ring introduced at calculated positions, not refined but constrained to ride their C atoms (C—H 0·95 Å; $B = 5$ Å^2). The last ΔF Fourier map exhibits poorly resolved peaks between the U and B atoms but a better peak for the terminal hydrogen HB(4) of the BH_4 group (height 0·8 e Å^{-3}), remaining H atoms located by imposing tetrahedral hydroborate geometry (B—H 1·15 Å and H—B—H 109°): H atoms constrained to ride their B atom. $R = 0{\cdot}037$, $wR = 0{\cdot}047$ ($w = 1$), $(\Delta/\sigma)_{\max} = 0{\cdot}06$, $S = 4{\cdot}15$, $(\Delta\rho)_{\max} = 0{\cdot}6$ e Å^{-3}; no extinction correction; *SDP* Enraf–Nonius programs (Frenz, 1983). Atomic parameters are given in Table 1, the coordination sphere of the U atom is in Fig. 1.*

Related literature. The bending of the line U—B—HB (terminal) of tridentate bonding was found in some other uranium hydroborates: $U(BH_4)_4$ form II (Charpin, Nierlich, Vigner, Lance & Baudry, 1987) and $U(BH_4)[OP(C_6H_5)_3]_2$ (Charpin, Nierlich, Chevrier, Vigner, Lance & Baudry, 1987).

Table 1. *Positional and equivalent isotropic thermal parameters*

	x	y	z	B(Å^2)
U	0·000	0·000	0·000	3·46 (4)*
B	0·174 (5)	0·217 (8)	−0·077 (5)	10 (2)
O	0·000	0·159 (4)	0·192 (3)	4·7 (7)
C(1)	0·000	0·08 (1)	0·307 (7)	12 (3)
C(2)	0·000	0·22 (1)	0·396 (8)	14 (3)
C(3)	0·000	0·386 (9)	0·331 (6)	10 (2)
C(4)	0·000	0·36 (1)	0·210 (8)	14 (3)
HB(1)	0·080	0·193	−0·127	
HB(2)	0·174	0·250	0·021	
HB(3)	0·236	0·083	−0·082	
HB(4)	0·229	0·340	−0·118	

* $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

References

Charpin, P., Nierlich, M., Chevrier, G., Vigner, D., Lance, M. & Baudry, D. (1987). *Acta Cryst.* C**43**, 1255–1258.

Charpin, P., Nierlich, M., Vigner, D., Lance, M. & Baudry, D. (1987). *Acta Cryst.* C**43**, 1465–1467.

Frenz, B. A. (1983). *Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Rietz, R., Edelstein, N., Ruben, H., Templeton, D. & Zalkin, A. (1978). *Inorg. Chem.* **17**, 658–660.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43859 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 1631–1633

Structure of a Benzoyloxytetraphenylazadiene

By F. Florencio, J. Mohedano and S. García-Blanco

X-ray Department, Institute Rocasolano, CSIC, Madrid, Spain

(*Received* 28 *May* 1986; *accepted* 2 *February* 1987)

Abstract. 2-Diphenylmethyleneamino-1-phenylstyryl benzoate, $C_{34}H_{25}NO_2$, $M_r = 479{\cdot}579$, monoclinic, $P2_1/n$, $a = 19{\cdot}364$ (1), $b = 9{\cdot}775$ (2), $c = 14{\cdot}683$ (1) Å, $\beta = 108{\cdot}38$ (1)°, $V = 2632{\cdot}1$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}2102$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}515$ cm^{-1}, $F(000) = 1008$, room temperature, final $R = 0{\cdot}060$, $wR = 0{\cdot}054$ for 3714 reflections. The azadiene chain is not planar. The benzoate group is very nearly planar with the carboxyl group rotated by 178·3 (4)° with respect to the benzene ring. The two phenyl rings attached to the C=C double bond in the azadiene chain are *trans*.

Experimental. Crystal 0·10 × 0·10 × 0·30 mm, Philips 1100 automatic four-circle diffractometer, graphite monochromator, Cu $K\alpha$ radiation. Lattice parameters determined by least-squares fit from 50 reflections with $\theta_{\max} = 24°$. Intensities measured with the same diffractometer, ω–2θ scan, $2 < \theta < 67°$; two reflections used as standard and remeasured after every 90 min, no decomposition observed. 4464 independent reflections ($-20 < h < 20$, $0 < k < 11$, $0 < l < 16$), 3714 observed with $I > 2\sigma(I)$, where $\sigma(I)$ is the standard deviation based on counting statistics. Lorentz and polarization corrections, no correction for absorption.

Structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by full-matrix least squares, $\sum w(|F_o| - |F_c|)^2$ minimized. After isotropic and anisotropic refinement, the resulting difference-density map revealed the positions of the H atoms. Further refinement with anisotropic tempera-

0108-2701/87/081631-03$01.50

ture factors for the non-H atoms and isotropic temperature factors for the H atoms (fixed and equal to those of the atoms to which they are bonded), except H11, H19 and H30 which were geometrically positioned, continued until the largest $\Delta/\sigma = 0{\cdot}03$. Max. heights in final difference map $= 0{\cdot}21$ e Å^{-3}. Final $R = 0{\cdot}060$ and $wR = \sum w[(|F_o| - |F_c|)^2/|F_o|]^{1/2} = 0{\cdot}054$ with $w = 1$.

The computations were made with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *PARST* (Nardelli, 1983) on a VAX11/750 computer.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^4)

$$U_{\rm eq} = \tfrac{1}{3}\sum U_{ij}a_i^*a_j^*a_ia_j\cos(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$U_{\rm eq}$
C1	0·57906 (14)	0·13841 (29)	0·89759 (20)	423 (10)
C2	0·60660 (14)	0·02832 (30)	0·86617 (19)	434 (10)
N3	0·68231 (12)	0·01677 (24)	0·88526 (16)	438 (9)
C4	0·70984 (14)	−0·03064 (27)	0·82234 (20)	394 (10)
O5	0·50480 (9)	0·12702 (20)	0·89226 (14)	449 (7)
O6	0·47271 (12)	0·28749 (28)	0·77663 (20)	806 (12)
C7	0·45626 (16)	0·20561 (31)	0·82666 (22)	497 (11)
C8	0·38091 (15)	0·17393 (31)	0·82471 (21)	466 (11)
C9	0·36598 (17)	0·07429 (37)	0·88359 (23)	562 (12)
C10	0·29423 (19)	0·04673 (45)	0·87730 (26)	682 (15)
C11	0·23924 (18)	0·11925 (48)	0·81464 (29)	753 (17)
C12	0·25308 (20)	0·21810 (44)	0·75628 (32)	752 (16)
C13	0·32435 (18)	0·24645 (38)	0·76091 (27)	645 (14)
C14	0·61551 (15)	0·26102 (30)	0·94859 (19)	427 (10)
C15	0·68139 (17)	0·30643 (33)	0·94113 (23)	515 (12)
C16	0·71404 (19)	0·42383 (38)	0·99061 (25)	640 (14)
C17	0·68213 (21)	0·49360 (39)	1·04804 (26)	683 (15)
C18	0·61717 (20)	0·44951 (37)	1·05673 (25)	641 (14)
C19	0·58382 (17)	0·33367 (35)	1·00735 (22)	544 (12)
C20	0·56117 (14)	−0·09403 (29)	0·82662 (20)	435 (10)
C21	0·49803 (17)	−0·08652 (35)	0·74922 (23)	535 (12)
C22	0·45632 (18)	−0·20351 (41)	0·71602 (26)	649 (14)
C23	0·47772 (20)	−0·32735 (41)	0·75925 (28)	670 (15)
C24	0·54079 (20)	−0·33731 (37)	0·83590 (26)	615 (14)
C25	0·58191 (17)	−0·22193 (32)	0·86978 (23)	509 (11)
C26	0·66919 (14)	−0·05901 (27)	0·72008 (20)	394 (10)
C27	0·63411 (17)	0·04556 (31)	0·65996 (23)	482 (11)
C28	0·59699 (18)	0·01859 (36)	0·56454 (24)	591 (13)
C29	0·59332 (19)	−0·11211 (37)	0·52930 (25)	608 (13)
C30	0·62759 (19)	−0·21745 (33)	0·58879 (24)	604 (13)
C31	0·66545 (16)	−0·19179 (30)	0·68419 (22)	497 (11)
C32	0·78944 (13)	−0·05952 (26)	0·85360 (19)	373 (9)
C33	0·82696 (16)	−0·08243 (33)	0·94958 (22)	501 (11)
C34	0·90193 (18)	−0·10620 (33)	0·97714 (25)	573 (12)
C35	0·93849 (17)	−0·10807 (32)	0·91101 (26)	566 (13)
C36	0·90179 (16)	−0·08507 (32)	0·81645 (26)	498 (12)
C37	0·82704 (15)	−0·06094 (27)	0·78717 (22)	426 (10)

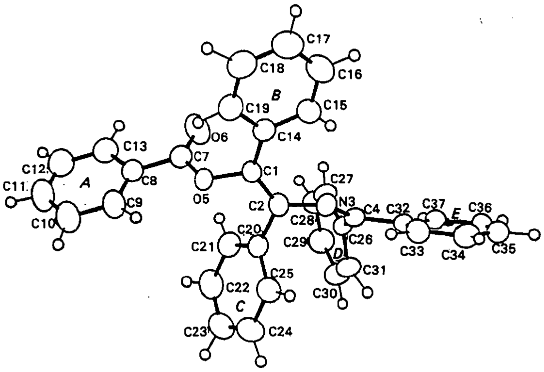

Fig. 1. A view of the molecule with the atomic labelling.

The anomalous-dispersion coefficients and scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The atomic parameters are listed in Table 1.* Fig. 1 displays a view of the molecule with the numbering used in the crystallographic study. Table 2 shows the bond lengths and angles with their e.s.d.'s.

Related literature. Bond lengths and angles in the chain are in good agreement with those found in analogous

Table 2. *Bond distances* (Å), *bond angles* (°) *and torsional angles* (°)

C1—C2	1·344 (4)	C1—O5	1·420 (3)
C1—C14	1·468 (4)	C2—N3	1·408 (4)
C2—C20	1·488 (4)	N3—C4	1·289 (4)
C4—C26	1·485 (4)	C4—C32	1·490 (4)
O5—C7	1·352 (3)	O6—C7	1·194 (5)
C7—C8	1·483 (4)	C8—C9	1·391 (5)
C8—C13	1·389 (4)	C9—C1)	1·389 (5)
C10—C11	1·365 (5)	C11—C12	1·371 (7)
C12—C13	1·388 (6)	C14—C15	1·387 (5)
C14—C19	1·399 (5)	C15—C16	1·396 (5)
C16—C17	1·373 (6)	C17—C18	1·373 (6)
C18—C19	1·387 (5)	C20—C21	1·384 (4)
C20—C25	1·400 (4)	C21—C22	1·394 (5)
C22—C23	1·367 (5)	C23—C24	1·379 (5)
C24—C25	1·378 (5)	C26—C27	1·380 (4)
C26—C31	1·392 (4)	C27—C28	1·383 (4)
C28—C29	1·369 (5)	C29—C30	1·376 (5)
C30—C31	1·384 (4)	C32—C33	1·385 (4)
C32—C37	1·389 (5)	C33—C34	1·398 (5)
C34—C35	1·370 (6)	C35—C36	1·364 (5)
C36—C37	1·394 (4)		
O5—C1—C14	114·1 (2)	C2—C1—C14	130·3 (3)
C2—C1—O5	115·0 (3)	C1—C2—C20	121·7 (3)
C1—C2—N3	119·8 (3)	N3—C2—C20	117·7 (2)
C2—N3—C4	121·7 (2)	N3—C4—C32	117·9 (3)
N3—C4—C26	125·8 (3)	C26—C4—C32	116·3 (2)
C1—O5—C7	117·4 (2)	O5—C7—O6	123·9 (3)
O6—C7—C8	125·4 (3)	O5—C7—C8	110·8 (3)
C7—C8—C13	117·9 (3)	C7—C8—C9	122·0 (3)
C9—C8—C13	120·1 (3)	C8—C9—C10	119·6 (3)
C9—C10—C11	119·7 (4)	C10—C11—C12	121·4 (4)
C11—C12—C13	119·8 (4)	C8—C13—C12	119·5 (4)
C1—C14—C19	119·6 (3)	C1—C14—C15	121·7 (3)
C15—C14—C19	118·7 (3)	C14—C15—C16	119·9 (3)
C15—C16—C17	120·6 (4)	C16—C17—C18	120·3 (4)
C17—C18—C19	119·7 (4)	C14—C19—C18	120·8 (3)
C2—C20—C25	119·2 (3)	C2—C20—C21	122·6 (3)
C21—C20—C25	118·2 (3)	C20—C21—C22	120·5 (3)
C21—C22—C23	120·3 (3)	C22—C23—C24	120·1 (4)
C23—C24—C25	120·0 (3)	C20—C25—C24	120·9 (3)
C4—C26—C31	120·3 (3)	C4—C26—C27	120·4 (3)
C27—C26—C31	119·3 (3)	C26—C27—C28	120·1 (3)
C27—C28—C29	120·6 (3)	C28—C29—C30	119·9 (3)
C29—C30—C31	120·2 (3)	C26—C31—C30	120·0 (3)
C4—C32—C37	120·4 (3)	C4—C32—C33	120·3 (3)
C33—C32—C37	119·3 (3)	C32—C33—C34	119·0 (3)
C33—C34—C35	121·2 (3)	C34—C35—C36	120·0 (3)
C35—C36—C37	119·9 (3)	C32—C37—C36	120·6 (3)
C2—C1—O5—C7	−110·1 (3)	O5—C1—C2—N3	−167·5 (2)
C1—C2—N3—C4	−140·6 (3)	C1—O5—C7—O6	−3·5 (4)
C1—O5—C7—C8	175·5 (2)	C14—C1—C2—C20	172·1 (3)
C14—C1—C2—N3	3·1 (5)	C14—C1—O5—C7	77·8 (3)
O5—C1—C2—C20	1·5 (4)	C20—C2—N3—C4	50·0 (4)
C2—N3—C4—C26	9·6 (4)	C2—N3—C4—C32	−170·7 (2)

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43777 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

compounds (Gunning, Neidle, Millican, Eaton, Mock & Mann, 1985; Bernardinelli & Giersch, 1985; Spek, Duisenberg, van Stein & van Koten, 1985; Armesto, Gallego, Horspool, Ortiz & Perez-Ossorio, 1987).

References

Armesto, D., Gallego, M. G., Horspool, W. M., Ortiz, M. J. & Perez-Ossorio, R. (1987). *J. Chem. Soc. Perkin Trans.* 1. In the press.

Bernardelli, G. & Giersch, W. (1985). *Acta Cryst.* C**41**, 746–749.

Gunning, J., Neidle, S., Millican, T. A., Eaton, M. A. W., Mock, G. A. & Mann, J. (1985). *Acta Cryst.* C**41**, 720–722.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Spek, A. L., Duisenberg, A. J. M., van Stein, G. C. & van Koten, G. (1985). *Acta Cryst.* C**41**, 374–377.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1987). C**43**, 1633–1634

Reinvestigation of the Structure of Ammonium Hydrogen D-Tartrate

By A. L. Spek

Vakgroep Algemene Chemie, Afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 *CH Utrecht, The Netherlands*

(*Received* 26 *January* 1987; *accepted* 25 *March* 1987)

Abstract. $NH_4^+.C_4H_5O_6^-$, $M_r = 167\cdot1$, orthorhombic, $P2_12_12_1$, $a = 7\cdot653\ (1)$, $b = 11\cdot072\ (1)$, $c = 7\cdot849\ (1)$ Å, $V = 665\cdot1\ (1)$ Å^3, $Z = 4$, $D_x = 1\cdot669$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot7$ cm^{-1}, $F(000) = 352$, $T = 295$ K, $R = 0\cdot029$ for 1043 observed reflections with $I > 2\cdot5\sigma(I)$. A more accurate and detailed structure of ammonium hydrogen D-tartrate is reported that includes the positions of the H atoms of the NH_4^+ cation that were not revealed in the previous investigation. All H atoms of NH_4^+ are involved in hydrogen bonding to O atoms of the distorted square-antiprism coordination polyhedron. The crystal structure consists of layers of hydrogen-bonded tartrate anions perpendicular to [010], separated by layers of NH_4^+ cations that are hydrogen-bonded to both sides resulting in a three-dimensional network.

Experimental. Diffraction data were collected on an Enraf–Nonius CAD-4*F* diffractometer using Zr-filtered Mo $K\alpha$ radiation for a spherical crystal (radius 0·2 mm; colourless; mounted on top of a glass fibre) that is in use as a CAD-4 test crystal. A redundant set of 7770 reflections (h −10:10; k −14:14; l −10:10; $\theta < 27\cdot5°$) was scanned in the ω-scan mode [$\Delta\omega = (0\cdot40 + 0\cdot35\tan\theta)°$; horizontal and vertical aperture 3 × 2 mm; crystal-to-detector distance 173 mm; max. time per reflection 60 s]. Eight reference reflections (±2, ±2, ±4) remained constant during the 103 h of exposure time. The observed values of $\sigma(I)/I$ were only slightly larger than the preset value 0·006 (maximum value 0·008). Cell dimensions were calculated from the setting angles of 18 reflections ($11 < \theta < 16°$) using an appropriate average of the setting angles for four alternative reflection settings. The reflection data were corrected for Lp effects and averaged into a unique set of 1091 reflections ($R_{\text{int}} = 0\cdot026$). Full-matrix least-squares refinement on F with a locally modified implementation of *SHELX76* (Sheldrick, 1976) on an Eclipse S/230 mini-computer was started from the published coordinates for the non-H atoms. All H atoms were located in a difference Fourier map including those on the N atom that could not be identified in the previous study and their positions refined. Convergence was reached at $R = 0\cdot029$ [$wR = 0\cdot032$; $S = 0\cdot32$; $(\Delta/\sigma)_{\text{av}} = 0\cdot05$, $(\Delta/\sigma)_{\text{max}} = 0\cdot04$] for 1043 reflections with $I > 2\cdot5\sigma(I)$, 130 parameters and weights based on counting statistics. An empirical correction for extinction was applied using the formula $F(\text{corr.}) = F(1 - 0\cdot0001g|F|^2/\sin\theta)$ with $g = 0\cdot0485\ (7)$. Maximum and minimum residual density in the final electron density difference map were 0·23 and −0·16 e Å^{-3}. All atoms were refined with anisotropic temperature factors except for the H atoms that were refined with one common isotropic temperature factor for those attached to O and one for those on N.

0108-2701/87/081633-02$01.50