Supplementary Material (ESI) for Chemical Communications


# Supporting Information

## for

# Coupling-isomerization-Claisen sequences – Mechanistic dichotomies in hetero domino reactions

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## SUPPORTING INFORMATION

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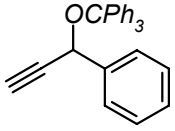
## I. Experimental section

General Considerations. All reactions involving water-sensitive compounds were carried out in oven-dried Schlenk glassware under a nitrogen atmosphere. The solvents were dried according to standard procedures[1] and were distilled prior to use. Column chromatography: silica gel 60 M (mesh 230-400) Macherey-Nagel. Thin layer chromatography (TLC): silica gel layered aluminium foil (60 $F_{254}$ Merck, Darmstadt). Melting points (uncorrected): Reichert-Jung Thermovar and Büchi Melting Point B-540. (Hetero)aryl halides **1** and Acid chlorides **5** were purchased from ACROS or Merck and used without further purification. Trityl propargyl ethers **2** were prepared from propargyl alcohols obtained by literature procedures.[2] $^{1}H$ and $^{13}C$ NMR spectra: Bruker ARX250, Bruker DRX 300 with $CDCl_3$ or as solvent. The assignments of quaternary C, CH, $CH_2$ and $CH_3$ were made on the basis of DEPT spectra. IR: Bruker Vector 22 FT-IR. UV/Vis: Hewlett Packard HP8452 A. MS: Jeol JMS-700 und Finnigan TSQ 700. Elemental analyses were carried out in the microanalitycal laboratory of the Department Chemie der Universität Heidelberg.

### General procedure for the preparation of 1-(hetero)aryl propargyltriphenylmethylethers 2

To a solution of 1.0 equiv of the propargyl alcohol in dichlormethane were successively added 10 mol% of DMAP, 1.5 equiv of pyridine, and 1.1 equiv of triphenylmethylchloride at room temp. Then the reaction mixture was heated to reflux temperature for 1 d. After cooling to room temp water was carefully added. The aqueous layer was extracted three times with dichloromethane. The combined organic layers were washed three times with brine and then dried with magnesium sulphate. After evaporation of the solvent in vacuo the residue was chromatographed on silica gel (hexane/ethylacetate 5:1) and crystallized from pentane/diethylether at –18 °C to give the 1-(hetero)aryl propargyltriphenylmethylethers **2** in good yields as crystalline solids.

### (1-Triphenylmethoxyprop-2-ynyl)-benzene (2a)

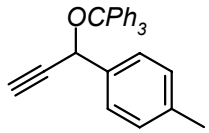



$C_{28}H_{22}O$
374.49
**2a**

$^{1}H$ NMR (300 MHz, $CDCl_3$): $\delta$ 2.29 (d, $J$ = 2.2 Hz, 1H), 5.13 (d, $J$ = 2.2 Hz, 1 H), 7.25-7.36 (m, 14 H), 7.56-7.57 (m, 3 H), 7.58-7.59 (m, 3H). $^{13}C$ NMR (75 MHz, $CDCl_3$): $\delta$ 66.5 (CH), 74.4 (CH), 83.6 ($C_{quat}$), 88.9 ($C_{quat}$), 126.6 (CH), 127.1 (CH), 127.7 (CH), 129.1 (CH), 140.7 ($C_{quat}$),

144.1 ($C_{quat}$). EI MS (70 eV, *m/z* (%)): 374 ($M^+$, 0.5), 244(($(C_6H_5)_3CH^+$, 84), 243 ($(C_6H_5)_3C^+$, 100), 115 ($M^+$-$(C_6H_5)_3CO$, 70). IR (KBr): $\tilde{\nu}$ = 3311 $cm^{-1}$ (m), 3289 (m), 3056 (w), 3032 (m), 1491 (s), 1448 (s), 1125 (m), 1040 (s), 1025 (s), 1003 (m), 993 (m), 773 (m), 745 (m), 709 (s), 696 (s), 657 (m), 646 (m). UV/Vis ($CH_2Cl_2$): $\lambda_{max}$ ($\varepsilon$) 260 nm (1700). Anal. calcd. for $C_{28}H_{22}O$ (374.5): C 89.81, H 5.92. Found: C 89.51, H 5.92.

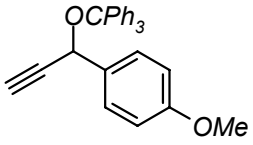
**1-Methyl-4-(1-trityloxyprop-2-ynyl)benzene (2b)**




$C_{29}H_{24}O$
388.51
**2b**

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.21 (d, *J* = 2.1 Hz, 1 H), 2.36 (s, 3 H), 5.08 (d, *J* = 2.1 Hz, 1 H), 7.12 (d, *J* = 8.3 Hz, 2 H), 7.24-7.32 (m, 11 H), 7.55-7.59 (m, 6 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 21.1 ($CH_3$), 66.3 (CH), 74.1 (CH), 83.7 ($C_{quat}$), 88.8 ($C_{quat}$), 126.6 (CH), 127.1 (CH), 127.7 (CH), 128.9 (CH), 129.1 (CH), 137.3 ($C_{quat}$), 137.8 ($C_{quat}$), 144.1 ($C_{quat}$). FAB MS (*m/z*): 411 ($M^+$+Na). FD MS (*m/z*): 388 ($M^+$). IR (KBr): $\tilde{\nu}$ = 3296 $cm^{-1}$ (s), 3022 (w), 3058 (w), 3031 (w), 2920 (w), 2890 (w), 2111 (w), 1613 (w), 1593 (m), 1491 (s), 1448 (s), 1037 (s), 821 (m), 704 (s), 631 (s). UV/Vis ($CH_2Cl_2$): $\lambda_{max}$ ($\varepsilon$) 262 nm (1200). Anal. calcd. for $C_{29}H_{24}O$ (388.5): C 89.66, H 6.23. Found: C 88.56, H 6.26.

**1-Methoxy-4-(1-triphenylmethoxyprop-2-ynyl)benzene (2c)**

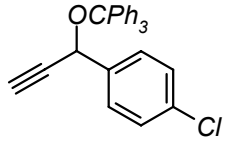



$C_{29}H_{24}O_2$
404.51
**2c**

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.25 (d, *J* = 2.2 Hz, 1 H), 3.81 (s, 3 H), 5.06 (d, *J* = 2.2 Hz, 1 H), 6.82 (d, *J* = 8.8 Hz, 2 H), 7.24-7.32 (m, 10 H), 7.54-7.57 (m, 7 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 55.1 ($CH_3$), 65.9 (CH), 73.9 (CH), 83.6 ($C_{quat}$), 88.6 ($C_{quat}$), 113.5 (CH), 126.9 (CH), 127.5 (CH), 127.7 (CH), 128.9 (CH), 132.9 ($C_{quat}$), 144.0 ($C_{quat}$), 158.9 ($C_{quat}$). EI MS (70 eV, *m/z* (%)): 404 ($M^+$, 4), 260 ($(C_6H_5)_3COH^+$, 15), 259 ($(C_6H_5)_3CO^+$, 75), 245 (100), 244 ($(C_6H_5)_3CH^+$, 100), 243 ($(C_6H_5)_3C^+$, 100). HRMS calcd. for $C_{29}H_{24}O_2$: 404.1776, Found: 404.1801. IR (KBr): $\tilde{\nu}$ = 3275 $cm^{-1}$ (s), 3085 (w), 3060 (w), 2905 (w), 2867 (w), 2835 (w), 2111 (w), 1611 (s), 1510 (s), 1491 (m), 1463 (s), 1449 (m), 1246 (s), 1171 (m), 1154 (m), 1053 (s), 1028 (s), 830 (m), 703

(s). UV/Vis ($CH_2Cl_2$): $\lambda_{max}$ (ε) 270 nm (1700), 280 (1300). Anal. calcd. for $C_{29}H_{24}O_2$ (404.5): C 86.11, H 5.98. Found: C 85.86, H 5.99.

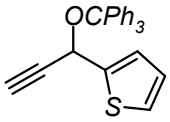
**1-Chloro-4-(1-triphenylmethoxyprop-2-ynyl)benzene (2d)**




$C_{28}H_{21}ClO$
408.93
**2d**

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.33 (d, $J$ = 2.2 Hz, 1 H), 5.08 (d, $J$ = 2.2 Hz, 1 H), 7.21 (s, 4 H), 7.23-7.31 (m, 9 H), 7.52-7.53 (m, 3 H), 7.54-7.55 (m, 3 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 65.7 (CH), 74.6 (CH), 83.0 ($C_{quat}$), 88.9 ($C_{quat}$) 127.1 (CH), 127.6 (CH), 127.8 (CH), 128.1 (CH), 128.8 (CH), 133.1 ($C_{quat}$), 139.1 ($C_{quat}$), 143.7 ($C_{quat}$). EI MS (70 eV, *m/z* (%)): 409 ($M^+$ ($^{37}$Cl), 0.7), 407 ($M^+$ ($^{35}$Cl), 0.3), 244 (($(C_6H_5)_3CH^{+\cdot}$, 71), 243 (($(C_6H_5)_3C^+$, 80), 165 ($M^+$-$(C_6H_5)_3C$, 31), 149 ($M^+$-$(C_6H_5)_3CO$, 42). IR (KBr): $\tilde{\nu}$ = 3296 cm$^{-1}$ (m), 3278 (m), 3082 (w), 3058 (w), 3033 (w), 2111 (w), 1593 (m), 1491 (s), 1448 (s), 1090 (s), 1014 (m), 703 (s). UV/Vis ($CH_2Cl_2$): $\lambda_{max}$ (ε) 260 nm (5400), 286 (1700). Anal. calcd. for $C_{28}H_{21}ClO$ (408.9): C 82.24, H 5.18, Cl 8.67. Found: C 82.02, H 5.17, Cl 8.53.

**2-(1-Trityloxyprop-2-ynyl)thiophene (2e)**




$C_{26}H_{20}OS$
380.51
**2e**

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.28 (d, $J$ = 2.2 Hz, 1 H), 5.37 (dd, $J$ = 2.2 Hz, $J$ = 0.7 Hz, 1 H), 6.82 (m, 1 H), 6.88 (dd, $J$ = 5.5 Hz, $J$ = 3.5 Hz, 1 H), 7.25-7.34 (m, 10 H), 7.57-7.61 (m, 6 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 62.3 (CH), 74.4 (CH), 82.5 ($C_{quat}$), 89.0 ($C_{quat}$), 124.8 (CH), 125.4 (CH), 126.1 (CH), 127.2 (CH), 127.7 (CH), 129.1 (CH), 143.9 ($C_{quat}$), 143.9 ($C_{quat}$). FAB MS (*m/z*): 404 ($M^+$+Na). FD MS (*m/z*): 380 ($M^+$). IR (KBr): $\tilde{\nu}$ = 3309 cm$^{-1}$ (s), 3288 (s), 3057 (s), 2216 (w), 1958 (m), 1593 (m), 1491 (s), 1448 (s), 1312 (m), 1279 (m), 1171 (m), 1155 (m), 1023 (s), 772 (s), 698 (s). UV/Vis ($CH_2Cl_2$): $\lambda_{max}$ (ε) 238 nm (14500). Anal. calcd. for $C_{26}H_{20}OS$ (380.5): C 82.07, H 5.30, S 8.43. Found: C 82.04, H 5.33, S 8.44.

**General procedure for the coupling-iosmerization-Claisen sequences to give the products 3, 4, 6, or 7**

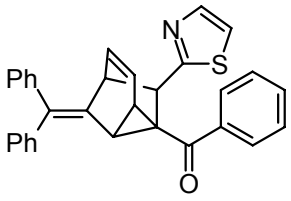
**Synthesis:** To a deaerated mixture of 6 mL of butyronitrile and 6 mL of triethylamine in an oven dried screw cap vessel were added successively 1.50 mmol of the electron deficient halide **8** (**1** or **5**), 1.65 mmol of the 1-aryl propargyl trityl ethers **2**, 53 mg (0.08 mmol) of $PdCl_2(PPh_3)_2$, and 14 mg (0.07 mmol) of CuI (For experimental details, see Table 1). The resulting mixture was stirred at room temperature for 1 h, and than heated at reflux temperature for 16-72 h. The reaction mixture was allowed to cool to room temperature, the solvents were removed in vacuo and the residue was chromatgraphed on silica gel (hexane/ethyl acetate, 2:1) and crystallized from pentane/diethylether to afford the products **3**, **4**, **6**, or **7** as colorless crystals.

Table 1. Experimental details of CI-Claisen sequences

| Entry | Electron deficient halide **8** (**1** or **5**) | Propargyl trityl ether **2** | Yield |
|---|---|---|---|
| 1[a] | 246 mg (1.50 mmol) of **1a** | 618 mg (1.65 mmol) of **2a** | 593 mg (87 %) of **3a** |
| 2[a] | 303 mg (1.50 mmol) of **1b** | 618 mg (1.65 mmol) of **2a** | 683 mg (91 %) of **3b** |
| 3[b] | 408 mg (1.50 mmol) of **1c** | 618 mg (1.65 mmol) of **2a** | 757 mg (97 %) of **3c** |
| 4[b] | 408 mg (1.50 mmol) of **1c** | 675 mg (1.65 mmol) of **2b** | 580 mg (76 %) of **3d** |
| 5[b] | 408 mg (1.50 mmol) of **1c** | 641 mg (1.65 mmol) of **2c** | 796 mg (99 %) of **3e** |
| 6[b] | 408 mg (1.50 mmol) of **1c** | 675 mg (1.65 mmol) of **2d** | 836 mg (99 %) of **3f** |
| 7[c] | 393 mg (1.50 mmol) of **1d** | 628 mg (1.65 mmol) of **2e** | 710 mg (92 %) of **3g** |
| 8[c] | 344 mg (1.50 mmol) of **1e** | 628 mg (1.65 mmol) of **2e** | 590 mg (82 %) of **3h** |
| 9[b] | 344 mg (1.50 mmol) of **1e** | 641 mg (1.65 mmol) of **2c** | 364 mg (50 %) of **4a** |
| 10[b] | 344 mg (1.50 mmol) of **1e** | 675 mg (1.65 mmol) of **2d** | 580 mg (76 %) of **4b** |
| 11[b] | 211 mg (1.50 mmol) of **5a** | 618 mg (1.65 mmol) of **2a** | 638 mg (89 %) of **6a** |
| 12[a] | 220 mg (1.50 mmol) of **5b** | 618 mg (1.65 mmol) of **2a** | 443 mg (61 %) of **6b** |
| 13[a] | 220 mg (1.50 mmol) of **5b** | 628 mg (1.65 mmol) of **2e** | 650 mg (88 %) of **6c** |
| 14[c] | 256 mg (1.50 mmol) of **5c** | 618 mg (1.65 mmol) of **2a** | 549 mg (72 %) of **7a** |
| 15[c] | 256 mg (1.50 mmol) of **5c** | 675 mg (1.65 mmol) of **2d** | 537 mg (66 %) of **7b** |

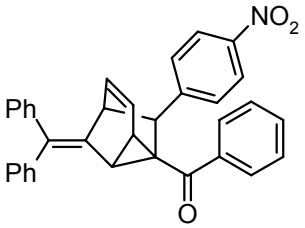
[a]Reaction for 16 h. [b]Reaction for 72 h. [c]Reaction for 24 h.

**(6-Benzhydrylidene-8-thiazol-2-yl-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl)-phenyl methanone (3a)**



$^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 2.17 (s, 6 H, acetone), 2.93 (dd, $J$ = 1.4, 7.6 Hz, 1 H), 3.24 (m, $J$ = 7.6 Hz, 1 H), 3.87 (m, $^3J$ = 5.1 Hz, $J$ = 1.4, 2.3 Hz, 1 H), 4.43 (d, $^3J$ = 5.1 Hz, 1 H, CH), 5.79 (dt, $J$ = 2.3, 7.0 Hz, 1 H), 6.25 (ddd, $J$ = 1.4, 5.6, 8.0 Hz, 1 H), 7.07 (d, $J$ = 3.3 Hz, 1 H), 7.23–7.36 (m, 11 H), 7.41 (m, 1 H), 7.47 (m, 1 H), 7.53 (d, $J$ = 3.3 Hz, 1 H), 7.71 (m, 2 H). $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 27.6 (CH), 30.9 ($CH_3$, acetone), 34.0 (CH), 44.7 ($C_{quat}$), 44.9 (CH), 45.7 (CH), 118.5 (CH), 125.1 (CH), 126.8 (CH), 127.1 (CH), 127.3 (CH), 127.6 (CH), 128.1 (CH), 128.2 (CH), 128.4 (CH), 129.7 (CH), 129.8 (CH), 131.9 (CH), 136.4 ($C_{quat}$), 136.6 ($C_{quat}$), 138.5 ($C_{quat}$), 141.6 ($C_{quat}$), 141.6 ($C_{quat}$), 141.8 (CH), 168.3 ($C_{quat}$), 199.6 ($C_{quat}$), 206.9 ($C_{quat}$, acetone). EI MS (70 eV), *m/z* (%): 457 ($M^+$, 100), 352 ($M^+ - COC_6H_5$, 81). HRMS calcd for $C_{31}H_{23}NOS$: 457.1500, found: 457.1515. Anal. calcd. for $C_{31}H_{23}NOS \times C_3H_6O$ (515.7): C 79.19, H 5.67, N 2.72, S 6.22; found: C 79.93, H 5.61, N 2.81, S 6.06.

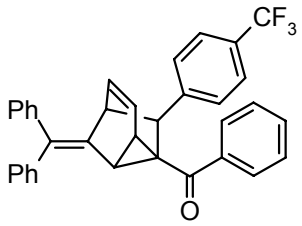
**[6-Benzhydrylidene-8-(4-nitro-phenyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl]-phenyl methanone (3b)**




$^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 2.17 (s, 6 H, acetone), 2.94 (dd, $J$ = 1.1, 7.6 Hz, 1 H), 3.24 (m, 1 H), 3.64 (m, 1 H), 4.15 (d, $J$ = 5.0 Hz, 1 H), 5.59 (m, 1 H), 6.25 (m, 1 H), 7.19 (d, $J$ = 8.6 Hz, 2 H), 7.25–7.29 (m, 5 H), 7.32–7.35 (m, 3 H), 7.37 (d, $J$ = 7.5 Hz, 2 H), 7.39–7.42 (m, 2 H), 7.49 (m, 1 H), 7.70 (m, 2 H), 7.98 (d, $J$ = 8.7 Hz, 2 H). $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 27.5 (CH), 30.9 ($CH_3$, acetone), 33.2 (CH), 43.3 ($C_{quat}$), 45.9 (CH), 46.8 (CH), 123.0 (CH), 125.2 (CH), 126.1 (CH), 127.2 (CH), 127.4 (CH), 127.5 (CH), 128.2 (CH), 128.3 (CH), 128.5 (CH), 129.1 (CH), 129.7 (CH), 129.8 (CH), 132.3 (CH), 136.3 ($C_{quat}$), 137.0 ($C_{quat}$), 138.2 ($C_{quat}$), 141.6 ($C_{quat}$), 141.6 ($C_{quat}$), 146.2 ($C_{quat}$), 146.5 ($C_{quat}$), 199.9 ($C_{quat}$), 206.8 ($C_{quat}$, acetone). EI MS

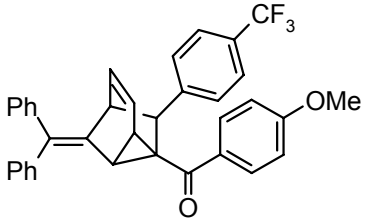
(70 eV), *m/z* (%): 495 ($M^+$, 69), 418 ($M^+ - C_6H_5$, 31), 390 (19), 360 (21), 359 (27), 358 (16), 328 (25), 265 (20), 254 (49), 243 (40), 167 (18), 164 (71), 105 ($COC_6H_5$, 100), 77 ($C_6H_5$, 25). HRMS calcd. for $C_{34}H_{25}NO_3$: 495.1834, found: 495.1847. Anal. calcd. for $C_{34}H_{25}NO_3 \times C_3H_6O$ (553.7): C 80.27, H 5.64, N 2.53; found: C 80.36, H 5.44, N 2.70.

**[6-Benzhydrylidene-8-(4-trifluoromethyl-phenyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl]-phenyl methanone (3c)**



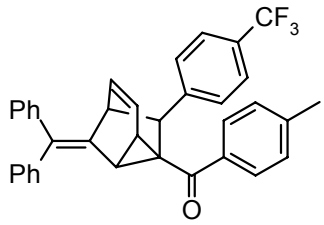

$^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 2.92 (dd, $J$ = 0.9, 7.6 Hz, 1 H), 3.24 (m, 1 H), 3.64 (dd, $J$ = 5.5, 6.4 Hz, 1 H), 4.15 (d, $J$ = 5.2 Hz, 1 H), 5.61 (m, 1 H), 6.25 (m, 1 H), 7.16 (d, $J$ = 7.9 Hz, 2 H), 7.28–7.30 (m, 5 H), 7.34 (d, $J$ = 7.3 Hz, 2 H), 7.37–7.43 (m, 7 H), 7.48–7.51 (m, 1 H), 7.74 (d, $J$ = 7.6 Hz, 2 H). $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 27.3 (CH), 33.2 (CH), 43.2 ($C_{quat}$), 45.8 (CH), 46.8 (CH), 124.1 (q, $^1J_{C\text{-}F}$ = 272.2 Hz, $C_{quat}$), 124.7 (q, $^3J_{C\text{-}F}$ = 3.8 Hz, CH), 125.1 (CH), 126.3 (CH), 127.1 (CH), 127.3 (CH), 127.6 (CH), 128.1 (CH), 128.3 (CH), 128.5 (q, $^2J_{C\text{-}F}$ = 33.0 Hz, $C_{quat}$), 128.5 (CH), 128.6 (CH), 129.8 (CH), 129.8 (CH), 132.2 (CH), 136.0 ($C_{quat}$), 137.6 ($C_{quat}$), 138.3 ($C_{quat}$), 141.7 ($C_{quat}$), 141.7 ($C_{quat}$), 142.4 (q, $^5J_{C\text{-}F}$ = 1.3 Hz, $C_{quat}$), 200.2 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 518 ($M^+$, 100), 441 ($M^+ - C_6H_5$, 36), 413 ($M^+ - COC_6H_5$, 45). HRMS calcd. for $C_{35}H_{25}F_3O$: 518.1858, found: 518.1838. Anal. calcd. for $C_{35}H_{25}F_3O$ (518.6): C 81.07, H 4.86; found: C 80.68, H 5.11.

**[6-Benzhydrylidene-8-(4-trifluoromethyl-phenyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl]-(4-methoxy-phenyl) methanone (3d)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.84 (dd, $J$ = 1.3, 7.6 Hz, 1 H), 3.14 (m, 1 H), 3.64 (m, 1 H), 3.85 (s, 3 H), 4.15 (d, $J$ = 4.8 Hz, 1 H), 5.59 (m, 1 H), 6.25 (ddd, $J$ = 1.3, 5.5, 6.9 Hz, 1 H), 6.92 (d, $J$ = 9.0 Hz, 2 H), 7.18 (d, $J$= 8.2 Hz, 2 H), 7.28–7.40 (m, 12 H), 7.84 (d, $J$ = 9.0 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 26.7 (CH), 32.0 (CH), 42.6 ($C_{quat}$), 45.7 (CH), 47.3 (CH), 55.2 ($CH_3$), 113.7 (CH), 124.2 (q, $^1J_{C\text{-}F}$ = 271.3 Hz, $C_{quat}$), 124.7 (q, $^3J_{C\text{-}F}$ = 3.8 Hz, CH), 125.3 (CH), 126.0 (CH), 127.1 (CH), 127.3 (CH), 128.1 (CH), 128.3 (CH), 128.5 (q, $^2J_{C\text{-}F}$ = 32.3 Hz, $C_{quat}$), 128.7 (CH), 129.8 (CH), 129.8 (CH), 130.4 (CH), 130.6 ($C_{quat}$), 135.8 ($C_{quat}$), 137.9 ($C_{quat}$), 141.8 ($C_{quat}$), 141.9 ($C_{quat}$), 142.5 (q, $^5J_{C\text{-}F}$ = 1.3 Hz, $C_{quat}$), 163.1 ($C_{quat}$), 197.8 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 548 ($M^+$, 32), 165 ($OCC_6H_4OCH_3^+$, 100). Anal. calcd. for $C_{36}H_{27}F_3O_2$ (548.6): C 78.82, H 4.96; found: C 78.63, H 5.01.

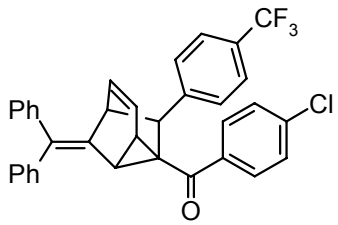
**[6-Benzhydrylidene-8-(4-trifluoromethyl-phenyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl]-*p*-tolyl methanone (3e)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.39 (s, 3 H), 2.89 (dd, $J$ = 1.3, 7.6 Hz, 1 H), 3.21 (m, 1 H), 3.66 (m, 1 H), 4.17 (d, $J$ = 4.9 Hz, 1 H), 5.61 (dq, $J$ = 1.3, 2.3 Hz, 1 H), 6.26 (dq, $J$ = 1.3, 2.4 Hz, 1 H), 7.18 (d, $J$ = 8.1 Hz, 2 H), 7.24 (d, $J$ = 7.9 Hz, 2 H), 7.29–7.41 (m, 12 H), 7.71 (d, $J$ = 8.1 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 21.6 ($CH_3$), 27.0 (CH), 32.7 (CH), 42.9 ($C_{quat}$), 45.8 (CH), 47.1 (CH), 124.1 (q, $^1J_{C\text{-}F}$ = 271.9 Hz, $C_{quat}$), 124.7 (q, $^3J_{C\text{-}F}$ = 3.7 Hz, CH), 125.1 (CH), 126.1 (CH), 127.1 (CH), 127.3 (CH), 127.9 (CH), 128.1 (CH), 128.2 (CH), 128.4 (q,

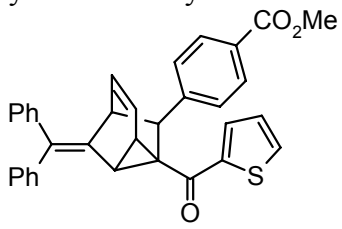
$^{2}J_{C\text{-}F}$ = 32.0 Hz, $C_{quat.}$), 128.6 (CH), 129.2 (CH), 129.8 (CH), 135.8 ($C_{quat}$), 137.7 ($C_{quat}$), 141.7 ($C_{quat}$), 141.8 ($C_{quat}$), 142.4 (q, $^{5}J_{C\text{-}F}$ = 1.4 Hz, $C_{quat}$), 143.2 ($C_{quat}$), 199.4[3] ($C_{quat}$). EI MS (70 eV), *m/z* (%): 532 ($M^+$, 100), 455 ($M^+ - C_6H_5$, 24), 413 ($M^+ - COC_6H_4CH_3$, 14). HRMS calcd. for $C_{36}H_{27}F_3O$: 532.2014, found: 532.2007. Anal. calcd. for $C_{36}H_{27}F_3O$ (532.6): C 81.19, H 5.11; found: C 81.16, H 5.12.

**[6-Benzhydrylidene-8-(4-trifluoromethyl-phenyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-1-yl]-(4-chloro-phenyl) methanone (3f)**



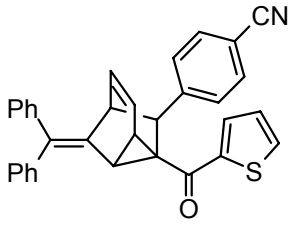

$^{1}$H NMR (300 MHz, $CDCl_3$): $\delta$2.88 (dd, *J* = 1.0, 7.6 Hz, 1 H), 3.22 (m, 1 H), 3.64 (m, 1 H), 4.07 (d, *J* = 4.8 Hz, 1 H), 5.60 (m, 1 H), 6.24 (m, 1 H), 7.14 (d, *J* = 7.8 Hz, 2 H), 7.26–7.40 (m, 14 H), 7.68 (d, *J* = 8.6 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$27.3 (CH), 33.2 (CH), 43.2 ($C_{quat}$), 45.8 (CH), 46.9 (CH), 124.1 (q, $^{1}J_{C\text{-}F}$ = 271.9 Hz, $C_{quat}$), 124.8 (CH), 124.8 (q, $^{3}J_{C\text{-}F}$ = 3.8 Hz, CH), 125.0 (CH), 126.3 (CH), 127.2 (CH), 127.4 (CH), 128.2 (CH), 128.3 (CH), 128.6 (CH), 128.7 (q, $^{2}J_{C\text{-}F}$ = 32.4 Hz, $C_{quat}$), 128.8 (CH), 129.1 (CH), 129.7 (CH), 136.2 ($C_{quat}$), 136.5 ($C_{quat}$), 137.3 ($C_{quat}$), 138.6 ($C_{quat}$), 141.6 ($C_{quat}$), 141.6 ($C_{quat}$), 142.1 (q, $^{5}J_{C\text{-}F}$ = 1.5 Hz, $C_{quat}$), 198.9 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 554 ($^{37}$Cl-$M^+$, 25), 552 ($^{35}$Cl-$M^+$, 45), 413 ($M^+ - COC_6H_4Cl$, 38). HRMS calcd. for $C_{35}H_{24}{}^{35}ClF_3O$: 552.1468, found: 552.1505. Anal. calcd. for $C_{35}H_{24}ClF_3O$ (553.0): C 76.02, H 4.37, Cl 6.41; found: C 75.93, H 4.50, Cl 6.37.

**Methyl 4-[8-Benzhydrylidene-7-(thiophene-2-carbonyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-6-yl]-benzoate (3g)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.95 (dd, $J$ = 1.4, 7.7 Hz, 1 H), 3.37 (m, 1 H), 3.65 (m, 1 H), 3.86 (s, 3 H), 4.30 (d, $J$ = 4.8 Hz, 1 H), 5.59 (m, 1 H), 6.23 (m, 1 H), 7.19 (s, 1 H), 7.02 (dd, $J$ = 4.0, 4.9 Hz, 1 H), 7.21–7.37 (m, 11 H), 7.54 (dd, $J$ = 0.9, 4.9 Hz, 1 H), 7.62 (dd, $J$ = 0.9, 3.9 Hz, 1 H), 7.86 (d, $J$ = 8.3 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 30.2 (CH), 34.9 (CH), 43.3 ($C_{quat}$), 46.3 (CH), 46.3 (CH), 51.9 ($CH_3$), 124.7 (CH), 126.5 (CH), 127.0 (CH), 127.2 (CH), 127.9 (CH), 128.1 (CH), 128.1 (CH), 128.3 ($C_{quat}$), 128.5 (CH), 129.2 (CH), 129.8 (CH), 129.9 (CH), 132.0 (CH), 133.1 (CH), 135.7 ($C_{quat}$), 137.2 ($C_{quat}$), 141.6 ($C_{quat}$), 141.8 ($C_{quat}$), 143.5 ($C_{quat}$), 143.8 ($C_{quat}$), 166.9 ($C_{quat}$), 190.5 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 514 ($M^+$, 100), 403 ($M^+ - COC_4H_3S$, 37), 379 ($M^+ - C_6H_4CO_2CH_3$, 10). HRMS calcd. for $C_{34}H_{26}O_3S$: 514.1603, found: 514.1583. Anal. calcd. for $C_{34}H_{26}O_3S$ (514.7): C 79.35, H 5.09, S 6.23; found: C 79.00, H 5.06, S 6.32.

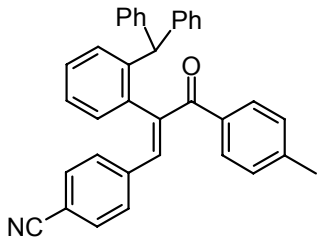
**4-[8-Benzhydrylidene-7-(thiophene-2-carbonyl)-tricyclo[3.2.1.0$^{2,7}$]oct-3-en-6-yl] benzonitrile (3h)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.98 (dd, $J$ = 7.7 Hz, $J$ = 1.5 Hz, 1 H), 3.33 (m, 1 H), 3.60 (m, 1 H), 4.25 (d, $J$ = 5.0 Hz, 1 H), 5.58 (ddd, $J$ = 6.9 Hz, $J$ = 5.7 Hz, $J$ = 2.2 Hz, 1 H), 6.22 (ddd, $J$ = 7.9 Hz, $J$ = 5.5 Hz, $J$ = 1.3 Hz, 1 H), 7.06 (dd, $J$ = 4.9 Hz, $J$ = 3.9 Hz, 1 H), 7.21-7.24 (m, 6 H), 7.28-7.37 (m, 6 H), 7.47 (d, $J$ = 8.3 Hz, 2 H), 7.57 (dd, $J$ = 5.0 Hz, $J$ = 1.0 Hz, 1 H), 7.62 (dd, $J$ = 3.9 Hz, $J$ = 1.0 Hz, 1 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 30.0 (CH), 34.2 (CH), 43.3

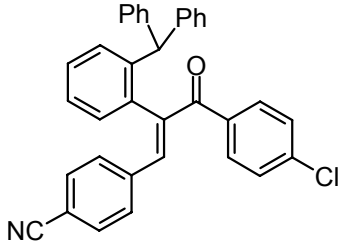
($C_{quat.}$), 46.1 (CH), 46.5 (CH), 110.2 ($C_{quat.}$), 118.9 ($C_{quat.}$), 124.8 (CH), 126.4 (CH), 127.1 (CH), 127.3 (CH), 128.0 (CH), 128.2 (CH), 128.2 (CH), 129.2 (CH), 129.7 (CH), 129.9 (CH), 131.7 (CH), 131.9 (CH), 133.3 (CH), 136.1 ($C_{quat.}$), 136.7 ($C_{quat.}$), 141.7 ($C_{quat.}$), 143.2 ($C_{quat.}$), 144.1 ($C_{quat.}$), 190.2 ($C_{quat.}$).[4] EI MS (70 eV), *m/z* (%): 481 ($M^+$, 100), 404 ($M^+$-$C_6H_5$, 17), 370 ($M^+$-$COC_4H_3S$, 35), 365 (32), 314 ($M^+$-$CH(C_6H_5)_2$, 30), 354 (41). HRMS calcd. for $C_{33}H_{23}NOS$: 481.1500, found: 481.1494.

**4-[(E)-2-(2-Benzhydryl-phenyl)-3-oxo-3-*p*-tolyl-propenyl] benzonitrile (4a)**



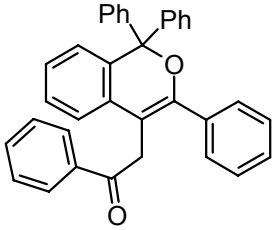

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.44 (s, 3 H), 5.53 (s, 1 H), 6.80 (d, $J$ = 8.2 Hz, 2 H), 6.90 (d, $J$ = 7.2 Hz, 2 H), 6.96 (d, $J$ = 7.2 Hz, 2 H), 7.08–7.18 (m, 7 H), 7.24 (m, 2 H), 7.27 (m, 4 H), 7.33 (dq, $J$ = 1.5, 7.5 Hz, 2 H), 7.71 (d, $J$ = 8.2 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 21.6 ($CH_3$), 53.8 (CH), 111.9 ($C_{quat}$), 118.4 ($C_{quat}$), 127.0 (CH), 128.1 (CH), 128.3 (CH),128.6 (CH), 129.1 (CH), 129.3 (CH), 129.7 (CH), 129.9 (CH), 130.3 (CH), 130.8 (CH), 131.6 (CH), 134.9 ($C_{quat}$), 136.0 ($C_{quat}$), 139.0 ($C_{quat}$), 140.1 (CH), 142.6 ($C_{quat}$), 142.9 ($C_{quat}$), 143.2 ($C_{quat}$), 143.6 ($C_{quat}$), 196.0 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 489 ($M^+$, 59), 370 ($M^+ - OCC_6H_4CH_3$, 11), 322 ($M^+ - CH(C_6H_5)_2$, 19), 119 ($OCC_6H_4CH_3^+$, 100). HRMS calcd. for $C_{36}H_{27}NO$: 489.2093, found: 489.2062. Anal. calcd. for $C_{36}H_{27}NO$ (489.6): C 88.31, H 5.56, N 2.86, found: C 88.00, H 5.62, N 2.90.

**4-[(E)-2-(2-Benzhydryl-phenyl)-3-(4-chloro-phenyl)-3-oxo-propenyl] benzonitrile (4b)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 5.46 (s, 1 H), 6.83–6.92 (m, 6 H), 7.07–7.19 (m, 8 H), 7.27–7.28 (m, 3 H), 7.31 (m, 1 H), 7.34 (dd, $J$ = 1.6, 7.5 Hz, 1 H), 7.39 (d, $J$ = 8.8 Hz, 2 H), 7.64 (d, $J$ = 8.8 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 53.9 (CH), 112.2 ($C_{quat}$), 118.3 ($C_{quat}$), 127.2 (CH), 128.1 (CH), 128.3 (CH), 128.6 (CH), 128.9 (CH), 129.2 (CH), 129.5 (CH), 130.4 (CH), 130.8 (CH), 130.9 (CH), 131.0 (CH), 131.7 (CH), 135.5 ($C_{quat}$), 135.8 ($C_{quat}$), 138.7 ($C_{quat}$), 138.7 ($C_{quat}$), 140.5 ($C_{quat}$), 142.5 ($C_{quat}$), 142.5 ($C_{quat}$), 195.0 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 509 ($^{35}$Cl-$M^+$, 100), 370 ($M^+$ – $C(O)C_6H_4Cl$, 29). HRMS calcd. for $C_{35}H_{24}{}^{35}ClNO$: 509.1546, found: 509.1558. Anal. calcd. for $C_{35}H_{24}ClNO$ (510.0): C 82.42, H 4.74, N 2.75, Cl 6.95, found: C 82.20, H 4.83, N 2.81,Cl 6.65.

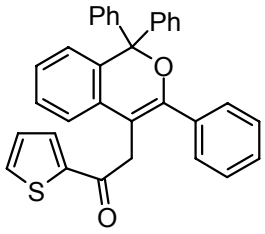
**1-Phenyl-2-(1,1,3-triphenyl-1*H*-isochromen-4-yl) ethanone (6a)**




$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 1.86 (m, 4 H, THF), 3.76 (m, 4 H, THF), 4.09 (s, 2 H), 6.63 (dd, $J$ = 0.9, 7.7 Hz, 1 H), 7.11 (dd, $J$ = 1.1, 7.5 Hz, 1 H), 7.16 (m, 1 H), 7.20 (m, 1 H), 7.23–7.29 (m, 11 H), 7.31 (m, 1 H), 7.33–7.35 (m, 3 H), 7.45–7.51 (m, 3 H), 7.76 (m, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 25.6 ($CH_2$, THF), 39.3 ($CH_2$), 67.9 ($CH_2$, THF), 86.8 ($C_{quat}$), 107.5 ($C_{quat}$), 122.0 (CH), 126.2 (CH), 127.3 (CH), 127.4 (CH), 127.7 (CH), 128.1 (CH), 128.1 (CH), 128.2 ($C_{quat}$), 128.3 (CH), 129.0 (CH), 129.1 (CH), 129.3 (CH), 132.3 (CH), 132.9 (CH), 133.2 ($C_{quat}$), 135.0 ($C_{quat}$), 136.2 ($C_{quat}$), 144.2 ($C_{quat}$), 151.4 ($C_{quat}$), 198.2 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 478 ($M^+$, 35), 373 ($M^+$ – $C(O)C_6H_5$, 28), 359 ($M^+$ – $CH_2COC_6H_5$, 20), 358 (57), 105

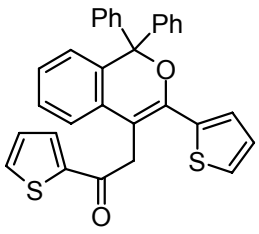
($OCC_6H_5^+$, 100). HRMS calcd. for $C_{35}H_{26}O_2$: 478.1933, found: 478.1926. Anal. calcd. for $C_{35}H_{26}O_2 \times C_4H_8O$ (550.7): C 85.06, H 6.22; found: C 85.11, H 6.14.

**1-Thiophen-2-yl-2-(1,1,3-triphenyl-1*H*-isochromen-4-yl) ethanone (6b)**



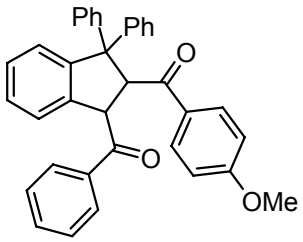

$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 2.18 (s, 3 H, 0.5 acetone), 4.02 (s, 2 H), 6.60 (d, $J$ = 7.9 Hz, 1 H), 6.83 (dd, $J$ = 3.9, 4.9 Hz, 1 H), 7.13 (m, 1 H), 7.20–7.23 (m, 5 H), 7.26–7.29 (m, 10 H), 7.34–7.36 (m, 2 H), 7.47–7.52 (m, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 30.9 ($CH_3$, acetone), 40.2 ($CH_2$), 86.8 ($C_{quat}$), 107.5 ($C_{quat}$), 122.1 (CH), 126.4 (CH), 127.3 (CH), 127.5 (CH), 127.8 (CH), 128.1 (CH), 128.2 (CH), 128.2 (CH), 129.0 (CH), 129.1 (CH), 129.4 (CH), 132.0 (CH), 132.2 ($C_{quat}$), 133.1 ($C_{quat}$), 133.3 (CH), 134.9 ($C_{quat}$), 143.1 ($C_{quat}$), 144.1 ($C_{quat}$), 151.6 ($C_{quat}$), 191.1 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 484 ($M^+$, 49), 373 ($M^+$ – $OCC_4H_3S$, ) 359 ($M^+$ – $CH_2COC_4H_3S$, 27), 358 (86), 105 ($COC_6H_5^+$, 100). HRMS calcd. for $C_{33}H_{24}O_2S$: 484.1497, found: 484.1526. Anal. calcd. for $C_{33}H_{24}O_2S \times 0.5$ acetone (519.5): C 80.46, H 5.36; found: C 80.15, H 4.95.

**2-(1,1-Diphenyl-3-thiophen-2-yl-1*H*-isochromen-4-yl)-1-thiophen-2-yl ethanone (6c)**



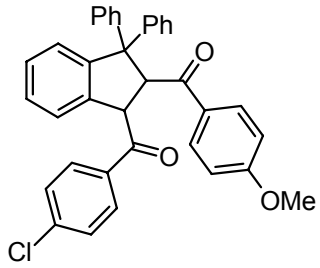
$^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 4.23 (s, 2 H), 6.61 (d, $J$ = 7.4 Hz, 1 H), 6.93 (dd, $J$ = 4.0, 4.8 Hz, 1 H), 7.00 (dd, $J$ = 3.8, 4.9 Hz, 1 H), 7.13 (dt, $J$ = 1.0, 7.4 Hz, 1 H), 7.21–7.32 (m, 13 H), 7.37 (dd, $J$ = 0.8, 5.1 Hz, 1 H), 7.44 (dd, $J$ = 0.7, 3.7 Hz, 1 H), 7.55 (dd, $J$ = 0.7, 4.8 Hz, 1 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 40.2 ($CH_2$), 87.0 ($C_{quat}$), 107.7 ($C_{quat}$), 122.6 (CH), 126.4 (CH), 127.1 (CH), 127.2 (CH), 127.5 (CH), 127.7 (CH), 127.8 (CH), 128.1 (CH), 128.3 (CH), 128.3 (CH), 128.8 (CH), 132.1 (CH), 132.5 ($C_{quat}$) 133.8 (CH), 137.8 ($C_{quat}$), 143.2 ($C_{quat}$), 134.9 ($C_{quat}$), 145.5 ($C_{quat}$), 190.5[5] ($C_{quat}$). EI MS (70 eV), *m/z* (%): 490 ($M^+$, 48), 379 ($M^+ - OCC_4H_3S$, 25), 365 ($M^+ - CH_2COC_4H_3S$, 27), 364 (97). HRMS calcd. for $C_{31}H_{22}O_2S_2$: 490.1061, found: 491.1068. Anal. calcd. for $C_{31}H_{22}O_2S_2$ (490.7): C 75.89, H 4.52, S 13.07; found: C 76.05, H 4.92, S 12.41.

**[2-(4-Methoxy-benzoyl)-3,3-diphenyl-indan-1-yl]-phenyl methanone (7a)**




$^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 3.79 (s, 3 H), 5.90 (d, $J$ = 9.4 Hz, 1 H), 5.96 (d, $J$ = 9.4 Hz, 1 H), 6.71 (m, 3 H), 6.75 (m, 2 H), 6.98 (d, $J$ = 7.7 Hz, 1 H), 7.14–7.24 (m, 5 H), 7.29 (d, $J$ = 7.1 Hz, 1 H), 7.34 (m, 2 H), 7.52–7.57 (m, 4 H), 7.64 (tt, $J$ = 1.4, 7.4 Hz, 1 H), 7.69 (d, $J$ = 9.0 Hz, 2 H), 8.17 (m, 2 H). $^{13}$C NMR (125 MHz, $CDCl_3$): $\delta$ 52.8 (CH), 55.3 ($CH_3$), 63.2 (CH), 65.3 ($C_{quat}$), 113.4 (CH), 123.7 (CH), 126.7 (CH), 126.9 (CH), 127.1 (CH), 127.4 (CH), 127.5 (CH), 128.0 (CH), 128.1 (CH), 128.8 (CH), 129.3 (CH), 129.3 (CH), 130.0 (CH), 131.1 (CH), 131.3 ($C_{quat}$), 133.6 (CH), 137.7 ($C_{quat}$), 140.2 ($C_{quat}$), 140.9 ($C_{quat}$), 146.0 ($C_{quat}$), 149.3 ($C_{quat}$), 163.2 ($C_{quat}$), 198.0 ($C_{quat}$), 199.7 ($C_{quat}$). EI MS (70 eV), *m/z* (%): 508 ($M^+$, 76), 373 ($M^+ - COC_6H_4OCH_3$), 265 ($M^+ - C_6H_4CH(C_6H_5)_2$, 25). HRMS calcd. for $C_{36}H_{28}O_3$: 508.2038, found: 508.2025. Anal. calcd. for $C_{36}H_{28}O$ (508.6): C 85.01, H 5.55; found: C 85.24, H 5.50.

**(4-Chloro-phenyl)-[2-(4-methoxy-benzoyl)-3,3-diphenyl-indan-1-yl] methanone (7b)**




$^{1}$H NMR (300 MHz, $CDCl_3$): $\delta$ 3.79 (s, 3 H), 5.82 (d, $J$ = 9.7 Hz, 1 H), 5.90 (d, $J$ = 9.7 Hz, 1 H), 6.68–6.74 (m, 5 H), 6.94 (m, 1 H), 7.14–7.22 (m, 5 H), 7.27–7.36 (m, 3 H), 7.49–7.54 (m, 4 H), 7.66 (d, $J$ = 8.8 Hz, 2 H), 8.10 (d, $J$ = 8.6 Hz, 2 H). $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 52.7 (CH), 55.4 ($CH_3$), 63.6 (CH), 65.4 ($C_{quat}$), 113.4 (CH), 123.5 (CH), 126.8 (CH), 127.0 (CH), 127.3 (CH), 127.5 (CH), 127.6 (CH), 128.2 (CH), 128.2 (CH), 129.2 (CH), 129.3 (CH), 130.0 (CH), 130.7 (CH), 131.1 (CH), 131.2 ($C_{quat}$), 136.0 ($C_{quat}$), 140.0 ($C_{quat}$), 140.2 ($C_{quat}$), 140.7 ($C_{quat}$), 145.9 ($C_{quat}$), 149.3 ($C_{quat}$), 163.3 ($C_{quat}$), 197.9 ($C_{quat}$), 198.7 ($C_{quat}$). FAB MS (*m/z*): 543 ($M^+$). EI MS (70 eV), *m/z* (%): 544 ($^{37}$Cl-$M^+$, 32), 542 ($^{35}$Cl-$M^+$, 78), 409 ($^{37}$Cl-$M^+$ – $CHCOC_6H_4OCH_3$, 22), 407 ($^{35}$Cl-$M^+$ – $OCC_6H_4OCH_3$, 63), 394 ($^{37}$Cl-$M^+$ – $CH_2COC_6H_4OCH_3$, 35), 392 ($^{35}$Cl-$M^+$ – $CH_2COC_6H_4OCH_3$ 90). HRMS calcd. for $C_{36}H_{27}{}^{35}ClO_3$: 542.1649, found: 542.1668. Anal. calcd. for $C_{36}H_{27}ClO_3$ (543.1): C 79.62, H 5.01, Cl 6.53; found: C 79.24, H 5.10, Cl 6.35.

**II. X-Ray structure data of 3a, 4b, 6b, and 7a**

Table 4: Crystal data and structure refinement for **3a**.

| | | |
|---|---|---|
| Identification code | dds10 (**3a**) | |
| Empirical formula | $C_{34}H_{29}NO_2S$ | |
| Formula weight | 515.64 | |
| Temperature | 200(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | triclinic | |
| Space group | $P\bar{1}$ | |
| Z | 2 | |
| Unit cell dimensions | a = 10.7645(4) Å | $\alpha$ = 78.687(1) deg. |
| | b = 10.8448(3) Å | $\beta$ = 69.861(1) deg. |
| | c = 13.3319(5) Å | $\gamma$ = 68.875(1) deg. |
| Volume | 1358.37(8) $Å^3$ | |
| Density (calculated) | 1.26 $g/cm^3$ | |
| Absorption coefficient | 0.15 $mm^{-1}$ | |
| Crystal shape | polyhedron | |
| Crystal size | .25 x .13 x .10 $mm^3$ | |
| Theta range for data collection | 1.6 to 25.7 deg. | |
| Index ranges | $-13\le h\le 13$, $-13\le k\le 13$, $-16\le l\le 16$ | |
| Reflections collected | 12447 | |
| Independent reflections | 5133 (R(int) = 0.0416) | |
| Observed reflections | 3360 ($I > 2\sigma(I)$) | |
| Absorption correction | Semi-empirical from equivalents | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data/restraints/parameters | 5133 / 0 / 369 | |
| Goodness-of-fit on $F^2$ | 1.01 | |
| Final R indices ($I>2\sigma(I)$) | R1 = 0.045, wR2 = 0.096 | |
| Largest diff. peak and hole | 0.17 and -0.32 $eÅ^{-3}$ | |

Table 5: Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for dds10 (**3a**). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.7689(1) | 0.4046(1) | 0.4238(1) | 0.0451(2) |
| C1 | 0.8060(2) | 0.5511(2) | 0.4021(2) | 0.0254(4) |
| N1 | 0.8274(2) | 0.5806(2) | 0.4835(1) | 0.0360(4) |
| C2 | 0.8142(2) | 0.4848(2) | 0.5672(2) | 0.0411(6) |
| C3 | 0.7837(2) | 0.3828(2) | 0.5497(2) | 0.0383(5) |
| C4 | 0.8106(2) | 0.6404(2) | 0.3007(2) | 0.0250(4) |
| H4 | 0.8842(17) | 0.6732(16) | 0.2868(13) | 0.013(4) |
| C5 | 0.8287(2) | 0.5811(2) | 0.1999(2) | 0.0246(4) |
| C6 | 0.9591(2) | 0.4746(2) | 0.1491(2) | 0.0280(5) |
| O6 | 0.9544(2) | 0.3806(1) | 0.1154(1) | 0.0438(4) |
| C7 | 0.6874(2) | 0.9438(2) | 0.1498(2) | 0.0241(4) |
| C11 | 1.0962(2) | 0.4877(2) | 0.1403(2) | 0.0286(5) |
| C12 | 1.1181(2) | 0.6095(2) | 0.1186(2) | 0.0374(5) |
| C13 | 1.2472(3) | 0.6182(3) | 0.1067(2) | 0.0523(7) |
| C14 | 1.3550(3) | 0.5045(3) | 0.1194(2) | 0.0567(7) |
| C15 | 1.3343(2) | 0.3828(3) | 0.1413(2) | 0.0524(7) |
| C16 | 1.2064(2) | 0.3733(2) | 0.1501(2) | 0.0393(6) |
| C21 | 0.7603(2) | 0.6978(2) | 0.1291(2) | 0.0270(5) |
| H21 | 0.7980(19) | 0.7017(18) | 0.0542(16) | 0.032(6) |
| C22 | 0.7043(2) | 0.8170(2) | 0.1895(2) | 0.0239(4) |

| | | | | |
|---|---|---|---|---|
| C23 | 0.6759(2) | 0.7629(2) | 0.3067(2) | 0.0252(5) |
| H23 | 0.6679(18) | 0.8215(18) | 0.3522(15) | 0.025(5) |
| C24 | 0.5522(2) | 0.7147(2) | 0.3361(2) | 0.0292(5) |
| H24 | 0.473(2) | 0.7455(18) | 0.3958(16) | 0.032(6) |
| C25 | 0.5634(2) | 0.6269(2) | 0.2743(2) | 0.0297(5) |
| H25 | 0.492(2) | 0.5909(18) | 0.2827(15) | 0.029(5) |
| C26 | 0.6927(2) | 0.5901(2) | 0.1834(2) | 0.0277(5) |
| H26 | 0.7034(18) | 0.5274(18) | 0.1333(14) | 0.028(5) |
| C31 | 0.7225(2) | 0.9816(2) | 0.0322(2) | 0.0274(5) |
| C32 | 0.8544(2) | 0.9330(2) | -0.0378(2) | 0.0452(6) |
| C33 | 0.8804(3) | 0.9688(2) | -0.1471(2) | 0.0601(8) |
| C34 | 0.7759(3) | 1.0538(2) | -0.1872(2) | 0.0561(7) |
| C35 | 0.6441(3) | 1.1041(2) | -0.1187(2) | 0.0474(6) |
| C36 | 0.6180(2) | 1.0694(2) | -0.0102(2) | 0.0352(5) |
| C41 | 0.6329(2) | 1.0555(2) | 0.2188(2) | 0.0253(4) |
| C42 | 0.5166(2) | 1.0692(2) | 0.3097(2) | 0.0324(5) |
| C43 | 0.4715(2) | 1.1728(2) | 0.3734(2) | 0.0397(6) |
| C44 | 0.5416(2) | 1.2642(2) | 0.3484(2) | 0.0404(6) |
| C45 | 0.6555(2) | 1.2538(2) | 0.2579(2) | 0.0387(6) |
| C46 | 0.6994(2) | 1.1514(2) | 0.1937(2) | 0.0317(5) |
| C50 | 0.1582(2) | 0.0380(2) | 0.4818(2) | 0.0408(6) |
| O50 | 0.2050(2) | -0.0739(2) | 0.4544(2) | 0.0756(6) |
| C51 | 0.1291(3) | 0.0648(3) | 0.5938(2) | 0.0710(8) |
| C52 | 0.1245(3) | 0.1548(2) | 0.4047(2) | 0.0538(7) |

Table 6: Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for dds10 (**3a**).

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H2 | 0.8258 | 0.4905 | 0.6335 | 0.049 |
| H3 | 0.7722 | 0.3094 | 0.5999 | 0.046 |
| H4 | 0.8842(17) | 0.6732(16) | 0.2868(13) | 0.013(4) |
| H12 | 1.0435 | 0.6879 | 0.1119 | 0.045 |
| H13 | 1.2621 | 0.7020 | 0.0897 | 0.063 |
| H14 | 1.4433 | 0.5106 | 0.1131 | 0.068 |
| H15 | 1.4083 | 0.3050 | 0.1504 | 0.063 |
| H16 | 1.1934 | 0.2888 | 0.1628 | 0.047 |
| H21 | 0.7980(19) | 0.7017(18) | 0.0542(16) | 0.032(6) |
| H23 | 0.6679(18) | 0.8215(18) | 0.3522(15) | 0.025(5) |
| H24 | 0.473(2) | 0.7455(18) | 0.3958(16) | 0.032(6) |
| H25 | 0.492(2) | 0.5909(18) | 0.2827(15) | 0.029(5) |
| H26 | 0.7034(18) | 0.5274(18) | 0.1333(14) | 0.028(5) |
| H32 | 0.9281 | 0.8746 | -0.0110 | 0.054 |
| H33 | 0.9715 | 0.9340 | -0.1945 | 0.072 |
| H34 | 0.7944 | 1.0781 | -0.2620 | 0.067 |
| H35 | 0.5711 | 1.1626 | -0.1462 | 0.057 |
| H36 | 0.5270 | 1.1059 | 0.0367 | 0.042 |
| H42 | 0.4677 | 1.0065 | 0.3282 | 0.039 |
| H43 | 0.3919 | 1.1810 | 0.4348 | 0.048 |
| H44 | 0.5117 | 1.3340 | 0.3931 | 0.048 |
| H45 | 0.7036 | 1.3171 | 0.2399 | 0.046 |
| H46 | 0.7768 | 1.1462 | 0.1309 | 0.038 |
| H51A | 0.0289 | 0.0848 | 0.6308 | 0.107 |
| H51B | 0.1581 | 0.1407 | 0.5939 | 0.107 |
| H51C | 0.1811 | -0.0135 | 0.6308 | 0.107 |
| H52A | 0.1910 | 0.1362 | 0.3333 | 0.081 |
| H52B | 0.1307 | 0.2321 | 0.4280 | 0.081 |
| H52C | 0.0297 | 0.1732 | 0.4019 | 0.081 |

Table 7: Anisotropic displacement parameters ($Å^2$) for dds10 (**3a**). The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$.

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0709(5) | 0.0318(3) | 0.0459(4) | 0.0083(3) | -0.0308(3) | -0.0248(3) |
| C1 | 0.0240(11) | 0.0230(10) | 0.0269(11) | -0.0046(9) | -0.0059(9) | -0.0049(8) |
| N1 | 0.0479(12) | 0.0344(10) | 0.0275(10) | -0.0012(8) | -0.0136(8) | -0.0136(9) |
| C2 | 0.0513(15) | 0.0425(14) | 0.0267(12) | 0.0002(10) | -0.0146(11) | -0.0104(11) |
| C3 | 0.0366(13) | 0.0354(13) | 0.0332(13) | 0.0086(10) | -0.0092(10) | -0.0072(10) |
| C4 | 0.0265(12) | 0.0232(11) | 0.0250(11) | -0.0033(8) | -0.0055(9) | -0.0090(9) |
| C5 | 0.0277(11) | 0.0220(10) | 0.0235(10) | -0.0032(8) | -0.0062(9) | -0.0077(9) |
| C6 | 0.0357(12) | 0.0208(11) | 0.0241(11) | -0.0016(9) | -0.0059(9) | -0.0079(9) |
| O6 | 0.0438(10) | 0.0314(9) | 0.0560(10) | -0.0197(8) | -0.0072(8) | -0.0108(7) |
| C7 | 0.0216(11) | 0.0242(11) | 0.0246(11) | -0.0020(8) | -0.0052(8) | -0.0065(8) |
| C11 | 0.0312(12) | 0.0301(12) | 0.0204(11) | -0.0054(9) | -0.0042(9) | -0.0067(9) |
| C12 | 0.0362(14) | 0.0351(13) | 0.0388(13) | -0.0042(10) | -0.0074(10) | -0.0120(10) |
| C13 | 0.0494(17) | 0.0627(18) | 0.0485(16) | -0.0089(13) | -0.0023(12) | -0.0316(15) |
| C14 | 0.0342(15) | 0.090(2) | 0.0496(16) | -0.0254(15) | -0.0053(12) | -0.0213(15) |
| C15 | 0.0342(15) | 0.0673(19) | 0.0485(16) | -0.0201(13) | -0.0165(12) | 0.0041(13) |
| C16 | 0.0439(15) | 0.0352(13) | 0.0335(13) | -0.0076(10) | -0.0144(11) | -0.0014(11) |
| C21 | 0.0340(12) | 0.0240(11) | 0.0221(12) | -0.0029(9) | -0.0082(9) | -0.0079(9) |
| C22 | 0.0224(11) | 0.0252(11) | 0.0225(10) | -0.0039(8) | -0.0047(8) | -0.0068(8) |
| C23 | 0.0317(12) | 0.0198(10) | 0.0217(11) | -0.0046(9) | -0.0067(9) | -0.0050(9) |
| C24 | 0.0276(12) | 0.0268(12) | 0.0256(12) | 0.0027(9) | -0.0057(10) | -0.0045(9) |
| C25 | 0.0271(12) | 0.0285(12) | 0.0342(12) | 0.0023(10) | -0.0112(10) | -0.0101(10) |
| C26 | 0.0330(12) | 0.0238(11) | 0.0295(12) | -0.0040(9) | -0.0110(10) | -0.0101(9) |
| C31 | 0.0361(13) | 0.0218(11) | 0.0258(11) | -0.0018(9) | -0.0082(9) | -0.0119(9) |
| C32 | 0.0495(15) | 0.0387(14) | 0.0310(13) | 0.0021(10) | -0.0045(11) | -0.0049(11) |
| C33 | 0.0703(19) | 0.0490(16) | 0.0321(14) | 0.0009(12) | 0.0060(13) | -0.0083(14) |
| C34 | 0.101(2) | 0.0439(15) | 0.0257(13) | 0.0065(11) | -0.0195(15) | -0.0307(16) |
| C35 | 0.0717(19) | 0.0401(14) | 0.0476(16) | 0.0129(12) | -0.0381(14) | -0.0270(13) |
| C36 | 0.0406(13) | 0.0331(12) | 0.0368(13) | 0.0037(10) | -0.0152(10) | -0.0174(10) |
| C41 | 0.0299(12) | 0.0211(10) | 0.0247(11) | -0.0012(8) | -0.0109(9) | -0.0057(9) |
| C42 | 0.0343(13) | 0.0243(11) | 0.0348(12) | -0.0036(9) | -0.0058(10) | -0.0088(9) |
| C43 | 0.0396(14) | 0.0352(13) | 0.0360(13) | -0.0098(10) | -0.0024(10) | -0.0071(11) |
| C44 | 0.0548(16) | 0.0263(12) | 0.0402(14) | -0.0109(10) | -0.0168(12) | -0.0063(11) |
| C45 | 0.0517(15) | 0.0251(12) | 0.0465(15) | -0.0013(10) | -0.0212(12) | -0.0150(10) |
| C46 | 0.0370(13) | 0.0280(12) | 0.0317(12) | 0.0014(9) | -0.0118(10) | -0.0125(10) |
| C50 | 0.0368(14) | 0.0476(15) | 0.0391(14) | -0.0039(12) | -0.0078(11) | -0.0177(12) |
| O50 | 0.1151(17) | 0.0445(12) | 0.0579(12) | -0.0025(10) | -0.0281(11) | -0.0138(11) |
| C51 | 0.085(2) | 0.099(2) | 0.0427(16) | -0.0050(16) | -0.0209(15) | -0.0438(19) |
| C52 | 0.0643(17) | 0.0458(15) | 0.0498(16) | -0.0032(12) | -0.0188(13) | -0.0147(13) |

Tabelle 8: Bond lengths (Å) and angles (deg) for dds10 (**3a**).

| | |
|---|---|
| S1-C3 | 1.703(2) |
| S1-C1 | 1.7208(19) |
| C1-N1 | 1.302(2) |
| C1-C4 | 1.497(3) |
| N1-C2 | 1.374(3) |
| C2-C3 | 1.343(3) |
| C4-C5 | 1.525(3) |
| C4-C23 | 1.565(3) |
| C5-C6 | 1.500(3) |
| C5-C26 | 1.520(3) |
| C5-C21 | 1.540(3) |
| C6-O6 | 1.215(2) |
| C6-C11 | 1.496(3) |

| | |
|---|---|
| C7-C22 | 1.343(2) |
| C7-C41 | 1.487(3) |
| C7-C31 | 1.491(3) |
| C11-C12 | 1.382(3) |
| C11-C16 | 1.395(3) |
| C12-C13 | 1.379(3) |
| C13-C14 | 1.385(4) |
| C14-C15 | 1.376(3) |
| C15-C16 | 1.381(3) |
| C21-C22 | 1.482(3) |
| C21-C26 | 1.528(3) |
| C22-C23 | 1.523(3) |
| C23-C24 | 1.504(3) |
| C24-C25 | 1.326(3) |
| C25-C26 | 1.480(3) |
| C31-C32 | 1.380(3) |
| C31-C36 | 1.395(3) |
| C32-C33 | 1.389(3) |
| C33-C34 | 1.368(4) |
| C34-C35 | 1.375(4) |
| C35-C36 | 1.377(3) |
| C41-C46 | 1.393(3) |
| C41-C42 | 1.397(3) |
| C42-C43 | 1.385(3) |
| C43-C44 | 1.377(3) |
| C44-C45 | 1.381(3) |
| C45-C46 | 1.380(3) |
| C50-O50 | 1.211(3) |
| C50-C51 | 1.482(3) |
| C50-C52 | 1.485(3) |
| C3-S1-C1 | 89.80(10) |
| N1-C1-C4 | 121.73(17) |
| N1-C1-S1 | 114.02(15) |
| C4-C1-S1 | 124.23(14) |
| C1-N1-C2 | 110.50(17) |
| C3-C2-N1 | 115.9(2) |
| C2-C3-S1 | 109.80(16) |
| C1-C4-C5 | 117.71(16) |
| C1-C4-C23 | 114.31(16) |
| C5-C4-C23 | 102.05(15) |
| C6-C5-C26 | 118.49(17) |
| C6-C5-C4 | 122.12(16) |
| C26-C5-C4 | 114.42(16) |
| C6-C5-C21 | 119.78(16) |
| C26-C5-C21 | 59.90(12) |
| C4-C5-C21 | 105.69(15) |
| O6-C6-C11 | 120.71(18) |
| O6-C6-C5 | 121.37(18) |
| C11-C6-C5 | 117.92(17) |
| C22-C7-C41 | 123.02(17) |
| C22-C7-C31 | 121.58(17) |
| C41-C7-C31 | 115.41(16) |
| C12-C11-C16 | 119.2(2) |
| C12-C11-C6 | 121.78(18) |
| C16-C11-C6 | 118.93(18) |
| C13-C12-C11 | 120.6(2) |
| C12-C13-C14 | 119.8(2) |
| C15-C14-C13 | 120.1(2) |
| C14-C15-C16 | 120.2(2) |
| C15-C16-C11 | 120.0(2) |
| C22-C21-C26 | 114.15(17) |
| C22-C21-C5 | 106.66(16) |
| C26-C21-C5 | 59.41(12) |
| C7-C22-C21 | 127.85(17) |

| | |
|---|---|
| C7-C22-C23 | 127.67(17) |
| C21-C22-C23 | 104.42(16) |
| C24-C23-C22 | 107.87(16) |
| C24-C23-C4 | 108.95(16) |
| C22-C23-C4 | 100.71(15) |
| C25-C24-C23 | 115.36(19) |
| C24-C25-C26 | 117.14(19) |
| C25-C26-C5 | 117.87(17) |
| C25-C26-C21 | 117.23(17) |
| C5-C26-C21 | 60.69(12) |
| C32-C31-C36 | 117.95(19) |
| C32-C31-C7 | 123.00(18) |
| C36-C31-C7 | 119.05(18) |
| C31-C32-C33 | 120.6(2) |
| C34-C33-C32 | 120.5(2) |
| C33-C34-C35 | 119.8(2) |
| C36-C35-C34 | 119.9(2) |
| C35-C36-C31 | 121.2(2) |
| C46-C41-C42 | 117.51(18) |
| C46-C41-C7 | 119.73(17) |
| C42-C41-C7 | 122.76(17) |
| C43-C42-C41 | 120.87(19) |
| C44-C43-C42 | 120.4(2) |
| C43-C44-C45 | 119.7(2) |
| C46-C45-C44 | 120.0(2) |
| C45-C46-C41 | 121.6(2) |
| O50-C50-C51 | 121.7(2) |
| O50-C50-C52 | 121.4(2) |
| C51-C50-C52 | 116.9(2) |

Table 9: Crystal data and structure refinement for dds25.

| | | |
|---|---|---|
| Identification code | dds25 (**4b**) | |
| Empirical formula | $C_{35}H_{24}ClNO$ | |
| Formula weight | 510.00 | |
| Temperature | 200(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | triclinic | |
| Space group | $P\bar{1}$ | |
| Z | 2 | |
| Unit cell dimensions | a = 9.1420(3) Å | $\alpha$ = 71.900(1) deg. |
| | b = 11.3244(4) Å | $\beta$ = 89.355(1) deg. |
| | c = 14.2565(5) Å | $\gamma$ = 77.085(1) deg. |
| Volume | 1364.72(8) $Å^3$ | |
| Density (calculated) | 1.24 $g/cm^3$ | |
| Absorption coefficient | 0.17 $mm^{-1}$ | |
| Crystal shape | polyhedron | |
| Crystal size | 0.32 x 0.22 x 0.14 $mm^3$ | |
| Theta range for data collection | 1.9 to 27.5 deg. | |
| Index ranges | $-11\le h\le 11$, $-14\le k\le 14$, $-18\le l\le 18$ | |
| Reflections collected | 14158 | |
| Independent reflections | 6191 (R(int) = 0.0405) | |
| Observed reflections | 3612 ($I > 2\sigma(I)$) | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.98 and 0.95 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data/restraints/parameters | 6191 / 0 / 343 | |
| Goodness-of-fit on $F^2$ | 1.01 | |
| Final R indices ($I>2\sigma(I)$) | R1 = 0.049, wR2 = 0.107 | |
| Largest diff. peak and hole | 0.18 and -0.45 $eÅ^{-3}$ | |

Tabelle 10: Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for dds25 (**4b**). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.6738(1) | 0.7682(1) | 0.0613(1) | 0.0770(2) |
| C1 | 0.7005(2) | 1.1151(2) | 0.3009(1) | 0.0354(4) |
| O1 | 0.6372(2) | 1.0949(1) | 0.3783(1) | 0.0462(4) |
| C2 | 0.7729(2) | 1.2264(2) | 0.2675(1) | 0.0322(4) |
| C3 | 0.8994(2) | 1.2157(2) | 0.2190(1) | 0.0363(4) |
| C4 | 0.8633(2) | 1.2386(2) | 0.4619(1) | 0.0316(4) |
| C11 | 0.7038(2) | 1.0291(2) | 0.2394(1) | 0.0357(4) |
| C12 | 0.6861(2) | 1.0762(2) | 0.1373(2) | 0.0496(5) |
| C13 | 0.6754(3) | 0.9961(2) | 0.0825(2) | 0.0567(6) |
| C14 | 0.6876(2) | 0.8685(2) | 0.1302(2) | 0.0477(5) |
| C15 | 0.7066(2) | 0.8191(2) | 0.2316(2) | 0.0470(5) |
| C16 | 0.7129(2) | 0.8998(2) | 0.2858(2) | 0.0410(5) |
| C21 | 0.7016(2) | 1.3380(2) | 0.3011(1) | 0.0306(4) |
| C22 | 0.7394(2) | 1.3431(2) | 0.3940(1) | 0.0313(4) |
| C23 | 0.6647(2) | 1.4475(2) | 0.4222(1) | 0.0377(5) |
| C24 | 0.5550(2) | 1.5442(2) | 0.3599(2) | 0.0418(5) |
| C25 | 0.5178(2) | 1.5381(2) | 0.2686(2) | 0.0419(5) |
| C26 | 0.5897(2) | 1.4353(2) | 0.2396(1) | 0.0384(5) |
| C31 | 0.8309(2) | 1.2090(2) | 0.5710(1) | 0.0363(5) |
| C32 | 0.9096(2) | 1.2418(2) | 0.6374(2) | 0.0506(6) |
| C33 | 0.8775(3) | 1.2099(3) | 0.7372(2) | 0.0690(7) |
| C34 | 0.7670(3) | 1.1452(2) | 0.7699(2) | 0.0698(8) |
| C35 | 0.6870(3) | 1.1123(2) | 0.7048(2) | 0.0606(7) |
| C36 | 0.7182(2) | 1.1442(2) | 0.6057(2) | 0.0467(5) |
| C41 | 1.0194(2) | 1.2632(2) | 0.4405(1) | 0.0309(4) |
| C42 | 1.1403(2) | 1.1601(2) | 0.4509(1) | 0.0391(5) |
| C43 | 1.2845(2) | 1.1766(2) | 0.4342(2) | 0.0456(5) |
| C44 | 1.3110(2) | 1.2975(2) | 0.4061(1) | 0.0438(5) |
| C45 | 1.1925(2) | 1.4010(2) | 0.3939(2) | 0.0476(5) |
| C46 | 1.0479(2) | 1.3845(2) | 0.4111(1) | 0.0420(5) |
| C51 | 0.9950(2) | 1.3076(2) | 0.1832(1) | 0.0345(4) |
| C52 | 0.9401(2) | 1.4397(2) | 0.1539(2) | 0.0441(5) |
| C53 | 1.0331(2) | 1.5207(2) | 0.1147(2) | 0.0465(5) |
| C54 | 1.1841(2) | 1.4716(2) | 0.1051(1) | 0.0408(5) |
| C55 | 1.2412(2) | 1.3411(2) | 0.1356(2) | 0.0473(5) |
| C56 | 1.1463(2) | 1.2609(2) | 0.1732(2) | 0.0442(5) |
| C57 | 1.2789(2) | 1.5571(2) | 0.0589(2) | 0.0508(6) |
| N57 | 1.3529(2) | 1.6246(2) | 0.0212(2) | 0.0763(7) |

Tabelle 11: Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for dds25 (**4b**).

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H3 | 0.9322 | 1.1372 | 0.2056 | 0.044 |
| H4 | 0.8625 | 1.1592 | 0.4453 | 0.038 |
| H12 | 0.6811 | 1.1641 | 0.1045 | 0.060 |
| H13 | 0.6599 | 1.0293 | 0.0125 | 0.068 |
| H15 | 0.7151 | 0.7305 | 0.2638 | 0.056 |
| H16 | 0.7236 | 0.8666 | 0.3559 | 0.049 |
| H23 | 0.6896 | 1.4522 | 0.4853 | 0.045 |
| H24 | 0.5056 | 1.6146 | 0.3802 | 0.050 |

| | | | | |
|---|---|---|---|---|
| H25 | 0.4429 | 1.6045 | 0.2256 | 0.050 |
| H26 | 0.5625 | 1.4308 | 0.1768 | 0.046 |
| H32 | 0.9862 | 1.2865 | 0.6152 | 0.061 |
| H33 | 0.9323 | 1.2330 | 0.7823 | 0.083 |
| H34 | 0.7457 | 1.1231 | 0.8377 | 0.084 |
| H35 | 0.6104 | 1.0677 | 0.7276 | 0.073 |
| H36 | 0.6622 | 1.1216 | 0.5609 | 0.056 |
| H42 | 1.1234 | 1.0763 | 0.4700 | 0.047 |
| H43 | 1.3656 | 1.1045 | 0.4421 | 0.055 |
| H44 | 1.4102 | 1.3091 | 0.3952 | 0.053 |
| H45 | 1.2100 | 1.4847 | 0.3736 | 0.057 |
| H46 | 0.9672 | 1.4569 | 0.4027 | 0.050 |
| H52 | 0.8374 | 1.4742 | 0.1610 | 0.053 |
| H53 | 0.9940 | 1.6105 | 0.0942 | 0.056 |
| H55 | 1.3447 | 1.3069 | 0.1307 | 0.057 |
| H56 | 1.1856 | 1.1712 | 0.1928 | 0.053 |

Tabelle 12: Anisotropic displacement parameters ($Å^2$) for dds25 (**4b**). The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.1038(6) | 0.0590(4) | 0.0779(5) | -0.0394(3) | -0.0092(4) | -0.0124(4) |
| C1 | 0.0309(10) | 0.0313(10) | 0.0414(11) | -0.0088(9) | 0.0014(9) | -0.0054(8) |
| O1 | 0.0503(9) | 0.0417(8) | 0.0496(9) | -0.0143(7) | 0.0174(7) | -0.0173(7) |
| C2 | 0.0338(11) | 0.0292(10) | 0.0332(10) | -0.0089(8) | 0.0025(8) | -0.0084(8) |
| C3 | 0.0412(12) | 0.0309(10) | 0.0388(11) | -0.0129(9) | 0.0064(9) | -0.0098(9) |
| C4 | 0.0324(10) | 0.0255(9) | 0.0361(10) | -0.0093(8) | 0.0023(8) | -0.0058(8) |
| C11 | 0.0329(11) | 0.0313(10) | 0.0425(12) | -0.0100(9) | 0.0016(8) | -0.0093(8) |
| C12 | 0.0680(15) | 0.0335(11) | 0.0455(13) | -0.0067(10) | -0.0017(11) | -0.0160(10) |
| C13 | 0.0815(17) | 0.0500(14) | 0.0395(12) | -0.0115(11) | -0.0019(11) | -0.0203(12) |
| C14 | 0.0510(13) | 0.0433(12) | 0.0554(14) | -0.0240(11) | 0.0015(10) | -0.0125(10) |
| C15 | 0.0520(13) | 0.0328(11) | 0.0559(14) | -0.0121(10) | 0.0019(10) | -0.0116(10) |
| C16 | 0.0423(12) | 0.0362(11) | 0.0424(11) | -0.0077(9) | 0.0015(9) | -0.0115(9) |
| C21 | 0.0277(10) | 0.0274(9) | 0.0361(10) | -0.0068(8) | 0.0055(8) | -0.0095(8) |
| C22 | 0.0276(10) | 0.0274(9) | 0.0373(11) | -0.0074(8) | 0.0049(8) | -0.0072(8) |
| C23 | 0.0399(11) | 0.0317(10) | 0.0399(11) | -0.0105(9) | 0.0060(9) | -0.0064(9) |
| C24 | 0.0416(12) | 0.0265(10) | 0.0519(13) | -0.0098(9) | 0.0117(10) | -0.0007(9) |
| C25 | 0.0333(11) | 0.0336(11) | 0.0465(13) | -0.0005(9) | 0.0020(9) | -0.0003(9) |
| C26 | 0.0366(11) | 0.0368(11) | 0.0371(11) | -0.0058(9) | 0.0004(9) | -0.0074(9) |
| C31 | 0.0334(11) | 0.0283(10) | 0.0387(11) | -0.0047(8) | 0.0041(9) | 0.0016(8) |
| C32 | 0.0486(13) | 0.0594(14) | 0.0400(12) | -0.0130(11) | 0.0051(10) | -0.0087(11) |
| C33 | 0.0713(18) | 0.0896(19) | 0.0398(14) | -0.0201(13) | 0.0020(13) | -0.0060(16) |
| C34 | 0.0692(18) | 0.0739(18) | 0.0423(14) | -0.0003(13) | 0.0174(13) | 0.0053(15) |
| C35 | 0.0524(15) | 0.0467(13) | 0.0614(16) | 0.0037(12) | 0.0223(13) | 0.0013(11) |
| C36 | 0.0427(12) | 0.0365(11) | 0.0533(13) | -0.0071(10) | 0.0109(10) | -0.0044(10) |
| C41 | 0.0334(10) | 0.0319(10) | 0.0267(9) | -0.0084(8) | 0.0019(8) | -0.0076(8) |
| C42 | 0.0357(11) | 0.0322(10) | 0.0437(12) | -0.0043(9) | 0.0077(9) | -0.0077(9) |
| C43 | 0.0335(12) | 0.0439(12) | 0.0492(12) | -0.0047(10) | 0.0082(9) | -0.0032(9) |
| C44 | 0.0336(11) | 0.0547(13) | 0.0425(12) | -0.0103(10) | 0.0074(9) | -0.0164(10) |
| C45 | 0.0479(13) | 0.0402(12) | 0.0580(14) | -0.0135(10) | 0.0030(11) | -0.0201(10) |
| C46 | 0.0373(12) | 0.0348(11) | 0.0539(13) | -0.0147(10) | 0.0022(10) | -0.0075(9) |
| C51 | 0.0413(12) | 0.0351(10) | 0.0308(10) | -0.0127(8) | 0.0087(8) | -0.0134(9) |
| C52 | 0.0429(12) | 0.0388(11) | 0.0488(12) | -0.0099(10) | 0.0105(10) | -0.0120(10) |
| C53 | 0.0549(14) | 0.0346(11) | 0.0462(12) | -0.0049(9) | 0.0049(10) | -0.0142(10) |
| C54 | 0.0446(13) | 0.0473(12) | 0.0306(10) | -0.0056(9) | 0.0000(9) | -0.0201(10) |
| C55 | 0.0384(12) | 0.0489(13) | 0.0530(13) | -0.0117(11) | 0.0070(10) | -0.0136(10) |
| C56 | 0.0435(13) | 0.0371(11) | 0.0520(13) | -0.0134(10) | 0.0101(10) | -0.0107(10) |
| C57 | 0.0485(14) | 0.0537(14) | 0.0414(12) | 0.0040(10) | -0.0109(10) | -0.0206(11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N57 | 0.0656(14) | 0.0744(14) | 0.0710(14) | 0.0185(11) | -0.0143(11) | -0.0379(12) |

Tabelle 13: Bond lengths (Å) and angles (deg) for dds25 (**4b**).

| | |
|---|---|
| Cl1-C14 | 1.742(2) |
| C1-O1 | 1.223(2) |
| C1-C11 | 1.495(3) |
| C1-C2 | 1.499(2) |
| C2-C3 | 1.343(2) |
| C2-C21 | 1.499(2) |
| C3-C51 | 1.474(2) |
| C4-C41 | 1.524(2) |
| C4-C31 | 1.527(2) |
| C4-C22 | 1.528(2) |
| C11-C12 | 1.382(3) |
| C11-C16 | 1.391(3) |
| C12-C13 | 1.387(3) |
| C13-C14 | 1.372(3) |
| C14-C15 | 1.375(3) |
| C15-C16 | 1.379(3) |
| C21-C22 | 1.395(2) |
| C21-C26 | 1.395(2) |
| C22-C23 | 1.398(2) |
| C23-C24 | 1.385(3) |
| C24-C25 | 1.375(3) |
| C25-C26 | 1.383(3) |
| C31-C32 | 1.380(3) |
| C31-C36 | 1.395(3) |
| C32-C33 | 1.400(3) |
| C33-C34 | 1.372(4) |
| C34-C35 | 1.374(4) |
| C35-C36 | 1.389(3) |
| C41-C42 | 1.387(2) |
| C41-C46 | 1.389(2) |
| C42-C43 | 1.381(3) |
| C43-C44 | 1.378(3) |
| C44-C45 | 1.374(3) |
| C45-C46 | 1.385(3) |
| C51-C56 | 1.389(3) |
| C51-C52 | 1.395(3) |
| C52-C53 | 1.377(3) |
| C53-C54 | 1.391(3) |
| C54-C55 | 1.380(3) |
| C54-C57 | 1.446(3) |
| C55-C56 | 1.380(3) |
| C57-N57 | 1.139(3) |
| O1-C1-C11 | 119.63(16) |
| O1-C1-C2 | 119.97(16) |
| C11-C1-C2 | 120.39(16) |
| C3-C2-C1 | 119.28(16) |
| C3-C2-C21 | 125.61(16) |
| C1-C2-C21 | 114.86(15) |
| C2-C3-C51 | 129.70(17) |
| C41-C4-C31 | 113.43(15) |
| C41-C4-C22 | 112.54(14) |
| C31-C4-C22 | 112.82(14) |
| C12-C11-C16 | 118.80(18) |
| C12-C11-C1 | 121.70(16) |
| C16-C11-C1 | 119.30(17) |
| C11-C12-C13 | 120.52(19) |
| C14-C13-C12 | 119.4(2) |
| C13-C14-C15 | 121.25(19) |
| C13-C14-Cl1 | 119.31(17) |
| C15-C14-Cl1 | 119.42(16) |
| C14-C15-C16 | 119.10(19) |
| C15-C16-C11 | 120.94(19) |
| C22-C21-C26 | 119.57(16) |
| C22-C21-C2 | 121.46(15) |

| | |
|---|---|
| C26-C21-C2 | 118.89(16) |
| C21-C22-C23 | 118.62(16) |
| C21-C22-C4 | 120.44(15) |
| C23-C22-C4 | 120.91(16) |
| C24-C23-C22 | 121.18(18) |
| C25-C24-C23 | 119.87(17) |
| C24-C25-C26 | 119.82(18) |
| C25-C26-C21 | 120.92(18) |
| C32-C31-C36 | 118.37(19) |
| C32-C31-C4 | 122.99(18) |
| C36-C31-C4 | 118.63(18) |
| C31-C32-C33 | 120.7(2) |
| C34-C33-C32 | 120.0(2) |
| C33-C34-C35 | 120.2(2) |
| C34-C35-C36 | 120.0(2) |
| C35-C36-C31 | 120.8(2) |
| C42-C41-C46 | 117.69(17) |
| C42-C41-C4 | 119.03(16) |
| C46-C41-C4 | 123.28(16) |
| C43-C42-C41 | 121.49(18) |
| C44-C43-C42 | 120.05(19) |
| C45-C44-C43 | 119.40(18) |
| C44-C45-C46 | 120.55(19) |
| C45-C46-C41 | 120.81(18) |
| C56-C51-C52 | 117.90(17) |
| C56-C51-C3 | 118.55(16) |
| C52-C51-C3 | 123.46(17) |
| C53-C52-C51 | 120.73(19) |
| C52-C53-C54 | 120.24(19) |
| C55-C54-C53 | 119.86(18) |
| C55-C54-C57 | 120.27(19) |
| C53-C54-C57 | 119.81(19) |
| C54-C55-C56 | 119.34(19) |
| C55-C56-C51 | 121.91(18) |
| N57-C57-C54 | 179.1(2) |

Table 14: Crystal data and structure refinement for dds21 (**4b**).

| | | |
|---|---|---|
| Identification code | dds21 (**4b**) | |
| Empirical formula | $C_{34}H_{25}Cl_3O_2S$ | |
| Formula weight | 603.95 | |
| Temperature | 200(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P&^-&B1 | |
| Z | 2 | |
| Unit cell dimensions | a = 8.9794(3) Å | $\alpha$ = 87.793(1) deg. |
| | b = 11.1754(4) Å | $\beta$ = 74.637(1) deg. |
| | c = 14.8724(6) Å | $\gamma$ = 88.191(1) deg. |
| Volume | 1437.67(9) $Å^3$ | |
| Density (calculated) | 1.39 $g/cm^3$ | |
| Absorption coefficient | 0.42 $mm^{-1}$ | |
| Crystal shape | polyhedron | |
| Crystal size | 0.30 x 0.22 x 0.08 $mm^3$ | |
| Theta range for data collection | 1.4 to 27.5 deg. | |
| Index ranges | $-11\le h\le 11$, $-14\le k\le 14$, $-19\le l\le 19$ | |
| Reflections collected | 15088 | |
| Independent reflections | 6549 (R(int) = 0.0349) | |
| Observed reflections | 4397 ($I > 2\sigma(I)$) | |
| Absorption correction | Semi-empirical from equivalents | |

| | |
|---|---|
| Max. and min. transmission | 0.97 and 0.88 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 6549 / 71 / 417 |
| Goodness-of-fit on $F^2$ | 1.01 |
| Final R indices (I>2σ(I)) | R1 = 0.043, wR2 = 0.104 |
| Largest diff. peak and hole | 0.33 and -0.44 eÅ$^{-3}$ |

Tabelle 15: Atomic coordinates and equivalent isotropic displacement parameters (Å$^2$) for dds21 (**4b**). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.)

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| C1 | -0.4637(2) | 0.9081(2) | 0.1968(1) | 0.0229(4) |
| O2 | -0.4446(1) | 0.8643(1) | 0.2867(1) | 0.0247(3) |
| C3 | -0.3073(2) | 0.8056(2) | 0.2862(1) | 0.0235(4) |
| C4 | -0.2260(2) | 0.7462(2) | 0.2105(1) | 0.0249(4) |
| C5 | -0.0646(2) | 0.6964(2) | 0.2024(1) | 0.0280(4) |
| O6 | -0.1593(2) | 0.4972(1) | 0.2416(1) | 0.0407(4) |
| C6 | -0.0497(2) | 0.5639(2) | 0.2254(1) | 0.0281(4) |
| C7 | 0.1044(2) | 0.5187(2) | 0.2277(1) | 0.0281(4) |
| S1 | 0.1308(1) | 0.3693(1) | 0.2492(1) | 0.0380(3) |
| C8 | 0.2414(9) | 0.5766(6) | 0.2151(8) | 0.0387(16) |
| C9 | 0.3660(2) | 0.5013(2) | 0.2241(2) | 0.0449(6) |
| C10 | 0.3184(3) | 0.3874(2) | 0.2427(2) | 0.0466(6) |
| C11 | -0.6327(2) | 0.9490(2) | 0.2171(1) | 0.0235(4) |
| C12 | -0.6794(2) | 1.0351(2) | 0.1593(1) | 0.0299(4) |
| C13 | -0.8342(2) | 1.0681(2) | 0.1746(1) | 0.0345(5) |
| C14 | -0.9434(2) | 1.0177(2) | 0.2485(2) | 0.0369(5) |
| C15 | -0.8977(2) | 0.9344(2) | 0.3079(1) | 0.0346(5) |
| C16 | -0.7434(2) | 0.8989(2) | 0.2913(1) | 0.0277(4) |
| C21 | -0.3558(2) | 1.0143(2) | 0.1627(1) | 0.0238(4) |
| C22 | -0.3693(2) | 1.1129(2) | 0.2194(1) | 0.0300(4) |
| C23 | -0.2777(2) | 1.2118(2) | 0.1900(2) | 0.0370(5) |
| C24 | -0.1717(2) | 1.2145(2) | 0.1029(2) | 0.0407(5) |
| C25 | -0.1573(2) | 1.1174(2) | 0.0466(2) | 0.0389(5) |
| C26 | -0.2491(2) | 1.0180(2) | 0.0762(1) | 0.0299(4) |
| C31 | -0.4266(2) | 0.8047(2) | 0.1299(1) | 0.0248(4) |
| C32 | -0.5065(2) | 0.7857(2) | 0.0640(1) | 0.0297(4) |
| C33 | -0.4602(2) | 0.6946(2) | 0.0007(1) | 0.0375(5) |
| C34 | -0.3336(3) | 0.6227(2) | 0.0036(1) | 0.0393(5) |
| C35 | -0.2536(2) | 0.6402(2) | 0.0698(1) | 0.0345(5) |
| C36 | -0.2993(2) | 0.7305(2) | 0.1350(1) | 0.0261(4) |
| C41 | -0.2747(2) | 0.8144(2) | 0.3781(1) | 0.0254(4) |
| C42 | -0.3176(2) | 0.9179(2) | 0.4290(1) | 0.0307(4) |
| C43 | -0.2906(2) | 0.9277(2) | 0.5156(1) | 0.0373(5) |
| C44 | -0.2197(3) | 0.8346(2) | 0.5533(1) | 0.0411(5) |
| C45 | -0.1789(3) | 0.7304(2) | 0.5049(2) | 0.0407(5) |
| C46 | -0.2073(2) | 0.7196(2) | 0.4184(1) | 0.0325(4) |
| C51 | 0.7457(3) | 0.3490(2) | 0.4398(2) | 0.0386(8) |
| Cl1 | 0.5529(2) | 0.3472(3) | 0.4350(2) | 0.0906(7) |
| Cl2 | 0.8264(2) | 0.2043(1) | 0.4270(1) | 0.0620(4) |
| Cl3 | 0.7617(2) | 0.4072(2) | 0.5440(2) | 0.0833(6) |
| C51B | 0.633(2) | 0.3079(12) | 0.4839(9) | 0.085(6) |
| Cl1B | 0.5832(10) | 0.4068(8) | 0.4071(5) | 0.075(2) |
| Cl2B | 0.682(3) | 0.3909(9) | 0.5682(8) | 0.192(7) |
| Cl3B | 0.8004(17) | 0.2268(13) | 0.4277(10) | 0.149(5) |
| S1B | 0.2544(14) | 0.6037(10) | 0.2118(16) | 0.040(2) |
| C8B | 0.151(2) | 0.4011(16) | 0.248(3) | 0.073(10) |

Tabelle 16: Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for dds21 (**4b**).

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| H5A | -0.0189 | 0.7416 | 0.2441 | 0.034 |
| H5B | -0.0019 | 0.7122 | 0.1377 | 0.034 |
| H8 | 0.2514 | 0.6602 | 0.2014 | 0.046 |
| H9 | 0.4682 | 0.5269 | 0.2181 | 0.054 |
| H10 | 0.3854 | 0.3230 | 0.2514 | 0.056 |
| H12 | -0.6047 | 1.0714 | 0.1090 | 0.036 |
| H13 | -0.8650 | 1.1257 | 0.1340 | 0.041 |
| H14 | -1.0494 | 1.0400 | 0.2587 | 0.044 |
| H15 | -0.9721 | 0.9015 | 0.3600 | 0.041 |
| H16 | -0.7134 | 0.8399 | 0.3311 | 0.033 |
| H22 | -0.4421 | 1.1122 | 0.2788 | 0.036 |
| H23 | -0.2874 | 1.2780 | 0.2295 | 0.044 |
| H24 | -0.1097 | 1.2826 | 0.0824 | 0.049 |
| H25 | -0.0842 | 1.1183 | -0.0127 | 0.047 |
| H26 | -0.2385 | 0.9519 | 0.0365 | 0.036 |
| H32 | -0.5936 | 0.8352 | 0.0621 | 0.036 |
| H33 | -0.5154 | 0.6820 | -0.0443 | 0.045 |
| H34 | -0.3012 | 0.5610 | -0.0400 | 0.047 |
| H35 | -0.1666 | 0.5902 | 0.0710 | 0.041 |
| H42 | -0.3661 | 0.9827 | 0.4037 | 0.037 |
| H43 | -0.3210 | 0.9988 | 0.5493 | 0.045 |
| H44 | -0.1992 | 0.8422 | 0.6123 | 0.049 |
| H45 | -0.1311 | 0.6658 | 0.5310 | 0.049 |
| H46 | -0.1807 | 0.6469 | 0.3862 | 0.039 |
| H51 | 0.8046 | 0.4009 | 0.3869 | 0.046 |
| H51B | 0.5462 | 0.2530 | 0.5128 | 0.103 |
| H8B | 0.0821 | 0.3364 | 0.2637 | 0.087 |

Tabelle 17:Anisotropic displacement parameters ($Å^2$) for dds21 (**4b**). The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ (h^2\ a^{*2}\ U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12})$)

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0252(9) | 0.0255(9) | 0.0191(9) | 0.0001(7) | -0.0082(7) | 0.0027(7) |
| O2 | 0.0243(6) | 0.0295(7) | 0.0201(6) | 0.0003(5) | -0.0064(5) | 0.0050(5) |
| C3 | 0.0232(9) | 0.0208(9) | 0.0261(9) | 0.0040(7) | -0.0067(7) | 0.0004(7) |
| C4 | 0.0277(9) | 0.0222(9) | 0.0238(9) | 0.0014(7) | -0.0059(8) | 0.0028(7) |
| C5 | 0.0261(9) | 0.0242(9) | 0.0320(10) | 0.0009(8) | -0.0051(8) | 0.0023(8) |
| O6 | 0.0333(8) | 0.0309(8) | 0.0569(10) | 0.0066(7) | -0.0111(7) | -0.0041(6) |
| C6 | 0.0302(10) | 0.0262(10) | 0.0260(10) | -0.0004(8) | -0.0043(8) | 0.0030(8) |
| C7 | 0.0316(10) | 0.0266(10) | 0.0242(9) | -0.0002(8) | -0.0049(8) | 0.0047(8) |
| S1 | 0.0404(4) | 0.0264(4) | 0.0423(5) | 0.0065(3) | -0.0046(4) | 0.0105(3) |
| C8 | 0.048(3) | 0.027(3) | 0.041(2) | -0.004(3) | -0.011(2) | 0.006(2) |
| C9 | 0.0325(11) | 0.0621(16) | 0.0412(13) | -0.0059(11) | -0.0118(10) | 0.0063(11) |
| C10 | 0.0441(13) | 0.0541(14) | 0.0383(12) | 0.0035(11) | -0.0088(10) | 0.0246(11) |
| C11 | 0.0239(9) | 0.0241(9) | 0.0237(9) | -0.0053(7) | -0.0083(7) | 0.0011(7) |
| C12 | 0.0293(10) | 0.0314(10) | 0.0292(10) | -0.0004(8) | -0.0088(8) | 0.0029(8) |
| C13 | 0.0351(11) | 0.0353(11) | 0.0381(11) | -0.0068(9) | -0.0193(9) | 0.0107(9) |
| C14 | 0.0242(10) | 0.0435(12) | 0.0461(12) | -0.0157(10) | -0.0137(9) | 0.0064(9) |
| C15 | 0.0245(10) | 0.0412(12) | 0.0358(11) | -0.0078(9) | -0.0029(8) | -0.0048(9) |
| C16 | 0.0288(10) | 0.0280(10) | 0.0280(10) | -0.0027(8) | -0.0099(8) | -0.0027(8) |
| C21 | 0.0220(9) | 0.0254(9) | 0.0252(9) | 0.0015(7) | -0.0092(7) | 0.0034(7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C22 | 0.0290(10) | 0.0310(10) | 0.0315(10) | -0.0006(8) | -0.0108(8) | 0.0026(8) |
| C23 | 0.0411(12) | 0.0292(11) | 0.0458(13) | -0.0012(9) | -0.0201(10) | -0.0015(9) |
| C24 | 0.0355(11) | 0.0328(11) | 0.0560(14) | 0.0122(10) | -0.0172(11) | -0.0079(9) |
| C25 | 0.0295(11) | 0.0426(13) | 0.0404(12) | 0.0092(10) | -0.0029(9) | -0.0038(9) |
| C26 | 0.0277(10) | 0.0326(10) | 0.0283(10) | 0.0020(8) | -0.0060(8) | 0.0017(8) |
| C31 | 0.0292(10) | 0.0242(9) | 0.0193(9) | 0.0009(7) | -0.0035(7) | -0.0010(7) |
| C32 | 0.0327(10) | 0.0319(10) | 0.0254(10) | -0.0005(8) | -0.0095(8) | 0.0002(8) |
| C33 | 0.0481(13) | 0.0406(12) | 0.0276(10) | -0.0066(9) | -0.0159(9) | -0.0015(10) |
| C34 | 0.0545(14) | 0.0354(11) | 0.0265(10) | -0.0107(9) | -0.0072(10) | 0.0045(10) |
| C35 | 0.0420(12) | 0.0314(11) | 0.0279(10) | -0.0045(8) | -0.0059(9) | 0.0088(9) |
| C36 | 0.0283(10) | 0.0256(9) | 0.0224(9) | 0.0008(7) | -0.0035(8) | 0.0012(8) |
| C41 | 0.0236(9) | 0.0302(10) | 0.0219(9) | 0.0027(7) | -0.0057(7) | -0.0017(8) |
| C42 | 0.0324(10) | 0.0322(10) | 0.0291(10) | -0.0007(8) | -0.0116(8) | 0.0033(8) |
| C43 | 0.0459(12) | 0.0377(12) | 0.0302(11) | -0.0047(9) | -0.0130(9) | -0.0004(10) |
| C44 | 0.0541(14) | 0.0453(13) | 0.0291(11) | 0.0021(10) | -0.0203(10) | -0.0048(11) |
| C45 | 0.0509(13) | 0.0405(12) | 0.0353(11) | 0.0089(10) | -0.0210(10) | 0.0019(10) |
| C46 | 0.0371(11) | 0.0307(10) | 0.0300(10) | 0.0017(8) | -0.0103(9) | 0.0029(9) |
| C51 | 0.0406(16) | 0.0343(15) | 0.0365(15) | 0.0063(12) | -0.0029(13) | -0.0073(12) |
| Cl1 | 0.0411(6) | 0.1320(18) | 0.0935(12) | -0.0303(12) | -0.0049(7) | -0.0029(8) |
| Cl2 | 0.0826(8) | 0.0407(6) | 0.0643(8) | -0.0130(5) | -0.0207(6) | 0.0014(6) |
| Cl3 | 0.1326(13) | 0.0527(8) | 0.0798(11) | -0.0256(8) | -0.0534(8) | 0.0146(7) |
| C51B | 0.142(16) | 0.069(11) | 0.063(11) | -0.001(7) | -0.059(11) | 0.005(11) |
| Cl1B | 0.066(4) | 0.094(5) | 0.067(3) | -0.020(3) | -0.017(3) | 0.005(3) |
| Cl2B | 0.46(2) | 0.084(5) | 0.071(5) | 0.002(4) | -0.148(10) | -0.009(9) |
| Cl3B | 0.150(8) | 0.166(10) | 0.140(8) | -0.019(6) | -0.062(6) | 0.060(6) |
| S1B | 0.035(3) | 0.025(4) | 0.062(5) | -0.006(3) | -0.013(3) | 0.005(2) |
| C8B | 0.071(10) | 0.044(10) | 0.12(3) | 0.022(15) | -0.049(16) | -0.031(8) |

Tabelle 18: (Bond lengths (Å) and angles (deg) for dds21 (**4b**).

| | |
|---|---|
| C1-O2 | 1.457(2) |
| C1-C31 | 1.525(2) |
| C1-C11 | 1.525(2) |
| C1-C21 | 1.539(3) |
| O2-C3 | 1.377(2) |
| C3-C4 | 1.353(2) |
| C3-C41 | 1.479(2) |
| C4-C36 | 1.462(3) |
| C4-C5 | 1.512(2) |
| C5-C6 | 1.517(3) |
| O6-C6 | 1.220(2) |
| C6-C7 | 1.466(3) |
| C7-C8 | 1.373(7) |
| C7-C8B | 1.414(16) |
| C7-S1B | 1.633(11) |
| C7-S1 | 1.709(2) |
| S1-C10 | 1.680(3) |
| C8-C9 | 1.411(8) |
| C9-C10 | 1.350(3) |
| C9-S1B | 1.536(12) |
| C10-C8B | 1.484(17) |
| C11-C16 | 1.387(3) |
| C11-C12 | 1.394(3) |
| C12-C13 | 1.387(3) |
| C13-C14 | 1.380(3) |
| C14-C15 | 1.385(3) |
| C15-C16 | 1.390(3) |
| C21-C26 | 1.387(3) |
| C21-C22 | 1.396(3) |
| C22-C23 | 1.385(3) |
| C23-C24 | 1.389(3) |

| | |
|---|---|
| C24-C25 | 1.378(3) |
| C25-C26 | 1.391(3) |
| C31-C32 | 1.385(3) |
| C31-C36 | 1.407(2) |
| C32-C33 | 1.391(3) |
| C33-C34 | 1.380(3) |
| C34-C35 | 1.387(3) |
| C35-C36 | 1.402(3) |
| C41-C42 | 1.395(3) |
| C41-C46 | 1.396(3) |
| C42-C43 | 1.381(3) |
| C43-C44 | 1.380(3) |
| C44-C45 | 1.381(3) |
| C45-C46 | 1.388(3) |
| C51-Cl3 | 1.745(3) |
| C51-Cl2 | 1.746(3) |
| C51-Cl1 | 1.752(3) |
| C51B-Cl1B | 1.694(12) |
| C51B-Cl2B | 1.741(12) |
| C51B-Cl3B | 1.756(13) |
| O2-C1-C31 | 107.99(13) |
| O2-C1-C11 | 104.59(13) |
| C31-C1-C11 | 112.20(14) |
| O2-C1-C21 | 108.46(13) |
| C31-C1-C21 | 112.29(14) |
| C11-C1-C21 | 110.90(14) |
| C3-O2-C1 | 116.73(13) |
| C4-C3-O2 | 121.11(16) |
| C4-C3-C41 | 128.88(16) |
| O2-C3-C41 | 109.94(14) |
| C3-C4-C36 | 117.87(16) |
| C3-C4-C5 | 122.21(16) |
| C36-C4-C5 | 119.91(15) |
| C4-C5-C6 | 116.78(15) |
| O6-C6-C7 | 120.93(17) |
| O6-C6-C5 | 122.43(17) |
| C7-C6-C5 | 116.63(16) |
| C8-C7-C8B | 100.0(9) |
| C8-C7-C6 | 131.1(3) |
| C8B-C7-C6 | 128.9(7) |
| C8-C7-S1B | 7.5(7) |
| C8B-C7-S1B | 107.4(7) |
| C6-C7-S1B | 123.6(4) |
| C8-C7-S1 | 109.8(3) |
| C8B-C7-S1 | 10.1(9) |
| C6-C7-S1 | 119.07(14) |
| S1B-C7-S1 | 117.3(4) |
| C10-S1-C7 | 91.78(11) |
| C7-C8-C9 | 114.2(5) |
| C10-C9-C8 | 109.9(3) |
| C10-C9-S1B | 121.6(5) |
| C8-C9-S1B | 11.7(6) |
| C9-C10-C8B | 101.7(7) |
| C9-C10-S1 | 114.29(16) |
| C8B-C10-S1 | 12.8(7) |
| C16-C11-C12 | 118.73(17) |
| C16-C11-C1 | 120.95(16) |
| C12-C11-C1 | 120.29(16) |
| C13-C12-C11 | 120.55(18) |
| C14-C13-C12 | 120.25(18) |
| C13-C14-C15 | 119.69(18) |
| C14-C15-C16 | 120.14(19) |
| C11-C16-C15 | 120.60(18) |
| C26-C21-C22 | 118.37(17) |

| | |
|---|---|
| C26-C21-C1 | 122.54(16) |
| C22-C21-C1 | 119.04(16) |
| C23-C22-C21 | 120.78(19) |
| C22-C23-C24 | 120.2(2) |
| C25-C24-C23 | 119.44(19) |
| C24-C25-C26 | 120.4(2) |
| C21-C26-C25 | 120.81(19) |
| C32-C31-C36 | 120.43(17) |
| C32-C31-C1 | 123.30(16) |
| C36-C31-C1 | 116.20(15) |
| C31-C32-C33 | 120.52(18) |
| C34-C33-C32 | 119.70(18) |
| C33-C34-C35 | 120.29(18) |
| C34-C35-C36 | 120.98(18) |
| C35-C36-C31 | 118.05(17) |
| C35-C36-C4 | 122.83(16) |
| C31-C36-C4 | 119.03(16) |
| C42-C41-C46 | 117.99(17) |
| C42-C41-C3 | 119.90(16) |
| C46-C41-C3 | 122.05(17) |
| C43-C42-C41 | 121.06(18) |
| C44-C43-C42 | 120.30(19) |
| C43-C44-C45 | 119.64(19) |
| C44-C45-C46 | 120.28(19) |
| C45-C46-C41 | 120.68(18) |
| Cl3-C51-Cl2 | 110.07(17) |
| Cl3-C51-Cl1 | 111.57(17) |
| Cl2-C51-Cl1 | 109.9(2) |
| Cl1B-C51B-Cl2B | 107.1(8) |
| Cl1B-C51B-Cl3B | 109.8(11) |
| Cl2B-C51B-Cl3B | 106.5(10) |
| C9-S1B-C7 | 95.0(6) |
| C7-C8B-C10 | 114.2(12) |

Table 18: Crystal data and structure refinement for dds22 (**7a**).

| | | |
|---|---|---|
| Identification code | dds22 (**7a**) | |
| Empirical formula | $C_{36}H_{28}O_3$ | |
| Formula weight | 508.58 | |
| Temperature | 200(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | monoclinic | |
| Space group | $P2_1/c$ | |
| Z | 4 | |
| Unit cell dimensions | a = 8.905(4) Å | $\alpha$ = 90 deg. |
| | b = 30.80(1) Å | $\beta$ = 99.91(1) deg. |
| | c = 10.012(5) Å | $\gamma$ = 90 deg. |
| Volume | 2705(2) $Å^3$ | |
| Density (calculated) | 1.25 $g/cm^3$ | |
| Absorption coefficient | 0.08 $mm^{-1}$ | |
| Crystal shape | irregular | |
| Crystal size | 0.24 x 0.14 x 0.10 $mm^3$ | |
| Theta range for data collection | 2.5 to 20.8 deg. | |
| Index ranges | $-8\le h\le 8$, $-30\le k\le 30$, $-9\le l\le 9$ | |
| Reflections collected | 14007 | |
| Independent reflections | 2777 (R(int) = 0.1048) | |
| Observed reflections | 1933 ($I > 2\sigma(I)$) | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.99 and 0.98 | |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 2777 / 0 / 464 |
| Goodness-of-fit on $F^2$ | 0.91 |
| Final R indices (I>2σ(I)) | R1 = 0.047, wR2 = 0.108 |
| Largest diff. peak and hole | 0.17 and -0.17 eÅ$^{-3}$ |

Tabelle 19: Atomic coordinates and equivalent isotropic displacement parameters (Å$^2$) for dds22 (**7a**). $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.)

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| O1 | 1.1064(3) | 0.6007(1) | 1.2654(2) | 0.0484(6) |
| O2 | 1.1361(3) | 0.6481(1) | 0.9595(2) | 0.0484(7) |
| O3 | 1.1984(3) | 0.4903(1) | 0.5771(2) | 0.0652(8) |
| C1 | 1.0570(4) | 0.6377(1) | 1.2467(3) | 0.0363(8) |
| C2 | 0.9295(4) | 0.6466(1) | 1.1280(3) | 0.0354(8) |
| C3 | 0.9184(4) | 0.6127(1) | 1.0149(3) | 0.0356(8) |
| C4 | 1.0540(4) | 0.6159(1) | 0.9406(3) | 0.0381(8) |
| C5 | 0.7480(4) | 0.6173(1) | 0.9350(3) | 0.0374(8) |
| C11 | 0.7705(4) | 0.6445(1) | 1.1629(3) | 0.0379(8) |
| C12 | 0.7219(4) | 0.6571(1) | 1.2815(4) | 0.0456(10) |
| C13 | 0.5696(5) | 0.6508(1) | 1.2917(4) | 0.0562(11) |
| C14 | 0.4691(5) | 0.6332(1) | 1.1865(4) | 0.0584(11) |
| C15 | 0.5177(4) | 0.6208(1) | 1.0674(4) | 0.0515(10) |
| C16 | 0.6691(4) | 0.6270(1) | 1.0565(3) | 0.0381(8) |
| C21 | 1.1163(4) | 0.6733(1) | 1.3391(3) | 0.0405(9) |
| C22 | 1.2175(5) | 0.6633(2) | 1.4569(4) | 0.0647(11) |
| C23 | 1.2786(6) | 0.6958(2) | 1.5454(5) | 0.0865(15) |
| C24 | 1.2395(6) | 0.7385(2) | 1.5166(5) | 0.0799(14) |
| C25 | 1.1394(6) | 0.7485(2) | 1.4017(5) | 0.0821(14) |
| C26 | 1.0797(5) | 0.7162(1) | 1.3137(4) | 0.0617(11) |
| C31 | 0.7343(4) | 0.6569(1) | 0.8408(3) | 0.0407(9) |
| C32 | 0.6356(5) | 0.6912(1) | 0.8541(4) | 0.0529(10) |
| C33 | 0.6256(6) | 0.7273(1) | 0.7699(5) | 0.0707(12) |
| C34 | 0.7123(6) | 0.7295(2) | 0.6690(5) | 0.0760(15) |
| C35 | 0.8102(6) | 0.6962(2) | 0.6526(4) | 0.0639(12) |
| C36 | 0.8206(5) | 0.6598(1) | 0.7361(3) | 0.0504(10) |
| C41 | 0.6881(4) | 0.5750(1) | 0.8650(3) | 0.0407(9) |
| C42 | 0.6459(4) | 0.5708(1) | 0.7257(4) | 0.0466(10) |
| C43 | 0.6043(5) | 0.5307(1) | 0.6674(5) | 0.0621(11) |
| C44 | 0.5979(5) | 0.4949(2) | 0.7466(5) | 0.0663(13) |
| C45 | 0.6339(4) | 0.4985(1) | 0.8855(5) | 0.0580(11) |
| C46 | 0.6767(4) | 0.5383(1) | 0.9431(4) | 0.0486(10) |
| C51 | 1.0881(4) | 0.5808(1) | 0.8499(3) | 0.0383(8) |
| C52 | 1.0287(4) | 0.5392(1) | 0.8486(3) | 0.0417(9) |
| C53 | 1.0631(4) | 0.5078(1) | 0.7606(3) | 0.0441(9) |
| C54 | 1.1596(4) | 0.5178(1) | 0.6713(3) | 0.0477(10) |
| C55 | 1.2218(5) | 0.5591(1) | 0.6720(4) | 0.0570(11) |
| C56 | 1.1887(4) | 0.5899(1) | 0.7601(3) | 0.0479(10) |
| C57 | 1.1481(8) | 0.4462(1) | 0.5802(6) | 0.0734(14) |

Tabelle 20: Hydrogen coordinates and isotropic displacement parameters (Å$^2$) for dds22 (**7a**).

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| H2 | 0.947(3) | 0.6760(9) | 1.092(3) | 0.032(8) |
| H3 | 0.922(3) | 0.5843(9) | 1.058(3) | 0.034(8) |
| H12 | 0.793(4) | 0.6674(9) | 1.358(3) | 0.040(9) |
| H13 | 0.544(4) | 0.6577(9) | 1.376(3) | 0.049(10) |
| H14 | 0.365(5) | 0.6269(11) | 1.197(3) | 0.073(12) |
| H15 | 0.444(4) | 0.6091(9) | 0.984(3) | 0.053(10) |
| H22 | 1.249(5) | 0.6316(14) | 1.467(4) | 0.094(14) |
| H23 | 1.340(5) | 0.6884(14) | 1.628(5) | 0.101(16) |
| H24 | 1.274(5) | 0.7606(14) | 1.581(4) | 0.101(15) |
| H25 | 1.118(6) | 0.7792(17) | 1.379(5) | 0.126(19) |
| H26 | 1.000(5) | 0.7243(13) | 1.231(4) | 0.091(14) |
| H32 | 0.575(4) | 0.6899(11) | 0.930(3) | 0.067(12) |
| H33 | 0.557(5) | 0.7521(14) | 0.791(4) | 0.102(15) |
| H34 | 0.710(5) | 0.7555(15) | 0.620(4) | 0.094(15) |
| H35 | 0.875(4) | 0.6985(10) | 0.580(3) | 0.060(11) |
| H36 | 0.895(4) | 0.6342(10) | 0.723(3) | 0.052(10) |
| H42 | 0.651(3) | 0.5952(10) | 0.669(3) | 0.049(10) |
| H43 | 0.573(4) | 0.5296(11) | 0.569(4) | 0.073(12) |
| H44 | 0.572(4) | 0.4688(11) | 0.712(3) | 0.052(10) |
| H45 | 0.625(4) | 0.4755(12) | 0.946(3) | 0.064(12) |
| H46 | 0.701(3) | 0.5406(9) | 1.039(3) | 0.048(10) |
| H52 | 0.959(3) | 0.5320(9) | 0.911(3) | 0.043(9) |
| H53 | 1.021(3) | 0.4803(10) | 0.765(3) | 0.039(9) |
| H55 | 1.286(4) | 0.5652(10) | 0.610(3) | 0.063(11) |
| H56 | 1.240(4) | 0.6206(11) | 0.767(3) | 0.057(10) |
| H57A | 1.189(5) | 0.4329(13) | 0.669(4) | 0.091(15) |
| H57B | 1.176(5) | 0.4327(13) | 0.496(4) | 0.097(14) |
| H57C | 1.044(6) | 0.4470(16) | 0.559(5) | 0.11(2) |

Tabelle 21: Anisotropic displacement parameters ($Å^2$) for dds22 (**7a**). The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0476(16) | 0.0418(15) | 0.0543(15) | 0.0020(12) | 0.0043(11) | 0.0019(12) |
| O2 | 0.0495(16) | 0.0403(14) | 0.0604(16) | -0.0046(11) | 0.0234(12) | -0.0106(13) |
| O3 | 0.091(2) | 0.0448(17) | 0.0716(17) | -0.0099(13) | 0.0489(16) | -0.0004(14) |
| C1 | 0.035(2) | 0.039(2) | 0.037(2) | -0.0014(17) | 0.0150(17) | -0.0016(17) |
| C2 | 0.034(2) | 0.034(2) | 0.040(2) | -0.0011(16) | 0.0099(17) | -0.0028(16) |
| C3 | 0.035(2) | 0.032(2) | 0.041(2) | -0.0026(17) | 0.0102(16) | -0.0037(16) |
| C4 | 0.041(2) | 0.036(2) | 0.039(2) | 0.0078(16) | 0.0125(17) | 0.0021(18) |
| C5 | 0.037(2) | 0.0369(19) | 0.0396(19) | -0.0035(16) | 0.0101(16) | -0.0016(16) |
| C11 | 0.039(2) | 0.0386(19) | 0.038(2) | -0.0017(16) | 0.0105(18) | 0.0023(16) |
| C12 | 0.042(3) | 0.050(2) | 0.045(2) | -0.0086(18) | 0.009(2) | 0.0000(18) |
| C13 | 0.048(3) | 0.068(3) | 0.057(3) | -0.014(2) | 0.023(2) | -0.001(2) |
| C14 | 0.042(3) | 0.067(3) | 0.071(3) | -0.013(2) | 0.024(3) | -0.004(2) |
| C15 | 0.042(3) | 0.055(2) | 0.058(3) | -0.011(2) | 0.010(2) | -0.0026(19) |
| C16 | 0.034(2) | 0.039(2) | 0.041(2) | -0.0021(16) | 0.0088(18) | 0.0031(16) |
| C21 | 0.040(2) | 0.048(2) | 0.035(2) | -0.0049(17) | 0.0099(17) | 0.0003(17) |
| C22 | 0.073(3) | 0.065(3) | 0.052(3) | -0.005(2) | 0.000(2) | -0.010(2) |
| C23 | 0.098(4) | 0.098(5) | 0.053(3) | -0.011(3) | -0.016(3) | -0.023(3) |
| C24 | 0.089(4) | 0.079(4) | 0.069(4) | -0.030(3) | 0.006(3) | -0.018(3) |
| C25 | 0.096(4) | 0.062(3) | 0.081(4) | -0.026(3) | -0.006(3) | -0.001(3) |
| C26 | 0.066(3) | 0.050(3) | 0.063(3) | -0.016(2) | -0.006(2) | 0.000(2) |
| C31 | 0.044(2) | 0.037(2) | 0.038(2) | -0.0046(16) | -0.0025(17) | -0.0062(18) |
| C32 | 0.056(3) | 0.045(2) | 0.052(3) | -0.005(2) | -0.006(2) | -0.003(2) |
| C33 | 0.080(3) | 0.050(3) | 0.072(3) | 0.004(3) | -0.015(3) | 0.002(2) |
| C34 | 0.102(4) | 0.047(3) | 0.066(3) | 0.015(3) | -0.024(3) | -0.016(3) |
| C35 | 0.078(3) | 0.065(3) | 0.042(3) | 0.006(2) | -0.005(2) | -0.025(3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C36 | 0.057(3) | 0.051(2) | 0.040(2) | 0.002(2) | 0.002(2) | -0.010(2) |
| C41 | 0.036(2) | 0.042(2) | 0.046(2) | -0.0096(18) | 0.0130(17) | -0.0044(16) |
| C42 | 0.050(2) | 0.045(2) | 0.048(3) | -0.007(2) | 0.0161(19) | -0.0064(19) |
| C43 | 0.070(3) | 0.060(3) | 0.061(3) | -0.018(3) | 0.023(2) | -0.015(2) |
| C44 | 0.069(3) | 0.049(3) | 0.086(4) | -0.031(3) | 0.029(3) | -0.022(2) |
| C45 | 0.060(3) | 0.045(3) | 0.073(3) | -0.005(3) | 0.026(2) | -0.010(2) |
| C46 | 0.052(3) | 0.042(3) | 0.054(3) | -0.005(2) | 0.018(2) | -0.0070(18) |
| C51 | 0.041(2) | 0.037(2) | 0.041(2) | 0.0015(16) | 0.0165(17) | 0.0019(16) |
| C52 | 0.046(2) | 0.037(2) | 0.046(2) | 0.0027(18) | 0.0202(19) | 0.0035(17) |
| C53 | 0.050(2) | 0.033(2) | 0.053(2) | 0.0018(19) | 0.018(2) | 0.0042(18) |
| C54 | 0.059(3) | 0.042(2) | 0.048(2) | -0.0032(18) | 0.027(2) | 0.0061(19) |
| C55 | 0.070(3) | 0.048(3) | 0.064(3) | -0.004(2) | 0.044(2) | -0.006(2) |
| C56 | 0.049(2) | 0.044(2) | 0.056(2) | -0.0026(19) | 0.023(2) | -0.0053(19) |
| C57 | 0.111(5) | 0.046(3) | 0.074(4) | -0.010(2) | 0.046(3) | -0.002(3) |

Tabelle 22: Bond lengths (Å) and angles (deg) for dds22 (**7a**).

| | | | |
|---|---|---|---|
| O1-C1 | 1.225(3) | C34-H34 | 0.94(4) |
| O2-C4 | 1.229(3) | C35-C36 | 1.392(5) |
| O3-C54 | 1.358(4) | C35-H35 | 1.00(3) |
| O3-C57 | 1.430(5) | C36-H36 | 1.05(3) |
| C1-C21 | 1.472(4) | C41-C42 | 1.386(5) |
| C1-C2 | 1.520(4) | C41-C46 | 1.389(5) |
| C2-C11 | 1.518(4) | C42-C43 | 1.391(5) |
| C2-C3 | 1.531(4) | C42-H42 | 0.94(3) |
| C2-H2 | 1.00(3) | C43-C44 | 1.365(6) |
| C3-C4 | 1.527(4) | C43-H43 | 0.98(4) |
| C3-C5 | 1.596(5) | C44-C45 | 1.376(6) |
| C3-H3 | 0.97(3) | C44-H44 | 0.89(3) |
| C4-C51 | 1.476(4) | C45-C46 | 1.381(5) |
| C5-C31 | 1.532(4) | C45-H45 | 0.94(3) |
| C5-C41 | 1.531(4) | C46-H46 | 0.95(3) |
| C5-C16 | 1.534(4) | C51-C52 | 1.385(4) |
| C11-C16 | 1.382(4) | C51-C56 | 1.402(4) |
| C11-C12 | 1.387(4) | C52-C53 | 1.379(4) |
| C12-C13 | 1.391(5) | C52-H52 | 0.98(3) |
| C12-H12 | 0.96(3) | C53-C54 | 1.378(4) |
| C13-C14 | 1.371(5) | C53-H53 | 0.93(3) |
| C13-H13 | 0.94(3) | C54-C55 | 1.386(5) |
| C14-C15 | 1.391(5) | C55-C56 | 1.362(5) |
| C14-H14 | 0.97(4) | C55-H55 | 0.93(3) |
| C15-C16 | 1.384(5) | C56-H56 | 1.05(3) |
| C15-H15 | 1.03(3) | C57-H57A | 0.99(4) |
| C21-C26 | 1.374(5) | C57-H57B | 1.01(4) |
| C21-C22 | 1.390(5) | C57-H57C | 0.92(5) |
| C22-C23 | 1.385(6) | C54-O3-C57 | 117.4(3) |
| C22-H22 | 1.02(4) | O1-C1-C21 | 121.1(3) |
| C23-C24 | 1.379(7) | O1-C1-C2 | 119.1(3) |
| C23-H23 | 0.94(5) | C21-C1-C2 | 119.8(3) |
| C24-C25 | 1.364(6) | C11-C2-C1 | 114.4(3) |
| C24-H24 | 0.95(5) | C11-C2-C3 | 101.4(2) |
| C25-C26 | 1.373(6) | C1-C2-C3 | 113.6(3) |
| C25-H25 | 0.98(5) | C11-C2-H2 | 109.6(15) |
| C26-H26 | 1.03(4) | C1-C2-H2 | 107.7(16) |
| C31-C32 | 1.396(5) | C3-C2-H2 | 110.0(14) |
| C31-C36 | 1.405(5) | C4-C3-C2 | 111.1(3) |
| C32-C33 | 1.389(6) | C4-C3-C5 | 120.9(3) |
| C32-H32 | 1.00(3) | C2-C3-C5 | 104.1(2) |
| C33-C34 | 1.375(6) | C4-C3-H3 | 107.6(16) |
| C33-H33 | 1.02(4) | C2-C3-H3 | 106.9(15) |
| C34-C35 | 1.375(6) | C5-C3-H3 | 105.3(17) |

| | | | |
|---|---|---|---|
| O2-C4-C51 | 120.4(3) | C42-C41-C5 | 123.6(3) |
| O2-C4-C3 | 118.5(3) | C46-C41-C5 | 119.3(3) |
| C51-C4-C3 | 121.1(3) | C41-C42-C43 | 120.8(4) |
| C31-C5-C41 | 114.2(3) | C41-C42-H42 | 119.6(18) |
| C31-C5-C16 | 109.6(2) | C43-C42-H42 | 119.5(18) |
| C41-C5-C16 | 110.9(2) | C44-C43-C42 | 120.6(4) |
| C31-C5-C3 | 110.4(3) | C44-C43-H43 | 122(2) |
| C41-C5-C3 | 112.3(2) | C42-C43-H43 | 117(2) |
| C16-C5-C3 | 98.4(2) | C43-C44-C45 | 119.8(4) |
| C16-C11-C12 | 120.7(3) | C43-C44-H44 | 122(2) |
| C16-C11-C2 | 110.5(3) | C45-C44-H44 | 118(2) |
| C12-C11-C2 | 128.8(3) | C44-C45-C46 | 119.5(4) |
| C11-C12-C13 | 118.4(4) | C44-C45-H45 | 124(2) |
| C11-C12-H12 | 120.9(18) | C46-C45-H45 | 117(2) |
| C13-C12-H12 | 120.4(17) | C45-C46-C41 | 122.0(4) |
| C14-C13-C12 | 121.0(4) | C45-C46-H46 | 119.1(19) |
| C14-C13-H13 | 124(2) | C41-C46-H46 | 118.8(19) |
| C12-C13-H13 | 115(2) | C52-C51-C56 | 117.6(3) |
| C13-C14-C15 | 120.6(4) | C52-C51-C4 | 124.3(3) |
| C13-C14-H14 | 120(2) | C56-C51-C4 | 118.1(3) |
| C15-C14-H14 | 119(2) | C53-C52-C51 | 121.9(3) |
| C16-C15-C14 | 118.7(4) | C53-C52-H52 | 119.0(16) |
| C16-C15-H15 | 118.6(17) | C51-C52-H52 | 119.0(16) |
| C14-C15-H15 | 122.5(18) | C54-C53-C52 | 119.3(3) |
| C11-C16-C15 | 120.6(3) | C54-C53-H53 | 122.5(17) |
| C11-C16-C5 | 111.1(3) | C52-C53-H53 | 118.3(18) |
| C15-C16-C5 | 128.3(3) | O3-C54-C53 | 124.9(3) |
| C26-C21-C22 | 117.9(4) | O3-C54-C55 | 115.4(3) |
| C26-C21-C1 | 123.5(3) | C53-C54-C55 | 119.7(3) |
| C22-C21-C1 | 118.6(3) | C56-C55-C54 | 120.8(4) |
| C23-C22-C21 | 120.6(5) | C56-C55-H55 | 121(2) |
| C23-C22-H22 | 124(2) | C54-C55-H55 | 118(2) |
| C21-C22-H22 | 115(2) | C55-C56-C51 | 120.6(3) |
| C24-C23-C22 | 119.9(5) | C55-C56-H56 | 122.2(17) |
| C24-C23-H23 | 120(3) | C51-C56-H56 | 117.2(16) |
| C22-C23-H23 | 120(3) | O3-C57-H57A | 110(2) |
| C25-C24-C23 | 119.8(5) | O3-C57-H57B | 105(2) |
| C25-C24-H24 | 120(3) | H57A-C57-H57B | 118(3) |
| C23-C24-H24 | 120(3) | O3-C57-H57C | 106(3) |
| C24-C25-C26 | 120.2(5) | H57A-C57-H57C | 115(4) |
| C24-C25-H25 | 119(3) | H57B-C57-H57C | 102(4) |
| C26-C25-H25 | 121(3) | | |
| C25-C26-C21 | 121.7(4) | | |
| C25-C26-H26 | 119(2) | | |
| C21-C26-H26 | 119(2) | | |
| C32-C31-C36 | 117.2(3) | | |
| C32-C31-C5 | 121.8(3) | | |
| C36-C31-C5 | 121.0(3) | | |
| C33-C32-C31 | 121.6(4) | | |
| C33-C32-H32 | 120(2) | | |
| C31-C32-H32 | 118(2) | | |
| C34-C33-C32 | 120.0(5) | | |
| C34-C33-H33 | 123(2) | | |
| C32-C33-H33 | 117(2) | | |
| C35-C34-C33 | 119.8(5) | | |
| C35-C34-H34 | 122(3) | | |
| C33-C34-H34 | 117(3) | | |
| C34-C35-C36 | 120.6(5) | | |
| C34-C35-H35 | 119.2(19) | | |
| C36-C35-H35 | 120(2) | | |
| C35-C36-C31 | 120.7(4) | | |
| C35-C36-H36 | 120.4(16) | | |
| C31-C36-H36 | 118.8(16) | | |
| C42-C41-C46 | 117.1(3) | | |

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[1] Various authors, *Organikum*, 14. edition, VEB Deutscher Verlag der Wissenschaften, Berlin, 1993.

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[3] Two quaternary carbon resonances overlap.

[4] One quaternary carbon resonance overlaps.

[5] One quaternary carbon resonance overlaps.