 55 10 35 26 11 24 -31 12 37 39 13 32 -33 | 6 96 03 5 10 -21 6 70 6 7 25 28 8 31 -28 | 1 204 205 2 248 273 3 44 40 4 29 -29 | 8 22 21 9 12 6 10 14 -15 11 34 29 | 6 19 18 7 22 23 4 15 13 |
| 8 32 3V 10 77 -76 12 96 93 14 109 -111 16 100 -104 | 2 200 -200 2 200 -200 5 6° -3 | 7 57 54 8 8 -6 9 22 -20 14 17 -13 11 87 -8 | 18 14 -8 19 23 -28 20 39 3 21 22 -20 | -9+2+L | 5 28 -28 6 12 -9 7 57 60 8 66 88 | 17 29 -11 18 39 39 19 17 15 | 11 3 33 12 8 0 13 0 -1 14 50 46 | 10 11 -0 11 14 -19 12 36 35 13 36 24 | | 15 146 158 16 91 -94 17 25 25 18 12 15 | 13 13 | 5 116 -118 6 105 -101 7 41 40 8 12 -20 9 10 -16 | 1 24 21 2 10 4 2 10 4 | 0 28 -24 5 •• 8 2 19 -18 5 •• 0 |
| 19 147 149 23 44 43 22 44 1 -4.0.0 | 8 34 -23 10 32 -30 12 100 -18 14 13 -8 | 12 32 34 13 132 130 14 51 -54 15 157 155 14 34 -30 | 3+1+L 0 77 -73 1 219 -200 2 201 275 | 2 40 -34 3 74 -0 3 21 -21 | 10 49 49 11 94 -5 12 34 35 13 54 66 14 35 -34 | 0 +0 -16 1 +3 +3 2 33 31 3 +1 -39 | 10 13 -13 17 104 -8 18 114 -4 -3+3+6 | 15 30 -26 16 39 -35 17 9• 1 18 31 -30 19 25 26 | 7 25 25 -8-4-C 1 30 30 | 0 151 158 | 1 238 -103 2 66 -65 3 67 -65 6 69 68 5 51 51 | 11 110 -0 12 00 0 13 18 20 15 15 -0 15 25 11 | 120 - 3 120 | · · · · · · · · · · · · · · · · · · · |
| 2 98 104 6 802 -870 6 140 128 8 773 818 | 8+0+1 0 [92 -184 2 84 -83 4 11 33 | 17 63 -63 18 176 171 19 61 -58 20 13 16 21 11 11 | 3 16 -16 4 358 -355 3 476 -76 6 428 -423 7 04 -1 | 7 12 -9 8 16 -13 9 6 -1 10 15 15 | 15 16 -11 16 58 6D 17 11 18 18 9 0 19 9 0 | • 12• -1 • 16 6 • 20 -18 7 61 58 • 26 13 | 1 3• 0 2 16 16 3 53 51 | 20 40 4 2030L 0 70 -10 | 2 27 79 3 25 25 4 22 -19 3 29 -29 4 23 23 | 2 76 -70 3 213 -215 4 87 71 5 105 -98 6 77 74 | 0 20 23 7 30 2 9.0.1 | 10 23 -23 17 110 -10 105.0 | 10 8• 8 11 17 19 12 46 48 11 39 -34 | |
| 12 127 -110 14 31 -30 14 323 343 18 34 54 20 110 -108 | 10 10 11 12 40 1 4.04 | -1-1-C | 9 350 -348 10 126 -125 11 36 -34 12 43 40 13 313 321 | 12 30 -6 13 16 18 10 40 -39 -8+2+1 | 21 60 -51 22 67 68 -1-2-1 | 10 67 65 11 10 8 12 76 73 13 61 34 16 18 -18 | 6 33 33 7 315 310 8 375 - 173 9 389 391 10 42 - 41 | 1 102 100 1 102 100 1 30 10 4 35 10 3 44 -19 6 97 -98 | 25 29 6 20 21 10 64 -8 11 10 -15 12 16 11 | 1 28 -28 4 89 -84 9 3) -39 10 98 -9 11 99 -23 12 47 -36 | 0 71 -60 1 74 -74 -8,5+1 | 0 210 204 1 112 111 2 63 -61 3 102 -168 4 206 -204 | - 3+6+1 1 16 - 31 2 24 - 3 3 30 - 28 | -3-246 1 18 -18 2 54 - |
| -3+0×L 2 241 -233 | 0 57 -60 2 51 -60 4 54 -53 6 29 25 8 36 -33 | 1 19 14 4 345 -353 5 12 14 6 186 191 7 44 44 8 340 351 | 14 93 -98 15 03 04 14 35 33 17 135 -135 18 86 80 18 95 -16 | 1 4 1 1 4 1 1 12 - 133 1 12 115 1 12 115 | 1 226 -218 2 385 401 3 287 -288 4 51 46 5 41 -41 | 15 9 0 16 02 01 17 22 21 18 15 -13 | 11 90 -90 12 233 225 13 54 -51 14 19 18 15 124 5 | 7 293 263 6 192 -193 9 71 68 10 126 126 11 98 -99 | -71416 1 109 -104 2 20 24 | 11 12* 19 14 67 -65 15 10* 4 16 27 24 17 40 -46 | 2 51 -50 3 35 35 5 35 35 6 20 19 | 6 0+ 0 7 66 63 8 68 63 9 33 36 10 129 -13 | 5 35 -29 6 70 -68 7 8 6 8 55 55 9 80 80 | |
| 6 8952 781 4 124 -131 10 155 -151 12 84 89 14 31 35 | 10+0+1 0 0+ -3 2 3+ -53 | • 5• • 10 75 -71 11 •1 •3 12 10• 19• 13 101 100 | 20 13 -10 21 27 20 4+1+L | 6 162 -168 7 20 21 8 87 -91 9 19 21 10 36 31 | 125 -125 4 37 -2 10 48 -64 11 28 14 | 2 17 18 1 27 -11 2 68 68 5 13 10 | 17 23 23 18 19 -16 19 35 -36 20 9 -6 | 12 68 66 13 18 18 16 10 - 6 15 32 30 16 0 56 17 6 0 | 3 56 -50 4 15 6 5 74 19 6 40 34 7 17 -11 8 65 -63 | 19 16 10 19 16 16 1.4 | · · · · · · · · · · · · · · · · · · · | 11 0• 1 12 18 -13 11 09 -05 14 20 24 15 10• 8 | 10 57 58 21 11 13 12 10 -13 13 14 13 | 24744 1 141 141 2 141 141 |
| 10 80 -3 10 110 -108 20 70 0 22 25 -21 | · 10 -28 · 10+1+L 1 21 -16 | 16 61 65 15 63 -66 16 51 56 17 58 -55 18 16 13 | 0 36 59 1 63 58 2 175 -164 3 240 218 6 292 -269 | 11 10+ 3 12 25 -26 13 8+ 5 14 15 15 15 11+ -8 | 12 36 -33 13 6* 0 14 112 114 15 15 20 16 70 73 | 6 33 -36 7 35 -61 8 10 -1 | -++3+L 1 1+ -6 2 34 33 3 18 -19 | 18 22 -20 19 16 -15 20 20 0 3+1+L | 9 88 -85 10 21 -16 11 26 29 12 64 1 11 94 8 | 1 46 | 3 42 40 4 45 48 3 46 -60 6 30 -49 7 64 1 | 17 19 16 2+3+1 0 61 -63 | -2-6-1 3 6* -3 2 26 28 3 21 23 | - 12 - 13 - 12 - 14 - 14 - 16 - 13 - 16 - 13 - 16 - 7 - 04 - 0 - 8 - 15 - 16 |
| 2 216 221 6 592 -563 6 35 -29 8 27 23 | 1 24 -6 26 -24 3 15 -16 6 114 -8 7 17 18 | 20 50 13 21 8 13 22 120 8 | 4 203 271 7 221 -210 8 58 50 9 40 48 | -7-2-0 | 10 00 -80 19 165 -168 20 59 58 21 60 -58 | 10 06 11 0 13 12 19 18 13 6 6 | 5 0+ 1 6 118 -114 7 81 75 8 49 -50 | 0 15 16 1 67 -70 2 5* 0 3 23 10 | 14 19 -14 15 94 -11 -6-4-0 | 6 66 68 7 56 1 8 61 61 9 15 15 10 67 -58 | 30 - 58 0 - 1 10 1 11 17 - 15 | 1 68 -86 2 33 -28 3 219 -223 4 62 68 3 87 83 | • 1• 18 • 10• 11 7 30 28 8 23 19 | યાત્ર હે. - હારના 1. કેર કહ |
| 10 +3 -48 12 202 -199 14 21 29 16 29 -34 18 22 -20 | * 105 100 * 31 10 10 * -3 11 17 -15 | 1 va -21 2 a0a a25 3 30 -30 4 71 73 | 11 230 234 12 210 215 13 44 45 14 84 1 15 130 -140 | 2 28 34 3 15 18 4 25 -28 5 97 98 6 182 -190 | 0 109 100 1 105 -103 | 15 21 19 16 16 -16 74746 | 10 431 435 11 122 -121 12 49 49 13 92 -89 14 258 -258 | 5 170 165 6 160 -163 7 19 20 6 123 118 9 231 -225 | 2 100 9 3 66 -63 6 25 21 5 50 -51 6 27 -25 | 12 00 5 11 70 5 14 30 31 15 21 15 16 110 5 | 1 17 19 7 25 71 3 22 71 | 7 10* -4 8 42 45 9 61 -63 10 11* 11 11 9* -4 | 10 19 -40 11 31 -31 12 8 - 1 13 7 - 5 14 19 19 | 7 70 -74 1 110 -11 4 15 -11 3 71 -21 6 40 -10 |
| -1.0 | -9+1+L 1 27 -24 7 104 -3 3 44 -3 | 6 96 -100 7 18 -24 8 62 53 9 75 -78 | 16 54 55 17 27 -26 18 13 14 19 21 23 20 14 13 | 7 35 35 8 104 110 9 45 45 10 108 108 11 104 -1 | 2 55 63 3 65 -65 6 767E -VU6 5 60 -35 6 235 -215 | 0 48 -43 1 14 -6 2 27 -29 1 116 -116 4 177 -181 | 15 56 -58 16 18 -18 17 6* 1 18 11* -3 19 37 -15 | 10 325 329 11 30 -40 12 84 -61 11 26 -20 14 35 -51 | 7 4 0 4 1 1 1 2 1 1 1 1 | 17 30 -36 18 70 6 19 36 -15 21416 | 5 113 -110 6 24 -20 7 10 -10 8 21 21 9 36 36 | 12 13 10 13 04 0 14 04 0 15 64 4 16 27 -25 | -1.6., 1 5° 6 2 50 | 8 11 -113 9 12 -11 9 12 -11 |
| 6 336 355 6 57 61 8 161 -161 12 65 65 12 28 28 | 103 -100 13 -13 1 -11 | 11 61 -39 12 16 -21 13 10 U 16 58 -31 15 100 U | 5+1+L U 16 -35 L 74 -74 2 192 191 | 13 6* 0 14 19 -11 15 0* -1 16 0* -1 16 0* -4 17 27 -24 | 1 431 437 9 207 196 10 76 76 11 92 -43 12 29 -29 | 3 37 39 6 37 39 7 116 123 8 96 98 9 110 0 10 60 | 20 64 3 •3+1+L 1 73 73 2 18 •18 | 15 63 -39 16 21 -20 17 6J -38 18 39 -39 19 19 -15 | 12 17 11 13 33 -33 14 74 23 15 46 65 16 27 -26 | 0 81 -61 1 166 -159 2 15 10 1 106 101 8 11 25 | 10 10• 16 11 49 48 12 29 -28 13 63 -63 14 13 16 | 1.5.1 0 51 51 1 54 -55 | | 1 14 11 2 16 13 3 69 -66 6 10- 8 3 90 -26 |
| 14 50 -45 16 24 -26 18 17 11 25 115 -114 22 74 76 | 10 13 11 70 4 12 00 -16 13 40 0 16 73 -20 | 16 41 43 17 15 413 18 35 -13 19 83 -74 23 60 63 | 3 4 -1 4 14 -11 5 14 11 6 79 73 7 34 -25 | 10 94 -V3 -6+2+L 1 25 29 | 13 4* 13 14 42 41 15 31 -34 16 78 -74 17 106 -148 | 11 19 -21 17 9* 5 13 17 13 14 18 19 | 3 22 -20 4 25 18 5 87 -83 4 25 30 7 94 11 | 4.1.1.L 0 27 24 1 47 49 | -5141L 1 80 -80 2 68 69 | 5 116 111 7 81 - 81 7 90 - 74 7 201 201 | -5.5.4 46 64 40 -0 5.162 -169 | 3 37 -35 4 27 -20 5 11 -11 6 14 -41 7 10 25 | | 7 22 -25 8 22 10 9 50 51 |
| 0.0.L 7 101 135 6 8705-1160 6 206 215 | -8+1+L 1 130 -126 2 105 -101 | 22 43 -14 23 39 -14 -1+1+L | ++++++++++++++++++++++++++++++++++++++ | 2 80 93 3 60 36 6 87 -84 5 76 -0 6 86 63 | 10 105 -108 19 12 11 20 14 15 21 115 125 22 63 -55 | 0 05 -46 1 07 -05 2 52 -05 | 8 66 68 9 60 -59 10 74 -6 11 95 -89 12 272 -268 | 3 28 -23 6 30 28 5 67 -68 6 13 10 7 101 -96 | 3 22 -23 6 04 -6 5 46 48 6 67 -53 7 67 -66 | 10 147 -148 11 48 69 12 40 18 19 40 -41 14 94 -6 | 4 105 108 5 58 -58 6 17 18 7 55 60 8 19 -18 | 15 145 10 10 10 10 1 | 14 16 -18 J+6+E 0 159 -163 | C 19 19 1 21 -20 2 21 28 1 13* -1 |
| 8 217 223 10 77 -76 12 25 -16 16 36 35 16 8+ 10 | 3 44 3 4 15 -24 5 83 83 6 27 -3 7 24 26 | 1 00 00 2 263 -275 3 25 -26 4 366 -356 3 115 -111 | 14 114 -10 15 24 -24 16 94 8 17 36 -34 18 19 -16 | 8 62 68 9 51 50 10 25 -26 11 65 -66 12 104 1 | 1.2.L N 20 -15 1 46 -45 2 109 -104 | 6 112 116 7 36 36 6 112 116 7 36 36 6 41 - 75 | | 9 99 -99 10 88 -86 11 86 -56 12 155 -150 | 10 80 -84 11 100 100 12 26 -26 | | | 10 - 10 10 - 10 10 - 10 10 - 10 | 1 120 10 1 101 20 1 101 | 5 16 15 6 13 13 7 56 56 8 56 58 |
| 18 57 58 23 81 79 27 101 103 1+2+L | * 52 51 7 4* 0 10 11* -71 11 4* -7 14 26 2* | 6 126 133 7 105 100 8 66 -51 7 60 3 10 83 -70 | 0-1+6 0-140 -140 1-10 -20 | 15 3* -3 16 3* -6 15 10* 8 16 3* 60 17 19 18 | 3 95 98 4 114 -111 5 132 128 6 445 448 7 288 293 | 9 29 -29 10 10+ -8 11 A+ -8 9+7+6 | 19 8+ 1 20 17 13 21 39 38 -2+3+6 | 14 30 28 15 64 13 16 61 -58 17 28 -25 18 104 8 | 14 22 -13 15 74 -4 16 04 5 17 37 36 18 49 48 | 0 11 21 1 32 13 2 52 -10 1 50 -46 | 15 25 25 -6.5.c 1 31 -31 | 0 114 10 1 40 -19 2 20 -15 1 04 n 4 93 -14 | 7 20 20 8 00 3 9 15 13 10 00 5 | 2000. 2010. 2011. |
| 0 99 -78 2 v226-1276 6 27 -26 6 218 -218 5 98 98 | 14 20 26 15 17 16 16 37 -16 17 16 -15 | 12 70 6) 13 70 65 14 26 26 15 00 0 16 98 -101 | 3 /8 -75 4 45 -43 5 50 -50 6 50 50 7 5 18 | -5+2+0 | 7 16 -36 10 92 -65 11 6 -53 12 26 -26 | 0 +2 +4 1 11 13 2 48 3 3 38 -18 | | 3+3+L 0 17 -18 1 33 29 | | 24 25 35 35 37 43 44 45 45 46 47 46 47 < | 2 100 100 3 55 -53 5 11 -10 5 23 -10 6 700 -100 | 5 103 105 6 40 40 7 44 175 8 46 45 4 14 -14 | 12 40 | 2 16 - 16 3 114 18 4 24 - 25 5 94 6 9 34 17 |
| 10 21 -1 12 18 -19 14 11 -35 16 56 56 19 12 -16 | -7+1-0 1 29 34 2 358 361 2 231 235 | 17 145 -153 18 04 0 17 32 -35 20 34 -38 21 142 -164 | 9 21 -50 9 10 4 10 10 -13 11 15 20 12 17 21 | 1 72 -64 2 01 86 3 1V -21 6 19 -6 3 21 10 | 14 44 -5 15 8* 15 16 27 -28 17 61 60 | 5 15 15 5 05 -1 7 08 0 8 17 16 | 6 36 -38 7 37 -30 8 37 -31 9 28 24 10 80 79 | 3 43 41 4 13 19 5 13* -13 6 81 -81 7 27 -28 | | 10 8* 6 11 185 -189 14 117 116 13 13 11 14 17 11 | | 11 20 -26 12 11 -29 14 17 16 14 1* 6 | 0 %6 51 1.10* 9% 2.116 119 3.127 119 6.11 175 | 1997 - 19 |
| 22 22 -21 2.0 0 234 - 214 | 3 4 44 3 4 23 7 3 6 33 7 3 6 33 7 3 6 33 | 0+1+L 1 62 65 7 9131 148 | 10 15 -16 15 50 -55 16 60 -61 17 77 -16 | * 124 -123 10 310 116 11 44 -4 | 20 30 33 21 74 3 2.2.L | 10+2+L 0 17 -33 L 11 -20 2 13 36 3 53 -50 | 11 00 -1 12 55 50 13 51 51 14 16 -40 15 124 123 | 5 56 58 10 30 33 11 21 23 12 9 10 | • 113 114 10 75 -73 11 • • -103 12 127 124 13 16 -16 | 15 61 61 16 41 18 17 16 11 | | 0 41 18 1 50 41 7 9+ 10 | 5 06 103 6 36 -33 7 176 8 .8 19 9 118 | F 1 5 76 7 8 - 6, 8 1, - 18 8 6• 9 8 7• 1, - |
| 7 315 - 125 - 511 - 529 + 34 - 53 4 51 - 53 4 51 - 53 17 189 - 138 | 10 11+ 15 11 8+ 8 12 5+ -+5 13 8+ -3 14 12+ + | 1 12 14 150 -151 5 220 220 5 208 213 7 231 234 | 2+1+L 0 192 188 1 212 -219 2 106 109 | 12 +3 -3V 13 +2 -43 14 77 -69 15 84 -79 16 30 28 | 0 136 130 1 25 -23 2 16 6 3 73 -71 4 25 -16 | -9.1.L 1 81 -LU 2 45 44 | 17 14 11 18 66 63 19 19 20 20 22 -21 21 14 5 | 15 35 -54 15 35 -74 16 39 18 | 15 15 11 16 00 35 17 51 30 18 07 -6 | 0 20 21 1 74 1 2 38 -76 3 95 98 | 1 0* -11 2 14 18 1 0* 0 | - 22 - 20 - 22 - 20 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 | 10 46 -43 11 25 28 12 59 -56 13 26 76 14 17 18 | 4 44 44 414 |
| 15 15 151 | | | · · · · · | 14 22 -24 | · · · · · · · | : :: .!! | -1+3+6 | 0 36 -60 | - 3 . 4 . 1 | 2 22 33 | 2 22 28 | 10 19 14 | 2.001 | |

fall into two bond types: (2)–(3) and (3)–(3). The following trend, though not very pronounced, is apparent in the C–C bond distance (3)-(3) > (2)-(3).

Table 6. Classification of bond lengths in terms of bond type

| | Bond type† | Length (Å) | Mean C–C distance |
|--|---------------|--|----------------------|
| C(11)-C(12) C(12)-C(13) C(12)-C(16) | (2)-(3) | 1·526‡ 1·523 1·531 | 1.527 |
| C(1)—C(16) C(7)—C(9) C(8)—C(9) C(10)–C(11) C(13)–C(14) | (2)–(3) | 1.524 1.517 1.516 1.534 1.532 | 1.525 |
| C(3)—C(7) C(6)—C(7) C(9)—C(10) C(9)—C(14) | (3)–(3) | 1·530 1·531 1·537 1·534 | 1.533 |
| $\begin{array}{c} C(1)C(2)\\ C(1)C(15)\\ C(2)C(3)\\ C(2)C(10)\\ C(6)C(15)\\ C(4)C(15) \end{array}$ | (3)-(3) | 1.533 1.535 1.544 1.541 1.545 1.545 | 1.539 |
| *C(4)C(5) | (2)–(2) | 1.552 | 1.552 |
| *C(3)—C(4) *C(5)—C(6) | (2)–(3) | 1·544 1·538 | 1.541 |

* Bonds in the bridge.

† Denotes nearest neighbor.

 \ddagger The average standard deviation in the bond lengths is 0.002 Å.

The longest C-C distance of 1.552 Å is associated with the bridge bond C(4)-C(5) [bond type (2)-(2)]. The marked lengthening in this bond is primarily due to the interaction between the eclipsed hydrogen atoms on C(4) and C(5). Similarly, the lengthening in the C(4)-C(3) and C(5)-C(6) bonds [bond type (2)-(3)] is due to the partially staggered bonds around them.

The C-C-C bond angles vary from 99.9 to 114.5°

with a mean of $109 \cdot 1^{\circ}$. The largest and smallest of these angles are associated with the atom C(7) at the bridgehead position of the ethano-bridged adamantane moiety. All of the endocyclic angles in the 5-membered ring are significantly smaller than the ideal tetrahedral value.

The C-H distances range from 0.96 to 1.09 Å with a mean of 1.02 ± 0.02 Å. The C-C-H and H-C-H angles have mean values of 109.7 ± 0.8 and $111 \pm 1^{\circ}$ respectively.

The molecule can be thought of as being composed of seven cyclohexane rings and one cyclopentane ring. The mean values of the endocyclic torsional angles of the cyclohexane ring systems together with the mean bond angles, are listed in Table 7. The mean torsional angles in all seven rings are greater than the mean value of 56° in cyclohexane itself (Altona & Sundaralingam, 1970). The greatest- and least-puckered cyclohexane rings are involved in the fusion with the ethano bridge. It is also seen that the mean bond angle decreases from the ideal tetrahedral value with increasing puckering of the ring.

| Table 7. The end | locyclic torsic | onal angl | es and l | bond | angl | es |
|------------------|-----------------|-----------|----------|------|------|----|
| in the val | rious cyclohe | xane ring | g syster | ms* | | |

| | | Torsion a | angle | Mean |
|-----|--|------------|-----------|------------|
| No. | Atoms | Range | Mean | bond angle |
| 1 | C(1), C(2), C(10), | 56·7–61·5° | 59·4 (2)° | 109·7 (1)° |
| 2 | C(11), C(12), C(16) C(9), C(10), C(11), | 58.0-61.7 | 59.8 (2) | 109.5 (1) |
| - | C(12), C(13), C(14) | | ¢, ¢ (_) | |
| 3 | C(1), C(15), C(14), | 56.8–60.7 | 59.1 (2) | 109.8 (1) |
| 4 | C(13), C(12), C(16) C(1), C(2), C(10), | 59·7-65·8 | 62.9 (2) | 108-2 (1) |
| 5 | C(9), C(14), C(15) C(1), C(2), C(3) | 56.1-69.3 | 64.2 (2) | 107.5 (1) |
| • | C(6), C(7), C(15) | 001 000 | ¢ · = (=) | |
| 6 | C(2), C(3), C(7), | 52.7-61.5 | 57.2 (2) | 110.5 (1) |
| 7 | C(8), C(9), (10) C(6), C(7), C(8), | 53-2-61-3 | 57.1 (2) | 110.6 (1) |
| | C(9), C(14), C(15) | | | |

* The six-membered rings 1, 2 and 3 form the top adamantane system (Fig. 3), while the rings 5, 6, and 7 form the ethanobridged adamantane ring. Ring 4 is common to both adamantane rings.



Fig. 1. A stereoscopic diagram of the molcule.



Fig.2. Bond lengths and bond angles in the carbon skeleton. The average standard deviation in the bond lengths is 0.002 Å and that in the bond angles is 0.13° .



Fig. 3. Packing diagram of the structure viewed down the b axis.

The cyclopentane ring is in the envelope form rather than the common half-chair form. The carbon atoms 3, 4, 5 and 6 lie on a plane. The methylene groups on C(4) and C(5) are eclipsed, with a twist of only 1° around the C(4)–C(5) bond. The smallest valence angle is at the puckered carbon atom C(7).

The carbon skeleton is not significantly twisted; the twists around the pseudo bonds C(2)-C(15), C(10)-C(14) and C(3)-C(6) are less than 0·1°. The H-C-C-H torsional angles around the C-C bonds in the cyclohexane ring systems vary from 52 to 70° with a mean of $59 \pm 2^{\circ}$. The smallest angle is around the C(2)-C(3) bond and the largest one is around the C(9)-C(10) bond. The H-C-C-H torsion angles around the C(3)-C(4) and C(5)-C(6) bonds in the cyclopentane ring are $(-31, 91^{\circ})$ and $(30, -87^{\circ})$ and are semistaggered.

The packing diagram of the structure as viewed down the b axis is shown in Fig. 3. There are no unusually short contacts in the structure, and the H---H distances less than 2.6 Å are shown in the Figure.

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Die Strukturen des Moleküls und des zweifach negativ geladenen Anions der *trans*-Cyclohexandicarbonsäure(1,4)

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(Eingegangen am 12. November 1970)

The structure of a potassium salt of *trans*-cyclohexane-1,4-dicarboxylic acid, chemical formula $2C_8H_{11}O_4K.C_8H_{12}O_4$ or $C_8H_{10}O_4K_2.2C_8H_{12}O_4$, was solved by X-ray analysis. Investigations of the bond lengths of the carboxylic groups led to the conclusion that the correct formula is $C_8H_{10}O_4K_2.2C_8H_{12}O_4$.

Wie wir bereits mitgeteilt haben (Luger, Plieth & Ruban, 1970) gelang uns im Rahmen einer Untersuchungsreihe an Cyclohexanderivaten die Darstellung des Mono-Kalium-sesqui[Cyclohexandicarbonsäure(1,4)]-salzes.

Mit den vorläufigen Ergebnissen einer röntgenographischen Strukturbestimmung konnten wir zeigen, dass die Substanz in der Raumgruppe $P\overline{1}$ mit zwei K⁺-Ionen und drei Säure- bzw. Säurerestmolekülen kristallisiert. Kristallographische Daten sind in Tabelle 1 enthalten.

Dabei befindet sich nicht nur der Schwerpunkt eines Cyclohexanringes in einem Symmetriezentrum, sondern alle drei Moleküle sind um die speziellen Lagen $\frac{1}{2}, 0, \frac{1}{2}; 0, 0, \frac{1}{2}$ bzw. $\frac{1}{2}, \frac{1}{2}, 0$ angeordnet.

Im Rahmen dieser Arbeit sollen die Ergebnisse einer

weiteren Verfeinerung der Struktur mitgeteilt werden. Es konnten die Parameter sämtlicher Wasserstoffatome bestimmt werden und aus den Bindungslängen an den Carboxylgruppen die Entscheidung zugunsten einer der beiden möglichen chemischen Formelngetroffenwerden. Das nach der Schweratommethode über die Kalium-Parameter bestimmte Strukturmodell wurde mit anisotropen Temperaturfaktoren für alle Atome durch das least-squares Programm *ORFLS* des Programmsystems *X-ray* 63 (1963) bis zu einem *R*-Wert von 8,3% verfeinert. Einer in diesem Stadium berechneten Differenzsynthese konnten sämtliche Wasserstoffatomlagen entnommen werden. Weitere Verfeinerungen, die bei den Wasserstoffatomen jedoch isotrop durchgeführt wurden, konvergierten bei einem *R*-Wert von 6,3%.