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3,8-Bis(4-chlorophenyl)-4,7-dimethyltricyclo[4.2.2.0^{2,5}]deca-3,7-diene

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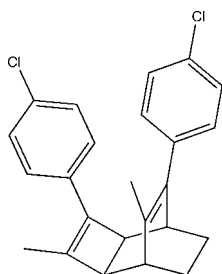
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.160; data-to-parameter ratio = 17.2.

The title tricyclic diene, $\text{C}_{24}\text{H}_{22}\text{Cl}_2$, is the product of thermal ring-opening of a corresponding basketane (pentacyclo[4.4.0.0^{2,5}.0^{3,8}.0^{4,7}]decane) derivative. The cyclobutene ring is planar to within 0.0032 (12) Å and its geometry is normal. The two 4-chlorophenyl groups are oriented in an approximately face-to-face conformation with a dihedral angle of 44.14 (6)° between them. The 4-chlorophenyl group bonded to the cyclobutene ring lies almost in the plane of the cyclobutene ring, with a dihedral angle of 8.29 (17)° between the ring planes. The average intramolecular C...C distance between the two C=C bonds is 2.92 Å. In the crystal structure, the molecules are well separated with no close C—H...Cl or C—H... π intermolecular interactions.

Related literature

For the preparation of the title compound, see: Tezuka *et al.* (1976); Mukai *et al.* (1981). For cage compounds, see: Osawa & Yonemitsu (1992). For the crystal structures of compounds with a tricyclo[4.2.2.0^{2,5}]deca-3,7-diene skeleton, see: Lemley *et al.* (1976); Hanson (1981); Mehta *et al.* (1990, 2003). For related literature, see: Allen (1984, 2002); Allen *et al.* (1987).



Experimental

Crystal data

| | |
|---|-----------------------------------|
| $\text{C}_{24}\text{H}_{22}\text{Cl}_2$ | $V = 1995.5$ (3) Å ³ |
| $M_r = 381.32$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Cu $K\alpha$ radiation |
| $a = 8.3389$ (7) Å | $\mu = 2.94$ mm ⁻¹ |
| $b = 21.2224$ (12) Å | $T = 295$ (1) K |
| $c = 11.6074$ (13) Å | $0.48 \times 0.35 \times 0.25$ mm |
| $\beta = 103.732$ (7)° | |

Data collection

| | |
|---|--|
| Enraf–Nonius CAD-4 diffractometer | 4070 independent reflections |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | 2872 reflections with $I > 2\sigma(I)$ |
| $T_{\min} = 0.328$, $T_{\max} = 0.484$ | $R_{\text{int}} = 0.021$ |
| 4348 measured reflections | 3 standard reflections |
| | frequency: 120 min |
| | intensity decay: 1.3% |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.050$ | 237 parameters |
| $wR(F^2) = 0.160$ | H-atom parameters constrained |
| $S = 1.04$ | $\Delta\rho_{\text{max}} = 0.20$ e Å ⁻³ |
| 4070 reflections | $\Delta\rho_{\text{min}} = -0.33$ e Å ⁻³ |

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *TEXSAN* (Rigaku/MS, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

We thank the Instrument Center of the Institute for Molecular Science for the X-ray structure analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2459).

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supplementary materials

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3,8-Bis(4-chlorophenyl)-4,7-dimethyltricyclo[4.2.2.0^{2,5}]deca-3,7-diene

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Comment

There is considerable interest in the chemistry of highly strained polycyclic "cage" compounds (Osawa & Yonemitsu, 1992). The title tricyclic diene, (I), Fig 1, 3,8-bis(4-chlorophenyl)-4,7-dimethyltricyclo[4.2.2.0^{2,5}]deca-3,7-diene, C₂₄H₂₂Cl₂, is the product of thermal ring-opening of a corresponding basketane (pentacyclo[4.4.0.0^{2,5}.0^{3,8}.0^{4,7}]decane) and reverts to the basketane derivative quantitatively upon irradiation (Tezuka *et al.*, 1976). A search for tricyclo[4.2.2.0^{2,5}]deca-3,7-diene skeleton in the Cambridge Structural Database (Version 5.29; Allen, 2002) gave only four examples, CTCYDD (Lemley *et al.*, 1976), CNUNDC (Hanson, 1981), KEVGEX (Mehta *et al.*, 1990) and GACFIA (Mehta *et al.*, 2003).

Bond lengths and angles in the molecule are within the normal ranges (Allen *et al.*, 1987) and the geometry of the cyclobutene ring is also similar to that of cyclobutene (Allen, 1984). The cyclobutene ring adopts a planar, rather than a puckered conformation, where the maximum deviation of the fitted atoms from the least-squares plane is 0.0032 (12) Å. The C3—C4 bond distance [1.562 (3) Å] in the cyclobutene ring is 1.8% shorter than the corresponding distance in the 2,3,4,5-tetrachloro derivative (Lemley *et al.*, 1976). The two 4-chlorophenyl groups are oriented in an approximately face-to-face conformation with a dihedral angle of 44.14 (6)° between them. The 4-chlorophenyl group bonded to the cyclobutene ring lies almost in the plane of the cyclobutene ring with a dihedral angle of 8.29 (17)° between the ring planes. Upon irradiation, an intramolecular photocyclization occurs between the C1=C2 and C6=C7 bonds. Intramolecular C1...C6 and C2...C7 distances are 2.918 (3) and 2.921 (3) Å, respectively, and the dihedral angle between the cyclobutene plane (C1—C4) and the C5—C8 plane is 60.89 (12)°. In the crystal structure the molecules are well separated with no close C—H...Cl or C—H...π intermolecular interactions.

Experimental

The compound (I) was synthesized according to a literature method (Tezuka *et al.*, 1976; Mukai *et al.*, 1981). Colorless crystals of (I) suitable for X-ray analysis were grown from a dichloromethane solution.

Refinement

All H atoms were placed in geometrically calculated positions and refined using a riding model, with C—H distances of 0.93, 0.96, 0.97 and 0.98 Å for aromatic, methyl, methylene and methine H atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic, methylene and methine or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

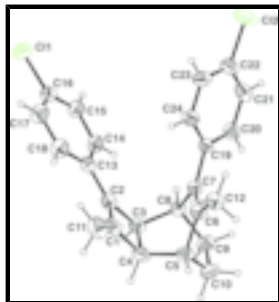


Fig. 1. The molecular structure of (I), with atom labels and 20% probability displacement ellipsoids for non-H atoms. H atoms are shown as small spheres with arbitrary radii.

3,8-Bis(4-chlorophenyl)-4,7-dimethyltricyclo[4.2.2.0^{2,5}]deca-3,7-diene

Crystal data

C₂₄H₂₂Cl₂

M_r = 381.32

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 8.3389 (7) Å

b = 21.2224 (12) Å

c = 11.6074 (13) Å

β = 103.732 (7)°

V = 1995.5 (3) Å³

Z = 4

*F*₀₀₀ = 800

D_x = 1.269 Mg m⁻³

Cu *K*α radiation

λ = 1.54178 Å

Cell parameters from 25 reflections

θ = 22.7–42.6°

μ = 2.94 mm⁻¹

T = 295 (1) K

Block, colorless

0.48 × 0.35 × 0.25 mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Monochromator: graphite

T = 295(1) K

ω–2θ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

*T*_{min} = 0.328, *T*_{max} = 0.484

4348 measured reflections

4070 independent reflections

2872 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.021

θ_{max} = 74.2°

θ_{min} = 4.2°

h = 0 → 10

k = 0 → 26

l = -14 → 14

3 standard reflections

every 120 min

intensity decay: 1.3%

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.050

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

| | |
|--|--|
| $wR(F^2) = 0.160$ | $w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.357P]$ |
| $S = 1.04$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 4070 reflections | $(\Delta/\sigma)_{\max} = 0.007$ |
| 237 parameters | $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$ |
| | Extinction correction: none |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|--------------|----------------------------------|
| C11 | 0.49013 (12) | 0.19930 (4) | 1.21790 (6) | 0.1028 (3) |
| C12 | 1.19192 (12) | 0.35095 (4) | 0.98762 (11) | 0.1347 (4) |
| C1 | 0.7177 (3) | 0.00447 (11) | 0.7920 (3) | 0.0761 (7) |
| C2 | 0.6407 (3) | 0.05850 (10) | 0.8097 (2) | 0.0661 (5) |
| C3 | 0.5989 (3) | 0.07597 (11) | 0.6796 (2) | 0.0652 (5) |
| H3 | 0.4802 | 0.0750 | 0.6432 | 0.078* |
| C4 | 0.6896 (3) | 0.01375 (11) | 0.6609 (2) | 0.0749 (7) |
| H4 | 0.6163 | -0.0179 | 0.6144 | 0.090* |
| C5 | 0.8386 (3) | 0.03011 (12) | 0.6096 (2) | 0.0753 (7) |
| H5 | 0.9014 | -0.0079 | 0.6011 | 0.090* |
| C6 | 0.9467 (3) | 0.07722 (11) | 0.6897 (2) | 0.0656 (5) |
| C7 | 0.8686 (2) | 0.13047 (10) | 0.70463 (19) | 0.0600 (5) |
| C8 | 0.6880 (2) | 0.13196 (11) | 0.63804 (19) | 0.0618 (5) |
| H8 | 0.6364 | 0.1719 | 0.6515 | 0.074* |
| C9 | 0.6785 (3) | 0.12218 (14) | 0.5049 (2) | 0.0793 (7) |
| H9A | 0.5639 | 0.1196 | 0.4614 | 0.095* |
| H9B | 0.7290 | 0.1576 | 0.4743 | 0.095* |
| C10 | 0.7692 (3) | 0.06101 (14) | 0.4880 (2) | 0.0858 (8) |
| H10A | 0.8585 | 0.0704 | 0.4503 | 0.103* |
| H10B | 0.6934 | 0.0323 | 0.4372 | 0.103* |
| C11 | 0.8109 (4) | -0.04637 (13) | 0.8691 (3) | 0.1037 (10) |
| H11A | 0.9228 | -0.0472 | 0.8609 | 0.156* |
| H11B | 0.7596 | -0.0863 | 0.8455 | 0.156* |
| H11C | 0.8102 | -0.0382 | 0.9503 | 0.156* |
| C12 | 1.1250 (3) | 0.06200 (12) | 0.7406 (3) | 0.0787 (7) |

supplementary materials

| | | | | |
|------|------------|--------------|--------------|------------|
| H12A | 1.1803 | 0.0985 | 0.7801 | 0.118* |
| H12B | 1.1756 | 0.0497 | 0.6779 | 0.118* |
| H12C | 1.1333 | 0.0281 | 0.7964 | 0.118* |
| C13 | 0.6085 (3) | 0.09224 (10) | 0.9117 (2) | 0.0617 (5) |
| C14 | 0.5426 (3) | 0.15260 (11) | 0.8976 (2) | 0.0721 (6) |
| H14 | 0.5194 | 0.1709 | 0.8227 | 0.086* |
| C15 | 0.5102 (3) | 0.18636 (12) | 0.9912 (2) | 0.0759 (6) |
| H15 | 0.4683 | 0.2271 | 0.9798 | 0.091* |
| C16 | 0.5404 (3) | 0.15904 (12) | 1.1008 (2) | 0.0710 (6) |
| C17 | 0.6047 (4) | 0.09927 (14) | 1.1188 (2) | 0.0875 (8) |
| H17 | 0.6248 | 0.0809 | 1.1936 | 0.105* |
| C18 | 0.6390 (4) | 0.06678 (12) | 1.0248 (2) | 0.0838 (7) |
| H18 | 0.6840 | 0.0266 | 1.0375 | 0.101* |
| C19 | 0.9420 (3) | 0.18508 (10) | 0.77687 (19) | 0.0574 (5) |
| C20 | 0.9320 (3) | 0.24474 (12) | 0.7278 (2) | 0.0772 (7) |
| H20 | 0.8739 | 0.2505 | 0.6496 | 0.093* |
| C21 | 1.0060 (4) | 0.29581 (13) | 0.7920 (3) | 0.0924 (9) |
| H21 | 0.9986 | 0.3355 | 0.7573 | 0.111* |
| C22 | 1.0901 (3) | 0.28762 (13) | 0.9069 (3) | 0.0810 (7) |
| C23 | 1.0954 (3) | 0.23022 (14) | 0.9607 (2) | 0.0826 (7) |
| H23 | 1.1486 | 0.2256 | 1.0403 | 0.099* |
| C24 | 1.0210 (3) | 0.17907 (12) | 0.8956 (2) | 0.0736 (6) |
| H24 | 1.0239 | 0.1400 | 0.9321 | 0.088* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.1386 (7) | 0.1011 (5) | 0.0714 (4) | -0.0031 (5) | 0.0303 (4) | -0.0133 (3) |
| C12 | 0.1042 (6) | 0.0980 (6) | 0.1974 (11) | -0.0306 (5) | 0.0271 (6) | -0.0685 (6) |
| C1 | 0.0663 (14) | 0.0549 (12) | 0.1071 (19) | 0.0000 (10) | 0.0209 (13) | -0.0022 (12) |
| C2 | 0.0562 (12) | 0.0574 (12) | 0.0851 (15) | 0.0019 (9) | 0.0176 (11) | 0.0013 (11) |
| C3 | 0.0487 (10) | 0.0662 (12) | 0.0783 (14) | 0.0005 (9) | 0.0103 (10) | -0.0110 (11) |
| C4 | 0.0571 (12) | 0.0666 (14) | 0.1002 (18) | -0.0060 (10) | 0.0171 (12) | -0.0246 (13) |
| C5 | 0.0591 (12) | 0.0737 (14) | 0.0931 (17) | 0.0031 (11) | 0.0181 (12) | -0.0285 (13) |
| C6 | 0.0520 (11) | 0.0691 (13) | 0.0746 (14) | 0.0025 (10) | 0.0127 (10) | -0.0114 (11) |
| C7 | 0.0524 (11) | 0.0636 (12) | 0.0625 (12) | 0.0005 (9) | 0.0110 (9) | -0.0040 (9) |
| C8 | 0.0530 (11) | 0.0680 (12) | 0.0626 (12) | 0.0069 (9) | 0.0104 (9) | -0.0029 (10) |
| C9 | 0.0639 (13) | 0.108 (2) | 0.0635 (13) | 0.0046 (13) | 0.0109 (11) | -0.0095 (13) |
| C10 | 0.0675 (14) | 0.113 (2) | 0.0764 (16) | -0.0002 (14) | 0.0156 (12) | -0.0309 (15) |
| C11 | 0.101 (2) | 0.0642 (15) | 0.150 (3) | 0.0211 (14) | 0.039 (2) | 0.0158 (17) |
| C12 | 0.0565 (12) | 0.0775 (15) | 0.0983 (18) | 0.0090 (11) | 0.0107 (12) | -0.0098 (13) |
| C13 | 0.0554 (11) | 0.0564 (11) | 0.0729 (13) | 0.0017 (9) | 0.0141 (10) | 0.0067 (10) |
| C14 | 0.0868 (16) | 0.0643 (13) | 0.0687 (13) | 0.0126 (11) | 0.0255 (12) | 0.0139 (11) |
| C15 | 0.0925 (17) | 0.0610 (13) | 0.0795 (15) | 0.0102 (12) | 0.0308 (13) | 0.0075 (11) |
| C16 | 0.0742 (14) | 0.0717 (14) | 0.0666 (13) | -0.0071 (11) | 0.0157 (11) | -0.0029 (11) |
| C17 | 0.105 (2) | 0.0858 (17) | 0.0691 (15) | 0.0102 (16) | 0.0156 (14) | 0.0184 (13) |
| C18 | 0.0937 (18) | 0.0691 (15) | 0.0869 (17) | 0.0185 (13) | 0.0180 (14) | 0.0162 (13) |
| C19 | 0.0526 (10) | 0.0575 (11) | 0.0636 (12) | 0.0002 (8) | 0.0163 (9) | -0.0031 (9) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C20 | 0.0820 (16) | 0.0708 (15) | 0.0762 (15) | -0.0063 (12) | 0.0137 (12) | 0.0103 (12) |
| C21 | 0.095 (2) | 0.0620 (14) | 0.121 (2) | -0.0147 (13) | 0.0270 (18) | 0.0067 (15) |
| C22 | 0.0630 (14) | 0.0708 (15) | 0.112 (2) | -0.0092 (11) | 0.0267 (14) | -0.0259 (15) |
| C23 | 0.0796 (17) | 0.0903 (18) | 0.0723 (15) | -0.0016 (13) | 0.0067 (12) | -0.0191 (14) |
| C24 | 0.0837 (16) | 0.0633 (13) | 0.0685 (14) | -0.0029 (11) | 0.0075 (12) | 0.0006 (11) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|---------------|-----------|
| C11—C16 | 1.739 (3) | C11—H11A | 0.9600 |
| C12—C22 | 1.740 (3) | C11—H11B | 0.9600 |
| C1—C2 | 1.353 (3) | C11—H11C | 0.9600 |
| C1—C11 | 1.496 (4) | C12—H12A | 0.9600 |
| C1—C4 | 1.496 (4) | C12—H12B | 0.9600 |
| C2—C13 | 1.462 (3) | C12—H12C | 0.9600 |
| C2—C3 | 1.513 (3) | C13—C18 | 1.386 (3) |
| C3—C8 | 1.538 (3) | C13—C14 | 1.388 (3) |
| C3—C4 | 1.562 (3) | C14—C15 | 1.381 (3) |
| C3—H3 | 0.9800 | C14—H14 | 0.9300 |
| C4—C5 | 1.540 (3) | C15—C16 | 1.366 (3) |
| C4—H4 | 0.9800 | C15—H15 | 0.9300 |
| C5—C6 | 1.510 (3) | C16—C17 | 1.374 (4) |
| C5—C10 | 1.539 (4) | C17—C18 | 1.377 (4) |
| C5—H5 | 0.9800 | C17—H17 | 0.9300 |
| C6—C7 | 1.336 (3) | C18—H18 | 0.9300 |
| C6—C12 | 1.499 (3) | C19—C20 | 1.383 (3) |
| C7—C19 | 1.476 (3) | C19—C24 | 1.385 (3) |
| C7—C8 | 1.521 (3) | C20—C21 | 1.376 (4) |
| C8—C9 | 1.543 (3) | C20—H20 | 0.9300 |
| C8—H8 | 0.9800 | C21—C22 | 1.363 (4) |
| C9—C10 | 1.538 (4) | C21—H21 | 0.9300 |
| C9—H9A | 0.9700 | C22—C23 | 1.365 (4) |
| C9—H9B | 0.9700 | C23—C24 | 1.383 (3) |
| C10—H10A | 0.9700 | C23—H23 | 0.9300 |
| C10—H10B | 0.9700 | C24—H24 | 0.9300 |
| C2—C1—C11 | 135.9 (3) | C1—C11—H11A | 109.5 |
| C2—C1—C4 | 94.5 (2) | C1—C11—H11B | 109.5 |
| C11—C1—C4 | 129.5 (2) | H11A—C11—H11B | 109.5 |
| C1—C2—C13 | 136.3 (2) | C1—C11—H11C | 109.5 |
| C1—C2—C3 | 93.5 (2) | H11A—C11—H11C | 109.5 |
| C13—C2—C3 | 130.17 (19) | H11B—C11—H11C | 109.5 |
| C2—C3—C8 | 119.23 (18) | C6—C12—H12A | 109.5 |
| C2—C3—C4 | 85.80 (18) | C6—C12—H12B | 109.5 |
| C8—C3—C4 | 108.89 (17) | H12A—C12—H12B | 109.5 |
| C2—C3—H3 | 113.3 | C6—C12—H12C | 109.5 |
| C8—C3—H3 | 113.3 | H12A—C12—H12C | 109.5 |
| C4—C3—H3 | 113.3 | H12B—C12—H12C | 109.5 |
| C1—C4—C5 | 118.5 (2) | C18—C13—C14 | 116.7 (2) |
| C1—C4—C3 | 86.23 (18) | C18—C13—C2 | 123.2 (2) |
| C5—C4—C3 | 109.0 (2) | C14—C13—C2 | 120.0 (2) |

supplementary materials

| | | | |
|---------------|-------------|-----------------|------------|
| C1—C4—H4 | 113.4 | C15—C14—C13 | 122.2 (2) |
| C5—C4—H4 | 113.4 | C15—C14—H14 | 118.9 |
| C3—C4—H4 | 113.4 | C13—C14—H14 | 118.9 |
| C6—C5—C10 | 108.8 (2) | C16—C15—C14 | 119.0 (2) |
| C6—C5—C4 | 109.20 (19) | C16—C15—H15 | 120.5 |
| C10—C5—C4 | 106.8 (2) | C14—C15—H15 | 120.5 |
| C6—C5—H5 | 110.6 | C15—C16—C17 | 120.9 (2) |
| C10—C5—H5 | 110.6 | C15—C16—C11 | 119.6 (2) |
| C4—C5—H5 | 110.6 | C17—C16—C11 | 119.5 (2) |
| C7—C6—C12 | 126.8 (2) | C16—C17—C18 | 119.3 (2) |
| C7—C6—C5 | 113.71 (19) | C16—C17—H17 | 120.4 |
| C12—C6—C5 | 119.4 (2) | C18—C17—H17 | 120.4 |
| C6—C7—C19 | 126.06 (19) | C17—C18—C13 | 122.0 (2) |
| C6—C7—C8 | 113.95 (19) | C17—C18—H18 | 119.0 |
| C19—C7—C8 | 119.98 (18) | C13—C18—H18 | 119.0 |
| C7—C8—C3 | 108.55 (18) | C20—C19—C24 | 117.5 (2) |
| C7—C8—C9 | 108.40 (18) | C20—C19—C7 | 120.6 (2) |
| C3—C8—C9 | 107.24 (19) | C24—C19—C7 | 121.9 (2) |
| C7—C8—H8 | 110.8 | C21—C20—C19 | 121.5 (2) |
| C3—C8—H8 | 110.8 | C21—C20—H20 | 119.2 |
| C9—C8—H8 | 110.8 | C19—C20—H20 | 119.2 |
| C10—C9—C8 | 109.4 (2) | C22—C21—C20 | 119.3 (3) |
| C10—C9—H9A | 109.8 | C22—C21—H21 | 120.3 |
| C8—C9—H9A | 109.8 | C20—C21—H21 | 120.3 |
| C10—C9—H9B | 109.8 | C21—C22—C23 | 121.0 (2) |
| C8—C9—H9B | 109.8 | C21—C22—C12 | 120.0 (2) |
| H9A—C9—H9B | 108.3 | C23—C22—C12 | 119.0 (2) |
| C9—C10—C5 | 109.39 (19) | C22—C23—C24 | 119.2 (2) |
| C9—C10—H10A | 109.8 | C22—C23—H23 | 120.4 |
| C5—C10—H10A | 109.8 | C24—C23—H23 | 120.4 |
| C9—C10—H10B | 109.8 | C23—C24—C19 | 121.2 (2) |
| C5—C10—H10B | 109.8 | C23—C24—H24 | 119.4 |
| H10A—C10—H10B | 108.2 | C19—C24—H24 | 119.4 |
| C11—C1—C2—C13 | -1.7 (5) | C4—C3—C8—C9 | 61.1 (2) |
| C4—C1—C2—C13 | -177.6 (3) | C7—C8—C9—C10 | 54.9 (3) |
| C11—C1—C2—C3 | 176.4 (3) | C3—C8—C9—C10 | -62.1 (2) |
| C4—C1—C2—C3 | 0.50 (19) | C8—C9—C10—C5 | 0.1 (3) |
| C1—C2—C3—C8 | -109.8 (2) | C6—C5—C10—C9 | -55.6 (3) |
| C13—C2—C3—C8 | 68.5 (3) | C4—C5—C10—C9 | 62.2 (3) |
| C1—C2—C3—C4 | -0.48 (18) | C1—C2—C13—C18 | -9.6 (4) |
| C13—C2—C3—C4 | 177.8 (2) | C3—C2—C13—C18 | 172.8 (2) |
| C2—C1—C4—C5 | 109.1 (2) | C1—C2—C13—C14 | 171.1 (3) |
| C11—C1—C4—C5 | -67.2 (3) | C3—C2—C13—C14 | -6.5 (4) |
| C2—C1—C4—C3 | -0.48 (18) | C18—C13—C14—C15 | 0.7 (4) |
| C11—C1—C4—C3 | -176.8 (3) | C2—C13—C14—C15 | -179.9 (2) |
| C2—C3—C4—C1 | 0.43 (16) | C13—C14—C15—C16 | -1.5 (4) |
| C8—C3—C4—C1 | 119.93 (19) | C14—C15—C16—C17 | 1.1 (4) |
| C2—C3—C4—C5 | -118.5 (2) | C14—C15—C16—C11 | -176.6 (2) |
| C8—C3—C4—C5 | 1.0 (3) | C15—C16—C17—C18 | 0.1 (4) |

| | | | |
|---------------|-------------|-----------------|------------|
| C1—C4—C5—C6 | -41.3 (3) | C11—C16—C17—C18 | 177.8 (2) |
| C3—C4—C5—C6 | 54.8 (3) | C16—C17—C18—C13 | -0.9 (5) |
| C1—C4—C5—C10 | -158.8 (2) | C14—C13—C18—C17 | 0.5 (4) |
| C3—C4—C5—C10 | -62.6 (2) | C2—C13—C18—C17 | -178.8 (3) |
| C10—C5—C6—C7 | 57.9 (3) | C6—C7—C19—C20 | 125.7 (3) |
| C4—C5—C6—C7 | -58.3 (3) | C8—C7—C19—C20 | -54.0 (3) |
| C10—C5—C6—C12 | -120.4 (2) | C6—C7—C19—C24 | -55.3 (3) |
| C4—C5—C6—C12 | 123.4 (2) | C8—C7—C19—C24 | 125.1 (2) |
| C12—C6—C7—C19 | -1.3 (4) | C24—C19—C20—C21 | 3.9 (4) |
| C5—C6—C7—C19 | -179.5 (2) | C7—C19—C20—C21 | -177.0 (2) |
| C12—C6—C7—C8 | 178.3 (2) | C19—C20—C21—C22 | -0.5 (4) |
| C5—C6—C7—C8 | 0.2 (3) | C20—C21—C22—C23 | -3.1 (4) |
| C6—C7—C8—C3 | 58.3 (3) | C20—C21—C22—C12 | 177.3 (2) |
| C19—C7—C8—C3 | -122.0 (2) | C21—C22—C23—C24 | 3.2 (4) |
| C6—C7—C8—C9 | -57.9 (3) | C12—C22—C23—C24 | -177.2 (2) |
| C19—C7—C8—C9 | 121.8 (2) | C22—C23—C24—C19 | 0.3 (4) |
| C2—C3—C8—C7 | 40.1 (3) | C20—C19—C24—C23 | -3.7 (4) |
| C4—C3—C8—C7 | -55.8 (2) | C7—C19—C24—C23 | 177.2 (2) |
| C2—C3—C8—C9 | 156.98 (19) | | |

Fig. 1

