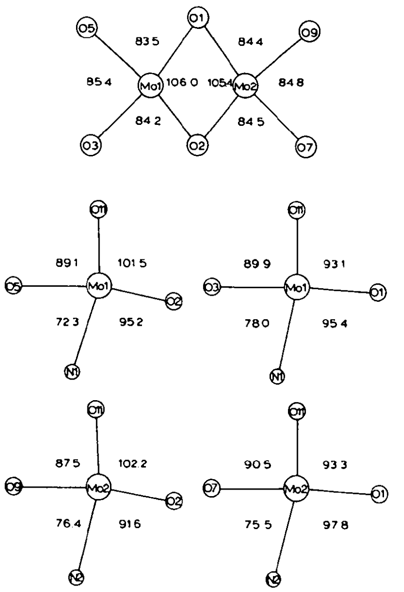

Fig. 2. Schematic views showing the coordination angles around the molybdenum atoms.

atoms are shown in Fig. 2. These vary from 90° by amounts between −18 and +12°; the largest distortions are shown by the angles which include the N atoms, despite the twist of the ethylene bridge. The Mo–Mo bond, 2·412 Å, is a strong metal–metal bond. The single bridge O has Mo–O = 1·899 and 1·900 Å with Mo–O–Mo = 163·6°, which are similar to the values found by Seleborg (1967) for $Na_2Mo_2O_7$ and Le Carpentier, Mitschler & Weiss (1972) for $(pyH^+)_2\{[MoO(O_2)_2H_2O]_2O\}$. The Mo–O distances for the double bridge O atoms are 1·93 and 2·08 Å; the latter, O(1), has a H atom attached.

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*Acta Cryst.* (1976). B32, 2886

# 6,10-Diaminododecafluorobicyclo[4,4,0]dec-1(10)-en-2-one

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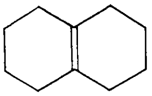
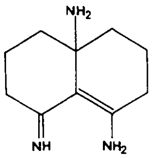
**Abstract.** $C_{10}H_4F_{12}N_2O$, monoclinic, $P2_1/c$; $a = 11{\cdot}705$ (10), $b = 9{\cdot}73$ (1), $c = 11{\cdot}67$ (1) Å, $\beta = 105{\cdot}91$ (5)°, $U = 1278$ Å$^3$; $Z = 4$, $D_c = 2{\cdot}058$ g cm$^{-3}$; $\mu$(Mo $K\alpha$) = 2·4 cm$^{-1}$. $R$ is 5·69% for 566 observed amplitudes. The measured bond lengths indicate that there is extensive conjugation between the 2-keto and 10-amino groups.

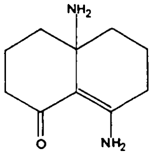
**Introduction.** Hexadecafluorobicyclo[4,4,0]dec-1(6)-ene (I) reacts readily with dry ammonia in ether to give the diaminoimine (II) which on hydrolysis with dilute acid yields the title compound (III) (Oliver, Stephens, Tatlow & Taylor, 1976). The X-ray analysis was undertaken to confirm the structural assignment and establish the geometry of the molecule.



(I)



(II)



(III)

(All unmarked substituents are fluorine)

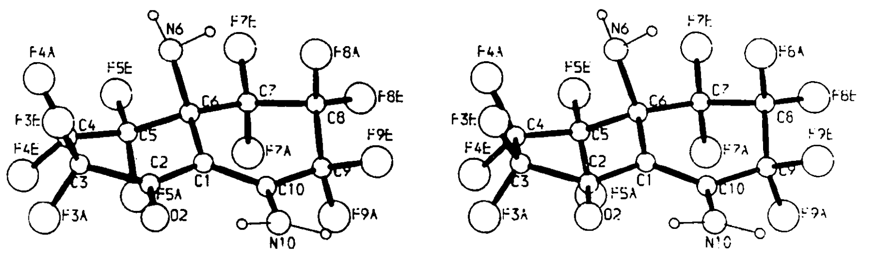


Fig. 1. Stereoscopic view of the molecule along the $z$ axis.

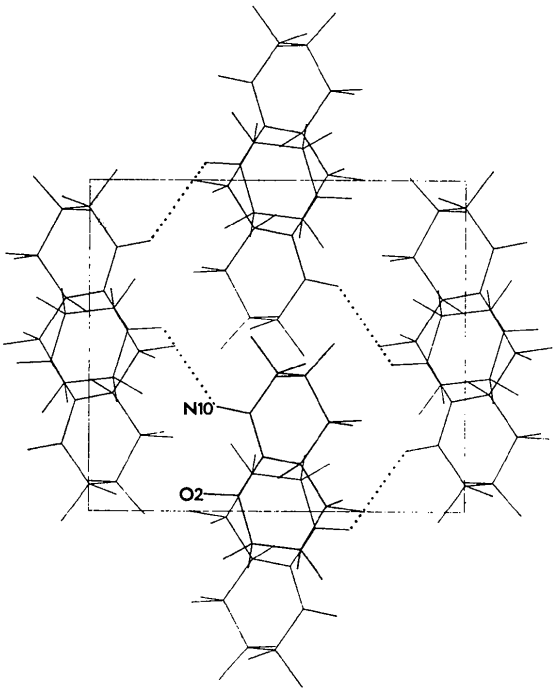


Fig. 2. Projection of the unit cell along the $x$ axis. The $y$ axis points up the page, the $z$ axis to the right. Intermolecular hydrogen bonds are indicated by dotted lines.

Crystals were grown from petroleum spirit – benzene in the form of thin plates of poor quality and unstable to the atmosphere. A crystal $0{\cdot}7\times0{\cdot}5\times0{\cdot}1$ mm was mounted about **c** and coated with varnish. Photographs showed absences $h0l$ with $l$ odd and $0k0$ with $k$ odd indicating the space group $P2_1/c$. The intensities of the reflexions fell off rapidly with increasing angle, due, as became evident later, to the high thermal motion of the atoms.

Intensities were measured with a Stoe computer-controlled two-circle diffractometer and Mo $K\alpha$ radiation, $\lambda=0{\cdot}71069$ Å, by the $\omega$ scan technique. 1607 reflexions were scanned in the range $0{\cdot}10<\sin\theta/\lambda<0{\cdot}54$, of which 566, with $I>2{\cdot}5\sigma(I)$, were considered to be observed. The intensities of the separate layers were placed on a common scale by the use of layer scale factors. No absorption corrections were applied.

The structure was solved by direct methods with *SHELX* (Sheldrick, 1975). With all the atoms attached to the decalin ring system treated as F atoms, $R$ was reduced from 36·7 to 16·2% by full-matrix least-squares refinement with isotropic thermal parameters. Consideration of bond lengths and temperature factors now indicated the O and N atom positions. After further refinement to $R=6{\cdot}4\%$ allowing the atoms to vibrate anisotropically, a difference synthesis indicated the position of the four H atoms consistent with these being bonded to the N atoms. Least-squares refinement was continued with the inclusion of the H

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and anisotropic thermal parameters* (Å$^2\times10^4$)

The anisotropic temperature factor is of the form $T=\exp\,[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*+\ldots)]$.

| | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| C(1) | 6900 (11) | 1663 (14) | 4625 (10) | 108 (82) | 155 (88) | 274 (65) | −38 (75) | −4 (57) | −32 (73) |
| C(2) | 6362 (13) | 494 (14) | 3976 (11) | 349 (104) | 198 (102) | 192 (68) | −8 (88) | −38 (69) | −47 (72) |
| C(3) | 6807 (16) | −965 (19) | 4357 (14) | 531 (141) | 397 (137) | 466 (91) | −143 (108) | −235 (88) | −38 (91) |
| C(4) | 7633 (17) | −1145 (17) | 5651 (14) | 488 (127) | 276 (120) | 657 (111) | 36 (102) | 214 (92) | −76 (90) |
| C(5) | 7900 (14) | 172 (16) | 6328 (12) | 390 (110) | 442 (122) | 353 (79) | −27 (94) | 77 (78) | −19 (85) |
| C(6) | 8037 (12) | 1440 (16) | 5626 (10) | 182 (96) | 333 (117) | 286 (77) | 85 (74) | 21 (68) | −10 (77) |
| C(7) | 8426 (14) | 2706 (17) | 6386 (15) | 320 (118) | 541 (140) | 632 (106) | 157 (107) | −31 (87) | −96 (105) |
| C(8) | 8223 (17) | 4109 (18) | 5734 (15) | 479 (146) | 347 (134) | 662 (111) | −92 (101) | 246 (108) | −142 (102) |
| C(9) | 6915 (15) | 4187 (16) | 5015 (16) | 429 (134) | 251 (110) | 687 (107) | 204 (91) | 180 (98) | −129 (94) |
| C(10) | 6396 (12) | 2973 (16) | 4276 (12) | 219 (91) | 268 (111) | 374 (85) | 35 (83) | 120 (73) | 53 (71) |
| F(3*a*) | 5863 (12) | −1723 (11) | 4260 (11) | 1105 (100) | 529 (79) | 1989 (140) | −496 (84) | −743 (101) | 578 (82) |
| F(3*e*) | 7325 (15) | −1460 (13) | 3583 (9) | 2593 (188) | 1214 (110) | 519 (67) | 1178 (120) | 104 (81) | −147 (66) |
| F(4*a*) | 8630 (9) | −1742 (9) | 5569 (8) | 655 (76) | 407 (66) | 1035 (79) | 244 (65) | −48 (54) | −113 (53) |
| F(4*e*) | 7130 (9) | −2052 (10) | 6226 (8) | 1080 (90) | 740 (82) | 842 (73) | −241 (73) | 319 (60) | 177 (58) |
| F(5*a*) | 6912 (8) | 374 (9) | 6756 (7) | 627 (70) | 897 (80) | 613 (55) | 125 (63) | 245 (48) | 120 (53) |
| F(5*e*) | 8822 (8) | −2 (9) | 7295 (6) | 751 (68) | 753 (81) | 412 (49) | 154 (61) | −214 (47) | 47 (48) |
| F(7*a*) | 7787 (8) | 2765 (9) | 7239 (7) | 1039 (79) | 760 (74) | 500 (53) | 162 (62) | 171 (50) | −161 (49) |
| F(7*e*) | 9560 (9) | 2598 (9) | 7026 (9) | 514 (65) | 641 (80) | 1170 (73) | 40 (58) | −567 (58) | −259 (56) |
| F(8*a*) | 8891 (9) | 4079 (10) | 4983 (9) | 491 (73) | 713 (80) | 1231 (90) | −155 (57) | 177 (63) | 120 (62) |
| F(8*e*) | 8532 (9) | 5096 (9) | 6507 (9) | 1131 (106) | 416 (68) | 1002 (77) | −25 (66) | −255 (66) | −296 (63) |
| F(9*a*) | 6278 (9) | 4491 (9) | 5746 (7) | 755 (73) | 825 (74) | 632 (57) | 365 (62) | 115 (49) | −271 (49) |
| F(9*e*) | 6827 (9) | 5303 (9) | 4291 (8) | 1015 (93) | 189 (65) | 831 (65) | −1 (56) | −173 (58) | −30 (52) |
| O(2) | 5536 (9) | 546 (10) | 3055 (8) | 605 (73) | 361 (73) | 400 (50) | −143 (61) | −95 (50) | −78 (47) |
| N(6) | 8983 (11) | 1019 (13) | 5044 (10) | 267 (85) | 697 (105) | 806 (91) | 101 (74) | 255 (71) | 123 (74) |
| N(10) | 5535 (10) | 3176 (10) | 3386 (9) | 474 (82) | 138 (74) | 230 (53) | 131 (63) | −169 (59) | 75 (48) |
| H1(N6) | 9760 | 688 | 5651 | 621 | | | | | |
| H2(N6) | 9170 | 1814 | 4533 | 621 | | | | | |
| H1(N10) | 5250 | 4152 | 3077 | 473 | | | | | |
| H2(N10) | 5342 | 2365 | 2786 | 473 | | | | | |

atoms with N–H constrained to 1·04 Å. Finally, the H atom parameters were fixed and the heavier atoms refined until all calculated shifts were $<0{\cdot}1\sigma$ and $R$ was 5·69% for the 566 observed amplitudes.*

Table 2. *Molecular dimensions*

(*a*) Bond distances (Å)

| | | | |
|---|---|---|---|
| C(1)—C(2) | 1·41$_5$ (2) | C(2)–O(2) | 1·23 (1) |
| C(2)—C(3) | 1·53$_5$ (2) | C(3)–F(3*a*) | 1·31 (2) |
| C(3)—C(4) | 1·56 (2) | C(3)–F(3*e*) | 1·31 (2) |
| C(4)—C(5) | 1·49 (2) | C(4)–F(4*a*) | 1·33 (2) |
| C(5)—C(6) | 1·51 (2) | C(4)–F(4*e*) | 1·34 (2) |
| C(6)—C(1) | 1·53 (2) | C(5)–F(5*a*) | 1·39 (2) |
| C(6)—C(7) | 1·51 (2) | C(5)–F(5*e*) | 1·34 (2) |
| C(7)—C(8) | 1·55 (2) | C(7)–F(7*a*) | 1·40 (2) |
| C(8)—C(9) | 1·53 (2) | C(7)–F(7*e*) | 1·34 (2) |
| C(9)—C(10) | 1·49 (2) | C(8)–F(8*a*) | 1·32$_5$ (2) |
| C(10)–C(1) | 1·42 (2) | C(8)–F(8*e*) | 1·30 (2) |
| C(6)—N(6) | 1·51 (2) | C(9)–F(9*a*) | 1·31 (2) |
| C(10)–N(10) | 1·25 (2) | C(9)–F(9*e*) | 1·36 (2) |

(*b*) Bond angles (°)

| | | | |
|---|---|---|---|
| C(2)—C(1)-C(6) | 117·5 (13) | C(5)—C(6)—C(7) | 114·0 (11) |
| C(2)—C(1)-C(10) | 118·7 (11) | C(1)—C(6)—N(6) | 106·8 (10) |
| C(6)—C(1)-C(10) | 123·7 (12) | C(5)—C(6)—N(6) | 102·9 (11) |
| C(1)—C(2)-C(3) | 121·5 (11) | C(7)—C(6)—N(6) | 110·2 (12) |
| C(1)—C(2)-O(2) | 124·1 (13) | C(6)—C(7)—C(8) | 116·7 (14) |
| C(3)—C(2)-O(2) | 114·4 (11) | F(7*a*)-C(7)—C(6) | 108·4 (12) |
| C(2)—C(3)-C(4) | 117·0 (13) | F(7*a*)-C(7)—C(8) | 105·9 (13) |
| F(3*a*)-C(3)-C(2) | 106·5 (14) | F(7*e*)-C(7)—C(6) | 110·9 (13) |
| F(3*a*)-C(3)-C(4) | 107·7 (14) | F(7*e*)-C(7)—C(8) | 109·8 (13) |
| F(3*e*)-C(3)-C(2) | 108·9 (13) | F(7*a*)-C(7)—F(7*e*) | 104·4 (13) |
| F(3*e*)-C(3)-C(4) | 110·1 (15) | C(7)—C(8)—C(9) | 107·9 (14) |
| F(3*a*)-C(3)-F(3*e*) | 105·8 (16) | F(8*a*)-C(8)—C(7) | 105·5 (14) |
| C(3)—C(4)-C(5) | 113·5 (14) | F(8*a*)-C(8)—C(9) | 108·7 (14) |
| F(4*a*)-C(4)-C(3) | 107·5 (13) | F(8*e*)-C(8)—C(7) | 109·6 (13) |
| F(4*a*)-C(4)-C(5) | 110·7 (15) | F(8*e*)-C(8)—C(9) | 112·9 (14) |
| F(4*e*)-C(4)-C(3) | 108·5 (14) | F(8*a*)-C(8)—F(8*e*) | 111·9 (16) |
| F(4*e*)-C(4)-C(5) | 110·0 (13) | C(8)—C(9)—C(10) | 117·7 (14) |
| F(4*a*)-C(4)-F(4*e*) | 105·3 (14) | F(9*a*)-C(9)—C(8) | 108·4 (14) |
| C(4)—C(5)-C(6) | 116·8 (12) | F(9*a*)-C(9)—C(10) | 109·9 (14) |
| F(5*a*)-C(5)-C(4) | 103·7 (13) | F(9*e*)-C(9)—C(8) | 105·9 (14) |
| F(5*a*)-C(5)-C(6) | 107·5 (12) | F(9*e*)-C(9)—C(10) | 108·9 (13) |
| F(5*e*)-C(5)-C(4) | 109·9 (13) | F(9*a*)-C(9)—F(9*e*) | 105·3 (12) |
| F(5*e*)-C(5)-C(6) | 112·1 (12) | C(1)—C(10)-C(9) | 118·4 (13) |
| F(5*a*)-C(5)-F(5*e*) | 105·7 (12) | C(1)—C(10)-N(10) | 124·0 (14) |
| C(1)—C(6)-C(5) | 109·2 (12) | C(9)—C(10)-N(10) | 117·6 (15) |
| C(1)—C(6)-C(7) | 113·0 (13) | | |

**Discussion.** The conformation of the molecule is shown in Fig. 1. Atomic coordinates and thermal parameters are listed in Table 1, and bond lengths, bond angles and selected torsion angles in Table 2.

C(2)–O(2) and C(10)–N(10) are close to the double-bond value (Sutton, 1965; Lofthus, 1959) while C(1)–C(2) and C(1)–C(10) are nearly equal and slightly (but not significantly) longer than expected for C–C bonds possessing *ca* 50% double-bond character. The shortness of C(2)–O(2) and C(10)–N(10) is difficult to rationalize but may be at least partially due to the effect of the highly electronegative F substituents at the adjacent C atoms (Gali, Miravitlles & Font-Altaba, 1975; Meresse, Courseille & Chanh, 1974; Yates, Ardao & Fieser, 1956). However, owing to the limited number of intensities available, the measured bond lengths are of relatively low reliability. The remaining C–C bonds range from 1·49 to 1·56, mean 1·52 Å, and C–F bonds range from 1·30 to 1·40, mean 1·34 Å, in reasonable agreement with expected values.

The six-atom grouping C(1), C(2), C(3), C(6), C(9), C(10) is approximately planar (maximum deviation 0·08 Å). C(4) and C(5) are displaced on the same side of this plane by 0·61 and 0·89 Å, while C(7) and C(8) lie on opposite sides at distances of 0·21 and 0·38 Å. Ring C(1), C(6)–C(10) thus adopts a distorted half-chair conformation. O(2) and N(10) are situated on the same side of the six-atom plane at distances of 0·20 and 0·16 Å. The five-atom grouping C(1), C(2), C(10), O(2), N(10) is planar to within 0·03 Å, consistent with extensive electron delocalization.

The F substituents generally occupy positions approximating to normal equatorial and axial, and have been labelled *e* and *a* accordingly. However, those at C(3) are oriented so that C(3)–F(3*e*) virtually eclipses C(4)–F(4*a*) and C(3)–F(3*a*) eclipses C(4)–F(4*e*). It is here that the shortest F···F contacts occur with F(3*a*)···F(4*e*) = 2·39 and F(3*e*)···F(4*a*) = 2·42 Å.

The distances O(2)···H2(N10) and O(2)···N(10) are 1·80 and 2·59 Å respectively, indicative of intramolecular hydrogen bonding. The angle H2(N10)–N(10)···O(2) is 32·4°.

The arrangement of molecules in the unit cell is shown in Fig. 2. Molecules related by the $2_1$ screw axis form zigzag chains parallel to $y$ linked by hydrogen bonds involving H1(N10) and O(2)$^{\rm i}$ where i denotes the equivalent position $1-x,\frac{1}{2}+y,\frac{1}{2}-z$. Relevant distances are H1(N10)···O(2)$^{\rm i}$, 1·94 and N(10)···O(2)$^{\rm i}$, 2·93 Å and the angle H1(N10)–N(10)···O(2)$^{\rm i}$ is 14·7°.

Table 2 (*cont.*)

(*c*) Selected torsion angles (°)

| | | | |
|---|---|---|---|
| C(1)–C(2)–C(3)—C(4) | 15·9 | C(9)—C(10)–C(1)—C(6) | −7·7 |
| C(2)–C(3)–C(4)—C(5) | −1·7 | C(10)–C(1)—C(6)—C(7) | 12·8 |
| C(3)–C(4)–C(5)—C(6) | −35·7 | C(1)—C(6)—C(7)—C(8) | −36·6 |
| C(4)–C(5)–C(6)—C(1) | 58·3 | C(2)—C(1)—C(6)—C(7) | −170·7 |
| C(5)–C(6)–C(1)—C(2) | −42·7 | C(2)—C(1)—C(10)–C(9) | 175·8 |
| C(6)–C(1)–C(2)—C(3) | 7·6 | C(3)—C(2)—C(1)—C(10) | −175·7 |
| C(6)–C(7)–C(8)—C(9) | 52·9 | C(4)—C(5)—C(6)—C(7) | −174·2 |
| C(7)–C(8)–C(9)—C(10) | −47·0 | C(5)—C(6)—C(7)—C(8) | −162·1 |
| C(8)–C(9)–C(10)–C(1) | 26·4 | C(5)—C(6)—C(1)—C(10) | 140·8 |

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31823 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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# Benzenephosphonic Acid

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**Abstract.** $C_6H_7O_3P$, *Pbca*; $a=11\cdot182$ (7), $b=8\cdot051$ (5), $c=15\cdot768$ (11) Å; $Z=8$, $D_m=1\cdot47$ (2), $D_x=1\cdot48$ g cm$^{-3}$; $\mu(\text{Cu } K\alpha)=28\cdot7$ cm$^{-1}$. Puckered layers at $z=0$ and $z=\frac{1}{2}$ result from intermolecular hydrogen bonding. Only weak contacts occur between layers.

**Introduction.** Crystals were from a commercial sample (plates, prominent {001} faces). Equi-inclination multi-film Weissenberg photographs were recorded (Cu radiation, $\lambda=1\cdot5418$ Å) for the levels *h*0–6*l*, 0–5*kl*, and *hk*0–3; all crystals measured about 0·3 × 0·3 × 0·05 mm. Intensities were measured by a scanning microdensitometer (S.R.C. Service, Atlas Computer Laboratory). Absorption corrections were applied. Following a symbolic addition procedure, an *E* map showed the P and the adjacent C and three O atoms. A difference map phased by these gave the positions of the remaining C atoms. Refinement was by full-matrix least squares.

A second difference map, when *R* had fallen to 0·062, showed all H atoms near their expected positions; these were refined in subsequent cycles. The final unweighted *R* was 0·048 (anisotropic P, O, and C; isotropic H; 119 parameters, 768 independent reflexions

Table 1. *Atomic parameters*

Parameters for non-hydrogen atoms are multiplied by $10^4$; those for hydrogen atoms, by $10^3$. The thermal function is $T=\exp\,[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{23}klb^*c^*)]$.

| | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| P | 1583 (1) | 484 (1) | 699 (1) | 332 (6) | 278 (7) | 494 (6) | 4 (5) | −6 (5) | 9 (5) |
| O(1) | 1254 (3) | −1154 (5) | 317 (2) | 420 (18) | 319 (20) | 465 (20) | 4 (16) | −120 (16) | −95 (17) |
| O(2) | 811 (3) | 1923 (5) | 343 (2) | 484 (17) | 308 (19) | 589 (20) | 34 (18) | −129 (15) | 52 (16) |
| O(3) | 2896 (3) | 927 (5) | 516 (2) | 381 (18) | 367 (20) | 764 (25) | −4 (15) | 113 (16) | 60 (20) |
| C(1) | 1406 (4) | 437 (5) | 1816 (3) | 380 (20) | 304 (27) | 538 (24) | 74 (19) | −4 (18) | 31 (24) |
| C(2) | 407 (5) | −358 (8) | 2165 (4) | 466 (28) | 562 (40) | 620 (31) | −98 (27) | 67 (24) | 38 (29) |
| C(3) | 223 (6) | −374 (9) | 3036 (4) | 753 (38) | 646 (49) | 702 (37) | 8 (36) | 226 (37) | 117 (37) |
| C(4) | 1039 (8) | 365 (9) | 3566 (4) | 1056 (57) | 685 (49) | 486 (29) | 249 (43) | 2 (35) | 129 (34) |
| C(5) | 2016 (7) | 1190 (9) | 3233 (5) | 876 (49) | 678 (45) | 650 (35) | 48 (44) | −231 (35) | −21 (40) |
| C(6) | 2210 (5) | 1213 (8) | 2357 (4) | 541 (29) | 537 (40) | 588 (34) | −55 (28) | −146 (25) | 6 (29) |

| | $x$ | $y$ | $z$ | $U$ | | $x$ | $y$ | $z$ | $U$ |
|---|---|---|---|---|---|---|---|---|---|
| H(1) | 316 (6) | 215 (10) | 48 (5) | 80 (21) | H(5) | 89 (9) | 23 (11) | 423 (7) | 129 (31) |
| H(2) | −2 (7) | 168 (11) | 16 (5) | 118 (31) | H(6) | 286 (7) | 167 (9) | 351 (5) | 108 (25) |
| H(3) | −20 (5) | −103 (8) | 181 (4) | 64 (16) | H(7) | 304 (5) | 175 (7) | 209 (3) | 53 (15) |
| H(4) | −58 (5) | −111 (9) | 331 (4) | 77 (19) | | | | | |