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Combined X-ray and Neutron Study of 1,1,2,2-Ethanetetracarbonitrile, C₆H₂N₄, at 158 K

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Abstract. $M_r = 130.11$. X-ray: monoclinic, $P2_1/n$, $a = 7.367$ (1), $b = 5.705$ (1), $c = 7.780$ (1) Å, $\beta = 95.02$ (1)°, $V = 325.8$ Å³, $Z = 2$, $D_x = 1.327$ g cm⁻³, $T = 158$ K, $\text{Mo } K\alpha$, $\lambda = 0.7107$ Å, $\mu = 0.99$ cm⁻¹, $F(000) = 132$. In order to explain the lengthening observed in the central bond of a number of symmetrically substituted ethanes, an analysis of the charge density of the title compound has been undertaken. The structure was redetermined at 158 K from neutron and X-ray diffraction data and refined to $R_w = 1.4$ and 2.8% respectively. $X-N$ and $X-X$ deformation maps show a weak density in the region of the central bond. A least-squares refinement including atomic charges was performed on the X-ray data and indicates two negatively charged C atoms at the ends of the central bond.

Introduction. This analysis of charge density in 1,1,2,2-ethanetetracarbonitrile (Fig. 1a) is part of a structural study of dehydro dimers corresponding to the formula $(R_1R_2R_3C)_2$. The structures of some of these dimers presented in Fig. 1 have been elucidated by X-ray diffraction at room temperature (Parfonry, Tinant, Declercq & Van Meerssche, 1982). We observed a significantly stretched central bond ($d \approx 1.59$ Å) for molecules like (b) and (c) (Tinant, Arte, Parfonry, Declercq, Germain & Van Meerssche, 1982) while the

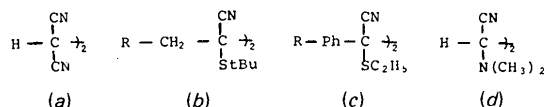


Fig. 1. Examples of dimers related to this study and whose structures have been determined by X-ray diffraction.

expected distance of 1.54 Å was found for molecule (d). A partial explanation of the bond lengthening in (b) and (c) can be found in the work of Rüchardt & Beckhaus (1980). They established quantitative correlations between steric strain, thermal stability and central bond lengths in a series of substituted alkanes. In ethane-tetracarbonitrile steric strain seems to be low and does not appear to explain the long bond length (1.561 Å) observed at room temperature. Moreover, *ab initio* calculations performed by Leroy, Wilante, Peeters & Khalil (1981) determine a central length of 1.584 Å. Electronic effects should be important and an experimental study of the charge density deformation must be of prime interest for the interpretation of the observed effects.

Experimental. Crystals grown by slow evaporation of a 3:2 solution of benzene and ethyl acetate. D_m not measured. Details of the data collections are summarized in Table 1.

X-ray data measured on a Syntex $P2_1$ diffractometer equipped with the Syntex LT-1 low-temperature attachment. Cell dimensions computed from the centring of 30 reflections. For all reflections with $\theta < 25^\circ$, four symmetry-equivalent intensities were collected. For the upper part of the data ($25^\circ < \theta \leq 50^\circ$), 657 independent reflections were selected on the basis of their intensity estimated from the known atomic parameters at room temperature (Piccinni-Leopardi, Tinant, Declercq & Germain, 1983). For each, two symmetry-related reflections were collected. A profile analysis based on the method described by Blessing, Coppens & Becker (1972) was applied. The data were not corrected for absorption, the transmission factors being nearly constant (see Table 1).

Neutron measurements made at the D9 diffractometer of the Institut Laue-Langevin high-flux reactor. Symmetry-related reflections for which $6 \leq I/\sigma_I \leq 90$ collected in the range $25^\circ < \theta < 50^\circ$. Data were reduced following the profile analysis of Lehmann & Larsen (1974) and corrected for absorption.

Refinements based on F were started with the atomic parameters determined at room temperature (Piccinni-Leopardi, Tinant, Declercq & Germain, 1983) and performed with the programs *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979) or *SHELX76* (Sheldrick, 1976).

Extinction parameters were refined for both data sets. Isotropic and anisotropic corrections, types I and II with Lorentzian and Gaussian distributions as described by Becker & Coppens (1974*a, b*, 1975), have been tested. There was no significant anisotropy of the extinction and the best results were obtained in both cases with an isotropic correction of type I and a Lorentzian distribution. As can be seen in Table 2 where the refinement parameters are listed the effects of extinction are weak.

Table 1. *Experimental conditions*

| | X-ray | Neutron |
|---|--|------------------|
| Wavelength (Å) | 0.7107 (Mo $K\alpha$) | 0.8457 |
| Temperature (K) | 158 | 158 |
| Crystal size (mm ³) | 5.2×10^{-3} | 9.5 |
| Scan mode | $\theta-2\theta$ | $\theta-2\theta$ |
| Scan width (°) | For $\theta < 25^\circ$: $\Delta\alpha_1 - \alpha_2 + 3.8$ For $\theta > 25^\circ$: $\Delta\alpha_1 - \alpha_2 + 4$ | From 0.8 to 3.5 |
| Maximum θ (°) | 50 | 50 |
| Reference reflection | 212 | 210 |
| Number of reflections between references | 50 | 40 |
| Total No. of reflections | 3692 | 2583 |
| No. of unique reflections | 1231 | 1604 |
| No. of unobserved ($I < 2.5\sigma_I$) | 41 | 296 |
| R factor between symmetry-related reflections (%) | 1.2 | 0.4 |
| μ (cm ⁻¹) | 0.99 | 0.41 |
| Absorption corrections | No | Yes |
| Min. and max. transmission factors | 0.984–0.990 | 0.913–0.954 |

Neutron. A refinement with the scattering lengths $b_C = 6.6484$, $b_N = 9.21$ and $b_H = -3.7409$ fm (Koester & Rauch, 1981) gave the results reported in Tables 3 and 4.

X-ray. Scattering factors computed by Fukamachi (1971) were used for C and N, and those in *International Tables for X-ray Crystallography* (1974) for H. Two separate refinements were performed. In the first case (X-ray 1) the refinement was based on the complete data set, following the κ formalism of Coppens *et al.* (1979). This technique incorporates expansion and contraction (κ) of the atomic valence shells together with a variation of their occupancy related to the charge q . The thermal parameters of the H atom were kept fixed at neutron values. A second refinement (X-ray 2) was performed using only high-order data ($\sin \theta/\lambda > 0.75 \text{ \AA}^{-1}$) and keeping H parameters at neutron values. The results of the refinements are given in Table 3, and Fig. 2 shows a stereoscopic view of the molecule.*

Discussion. As can be seen from Table 3, there is good agreement between atomic coordinates derived from neutron and X-ray data. The corresponding bond distances and angles are given in Table 4. The geometry

* A description of the crystal used for the neutron data collection and lists of structure factors for the neutron and X-ray experiments have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38648 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Least-squares refinements*

| | Neutron | X-ray 1 (κ refinement) | X-ray 2 (high-order) |
|---|--------------------------|-----------------------------------|-------------------------|
| $\sin \theta/\lambda$ (min.) (Å ⁻¹) | 0 | 0 | 0.75 |
| $\sin \theta/\lambda$ (max.) (Å ⁻¹) | 0.91 | 1.08 | 1.08 |
| Number of observations | 1308 | 1190 | 364 |
| Number of variables | 56 | 62 | 46 |
| Width parameters of the mosaic distribution (g) | $5.8 (1) \times 10^{-4}$ | $1.8 (3) \times 10^{-4}$ | Fixed |
| Most severe extinction correction (v) | 0.87 | 0.94 | — |
| $R(F)$ (%) | 2.9 | 4.0 | 2.8 |
| $R_w(F)$ (%) $ w = 1/\sigma^2(F) $ | 1.4 | 2.8 | 2.6 |

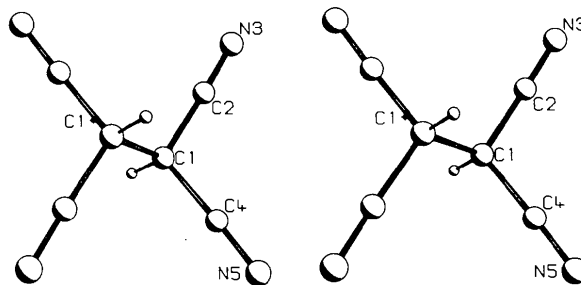


Fig. 2. Stereoscopic view of the molecule and numbering of the atoms.

Table 3. Atomic positional and thermal parameters ($\times 10^4$)The temperature factor used is $\exp[-2\pi^2(h^2a^*U_{11} + \dots + 2hka^*b^*U_{12} + \dots)]$. (U_{ij} in \AA^2). N^* denotes neutron temperature factors multiplied by 0.836.

| | | <i>x</i> | <i>y</i> | <i>z</i> | U_{11} | U_{22} | U_{33} | U_{12} | U_{23} | U_{13} |
|------|-----------|-----------|-----------|-----------|-------------------------|----------|----------|----------|----------|----------|
| C(1) | <i>N</i> | 4089 (1) | 47 (1) | 5431 (1) | 162 (2) | 138 (2) | 182 (2) | -4 (1) | 13 (2) | 17 (1) |
| | <i>X1</i> | 4082 (1) | 51 (1) | 5430 (1) | 155 (3) | 121 (3) | 147 (3) | -4 (2) | 0 (2) | 16 (2) |
| | <i>X2</i> | 4082 (2) | 53 (2) | 5431 (1) | 137 (3) | 107 (3) | 132 (3) | -6 (3) | 7 (2) | 18 (2) |
| | N^* | | | | 135 | 115 | 152 | -3 | 11 | 14 |
| C(2) | <i>N</i> | 2655 (1) | 1105 (1) | 4252 (1) | 179 (2) | 166 (2) | 243 (2) | 14 (2) | 15 (2) | -17 (2) |
| | <i>X1</i> | 2642 (1) | 1114 (1) | 4238 (1) | 185 (3) | 141 (3) | 228 (4) | -15 (2) | -14 (2) | 27 (2) |
| | <i>X2</i> | 2647 (2) | 1106 (2) | 4244 (2) | 159 (4) | 142 (3) | 191 (5) | 9 (3) | 8 (3) | -7 (3) |
| | N^* | | | | 150 | 139 | 203 | 12 | 13 | -14 |
| N(3) | <i>N</i> | 1568 (1) | 1999 (1) | 3332 (1) | 264 (2) | 254 (2) | 371 (2) | 48 (1) | 47 (2) | -100 (2) |
| | <i>X1</i> | 1564 (1) | 1994 (1) | 3318 (1) | 250 (4) | 213 (4) | 331 (4) | 14 (2) | 20 (2) | -46 (3) |
| | <i>X2</i> | 1565 (3) | 1997 (2) | 3309 (3) | 231 (5) | 218 (4) | 321 (7) | 41 (3) | 38 (4) | -84 (4) |
| | N^* | | | | 221 | 212 | 310 | 40 | 39 | -84 |
| C(4) | <i>N</i> | 3570 (1) | -2309 (1) | 5950 (1) | 208 (2) | 149 (2) | 231 (2) | -16 (2) | 34 (2) | 52 (2) |
| | <i>X1</i> | 3557 (1) | -2327 (1) | 5944 (1) | 174 (3) | 183 (3) | 180 (3) | 6 (2) | 1 (2) | 39 (2) |
| | <i>X2</i> | 3558 (2) | -2312 (2) | 5942 (2) | 178 (4) | 127 (3) | 183 (4) | -10 (3) | 26 (3) | 50 (3) |
| | N^* | | | | 174 | 125 | 193 | -13 | 28 | 43 |
| N(5) | <i>N</i> | 3215 (1) | -4162 (1) | 6380 (1) | 339 (2) | 159 (1) | 350 (2) | -31 (1) | 56 (1) | 125 (2) |
| | <i>X1</i> | 3200 (1) | -4169 (1) | 6366 (1) | 274 (4) | 184 (3) | 278 (4) | -8 (2) | 27 (2) | 97 (2) |
| | <i>X2</i> | 3202 (2) | -4176 (2) | 6368 (2) | 285 (5) | 143 (3) | 287 (5) | -25 (3) | 44 (3) | 105 (4) |
| | N^* | | | | 283 | 133 | 293 | -26 | 47 | 105 |
| H | <i>N</i> | 4271 (1) | 1157 (2) | 6599 (1) | 369 (5) | 298 (5) | 286 (5) | -1 (4) | -71 (4) | 41 (4) |
| | <i>X1</i> | 4270 (10) | 1030 (10) | 6470 (10) | Fixed at neutron values | | | | | |
| | <i>X2</i> | | | | 308 | 249 | 239 | -1 | -59 | 34 |
| | N^* | | | | | | | | | |

of the two cyano groups which are independent from a crystallographic point of view are quite comparable. The largest discrepancy between the neutron and X-ray high-order refinement occurs for the central distance C(1)—C(1*). This can be partially explained by the fact that the two atoms are related by a centre of symmetry. Any difference in the atomic coordinates produces a difference in the bond length larger than for a bond between two independent atoms. By a librational analysis of rigid-molecule motion (Schomaker & Trueblood, 1968) the shortening of the bond lengths due to thermal motion has been estimated to be between 0.002 and 0.003 \AA . The agreement is less satisfactory between anisotropic temperature factors, the neutron values being systematically larger than the X-ray high-order values. Such differences are not unusual in the literature and Coppens & Vos (1971) suggested in a similar situation that the neutron data collection temperature was higher than the X-ray temperature. According to Coppens (1978), the neutron temperature factors were scaled to high-order results by multiplication with the ratio $\langle U_{ii}(X)/U_{ii}(N) \rangle$, which in the present case has the value 0.836.

Such 'corrected' temperature factors are presented in Table 3 on the lines denoted N^* and it can be seen that the agreement with X-ray high-order values is now nearly perfect. These values have been used for the production of $X-N$ maps, together with the neutron coordinates, for which the difference due to temperature mismatch is negligibly small.

Charge distribution

Table 5 shows the charges (q) and the expansion-contraction term (κ) derived from the 'X-ray 1' refinement. The two equivalent cyano groups have similar charges. For C(1) and the N atoms, the relation between κ and q conforms very well with Figs. 4 and 5 of Coppens *et al.* (1979), while for C(2) and C(4) one would expect less contracted atoms ($\kappa \approx 1.05$); but,

Table 4. Bond distances (\AA) and angles ($^\circ$)

| | <i>N</i> | <i>X1</i> | <i>X2</i> |
|-----------------|-----------|-----------|-----------|
| C(1)—C(1*) | 1.553 (1) | 1.562 (1) | 1.563 (2) |
| C(1)—C(2) | 1.467 (1) | 1.477 (1) | 1.470 (2) |
| C(1)—C(4) | 1.464 (1) | 1.476 (1) | 1.468 (2) |
| C(1)—H | 1.107 (1) | 0.982 (7) | 1.104 (1) |
| C(2)—N(3) | 1.146 (1) | 1.138 (1) | 1.150 (2) |
| C(4)—N(5) | 1.146 (1) | 1.139 (1) | 1.151 (2) |
| C(4)—C(1)—C(2) | 111.1 (1) | 110.9 (1) | 110.7 (1) |
| H—C(1)—C(2) | 107.9 (1) | 109.0 (4) | 108.4 (1) |
| H—C(1)—C(4) | 108.3 (1) | 108.6 (4) | 108.6 (1) |
| C(2)—C(1)—C(1*) | 110.3 (1) | 110.3 (1) | 110.4 (1) |
| C(4)—C(1)—C(1*) | 110.2 (1) | 109.8 (1) | 109.8 (1) |
| H—C(1)—C(1*) | 109.1 (1) | 108.0 (4) | 108.9 (1) |
| N(3)—C(2)—C(1) | 177.8 (2) | 177.9 (2) | 177.6 (2) |
| N(5)—C(4)—C(1) | 178.0 (2) | 178.0 (2) | 177.8 (3) |

Table 5. Net charges and κ values

| | q | κ |
|------|-----------|----------|
| C(1) | -0.15 (4) | 1.03 (1) |
| C(2) | 0.20 (4) | 1.10 (1) |
| N(3) | -0.27 (3) | 1.01 (1) |
| C(4) | 0.33 (4) | 1.11 (1) |
| N(5) | -0.30 (3) | 1.03 (1) |
| H | 0.19 (3) | 1.46 (4) |

according to the comments of the authors, the curve for C atoms is less well defined. They observe a much clearer correlation considering only points for which neutron thermal parameters are available, and this curve gives a better estimate ($\kappa \approx 1.09$) of our values. The H atom agrees with the mean value of $\kappa = 1.4$ given in the same paper. A schematic representation of the charge distribution in the molecule is given in Fig. 3.

There is an alternance of positive and negative atomic charges everywhere in the molecule except at the ends of the central bond where the two C atoms are negative. The repulsion of these charges could explain, at least partially, the stretching of the central bond.

Deformation density maps

Deformation electron density maps have been produced by both $X-N$ and $X-X$ methods. The X-ray data were limited to $\sin \theta/\lambda = 0.6 \text{ \AA}^{-1}$ because this is the value to which the full data set was collected and with the highest accuracy. The scale factor was obtained from a least-squares refinement using other fixed parameters. In both cases the neutron parameters were used for the H atom. Some sections are presented in Fig. 4. Only one of the $-C \equiv N$ bonds is given, the other being quite comparable. It can be seen that the maps produced by the two methods look very similar except in the lone-pair region which is, as usual, underestimated by the $X-X$ method (Coppens, 1978). The cross sections perpendicular to the bonds through their midpoints show a quasi cylindrical symmetry. Because of the relatively limited data range, the peak heights are slightly reduced (Lehmann & Coppens, 1977). According to these authors, the observed maximum density $\rho'(0)$ is related to the density at infinite resolution $\rho(0)$ by the relation $\rho'(0) = \rho(0)4\pi^{-1/2} \int_0^v t^2 \exp(-t^2) dt$, with $v = 2^{1/2} \pi (2 \sin \theta/\lambda)_{\max} \sigma_T$ describes the width of the thermally smeared density. $\rho(0)$ and σ_T may be evaluated by fitting the observed and calculated $\rho'(0)$ for different resolutions. The results are given for each type of bond and the lone pairs in Table 6. It may be noticed that the density in the central bond is low compared to the other C-C bonds. This density will be compared later, when the data become available, with the value observed in differently substituted ethanes.

We wish to thank Professor G. Leroy (Laboratoire de Chimie Quantique, Université de Louvain) for supplying the crystals.

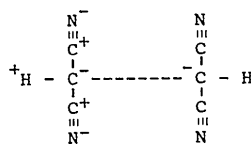


Fig. 3. Schematic representation of the charge distribution.

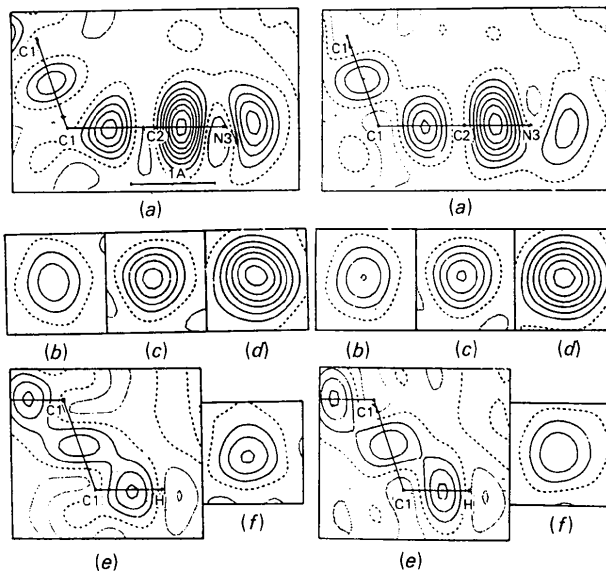


Fig. 4. Sections in the deformation density maps. $X-N$ maps are on the left and $X-X$ on the right. Contours are at $0.1 e \text{ \AA}^{-3}$ (— positive, - - zero, ... negative). (a) Section in the plane of the central bond and the $C(2) \equiv N(3)$ cyano group. (b), (c), (d) Cross sections through the midpoints of the central bond, $C(1)-C(2)$ and $C(2) \equiv N(3)$ respectively. (e) The plane of the $C(1^*)-C(1)-H$ bond. (f) Cross section through the midpoint of $C-H$.

Table 6. Evaluation of the densities ($e \text{ \AA}^{-3}$) at infinite resolution

| | $X-N$ | $X-X$ |
|------------------|-------|-------|
| C-C (central) | 0.31 | 0.31 |
| C-C(\equiv N) | 0.44 | 0.45 |
| C \equiv N | 0.90 | 0.83 |
| C-H | 0.39 | 0.42 |
| Lone pair | 0.57 | 0.37 |

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Structure of *cis*-2,3-Tetralindiol, *cis*-1,2,3,4-Tetrahydro-2,3-naphthalenediol, C₁₀H₁₂O₂

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Abstract. $M_r = 164.0$, monoclinic, $P2_1/c$, $a = 9.401$ (4), $b = 11.664$ (1), $c = 17.632$ (9) Å, $\beta = 117.97$ (4)°, $V = 1707.5$ Å³, $Z = 8$, $D_m = 1.29$, $D_x = 1.28$ g cm⁻³, $\text{Cu } K\alpha_1$ $\lambda = 1.54051$ Å, $\mu = 7.27$ cm⁻¹, $F(000) = 704$, $T = 300$ K, $R = 0.053$ for 1088 reflections. The title compound is a naphthalene derivative with adjacent molecules linked by intermolecular hydrogen bonds.

Introduction. Many polycyclic aromatic hydrocarbons are common environmental pollutants and potent chemical carcinogens. The active forms are believed to be metabolic derivatives of the parent hydrocarbons (Harvey, 1981, 1982). A series of naphthalene derivatives which model various metabolites of other carcinogenic hydrocarbons and are, in some cases, themselves natural metabolites of naphthalene (Tsang, Griffin, Horning & Stillwell, 1982) have been chosen for study. The structure of *cis*-2,3-tetralindiol confirms a stereochemical assignment made on the basis of its NMR spectra.

Experimental. Colorless, needle-like crystal, approximately $0.6 \times 0.1 \times 0.1$ mm, D_m by flotation in dioxane/CHCl₃; unit-cell dimensions and systematic absences $h0l$, $l = 2n$, and $0k0$, $k = 2n$, indicated monoclinic, $P2_1/c$ confirmed by successful determination of structure; lattice constants from least-squares fit of 37 carefully measured 2θ values (2° take-off angle, 0.05° slit) for $2\theta > 61^\circ$; General Electric XRD-490 automated diffractometer, station-

ary-crystal-stationary-counter, balanced Ni and Co filters, 2648 reflections measured, $2\theta_{\text{max}} = 120^\circ$, 1088 (41%) considered statistically significant by $[I_{\text{Ni}} - 2\sigma(I_{\text{Ni}})] - [I_{\text{Co}} - 2\sigma(I_{\text{Co}})] > 500$ with σ 's based entirely on counting statistics and $F > 68$, data corrected for polarization and background as a function of 2θ , φ absorption minimal and not corrected for, three standard reflections after every 100 reflections, no significant change in intensity; direct methods, normalized structure factors, scaled by a k curve (Karle, Hauptman & Christ, 1958), phases determined with *MULTAN* (Germain, Main & Woolfson, 1971); initial E map contained twenty-four non-hydrogen peaks corresponding to two asymmetric molecular units; these 24 atoms refined isotropically on F , block-diagonal least squares, $R = 0.154$; ring hydrogen atoms calculated on the basis of sp^2 or sp^3 geometry and C–H bond distance of 1.0 Å; refinement of hydrogen-atom thermal parameters resulted in one set of temperature factors approximately twice as large as another set so average value in each set was calculated and hydrogen atoms assigned values for U of 0.0569 or 0.1139 Å²; continued block-diagonal least squares of nonhydrogen atom positions (anisotropic) and hydrogen-atom positions (fixed isotropic) resulted in C–H bond distances between 0.93 (7) and 1.15 (6) Å; all four hydroxyl hydrogen atoms participate in an intermolecular hydrogen-bond network; coordinates for two of the hydroxyl hydrogen atoms [H(B1) and H(B1')] found on a difference map, coordinates for remaining two hydroxyl hydrogen atoms [H(B2) and H(B2')]