# organic compounds

# 2,4-Dibenzoyl-1,3,5-triphenylcyclo-hexan-1-ol dichloromethane hemisolvate

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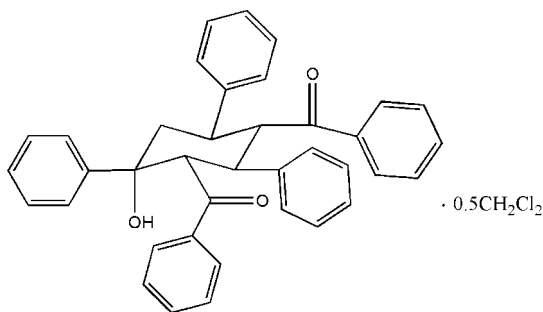Correspondence e-mail: hxqqxh2008@163.com

The asymmetric unit of the title compound, $C_{38}H_{32}O_3 \cdot 0.5CH_2Cl_2$, which was synthesized by the reaction of benzaldehyde with acetophenone and NaOH under phase-transfer catalysis and solvent-free conditions, contains two independent molecules of 2,4-dibenzoyl-1,3,5-triphenylcyclo-hexan-1-ol and a disordered dichloromethane molecule. In both independent cyclohexanol molecules, the central six-membered ring adopts a chair conformation and most of the bulky side groups are located in equatorial positions. The hydroxyl groups are involved in weak intramolecular hydrogen bonding.

## Related literature

The crystal structures of 2,4-dibenzoyl-3,5-bis(4-methoxy-lphenyl)-1-phenylcyclohexanol and 2,4-dibenzoyl-3,5-bis(2-thienyl)-1-phenylcyclohexanol were reported by Luo & Shan (2006) and Huang & Wang (2007), respectively.



· 0.5CH₂Cl₂

## Experimental

### Crystal data

| | |
|---|---|
| $C_{38}H_{31}O_3 \cdot 0.5CH_2Cl_2$ | $\gamma = 100.908\ (14)°$ |
| $M_r = 579.1$ | $V = 3116\ (5)$ Å³ |
| Triclinic, $P\bar{1}$ | $Z = 4$ |
| $a = 12.452\ (11)$ Å | Mo $K\alpha$ radiation |
| $b = 12.527\ (11)$ Å | $\mu = 0.16$ mm⁻¹ |
| $c = 20.631\ (18)$ Å | $T = 298\ (2)$ K |
| $\alpha = 94.152\ (15)°$ | $0.44 \times 0.39 \times 0.33$ mm |
| $\beta = 97.683\ (14)°$ | |

### Data collection

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 16393 measured reflections |
| | 10835 independent reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 5265 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.933$, $T_{max} = 0.949$ | $R_{int} = 0.027$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | 108 restraints |
| $wR(F^2) = 0.175$ | H-atom parameters constrained |
| $S = 1.00$ | $\Delta\rho_{max} = 0.40$ e Å⁻³ |
| 10835 reflections | $\Delta\rho_{min} = -0.28$ e Å⁻³ |
| 784 parameters | |

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| O1—H1⋯O2 | 0.82 | 2.12 | 2.701 (4) | 128 |
| O4—H4⋯O5 | 0.82 | 2.19 | 2.759 (4) | 127 |

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2326).

## References

Huang, X.-Q. & Wang, J.-X. (2007). Acta Cryst. E**63**, o4168.
Luo, X. & Shan, Z. (2006). Acta Cryst. E**62**, o1631–o1632.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
Sheldrick, G. M. (1997b). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

**supplementary materials**

## 2,4-Dibenzoyl-1,3,5-triphenylcyclohexan-1-ol dichloromethane hemisolvate

**J.-H. Zhang, Q.-P. He, Y. Wang and D.-Q. Wang**

### Comment

In this paper, we present the crystal structure of 2,4-Dibenzoyl-1,3,5-triphenyl-1-cyclohexanol (I) dichloromethane 2:1 solvate, which was synthesized through the condensation and Micheal addition of benzaldehyde with acetophenone under tetrabutyl ammonium bromide and solvent-free conditions.

In the crystal structure, the asymmetric unit contains two independent molecules of (I) (Fig. 1) with normal values of bond lengths and angles corresponding to the published ones (Luo *et al.*, 2006; Huang *et al.*, 2007), and one disordered dichloromethane solvent molecule. The hydroxyl groups in two independent molecules are involved in weak intramolecular hydrogen bonding (Table 1).

### Experimental

Acetophenone (6.25 mmol), freshly distilled benzaldehyde (3.125 mmol), NaOH (6.25 mmol) and tetrabutyl ammonium bromide (1 mmol) were aggregated with a glass paddle in an open flask. The resulting mixture was washed with water several times to remove NaOH and was recrystallized from ethanol and $CH_2Cl_2$, affording the title compound as a crystalline solid. Elemental analysis: calculated for $C_{77}H_{66}O_6Cl_2$: C 79.85, H 5.74%; found: C 79.78, H 5.83%.

### Refinement

C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.98 Å and $U_{iso}(H) = U_{eq}(C)$. The H atoms of hydroxyl were placed in idealized positions and constrained to ride on their parent atoms with O—H distances of 0.82%/A, and with $U_{iso}(H) = 1.2\ U_{eq}(O)$. The dichloromethane molecule was treated as disordered between two positions with the occupancies fixed to 0.6 and 0.4, respectively.
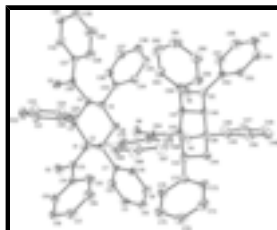
### Figures



Fig. 1. The content of asymmetric unit of the title compound showing the atomic numbering scheme and 20% probabilty displacement ellipsoids. Hydrogen atoms and dichloromethane solvent molecule are omitted for clarity.

# supplementary materials

**2,4-Dibenzoyl-1,3,5-triphenylcyclohexan-1-ol dichloromethane hemisolvate**

*Crystal data*

| | |
|---|---|
| $C_{38}H_{31}O_3 \cdot 0.5CH_2Cl_2$ | $Z = 4$ |
| $M_r = 579.1$ | $F_{000} = 1220$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.234$ Mg m$^{-3}$ |
| $a = 12.452$ (11) Å | Mo $K\alpha$ radiation $\lambda = 0.71073$ Å |
| $b = 12.527$ (11) Å | Cell parameters from 3197 reflections |
| $c = 20.631$ (18) Å | $\theta = 2.5$–22.0º |
| $\alpha = 94.152$ (15)º | $\mu = 0.16$ mm$^{-1}$ |
| $\beta = 97.683$ (14)º | $T = 298$ (2) K |
| $\gamma = 100.908$ (14)º | Block, colourless |
| $V = 3116$ (5) Å$^3$ | $0.44 \times 0.39 \times 0.33$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 10835 independent reflections |
| Radiation source: fine-focus sealed tube | 5265 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.027$ |
| $T = 298(2)$ K | $\theta_{max} = 25.0$º |
| phi and $\omega$ scans | $\theta_{min} = 1.8$º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -13 \rightarrow 14$ |
| $T_{min} = 0.933$, $T_{max} = 0.949$ | $k = -14 \rightarrow 14$ |
| 16393 measured reflections | $l = -24 \rightarrow 21$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | H-atom parameters constrained |
| $wR(F^2) = 0.175$ | $w = 1/[\sigma^2(F_o^2) + (0.0761P)^2 + 0.1993P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.01$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 10835 reflections | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| 784 parameters | $\Delta\rho_{min} = -0.27$ e Å$^{-3}$ |
| 108 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)*

|       | $x$         | $y$         | $z$         | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|-------|-------------|-------------|-------------|--------------------|-----------|
| Cl1   | 0.6656 (2)  | 0.7680 (2)  | 0.36319 (11)| 0.1137 (8)         | 0.60      |
| Cl2   | 0.7072 (2)  | 0.8105 (4)  | 0.49918 (13)| 0.1926 (15)        | 0.60      |
| Cl1'  | 0.6733 (8)  | 0.7238 (8)  | 0.4053 (6)  | 0.320 (5)          | 0.40      |
| Cl2'  | 0.6652 (5)  | 0.9270 (4)  | 0.4809 (2)  | 0.189 (2)          | 0.40      |
| O1    | 0.36092 (18)| 0.40310 (17)| 0.36048 (10)| 0.0615 (6)         |           |
| H1    | 0.3285      | 0.3744      | 0.3888      | 0.092*             |           |
| O2    | 0.1561 (2)  | 0.3367 (2)  | 0.39103 (12)| 0.0739 (7)         |           |
| O3    | 0.2578 (2)  | 0.24637 (19)| 0.16499 (11)| 0.0682 (7)         |           |
| O4    | 0.43540 (19)| 0.90376 (19)| 0.31106 (12)| 0.0751 (7)         |           |
| H4    | 0.4821      | 0.8848      | 0.2908      | 0.113*             |           |
| O5    | 0.47955 (19)| 0.8726 (2)  | 0.18429 (12)| 0.0724 (7)         |           |
| O6    | 0.10992 (18)| 0.72014 (18)| 0.24378 (12)| 0.0683 (7)         |           |
| C1    | 0.3194 (2)  | 0.4986 (2)  | 0.34476 (14)| 0.0447 (8)         |           |
| C2    | 0.1943 (2)  | 0.4668 (2)  | 0.31583 (13)| 0.0427 (7)         |           |
| H2    | 0.1683      | 0.5330      | 0.3041      | 0.051*             |           |
| C3    | 0.1743 (2)  | 0.3851 (2)  | 0.25358 (14)| 0.0438 (8)         |           |
| H3    | 0.1951      | 0.3176      | 0.2672      | 0.053*             |           |
| C4    | 0.2475 (2)  | 0.4258 (2)  | 0.20236 (14)| 0.0441 (8)         |           |
| H4A   | 0.2213      | 0.4871      | 0.1830      | 0.053*             |           |
| C5    | 0.3712 (2)  | 0.4628 (2)  | 0.23160 (14)| 0.0469 (8)         |           |
| H5    | 0.3975      | 0.3983      | 0.2462      | 0.056*             |           |
| C6    | 0.3842 (2)  | 0.5448 (2)  | 0.29221 (14)| 0.0490 (8)         |           |
| H6A   | 0.4619      | 0.5661      | 0.3106      | 0.059*             |           |
| H6B   | 0.3591      | 0.6098      | 0.2790      | 0.059*             |           |
| C7    | 0.3379 (2)  | 0.5823 (3)  | 0.40493 (14)| 0.0460 (8)         |           |
| C8    | 0.3091 (3)  | 0.6832 (3)  | 0.40019 (16)| 0.0623 (10)        |           |
| H8    | 0.2759      | 0.6996      | 0.3600      | 0.075*             |           |
| C9    | 0.3289 (3)  | 0.7599 (3)  | 0.45408 (17)| 0.0685 (10)        |           |
| H9    | 0.3092      | 0.8274      | 0.4500      | 0.082*             |           |
| C10   | 0.3779 (3)  | 0.7368 (3)  | 0.51393 (17)| 0.0658 (10)        |           |
| H10   | 0.3919      | 0.7885      | 0.5503      | 0.079*             |           |
| C11   | 0.4055 (3)  | 0.6376 (3)  | 0.51921 (16)| 0.0641 (10)        |           |
| H11   | 0.4384      | 0.6218      | 0.5596      | 0.077*             |           |

| C12 | 0.3856 (3) | 0.5597 (3) | 0.46554 (15) | 0.0560 (9) |
| H12 | 0.4043 | 0.4919 | 0.4703 | 0.067* |
| C13 | 0.1275 (3) | 0.4156 (3) | 0.36602 (15) | 0.0490 (8) |
| C14 | 0.0314 (3) | 0.4560 (3) | 0.38562 (15) | 0.0507 (8) |
| C15 | −0.0007 (3) | 0.5486 (3) | 0.36472 (17) | 0.0656 (10) |
| H15 | 0.0393 | 0.5895 | 0.3368 | 0.079* |
| C16 | −0.0920 (3) | 0.5811 (3) | 0.3849 (2) | 0.0803 (12) |
| H16 | −0.1133 | 0.6435 | 0.3702 | 0.096* |
| C17 | −0.1508 (3) | 0.5225 (4) | 0.4259 (2) | 0.0849 (12) |
| H17 | −0.2115 | 0.5454 | 0.4398 | 0.102* |
| C18 | −0.1206 (3) | 0.4297 (4) | 0.44666 (18) | 0.0773 (12) |
| H18 | −0.1620 | 0.3889 | 0.4740 | 0.093* |
| C19 | −0.0299 (3) | 0.3961 (3) | 0.42748 (16) | 0.0642 (10) |
| H19 | −0.0093 | 0.3335 | 0.4424 | 0.077* |
| C20 | 0.0529 (3) | 0.3561 (2) | 0.22293 (14) | 0.0455 (8) |
| C21 | −0.0046 (3) | 0.2497 (3) | 0.21561 (17) | 0.0652 (10) |
| H21 | 0.0310 | 0.1948 | 0.2298 | 0.078* |
| C22 | −0.1147 (3) | 0.2233 (3) | 0.1873 (2) | 0.0848 (13) |
| H22 | −0.1523 | 0.1509 | 0.1828 | 0.102* |
| C23 | −0.1684 (3) | 0.3017 (4) | 0.16631 (18) | 0.0781 (12) |
| H23 | −0.2425 | 0.2834 | 0.1476 | 0.094* |
| C24 | −0.1132 (3) | 0.4080 (3) | 0.17269 (17) | 0.0714 (11) |
| H24 | −0.1496 | 0.4621 | 0.1581 | 0.086* |
| C25 | −0.0029 (3) | 0.4352 (3) | 0.20089 (16) | 0.0570 (9) |
| H25 | 0.0341 | 0.5077 | 0.2051 | 0.068* |
| C26 | 0.2361 (3) | 0.3329 (3) | 0.14882 (15) | 0.0493 (8) |
| C27 | 0.2015 (3) | 0.3452 (3) | 0.07848 (16) | 0.0538 (9) |
| C28 | 0.1639 (4) | 0.4334 (3) | 0.05594 (19) | 0.0923 (14) |
| H28 | 0.1616 | 0.4920 | 0.0857 | 0.111* |
| C29 | 0.1288 (5) | 0.4374 (4) | −0.0105 (2) | 0.1158 (18) |
| H29 | 0.1021 | 0.4976 | −0.0246 | 0.139* |
| C30 | 0.1335 (4) | 0.3542 (4) | −0.0546 (2) | 0.0992 (16) |
| H30 | 0.1111 | 0.3573 | −0.0991 | 0.119* |
| C31 | 0.1707 (4) | 0.2667 (4) | −0.0337 (2) | 0.0949 (15) |
| H31 | 0.1737 | 0.2092 | −0.0641 | 0.114* |
| C32 | 0.2046 (3) | 0.2609 (3) | 0.03230 (19) | 0.0770 (11) |
| H32 | 0.2297 | 0.1995 | 0.0457 | 0.092* |
| C33 | 0.4386 (3) | 0.5055 (3) | 0.17995 (15) | 0.0478 (8) |
| C34 | 0.4320 (3) | 0.6037 (3) | 0.15533 (17) | 0.0644 (10) |
| H34 | 0.3875 | 0.6469 | 0.1726 | 0.077* |
| C35 | 0.4894 (3) | 0.6397 (3) | 0.10586 (19) | 0.0757 (11) |
| H35 | 0.4827 | 0.7058 | 0.0896 | 0.091* |
| C36 | 0.5563 (3) | 0.5777 (4) | 0.08089 (19) | 0.0773 (12) |
| H36 | 0.5949 | 0.6013 | 0.0472 | 0.093* |
| C37 | 0.5665 (3) | 0.4810 (3) | 0.10538 (19) | 0.0764 (11) |
| H37 | 0.6128 | 0.4391 | 0.0888 | 0.092* |
| C38 | 0.5081 (3) | 0.4456 (3) | 0.15460 (16) | 0.0618 (9) |
| H38 | 0.5159 | 0.3798 | 0.1711 | 0.074* |
| C39 | 0.4095 (3) | 1.0040 (3) | 0.28854 (15) | 0.0497 (8) |

| | | | |
|---|---|---|---|
| C40 | 0.3593 (2) | 0.9856 (2) | 0.21476 (14) | 0.0435 (7) |
| H40 | 0.3385 | 1.0533 | 0.2013 | 0.052* |
| C41 | 0.2570 (2) | 0.8929 (2) | 0.20069 (15) | 0.0462 (8) |
| H41 | 0.2808 | 0.8252 | 0.2113 | 0.055* |
| C42 | 0.1701 (2) | 0.9110 (2) | 0.24427 (14) | 0.0446 (8) |
| H42 | 0.1362 | 0.9713 | 0.2295 | 0.054* |
| C43 | 0.2189 (2) | 0.9378 (3) | 0.31802 (15) | 0.0504 (8) |
| H43 | 0.2420 | 0.8724 | 0.3337 | 0.060* |
| C44 | 0.3210 (2) | 1.0300 (3) | 0.32778 (15) | 0.0528 (8) |
| H44A | 0.2992 | 1.0964 | 0.3145 | 0.063* |
| H44B | 0.3516 | 1.0429 | 0.3741 | 0.063* |
| C45 | 0.5130 (3) | 1.0956 (3) | 0.29897 (15) | 0.0520 (8) |
| C46 | 0.6171 (3) | 1.0724 (3) | 0.31124 (18) | 0.0731 (11) |
| H46 | 0.6252 | 1.0008 | 0.3156 | 0.088* |
| C47 | 0.7105 (3) | 1.1560 (4) | 0.3171 (2) | 0.0887 (13) |
| H47 | 0.7805 | 1.1395 | 0.3251 | 0.106* |
| C48 | 0.7006 (4) | 1.2612 (4) | 0.31141 (18) | 0.0806 (12) |
| H48 | 0.7633 | 1.3162 | 0.3147 | 0.097* |
| C49 | 0.5980 (3) | 1.2851 (3) | 0.30080 (17) | 0.0745 (11) |
| H49 | 0.5904 | 1.3571 | 0.2977 | 0.089* |
| C50 | 0.5043 (3) | 1.2025 (3) | 0.29459 (16) | 0.0623 (10) |
| H50 | 0.4347 | 1.2199 | 0.2873 | 0.075* |
| C51 | 0.4469 (3) | 0.9577 (3) | 0.17487 (16) | 0.0530 (9) |
| C52 | 0.4938 (3) | 1.0293 (3) | 0.12746 (15) | 0.0496 (8) |
| C53 | 0.4558 (3) | 1.1216 (3) | 0.10951 (17) | 0.0645 (10) |
| H53 | 0.3984 | 1.1423 | 0.1286 | 0.077* |
| C54 | 0.5018 (3) | 1.1827 (3) | 0.06389 (19) | 0.0780 (11) |
| H54 | 0.4747 | 1.2438 | 0.0519 | 0.094* |
| C55 | 0.5872 (3) | 1.1544 (4) | 0.03611 (19) | 0.0813 (13) |
| H55 | 0.6187 | 1.1965 | 0.0056 | 0.098* |
| C56 | 0.6260 (3) | 1.0641 (4) | 0.0533 (2) | 0.0830 (13) |
| H56 | 0.6843 | 1.0450 | 0.0345 | 0.100* |
| C57 | 0.5800 (3) | 1.0009 (3) | 0.09827 (17) | 0.0653 (10) |
| H57 | 0.6067 | 0.9390 | 0.1091 | 0.078* |
| C58 | 0.2059 (2) | 0.8773 (3) | 0.12931 (16) | 0.0491 (8) |
| C59 | 0.1685 (3) | 0.9616 (3) | 0.09842 (16) | 0.0598 (9) |
| H59 | 0.1757 | 1.0289 | 0.1225 | 0.072* |
| C60 | 0.1211 (3) | 0.9480 (4) | 0.03316 (19) | 0.0761 (11) |
| H60 | 0.0984 | 1.0061 | 0.0134 | 0.091* |
| C61 | 0.1077 (3) | 0.8484 (5) | −0.0023 (2) | 0.0906 (14) |
| H61 | 0.0738 | 0.8378 | −0.0459 | 0.109* |
| C62 | 0.1442 (4) | 0.7648 (4) | 0.0267 (2) | 0.0916 (14) |
| H62 | 0.1361 | 0.6975 | 0.0024 | 0.110* |
| C63 | 0.1931 (3) | 0.7790 (3) | 0.0918 (2) | 0.0720 (11) |
| H63 | 0.2179 | 0.7211 | 0.1106 | 0.086* |
| C64 | 0.0804 (3) | 0.8076 (3) | 0.23779 (15) | 0.0485 (8) |
| C65 | −0.0391 (3) | 0.8106 (3) | 0.22748 (15) | 0.0489 (8) |
| C66 | −0.0807 (3) | 0.9029 (3) | 0.21618 (19) | 0.0741 (11) |
| H66 | −0.0322 | 0.9683 | 0.2136 | 0.089* |

| | | | | | |
|---|---|---|---|---|---|
| C67 | −0.1927 (3) | 0.9005 (3) | 0.2086 (2) | 0.0922 (14) | |
| H67 | −0.2191 | 0.9639 | 0.2016 | 0.111* | |
| C68 | −0.2644 (3) | 0.8055 (4) | 0.2114 (2) | 0.0830 (12) | |
| H68 | −0.3401 | 0.8037 | 0.2055 | 0.100* | |
| C69 | −0.2262 (3) | 0.7127 (3) | 0.22272 (18) | 0.0731 (11) | |
| H69 | −0.2755 | 0.6478 | 0.2252 | 0.088* | |
| C70 | −0.1142 (3) | 0.7154 (3) | 0.23058 (16) | 0.0588 (9) | |
| H70 | −0.0886 | 0.6516 | 0.2382 | 0.071* | |
| C71 | 0.1287 (3) | 0.9628 (3) | 0.35635 (15) | 0.0514 (8) | |
| C72 | 0.0953 (3) | 1.0615 (3) | 0.35598 (17) | 0.0651 (10) | |
| H72 | 0.1337 | 1.1175 | 0.3357 | 0.078* | |
| C73 | 0.0060 (3) | 1.0784 (4) | 0.3852 (2) | 0.0820 (12) | |
| H73 | −0.0158 | 1.1453 | 0.3841 | 0.098* | |
| C74 | −0.0505 (3) | 0.9978 (4) | 0.4156 (2) | 0.0910 (14) | |
| H74 | −0.1115 | 1.0092 | 0.4346 | 0.109* | |
| C75 | −0.0171 (3) | 0.8993 (4) | 0.41813 (19) | 0.0854 (13) | |
| H75 | −0.0545 | 0.8444 | 0.4396 | 0.102* | |
| C76 | 0.0716 (3) | 0.8826 (3) | 0.38880 (17) | 0.0663 (10) | |
| H76 | 0.0939 | 0.8159 | 0.3907 | 0.080* | |
| C77 | 0.6124 (5) | 0.8111 (7) | 0.4318 (3) | 0.172 (3) | 0.60 |
| H77A | 0.5982 | 0.8842 | 0.4284 | 0.207* | 0.60 |
| H77B | 0.5433 | 0.7624 | 0.4352 | 0.207* | 0.60 |
| C77' | 0.6124 (5) | 0.8111 (7) | 0.4318 (3) | 0.172 (3) | 0.40 |
| H77C | 0.5560 | 0.7712 | 0.4541 | 0.207* | 0.40 |
| H77D | 0.5730 | 0.8337 | 0.3930 | 0.207* | 0.40 |

*Atomic displacement parameters ($Å^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.1009 (17) | 0.148 (2) | 0.0975 (16) | 0.0285 (15) | 0.0339 (13) | 0.0032 (14) |
| Cl2 | 0.110 (2) | 0.346 (5) | 0.0979 (19) | −0.011 (3) | 0.0096 (14) | 0.027 (3) |
| Cl1' | 0.198 (7) | 0.340 (11) | 0.414 (13) | 0.051 (7) | 0.046 (9) | −0.003 (9) |
| Cl2' | 0.222 (5) | 0.190 (4) | 0.115 (3) | −0.060 (4) | 0.064 (3) | −0.051 (3) |
| O1 | 0.0713 (16) | 0.0643 (15) | 0.0539 (14) | 0.0289 (13) | 0.0042 (11) | 0.0072 (11) |
| O2 | 0.0840 (19) | 0.0666 (17) | 0.0784 (17) | 0.0199 (14) | 0.0186 (13) | 0.0305 (14) |
| O3 | 0.0891 (19) | 0.0502 (15) | 0.0669 (16) | 0.0242 (13) | 0.0103 (13) | −0.0080 (12) |
| O4 | 0.0585 (16) | 0.0811 (18) | 0.0909 (18) | 0.0220 (13) | 0.0096 (13) | 0.0248 (15) |
| O5 | 0.0629 (17) | 0.0610 (16) | 0.103 (2) | 0.0278 (13) | 0.0258 (13) | 0.0051 (14) |
| O6 | 0.0562 (15) | 0.0432 (14) | 0.109 (2) | 0.0111 (12) | 0.0219 (13) | 0.0120 (13) |
| C1 | 0.048 (2) | 0.0444 (19) | 0.0437 (18) | 0.0154 (15) | 0.0031 (14) | 0.0059 (15) |
| C2 | 0.0446 (19) | 0.0406 (18) | 0.0414 (17) | 0.0079 (14) | 0.0043 (14) | 0.0008 (14) |
| C3 | 0.0470 (19) | 0.0393 (18) | 0.0441 (18) | 0.0092 (15) | 0.0047 (14) | 0.0009 (14) |
| C4 | 0.0487 (19) | 0.0397 (18) | 0.0434 (18) | 0.0115 (15) | 0.0040 (14) | 0.0000 (14) |
| C5 | 0.046 (2) | 0.0470 (19) | 0.0495 (19) | 0.0147 (15) | 0.0076 (15) | 0.0016 (15) |
| C6 | 0.0427 (19) | 0.054 (2) | 0.0480 (19) | 0.0102 (15) | 0.0017 (15) | −0.0023 (16) |
| C7 | 0.0394 (19) | 0.056 (2) | 0.0404 (18) | 0.0090 (16) | 0.0027 (14) | 0.0004 (15) |
| C8 | 0.078 (3) | 0.058 (2) | 0.047 (2) | 0.020 (2) | −0.0070 (17) | −0.0056 (18) |
| C9 | 0.083 (3) | 0.061 (2) | 0.057 (2) | 0.017 (2) | 0.002 (2) | −0.0112 (19) |

| | | | | | | |
|------|-------------|-------------|-------------|--------------|--------------|---------------|
| C10 | 0.060 (2) | 0.077 (3) | 0.053 (2) | 0.004 (2) | 0.0111 (18) | −0.018 (2) |
| C11 | 0.060 (2) | 0.089 (3) | 0.039 (2) | 0.013 (2) | 0.0027 (16) | 0.002 (2) |
| C12 | 0.052 (2) | 0.072 (2) | 0.045 (2) | 0.0177 (18) | 0.0030 (16) | 0.0051 (18) |
| C13 | 0.055 (2) | 0.046 (2) | 0.0426 (18) | 0.0047 (17) | 0.0022 (15) | 0.0049 (16) |
| C14 | 0.050 (2) | 0.053 (2) | 0.0437 (19) | −0.0009 (17) | 0.0074 (16) | −0.0043 (16) |
| C15 | 0.062 (3) | 0.071 (3) | 0.070 (2) | 0.018 (2) | 0.0220 (19) | 0.009 (2) |
| C16 | 0.076 (3) | 0.087 (3) | 0.090 (3) | 0.029 (2) | 0.033 (2) | 0.013 (2) |
| C17 | 0.066 (3) | 0.101 (4) | 0.085 (3) | 0.009 (3) | 0.025 (2) | −0.012 (3) |
| C18 | 0.065 (3) | 0.092 (3) | 0.064 (3) | −0.016 (2) | 0.025 (2) | −0.008 (2) |
| C19 | 0.063 (2) | 0.066 (2) | 0.056 (2) | −0.008 (2) | 0.0125 (18) | 0.0002 (18) |
| C20 | 0.050 (2) | 0.044 (2) | 0.0397 (18) | 0.0044 (16) | 0.0059 (14) | 0.0002 (15) |
| C21 | 0.070 (3) | 0.048 (2) | 0.070 (2) | 0.0027 (19) | 0.0000 (19) | 0.0043 (18) |
| C22 | 0.072 (3) | 0.067 (3) | 0.097 (3) | −0.020 (2) | −0.005 (2) | 0.007 (2) |
| C23 | 0.053 (2) | 0.094 (3) | 0.071 (3) | −0.011 (2) | −0.0064 (19) | −0.002 (2) |
| C24 | 0.057 (2) | 0.079 (3) | 0.073 (3) | 0.014 (2) | −0.0062 (19) | 0.004 (2) |
| C25 | 0.051 (2) | 0.050 (2) | 0.064 (2) | 0.0066 (17) | −0.0021 (17) | 0.0014 (17) |
| C26 | 0.049 (2) | 0.044 (2) | 0.055 (2) | 0.0115 (16) | 0.0096 (15) | −0.0030 (17) |
| C27 | 0.057 (2) | 0.052 (2) | 0.047 (2) | −0.0010 (17) | 0.0118 (16) | −0.0037 (17) |
| C28 | 0.144 (4) | 0.075 (3) | 0.054 (3) | 0.032 (3) | −0.006 (2) | −0.004 (2) |
| C29 | 0.190 (6) | 0.103 (4) | 0.052 (3) | 0.041 (4) | −0.008 (3) | 0.005 (3) |
| C30 | 0.124 (4) | 0.109 (4) | 0.051 (3) | −0.007 (3) | 0.009 (2) | 0.002 (3) |
| C31 | 0.122 (4) | 0.094 (4) | 0.056 (3) | −0.008 (3) | 0.026 (2) | −0.025 (2) |
| C32 | 0.093 (3) | 0.073 (3) | 0.061 (3) | 0.007 (2) | 0.020 (2) | −0.013 (2) |
| C33 | 0.047 (2) | 0.047 (2) | 0.0470 (19) | 0.0075 (16) | 0.0050 (15) | −0.0030 (16) |
| C34 | 0.075 (3) | 0.057 (2) | 0.065 (2) | 0.0151 (19) | 0.0217 (19) | 0.0097 (19) |
| C35 | 0.089 (3) | 0.064 (3) | 0.073 (3) | 0.007 (2) | 0.015 (2) | 0.013 (2) |
| C36 | 0.076 (3) | 0.079 (3) | 0.068 (3) | −0.010 (2) | 0.022 (2) | −0.001 (2) |
| C37 | 0.074 (3) | 0.078 (3) | 0.082 (3) | 0.015 (2) | 0.032 (2) | 0.004 (2) |
| C38 | 0.066 (2) | 0.062 (2) | 0.061 (2) | 0.0148 (19) | 0.0223 (19) | 0.0035 (18) |
| C39 | 0.0421 (19) | 0.052 (2) | 0.057 (2) | 0.0164 (16) | 0.0040 (15) | 0.0090 (16) |
| C40 | 0.0374 (18) | 0.0410 (18) | 0.0542 (19) | 0.0118 (14) | 0.0103 (14) | 0.0027 (15) |
| C41 | 0.0410 (19) | 0.0382 (18) | 0.060 (2) | 0.0107 (15) | 0.0085 (15) | 0.0003 (15) |
| C42 | 0.0398 (18) | 0.0400 (18) | 0.055 (2) | 0.0113 (14) | 0.0078 (14) | 0.0027 (15) |
| C43 | 0.0434 (19) | 0.053 (2) | 0.055 (2) | 0.0094 (16) | 0.0088 (15) | 0.0070 (16) |
| C44 | 0.045 (2) | 0.062 (2) | 0.0478 (19) | 0.0059 (17) | 0.0056 (15) | 0.0008 (16) |
| C45 | 0.041 (2) | 0.063 (2) | 0.050 (2) | 0.0088 (17) | 0.0030 (15) | 0.0015 (17) |
| C46 | 0.044 (2) | 0.088 (3) | 0.086 (3) | 0.011 (2) | 0.0070 (19) | 0.002 (2) |
| C47 | 0.042 (2) | 0.123 (4) | 0.097 (3) | 0.013 (3) | 0.006 (2) | 0.001 (3) |
| C48 | 0.061 (3) | 0.100 (4) | 0.067 (3) | −0.011 (3) | 0.007 (2) | −0.006 (2) |
| C49 | 0.067 (3) | 0.076 (3) | 0.069 (3) | −0.006 (2) | 0.003 (2) | 0.001 (2) |
| C50 | 0.045 (2) | 0.070 (3) | 0.066 (2) | 0.0018 (19) | 0.0025 (17) | 0.0046 (19) |
| C51 | 0.040 (2) | 0.050 (2) | 0.066 (2) | 0.0099 (17) | 0.0045 (16) | −0.0071 (18) |
| C52 | 0.0409 (19) | 0.048 (2) | 0.056 (2) | 0.0024 (16) | 0.0101 (15) | −0.0096 (17) |
| C53 | 0.070 (3) | 0.057 (2) | 0.067 (2) | 0.005 (2) | 0.0259 (19) | −0.0013 (19) |
| C54 | 0.091 (3) | 0.066 (3) | 0.077 (3) | 0.005 (2) | 0.027 (2) | 0.006 (2) |
| C55 | 0.069 (3) | 0.100 (4) | 0.064 (3) | −0.014 (3) | 0.023 (2) | −0.004 (2) |
| C56 | 0.051 (3) | 0.117 (4) | 0.078 (3) | 0.009 (3) | 0.023 (2) | −0.009 (3) |
| C57 | 0.044 (2) | 0.085 (3) | 0.065 (2) | 0.0114 (19) | 0.0137 (18) | −0.008 (2) |
| C58 | 0.0400 (19) | 0.049 (2) | 0.058 (2) | 0.0074 (16) | 0.0110 (15) | −0.0046 (17) |

| C59 | 0.051 (2) | 0.070 (2) | 0.056 (2) | 0.0104 (18) | 0.0050 (17) | −0.0013 (19) |
| C60 | 0.057 (2) | 0.099 (3) | 0.065 (3) | 0.007 (2) | −0.001 (2) | 0.008 (2) |
| C61 | 0.060 (3) | 0.130 (4) | 0.065 (3) | −0.011 (3) | 0.004 (2) | −0.012 (3) |
| C62 | 0.082 (3) | 0.089 (3) | 0.088 (4) | −0.007 (3) | 0.017 (3) | −0.037 (3) |
| C63 | 0.067 (3) | 0.062 (2) | 0.080 (3) | 0.004 (2) | 0.013 (2) | −0.021 (2) |
| C64 | 0.052 (2) | 0.042 (2) | 0.054 (2) | 0.0128 (17) | 0.0129 (15) | 0.0032 (16) |
| C65 | 0.044 (2) | 0.049 (2) | 0.0504 (19) | 0.0066 (17) | 0.0039 (15) | −0.0050 (16) |
| C66 | 0.046 (2) | 0.054 (2) | 0.116 (3) | 0.0093 (18) | −0.006 (2) | −0.001 (2) |
| C67 | 0.050 (3) | 0.072 (3) | 0.147 (4) | 0.021 (2) | −0.015 (2) | −0.009 (3) |
| C68 | 0.042 (2) | 0.091 (3) | 0.108 (3) | 0.013 (2) | −0.004 (2) | −0.013 (3) |
| C69 | 0.045 (2) | 0.076 (3) | 0.088 (3) | −0.006 (2) | 0.0056 (19) | −0.003 (2) |
| C70 | 0.053 (2) | 0.056 (2) | 0.064 (2) | 0.0072 (18) | 0.0071 (17) | 0.0012 (17) |
| C71 | 0.044 (2) | 0.056 (2) | 0.052 (2) | 0.0070 (17) | 0.0064 (16) | 0.0016 (17) |
| C72 | 0.056 (2) | 0.064 (3) | 0.076 (3) | 0.0132 (19) | 0.0170 (19) | −0.002 (2) |
| C73 | 0.062 (3) | 0.080 (3) | 0.103 (3) | 0.017 (2) | 0.021 (2) | −0.018 (3) |
| C74 | 0.064 (3) | 0.110 (4) | 0.096 (3) | 0.007 (3) | 0.034 (2) | −0.022 (3) |
| C75 | 0.072 (3) | 0.101 (4) | 0.081 (3) | −0.001 (3) | 0.036 (2) | 0.003 (3) |
| C76 | 0.062 (2) | 0.071 (3) | 0.065 (2) | 0.005 (2) | 0.0184 (19) | 0.007 (2) |
| C77 | 0.113 (5) | 0.272 (8) | 0.136 (5) | 0.037 (5) | 0.043 (4) | 0.005 (5) |
| C77' | 0.113 (5) | 0.272 (8) | 0.136 (5) | 0.037 (5) | 0.043 (4) | 0.005 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl1—C77 | 1.736 (7) | C36—C37 | 1.368 (5) |
| Cl2—C77 | 1.700 (7) | C36—H36 | 0.9300 |
| O1—C1 | 1.432 (3) | C37—C38 | 1.377 (5) |
| O1—H1 | 0.8200 | C37—H37 | 0.9300 |
| O2—O2 | 0.000 (6) | C38—H38 | 0.9300 |
| O2—C13 | 1.236 (4) | C39—C44 | 1.521 (4) |
| O3—C26 | 1.224 (3) | C39—C45 | 1.534 (4) |
| O4—C39 | 1.446 (4) | C39—C40 | 1.550 (4) |
| O4—H4 | 0.8200 | C40—C51 | 1.526 (4) |
| O5—O5 | 0.000 (8) | C40—C41 | 1.533 (4) |
| O5—C51 | 1.230 (4) | C40—H40 | 0.9800 |
| O6—C64 | 1.229 (3) | C41—C58 | 1.507 (4) |
| C1—C6 | 1.520 (4) | C41—C42 | 1.535 (4) |
| C1—C7 | 1.529 (4) | C41—H41 | 0.9800 |
| C1—C2 | 1.557 (4) | C42—C64 | 1.527 (4) |
| C2—C13 | 1.517 (4) | C42—C43 | 1.549 (4) |
| C2—C3 | 1.545 (4) | C42—H42 | 0.9800 |
| C2—H2 | 0.9800 | C43—C71 | 1.524 (4) |
| C3—C20 | 1.524 (4) | C43—C44 | 1.527 (4) |
| C3—C4 | 1.538 (4) | C43—H43 | 0.9800 |
| C3—H3 | 0.9800 | C44—H44A | 0.9700 |
| C4—C26 | 1.518 (4) | C44—H44B | 0.9700 |
| C4—C5 | 1.547 (4) | C45—C50 | 1.372 (5) |
| C4—H4A | 0.9800 | C45—C46 | 1.377 (5) |
| C5—C33 | 1.509 (4) | C46—C47 | 1.395 (5) |
| C5—C6 | 1.531 (4) | C46—H46 | 0.9300 |

| | | | |
|---|---|---|---|
| C5—H5 | 0.9800 | C47—C48 | 1.359 (5) |
| C6—H6A | 0.9700 | C47—H47 | 0.9300 |
| C6—H6B | 0.9700 | C48—C49 | 1.360 (5) |
| C7—C12 | 1.383 (4) | C48—H48 | 0.9300 |
| C7—C8 | 1.384 (4) | C49—C50 | 1.390 (5) |
| C8—C9 | 1.379 (4) | C49—H49 | 0.9300 |
| C8—H8 | 0.9300 | C50—H50 | 0.9300 |
| C9—C10 | 1.378 (5) | C51—O5 | 1.230 (4) |
| C9—H9 | 0.9300 | C51—C52 | 1.477 (5) |
| C10—C11 | 1.358 (5) | C52—C53 | 1.385 (4) |
| C10—H10 | 0.9300 | C52—C57 | 1.388 (4) |
| C11—C12 | 1.386 (4) | C53—C54 | 1.372 (5) |
| C11—H11 | 0.9300 | C53—H53 | 0.9300 |
| C12—H12 | 0.9300 | C54—C55 | 1.364 (5) |
| C13—O2 | 1.236 (4) | C54—H54 | 0.9300 |
| C13—C14 | 1.477 (5) | C55—C56 | 1.363 (5) |
| C14—C15 | 1.375 (4) | C55—H55 | 0.9300 |
| C14—C19 | 1.394 (4) | C56—C57 | 1.375 (5) |
| C15—C16 | 1.381 (5) | C56—H56 | 0.9300 |
| C15—H15 | 0.9300 | C57—H57 | 0.9300 |
| C16—C17 | 1.358 (5) | C58—C63 | 1.376 (4) |
| C16—H16 | 0.9300 | C58—C59 | 1.393 (4) |
| C17—C18 | 1.368 (5) | C59—C60 | 1.381 (5) |
| C17—H17 | 0.9300 | C59—H59 | 0.9300 |
| C18—C19 | 1.373 (5) | C60—C61 | 1.370 (6) |
| C18—H18 | 0.9300 | C60—H60 | 0.9300 |
| C19—H19 | 0.9300 | C61—C62 | 1.364 (6) |
| C20—C21 | 1.376 (4) | C61—H61 | 0.9300 |
| C20—C25 | 1.383 (4) | C62—C63 | 1.383 (6) |
| C21—C22 | 1.385 (5) | C62—H62 | 0.9300 |
| C21—H21 | 0.9300 | C63—H63 | 0.9300 |
| C22—C23 | 1.354 (5) | C64—C65 | 1.482 (4) |
| C22—H22 | 0.9300 | C65—C66 | 1.377 (4) |
| C23—C24 | 1.366 (5) | C65—C70 | 1.381 (4) |
| C23—H23 | 0.9300 | C66—C67 | 1.377 (5) |
| C24—C25 | 1.386 (4) | C66—H66 | 0.9300 |
| C24—H24 | 0.9300 | C67—C68 | 1.356 (5) |
| C25—H25 | 0.9300 | C67—H67 | 0.9300 |
| C26—C27 | 1.485 (5) | C68—C69 | 1.361 (5) |
| C27—C28 | 1.366 (5) | C68—H68 | 0.9300 |
| C27—C32 | 1.380 (5) | C69—C70 | 1.376 (5) |
| C28—C29 | 1.390 (5) | C69—H69 | 0.9300 |
| C28—H28 | 0.9300 | C70—H70 | 0.9300 |
| C29—C30 | 1.347 (6) | C71—C72 | 1.376 (4) |
| C29—H29 | 0.9300 | C71—C76 | 1.385 (4) |
| C30—C31 | 1.346 (6) | C72—C73 | 1.376 (5) |
| C30—H30 | 0.9300 | C72—H72 | 0.9300 |
| C31—C32 | 1.381 (5) | C73—C74 | 1.359 (5) |
| C31—H31 | 0.9300 | C73—H73 | 0.9300 |

| | | | |
|---|---|---|---|
| C32—H32 | 0.9300 | C74—C75 | 1.377 (6) |
| C33—C34 | 1.377 (4) | C74—H74 | 0.9300 |
| C33—C38 | 1.378 (4) | C75—C76 | 1.370 (5) |
| C34—C35 | 1.377 (5) | C75—H75 | 0.9300 |
| C34—H34 | 0.9300 | C76—H76 | 0.9300 |
| C35—C36 | 1.365 (5) | C77—H77A | 0.9700 |
| C35—H35 | 0.9300 | C77—H77B | 0.9700 |
| | | | |
| C1—O1—H1 | 109.5 | O4—C39—C45 | 111.1 (3) |
| O2—O2—C13 | 0(10) | C44—C39—C45 | 112.5 (3) |
| C39—O4—H4 | 109.5 | O4—C39—C40 | 110.4 (2) |
| O5—O5—C51 | 0(10) | C44—C39—C40 | 108.2 (2) |
| O1—C1—C6 | 105.1 (2) | C45—C39—C40 | 109.5 (2) |
| O1—C1—C7 | 111.3 (2) | C51—C40—C41 | 109.1 (2) |
| C6—C1—C7 | 110.4 (2) | C51—C40—C39 | 108.7 (2) |
| O1—C1—C2 | 110.2 (2) | C41—C40—C39 | 112.2 (2) |
| C6—C1—C2 | 109.2 (2) | C51—C40—H40 | 109.0 |
| C7—C1—C2 | 110.5 (2) | C41—C40—H40 | 109.0 |
| C13—C2—C3 | 108.6 (2) | C39—C40—H40 | 109.0 |
| C13—C2—C1 | 110.7 (2) | C58—C41—C40 | 112.3 (2) |
| C3—C2—C1 | 111.3 (2) | C58—C41—C42 | 110.0 (2) |
| C13—C2—H2 | 108.7 | C40—C41—C42 | 111.5 (2) |
| C3—C2—H2 | 108.7 | C58—C41—H41 | 107.6 |
| C1—C2—H2 | 108.7 | C40—C41—H41 | 107.6 |
| C20—C3—C4 | 110.7 (2) | C42—C41—H41 | 107.6 |
| C20—C3—C2 | 112.2 (2) | C64—C42—C41 | 109.4 (2) |
| C4—C3—C2 | 112.2 (2) | C64—C42—C43 | 107.6 (2) |
| C20—C3—H3 | 107.1 | C41—C42—C43 | 113.0 (2) |
| C4—C3—H3 | 107.1 | C64—C42—H42 | 108.9 |
| C2—C3—H3 | 107.1 | C41—C42—H42 | 108.9 |
| C26—C4—C3 | 108.1 (2) | C43—C42—H42 | 108.9 |
| C26—C4—C5 | 108.5 (2) | C71—C43—C44 | 113.1 (3) |
| C3—C4—C5 | 113.0 (2) | C71—C43—C42 | 109.0 (2) |
| C26—C4—H4A | 109.0 | C44—C43—C42 | 111.0 (2) |
| C3—C4—H4A | 109.0 | C71—C43—H43 | 107.9 |
| C5—C4—H4A | 109.0 | C44—C43—H43 | 107.9 |
| C33—C5—C6 | 113.3 (3) | C42—C43—H43 | 107.9 |
| C33—C5—C4 | 110.9 (2) | C39—C44—C43 | 112.3 (3) |
| C6—C5—C4 | 110.0 (2) | C39—C44—H44A | 109.2 |
| C33—C5—H5 | 107.5 | C43—C44—H44A | 109.2 |
| C6—C5—H5 | 107.5 | C39—C44—H44B | 109.2 |
| C4—C5—H5 | 107.5 | C43—C44—H44B | 109.2 |
| C1—C6—C5 | 112.4 (3) | H44A—C44—H44B | 107.9 |
| C1—C6—H6A | 109.1 | C50—C45—C46 | 118.2 (3) |
| C5—C6—H6A | 109.1 | C50—C45—C39 | 120.9 (3) |
| C1—C6—H6B | 109.1 | C46—C45—C39 | 120.8 (3) |
| C5—C6—H6B | 109.1 | C45—C46—C47 | 120.1 (4) |
| H6A—C6—H6B | 107.8 | C45—C46—H46 | 119.9 |
| C12—C7—C8 | 118.2 (3) | C47—C46—H46 | 119.9 |
| C12—C7—C1 | 120.7 (3) | C48—C47—C46 | 120.9 (4) |

| | | | |
|---|---|---|---|
| C8—C7—C1 | 121.1 (3) | C48—C47—H47 | 119.5 |
| C9—C8—C7 | 121.1 (3) | C46—C47—H47 | 119.5 |
| C9—C8—H8 | 119.5 | C47—C48—C49 | 119.3 (4) |
| C7—C8—H8 | 119.5 | C47—C48—H48 | 120.4 |
| C10—C9—C8 | 120.1 (4) | C49—C48—H48 | 120.4 |
| C10—C9—H9 | 120.0 | C48—C49—C50 | 120.3 (4) |
| C8—C9—H9 | 120.0 | C48—C49—H49 | 119.9 |
| C11—C10—C9 | 119.3 (3) | C50—C49—H49 | 119.9 |
| C11—C10—H10 | 120.4 | C45—C50—C49 | 121.1 (4) |
| C9—C10—H10 | 120.4 | C45—C50—H50 | 119.4 |
| C10—C11—C12 | 121.2 (3) | C49—C50—H50 | 119.4 |
| C10—C11—H11 | 119.4 | O5—C51—O5 | 0.0 (2) |
| C12—C11—H11 | 119.4 | O5—C51—C52 | 120.0 (3) |
| C7—C12—C11 | 120.2 (3) | O5—C51—C52 | 120.0 (3) |
| C7—C12—H12 | 119.9 | O5—C51—C40 | 117.3 (3) |
| C11—C12—H12 | 119.9 | O5—C51—C40 | 117.3 (3) |
| O2—C13—O2 | 0.0 (2) | C52—C51—C40 | 122.7 (3) |
| O2—C13—C14 | 119.7 (3) | C53—C52—C57 | 118.1 (3) |
| O2—C13—C14 | 119.7 (3) | C53—C52—C51 | 124.1 (3) |
| O2—C13—C2 | 117.5 (3) | C57—C52—C51 | 117.8 (3) |
| O2—C13—C2 | 117.5 (3) | C54—C53—C52 | 120.8 (4) |
| C14—C13—C2 | 122.8 (3) | C54—C53—H53 | 119.6 |
| C15—C14—C19 | 118.7 (3) | C52—C53—H53 | 119.6 |
| C15—C14—C13 | 123.7 (3) | C55—C54—C53 | 120.4 (4) |
| C19—C14—C13 | 117.6 (3) | C55—C54—H54 | 119.8 |
| C14—C15—C16 | 120.4 (4) | C53—C54—H54 | 119.8 |
| C14—C15—H15 | 119.8 | C56—C55—C54 | 119.6 (4) |
| C16—C15—H15 | 119.8 | C56—C55—H55 | 120.2 |
| C17—C16—C15 | 120.5 (4) | C54—C55—H55 | 120.2 |
| C17—C16—H16 | 119.8 | C55—C56—C57 | 120.9 (4) |
| C15—C16—H16 | 119.8 | C55—C56—H56 | 119.6 |
| C16—C17—C18 | 119.9 (4) | C57—C56—H56 | 119.6 |
| C16—C17—H17 | 120.1 | C56—C57—C52 | 120.2 (4) |
| C18—C17—H17 | 120.1 | C56—C57—H57 | 119.9 |
| C17—C18—C19 | 120.6 (4) | C52—C57—H57 | 119.9 |
| C17—C18—H18 | 119.7 | C63—C58—C59 | 116.9 (3) |
| C19—C18—H18 | 119.7 | C63—C58—C41 | 121.7 (3) |
| C18—C19—C14 | 119.9 (4) | C59—C58—C41 | 121.4 (3) |
| C18—C19—H19 | 120.1 | C60—C59—C58 | 121.9 (3) |
| C14—C19—H19 | 120.1 | C60—C59—H59 | 119.0 |
| C21—C20—C25 | 117.8 (3) | C58—C59—H59 | 119.0 |
| C21—C20—C3 | 120.9 (3) | C61—C60—C59 | 119.5 (4) |
| C25—C20—C3 | 121.4 (3) | C61—C60—H60 | 120.2 |
| C20—C21—C22 | 120.8 (3) | C59—C60—H60 | 120.2 |
| C20—C21—H21 | 119.6 | C62—C61—C60 | 119.6 (4) |
| C22—C21—H21 | 119.6 | C62—C61—H61 | 120.2 |
| C23—C22—C21 | 120.7 (4) | C60—C61—H61 | 120.2 |
| C23—C22—H22 | 119.6 | C61—C62—C63 | 120.7 (4) |
| C21—C22—H22 | 119.6 | C61—C62—H62 | 119.6 |

| | | | |
|---|---|---|---|
| C22—C23—C24 | 119.7 (4) | C63—C62—H62 | 119.6 |
| C22—C23—H23 | 120.2 | C58—C63—C62 | 121.2 (4) |
| C24—C23—H23 | 120.2 | C58—C63—H63 | 119.4 |
| C23—C24—C25 | 120.0 (4) | C62—C63—H63 | 119.4 |
| C23—C24—H24 | 120.0 | O6—C64—C65 | 119.9 (3) |
| C25—C24—H24 | 120.0 | O6—C64—C42 | 117.9 (3) |
| C20—C25—C24 | 121.0 (3) | C65—C64—C42 | 122.2 (3) |
| C20—C25—H25 | 119.5 | C66—C65—C70 | 117.3 (3) |
| C24—C25—H25 | 119.5 | C66—C65—C64 | 124.3 (3) |
| O3—C26—C27 | 120.0 (3) | C70—C65—C64 | 118.4 (3) |
| O3—C26—C4 | 118.0 (3) | C65—C66—C67 | 121.3 (4) |
| C27—C26—C4 | 121.9 (3) | C65—C66—H66 | 119.4 |
| C28—C27—C32 | 117.1 (3) | C67—C66—H66 | 119.4 |
| C28—C27—C26 | 124.7 (3) | C68—C67—C66 | 120.0 (4) |
| C32—C27—C26 | 118.2 (3) | C68—C67—H67 | 120.0 |
| C27—C28—C29 | 121.5 (4) | C66—C67—H67 | 120.0 |
| C27—C28—H28 | 119.3 | C67—C68—C69 | 120.3 (4) |
| C29—C28—H28 | 119.3 | C67—C68—H68 | 119.8 |
| C30—C29—C28 | 120.1 (5) | C69—C68—H68 | 119.8 |
| C30—C29—H29 | 119.9 | C68—C69—C70 | 119.6 (4) |
| C28—C29—H29 | 119.9 | C68—C69—H69 | 120.2 |
| C31—C30—C29 | 119.6 (4) | C70—C69—H69 | 120.2 |
| C31—C30—H30 | 120.2 | C69—C70—C65 | 121.5 (3) |
| C29—C30—H30 | 120.2 | C69—C70—H70 | 119.3 |
| C30—C31—C32 | 121.0 (4) | C65—C70—H70 | 119.3 |
| C30—C31—H31 | 119.5 | C72—C71—C76 | 117.8 (3) |
| C32—C31—H31 | 119.5 | C72—C71—C43 | 121.9 (3) |
| C27—C32—C31 | 120.7 (4) | C76—C71—C43 | 120.1 (3) |
| C27—C32—H32 | 119.7 | C73—C72—C71 | 120.9 (4) |
| C31—C32—H32 | 119.7 | C73—C72—H72 | 119.5 |
| C34—C33—C38 | 117.3 (3) | C71—C72—H72 | 119.5 |
| C34—C33—C5 | 122.1 (3) | C74—C73—C72 | 120.4 (4) |
| C38—C33—C5 | 120.6 (3) | C74—C73—H73 | 119.8 |
| C35—C34—C33 | 121.8 (4) | C72—C73—H73 | 119.8 |
| C35—C34—H34 | 119.1 | C73—C74—C75 | 119.8 (4) |
| C33—C34—H34 | 119.1 | C73—C74—H74 | 120.1 |
| C36—C35—C34 | 119.6 (4) | C75—C74—H74 | 120.1 |
| C36—C35—H35 | 120.2 | C76—C75—C74 | 119.6 (4) |
| C34—C35—H35 | 120.2 | C76—C75—H75 | 120.2 |
| C35—C36—C37 | 120.0 (4) | C74—C75—H75 | 120.2 |
| C35—C36—H36 | 120.0 | C75—C76—C71 | 121.4 (4) |
| C37—C36—H36 | 120.0 | C75—C76—H76 | 119.3 |
| C36—C37—C38 | 119.9 (4) | C71—C76—H76 | 119.3 |
| C36—C37—H37 | 120.0 | Cl2—C77—Cl1 | 107.9 (4) |
| C38—C37—H37 | 120.0 | Cl2—C77—H77A | 110.1 |
| C37—C38—C33 | 121.4 (4) | Cl1—C77—H77A | 110.1 |
| C37—C38—H38 | 119.3 | Cl2—C77—H77B | 110.1 |
| C33—C38—H38 | 119.3 | Cl1—C77—H77B | 110.1 |
| O4—C39—C44 | 105.1 (3) | H77A—C77—H77B | 108.4 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O2 | 0.82 | 2.12 | 2.701 (4) | 128 |
| O4—H4···O5 | 0.82 | 2.19 | 2.759 (4) | 127 |

**Fig. 1**