

symmetry (see Fig. 2). There are three anions [see Fig. 1(b)], one on a twofold axis with statistical occupancy (see also Fig. 2).

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Redetermination of the Crystal Structure of 3,5,7-Trimethyl-1-adamantyl Undecafluorodiantimonate(V)

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Abstract

The crystal structure of the 3,5,7-trimethyl-1-adamantyl cation (1) has been redetermined by X-ray diffraction at 103 K, since the structural data already published [Laube (1986a). *Angew. Chem.* **98**, 368–369; Laube (1986b). *Angew. Chem. Int. Ed. Engl.* **25**, 349–350] showed a poor *R*-value (0.070) and exhibited a rather large variety of lengths of chemically but not crystallographically equivalent bonds, which is no longer present in the newly determined structure with $R_1 = 0.0452$. A partial disorder model of the anion could be refined and the experimental data of (1) are compared with results of semi-empirical and *ab initio* calculations. The results are interpreted in terms of stabilization of (1) by C,C-hyperconjugation. The detailed experimental procedure for the synthesis and crystallization of (1).Sb₂F₁₁ is described.

Introduction

The 3,5,7-trimethyl-1-adamantyl cation (1) is the only clear example yet known of C,C-hyperconjugation (Olah *et al.*, 1985) in a crystal structure where both C_α–C_β shortening and C_β–C_γ elongation could be proven experimentally [Fig. 1 (Laube, 1986a,b)]. In the recently determined structure of the *tert*-butyl cation (Hollenstein & Laube, 1993), only the C_α–C_β shortening could be observed, because the H atoms were partially disor-

dered and refined with constraints. The poor original refinement of (1).Sb₂F₁₁ and the availability of high-quality *ab initio* data for comparison (Buzek, Schleyer & Sieber, 1992) made a more precise crystal structure determination necessary.

Experimental

Synthesis and crystallization of (1).Sb₂F₁₁

The reaction was carried out in a special apparatus (see Laube, 1994, Fig. 6 and *Experimental* section). In the reaction vessel, a solution of 413 mg of 1-

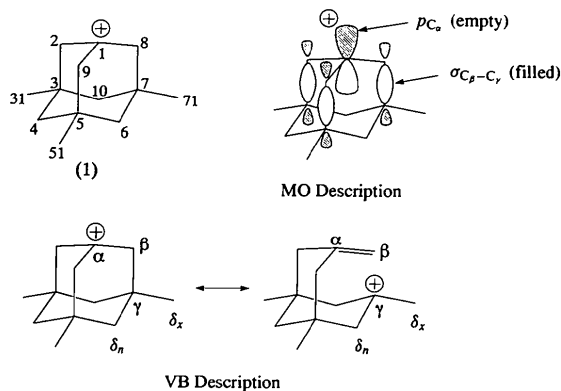


Fig. 1. Constitution and numbering of (1) and hyperconjugation in the MO and VB description (n = endocyclic, x = exocyclic).

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fluoro-3,5,7-trimethyladamantane [2.11 mmol; prepared analogously to Fort & Schleyer (1965)] in 35 ml of $\text{CCl}_2\text{F}-\text{CClF}_2$ was prepared at 240 K. From the PTFE dropping funnel, two equivalents of antimony pentafluoride (964 mg, 4.45 mmol; FLUKA) in 8 ml of $\text{CCl}_2\text{F}-\text{CClF}_2$ were slowly added. After 30 min, the brownish-pink reaction mixture was filtered at 240 K, the solid raw product washed with 3 ml of $\text{CCl}_2\text{F}-\text{CClF}_2$ and 2×3 ml of CH_2Cl_2 , and dried in the high vacuum (HV) at 240 K. The dry colourless filter cake was coarsely powdered under argon in the reaction vessel, and transferred into a two-neck flask (with an argon frit on its second neck), connected at joint 5 (instead of the 'filtration vessel'). CH_2Cl_2 (20 ml) was added slowly at 238 K, the suspension rapidly filtered through the argon frit, and the clear yellow filtrate was collected in a two-neck flask at 238 K under argon. The stoppered flask was cooled from 238 to 203 K within 35 h (in a programmable cryostat). The mother liquor was removed at 203 K with a syringe, and the crystals (slightly intergrown prisms, length up to 3 mm) were dried in the HV at 233 K.

X-ray measurement

A suitable crystal was selected and mounted on an Enraf-Nonius CAD-4 diffractometer (MoK α radiation with $\lambda = 0.71069$ Å, graphite monochromator), as described by Laube (1994). The final data of the orthorhombic unit cell [space group $Pbca$, $M_r(\text{C}_{13}\text{H}_{21}\text{Sb}_2\text{F}_{11}) = 629.791$, $Z = 8$, $F(000) = 2400$] at 103 K [$a = 11.527$ (6), $b = 16.228$ (8), $c = 20.450$ (8) Å, $V = 3825$ (3) Å³, $\rho_x = 2.187$ g cm⁻³, $\mu = 2.921$ mm⁻¹] were obtained from 25 reflections in the range $\theta = 8.1$ – 10.3° . A total of 4705 reflections (3593 with $F > 4\sigma F_{\text{obs}}$) up to $\theta = 28^\circ$ were measured (ω – 2θ scans; $0 \leq h \leq 15$, $0 \leq k \leq 21$, $0 \leq l \leq 26$). The $\bar{3}17$ reflection ($\chi_{\text{Euler}} = 87.7^\circ$) was measured 36 times at different φ -angles for an empirical absorption correction.

Solution and refinement

The data reduction was carried out with *DIFDAT* (Stewart *et al.*, 1992) and the absorption correction was applied with *ABSCAL* (Watenpaugh & Stewart, 1992) from the *XTAL3.2* system (update August 1993; Hall, Flack & Stewart, 1992). The structure was solved using the Patterson option of *SHELXS86* (Sheldrick, 1985, 1990) and refined on F^2 (full matrix) with *SHELXL93* (Sheldrick, 1993, 1995). A disorder model for one part of the anion could be refined [major orientation: F21–F25, anisotropic, $p = 0.800$ (8); minor orientation: F211–F251, isotropic, $p = 0.200$ (8)] using SADI restraints, so that F1 and F211–F251 keep approximate $4mm$ (C_{4v} ; fourfold axis through F1 and F211) symmetry as a pseudo-octahedron centred at Sb2. The isotropic U 's of F211 and F231 were constrained to be equal to the equivalent U 's of F21 and F23. The

disorder of Sb2 is indicated by the high anisotropy of its p.d.f. [eigenvalues of the displacement tensors (in Å²) of Sb1: 0.0350 (2), 0.0225 (2), 0.0172 (2); of Sb2: 0.0620 (2), 0.0214 (2), 0.0169 (2)] and strong difference-density peaks and holes near Sb2, but the disorder could not be resolved (the distance between the two split Sb2 positions is estimated to be *ca* 0.30 Å, see supplementary material*). The H atoms attached to the following C atoms were refined with constraints: one common C–H length and one $U_{\text{iso}}(\text{H})$ at each C atom; C6 and C9: idealized CH_2 groups with riding H positions, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ at each C atom; C51 and C71: one common $U_{\text{iso}}(\text{H})$ at each C atom. The final weights were $w = 1/[(\sigma F_{\text{obs}})^2 + (0.0780P)^2 + 19.48P]$, with $P = [\max(F_{\text{obs}}^2, 0) + 2F_{\text{calc}}^2]/3$; the extinction parameter converged to 0.0006 (1). The final R -values are ($F > 4\sigma F_{\text{obs}}$): $R_1 = 0.0452$, $wR_2 = 0.1174$; for all data: $R_1 = 0.0574$, $wR_2 = 0.1289$ (4600 data, 24 restraints, 307 parameters). The highest difference-density maxima (up to $2.97 \text{ e} \text{ \AA}^{-3}$) and minima (up to $-2.84 \text{ e} \text{ \AA}^{-3}$) occur around the Sb atoms; the highest maxima and minima in the cation region are 0.57 and $-0.49 \text{ e} \text{ \AA}^{-3}$ (r.m.s. deviation of the difference density from the mean: $0.20 \text{ e} \text{ \AA}^{-3}$). The data thus obtained are considered as final (see Fig. 2 and Table 1) and used for the preparation of the tables [in most cases in the supplementary material with a modified version of *PARST88* (Nardelli, 1983, 1988)] and figures [with *ORTEP* (Davenport, Hall & Dreissig, 1992; Johnson, 1976) from the *XTAL3.2* system or with *SYBYL* (Tripos Associates Inc., 1992)].

The thermal motion analysis was carried out with *THMA11* (Trueblood, 1988). In order to obtain electron

*Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, and crystal packing and difference-density diagrams have been deposited with the IUCr (Reference HU0423). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

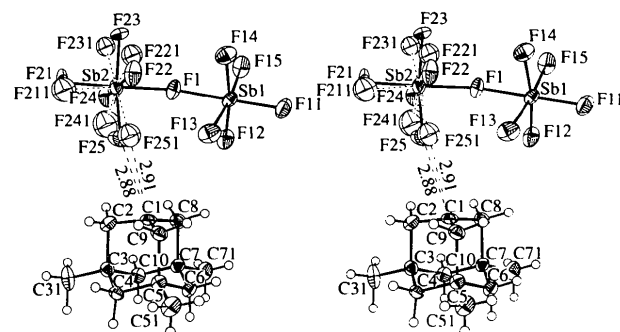


Fig. 2. ORTEP stereodiagram of the asymmetric unit of (1). Sb_2F_{11} . The displacement ellipsoids are drawn at the 50% probability level, and the H atoms are represented by spheres with radius 0.1 Å. The F atoms of the minor orientation of the disordered part of the anion (F211–F251) are drawn with dashed bonds to Sb2. Distances are given in Å.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{eq}
Sb1	0.13593 (3)	0.02241 (2)	0.19756 (2)	0.02489 (13)
Sb2	0.37921 (3)	-0.09929 (3)	0.11100 (2)	0.03342 (14)
F1	0.2878 (3)	-0.0085 (2)	0.1547 (2)	0.0359 (8)
F11	-0.0030 (3)	0.0527 (3)	0.2367 (2)	0.0430 (8)
F12	0.2251 (3)	0.0413 (2)	0.2720 (2)	0.0421 (9)
F13	0.1250 (3)	-0.0873 (2)	0.2216 (2)	0.0436 (9)
F14	0.0667 (4)	-0.0029 (2)	0.1184 (2)	0.0418 (8)
F15	0.1666 (3)	0.1295 (2)	0.1683 (2)	0.0444 (9)
F21	0.4623 (7)	-0.1810 (6)	0.0683 (4)	0.066 (2)
F211	0.4483 (35)	-0.1943 (35)	0.0784 (18)	0.066
F22	0.2391 (4)	-0.1428 (3)	0.0879 (3)	0.0428 (14)
F221	0.2424 (17)	-0.1235 (13)	0.0624 (10)	0.045 (7)
F23	0.3777 (5)	-0.0307 (3)	0.0370 (2)	0.0447 (14)
F231	0.4489 (18)	-0.0320 (11)	0.0506 (9)	0.045
F24	0.5146 (4)	-0.0418 (3)	0.1360 (2)	0.0445 (14)
F241	0.4974 (18)	-0.0888 (16)	0.1742 (12)	0.097 (11)
F25	0.3765 (6)	-0.1504 (4)	0.1896 (3)	0.059 (2)
F251	0.3019 (19)	-0.1752 (12)	0.1796 (10)	0.058 (6)
C1	0.3186 (5)	-0.2033 (3)	0.3198 (2)	0.0267 (10)
C2	0.4304 (5)	-0.2391 (3)	0.3081 (3)	0.0288 (11)
C3	0.4572 (4)	-0.2769 (3)	0.3793 (3)	0.0253 (10)
C4	0.3608 (4)	-0.3367 (3)	0.3956 (2)	0.0236 (10)
C5	0.2416 (4)	-0.2950 (3)	0.3961 (2)	0.0237 (9)
C6	0.2429 (5)	-0.2233 (3)	0.4447 (3)	0.0261 (10)
C7	0.3389 (5)	-0.1617 (3)	0.4286 (2)	0.0248 (10)
C8	0.3143 (5)	-0.1267 (3)	0.3564 (3)	0.0291 (11)
C9	0.2202 (5)	-0.2570 (3)	0.3239 (3)	0.0278 (10)
C10	0.4567 (5)	-0.2041 (3)	0.4270 (3)	0.0272 (10)
C31	0.5753 (5)	-0.3184 (4)	0.3761 (4)	0.0413 (15)
C51	0.1443 (6)	-0.3548 (4)	0.4090 (3)	0.0362 (13)
C71	0.3386 (6)	-0.0884 (4)	0.4756 (3)	0.0358 (13)

and difference-density contour diagrams, the refinement [based on F^2 , full matrix, $w = 1/(\sigma F_{\text{obs}}^2)^2$] was repeated with *CRYLSQ* (Olthof-Hazekamp, 1992) from the *XTAL3.2* system, where all H parameters, positions of F211–F251 and U_{iso} -values of F211 and F231 were not refined. An isotropic extinction correction according to Becker & Coppens [1974 (type I, Gaussian distribution)] was applied and refined in the last cycles of the refinement with *CRYLSQ* [$g = 0.31$ (4); final R -values for reflections with $F^2 > 2\sigma F_{\text{obs}}^2$: $R = 0.046$ (using $|F|$), $wR = 0.112$ (using $|F^2|$)]. The difference-density contour maps in the three planes defined by C1 and the three possibilities of C_β and C_γ [Figs. 3(a)–(c)] were first generated with the program sequence *FOURR* (Stewart, Holden, Doherty & Hall, 1992), *SLANT* (Spadaccini & Alden, 1992), *CONTRS* (Spackman, 1992), *PREVUE* (Streltsov, 1993), *PLOTX* (Hall, Olthof-Hazekamp, Flack, Hester & Bartsch, 1992), all from *XTAL3.2*. A *FORTTRAN77* program was written, which generated the same diagrams and their arithmetic average [Fig. 3(d)] from the *ASCII* data of the *SLANT* outputs.

Results

The structure of the closest ion pair [C1–F25 = 2.877 (8), C1–F251 = 2.91 (2) \AA] is shown in Fig. 2, and the most important structural parameters of (1) are given in Table 2.

Table 2. Selected geometric parameters (\AA , $^\circ$)

C1–C2	1.433 (8)	C5–C6	1.531 (7)
C1–C9	1.433 (8)	C5–C9	1.619 (7)
C1–C8	1.451 (7)	C6–C7	1.527 (7)
C2–C3	1.610 (7)	C7–C10	1.523 (8)
C3–C4	1.513 (7)	C7–C71	1.529 (7)
C3–C31	1.520 (8)	C7–C8	1.607 (7)
C3–C10	1.532 (7)	[C3–P(4,10,31)]	0.450 (5)
C4–C5	1.533 (7)	[C5–P(4,6,51)]	0.453 (5)
C5–C51	1.506 (8)	[C7–P(6,10,71)]	0.459 (5)
C2–C1–C9	118.4 (5)	C9–C5–C51	106.9 (5)
C2–C1–C8	117.6 (5)	C6–C5–C9	107.7 (4)
C8–C1–C9	117.6 (5)	C4–C5–C9	107.4 (4)
C1–C2–C3	100.1 (4)	C5–C6–C7	111.3 (4)
C4–C3–C31	112.5 (5)	C10–C7–C71	111.5 (5)
C4–C3–C10	110.6 (4)	C6–C7–C10	110.8 (4)
C10–C3–C31	111.9 (5)	C6–C7–C71	111.8 (5)
C2–C3–C4	107.6 (4)	C8–C7–C10	107.3 (4)
C2–C3–C31	107.6 (5)	C8–C7–C71	107.6 (4)
C2–C3–C10	106.4 (4)	C6–C7–C8	107.6 (4)
C3–C4–C5	112.1 (4)	C1–C8–C7	99.5 (4)
C6–C5–C51	112.6 (5)	C1–C9–C5	99.5 (4)
C4–C5–C51	112.6 (5)	C3–C10–C7	111.4 (4)
C4–C5–C6	109.4 (4)		
C4–C3–C10–C7	-55.2 (6)	C4–C5–C6–C7	56.6 (6)
C10–C3–C4–C5	56.3 (6)	C5–C6–C7–C10	-56.7 (6)
C3–C4–C5–C6	-56.9 (6)	C6–C7–C10–C3	55.5 (6)

$P(i,j,k)$ is the plane through C_i, C_j, C_k .

The average C_α – C_β bond length is 1.439 (6) \AA [*i.e.* 0.063 (6) \AA smaller than the reference value for a Csp^3 – CH_2 – Csp^2 bond, 1.502 (1) \AA (Allen *et al.*, 1992)], and the average C_β – C_γ bond length is 1.612 (4) \AA [*i.e.* 0.074 (4) \AA greater than the (not perfectly suitable) reference value for a $(Csp^3)_3C$ – CH_2 – Csp^3 bond, 1.538 (1) \AA (Allen *et al.*, 1992)]. The pyramidalization of C1 [0.212 (5) \AA] is remarkable for an sp^2 -like C atom, and the quaternary C_γ atoms are slightly flattened [average pyramidalization $|\Delta(C_\gamma)|$, ignoring the C_β – C_γ bond: 0.454 (3) \AA], if compared with the reference value of 0.510 \AA [$\frac{1}{3} \times Csp^3$ – Csp^3 ; Allen *et al.* (1992)]. The average C_γ – C_{δ_n} and C_γ – C_{δ_x} bond lengths [1.527 (3) and 1.519 (7) \AA] are slightly smaller [by 0.011 (3) and 0.015 (7) \AA] than their reference values [(Csp^3) $_3C$ – CH_2 – Csp^3 : 1.538 (1) and (Csp^3) $_3C$ – CH_3 : 1.534 (1) \AA (Allen *et al.*, 1992)]. The average C_β – C_α – $C_{\beta'}$ and C_α – C_β – C_γ bond angles [117.9 (3) and 99.7 (2) $^\circ$] also indicate the unusual geometry about C1. In agreement with the slight flattening of the C_γ atoms, the average C_β – C_γ – C_{δ_n} and C_β – C_γ – C_{δ_x} angles [both 107.4 (2) $^\circ$] are slightly smaller, whereas the average C_{δ_n} – C_γ – $C_{\delta_n'}$ and C_{δ_n} – C_γ – C_{δ_x} angles [110.9 (4) and 112.2 (2) $^\circ$] are slightly larger than the ideal tetrahedral angle of 109.5 $^\circ$. Also, the average absolute C_γ – C_{δ_n} – $C_{\gamma'}$ – $C_{\delta_n'}$ torsion angle [56.2 (3) $^\circ$] is smaller than the ideal value of 60 $^\circ$.

Although disorder and the presence of heavy atoms are not ideal prerequisites for the analysis of the difference electron density, the average map of the three chemically equivalent C_α – C_β – C_γ bridges [Fig. 3(d)] shows that the bond density in the C_β – C_γ bond is smaller than in the C_α – C_β and C_γ – C_{δ_x} bonds. The

two local maxima above and below C1 in Fig. 3(d) may be due to an error in the probability distribution function (p.d.f.) caused by the electron deficiency at C1 (neutral and isotropic scattering factors were used throughout this work).

The thermal motion analysis of (1) shows that the displacement parameters of the cation may be roughly explained by the motion of a rigid body. Using the σ_U -values for weights, one obtains a weighted R of 0.082 for all U 's and 0.056 for the diagonal U 's only. The roots of the eigenvalues of the T and L tensors are 0.160, 0.139, 0.127 Å and 4.4, 2.8, 2.0°. The largest differences between the mean-square displacement amplitudes of bonded atoms occur between C1 and C8 [-0.006 (4) Å²], and between C5 and C51 [0.006 (4) Å²]. The rigid-body corrections of the bond lengths are less than 0.007 Å and are thus negligible.

Discussion

The structural features of (1) may be easily explained in terms of stabilization by C,C-hyperconjugation, *i.e.* a delocalization of the C_β — C_γ bond electron density towards the electron deficient C1. This was already concluded by Olah *et al.* (1985) on the basis of NMR chemical shifts of several 1-adamantyl cations (C_α and also C_γ show strong downfield shifts, if compared with neutral molecules), and also Buzek, Schleyer & Sieber (1992) explained the computed structure of the unsubstituted 1-adamantyl cation by hyperconjugation (see Fig. 4).

Agreement between the experimental data of (1) presented here with the *ab initio* data of Buzek, Schleyer & Sieber (1992) about the unsubstituted 1-adamantyl cation is very good. The shortening of the C_α — C_β bond agrees

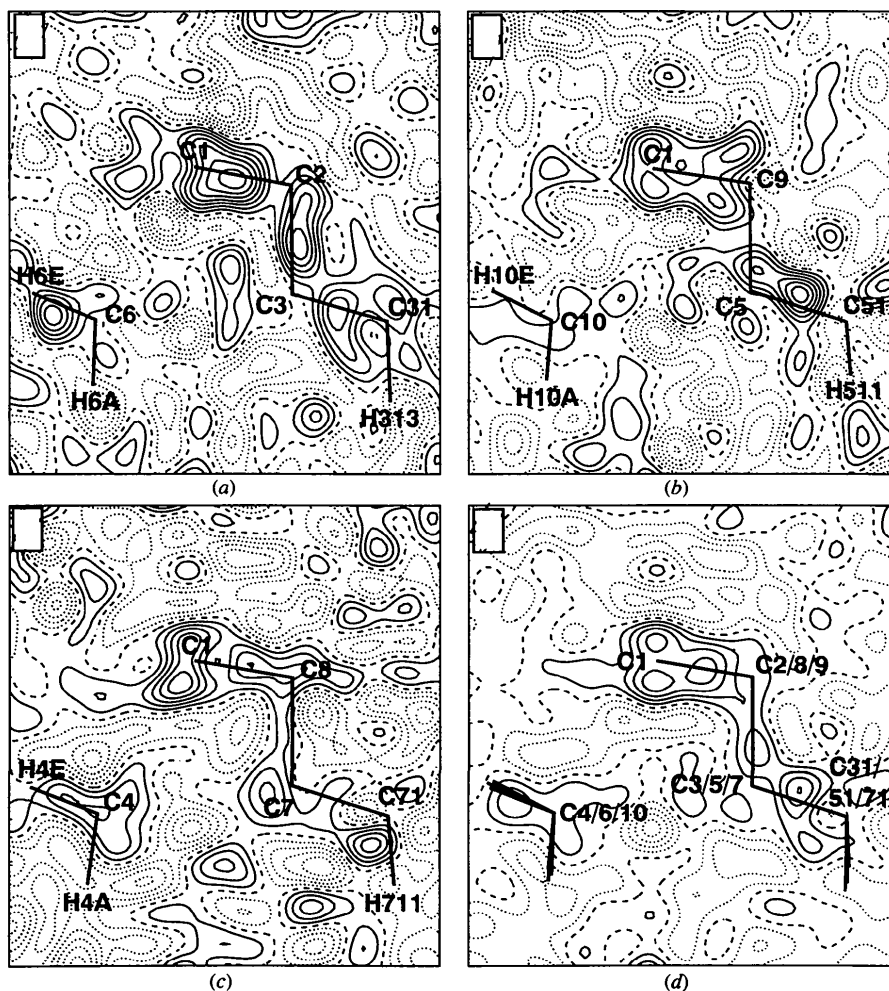


Fig. 3. Difference density in the planes containing the bridges emanating from C1 of (1). Chemical bonds in or near the planes are indicated by thick solid lines. Density difference of the contour lines: $0.1 \text{ e} \text{ \AA}^{-3}$. Density and contour line types: positive = solid, zero = dashed, negative = dotted. (a)–(c) The three crystallographically independent planes; (d) averaged difference density of the three planes.

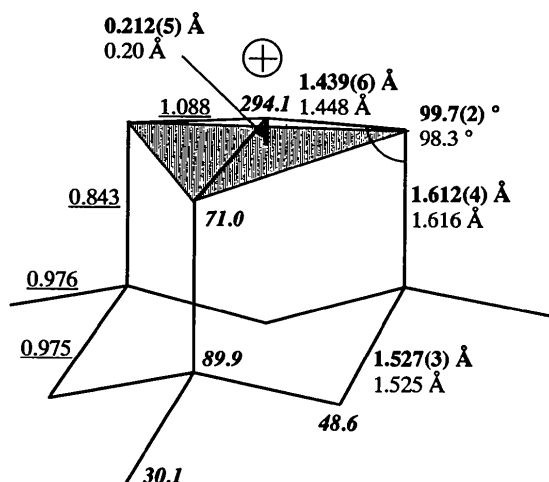


Fig. 4. Comparison of the experimental structure of (1) (averaged data; bold type) with *ab initio* data [regular type; Buzek, Schleyer & Sieber (1992)]. The ^{13}C NMR chemical shifts were determined by Olah *et al.* [(1985); bold italics], and average bond orders computed on the experimental structure with MOPAC [Stewart (1992); underline].

with a partial double-bond character, the elongation of the $\text{C}_\beta\text{--C}_\gamma$ bond with a weakened single bond (see VB description in Fig. 1). The average difference density shown in Fig. 3 and the computed bond orders shown in Fig. 4 also support this interpretation. The ^{13}C data of Olah *et al.* (1985) indicate that a significant amount of the positive charge is found on the C_γ atoms. Accordingly, these atoms are also slightly flattened, and all $\text{C}_\gamma\text{--C}_\delta$ bonds are very slightly shortened.

Concluding remarks

The structural parameters of (1) show strong deviations from reference values, which may be explained by C,C-hyperconjugation, *i.e.* a delocalization of the positive charge from C1 to the C_γ atoms. These results agree well with ^{13}C NMR and *ab initio* data obtained by other authors for (1) and related carbocations.

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