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SUPPORTING INFORMATION

Supplementary Material

Synthesis of Organometallic Dehydroannulens Containing Ferrocene or (Cyclopentadienylcobalt)cyclobutadiene Moieties

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Received

Materials: The synthesis of **1**, **2**, **8** and **9** is described elsewhere[1]. Copper(I)chloride, TMEDA and tetramethylammonium fluoride were used as purchased from Aldrich. Pentane, heptane, dichloromethane and acetone were purchased from Riedel de Haen and used for HPLC in *pa* quality. ^{1}H NMR and ^{13}C NMR spectra were measured on a Bruker AM 300 with 300 and 75 MHz respectively in dichloromethane-d_2 relative to the chemical shift of residual CH_2Cl_2 signals. IR: Nicolet DX 320. UV: Cary 219. HPLC: Gilson Abimed 305 with a Merck LiCroSorb CN (10 μm) cyanophase column (21 x 5 cm) was used with a 19 : 1 mixture of heptane and acetone as eluent (flow rate 20 mL/min, pressure 80 bar). Preparative gel permeation chromatography: Abimed M305, polystyrene gel; eluent chloroform (flow rate 7 mL/min, pressure 80 bar). Column Chromatography: Neutral aluminum oxide activity III (Riedel de Haen), Baker silica gel 600 - 200 mesh. Melting points are uncorrected. Elemental analyses: Mikroanalytisches Laboratorium des Organischen Institutes der Universität Mainz. Cyclic voltammetry: potentiostat/galvanostat EG&G, M 273, Princeton Applied Research. Oxidations were carried out in dichloromethane,

reductions in dry THF (from potassium benzophenone). Tetrabutylammonium hexafluorophosphate was used as supporting electrolyte (Fluka, electrochemical grade). Working electrodes: gold wire 1 mm o.d. sealed in glass; counter electrodes platinum wire; silver wire as pseudo-reference; the potentials were calibrated against SCE with the ferrrocenium/ferrocene redox couple: + 0.310 V vs. SCE. The potential ramp was 0.1 V s^{-1} unless otherwise stated. In case of the irreversible electron transfer, the peak potentials are given.

Crystal structure determination: Suitable crystals were grown by slow concentration of diluted solutions of **3a** from dichloromethane/pentane. Crystal data for $(C_{57}H_{69}Si_6Co_3)*(CH_2Cl_2)_4*(C_5H_{12})_2$: Enraf-Nonius-CAD4-diffractometer, CuKα-radiation. The structure was solved by heavy atom methods (Patterson). Parameters of the H atoms were refined in the *riding mode* with isotropic temperature factors. An empirical absorption correction was performed. Program used: Molen. a = 16.697(8), b = 23.946(9), c = 18.104(8), α=β=γ=90°, *Pnma* (orthorhombic), Z = 4, V = 7238.4 Å^3, $\rho_{calcd.}$ = 1.087 gcm^{-3}, μ = 76.96 cm^{-1}; 5455 obtained reflexes, 1997 observed (I > 3σ(I)), R = 0.082, R_w = 0.086. The ring was located on a mirror plane. The crystals contained solvent in the asymmetric unit cell (2 molecule dichloromethane, 1 pentane), which evaporated or desolvated quickly with destruction of the crystals; thus the X-ray measurment had to be performed in a sealed capillary. The solvates were disordered and were refined isotropically. The relatively high R-value arises mostly from the disorder of the solvent molecules and disorder in four of the six TMS-groups (each has two roughly equally occupied positions).

Hay-Coupling of 1a: A solution of 1.08 g (2.93 mmol) **1a** and 0.594 g (6.00 mmol) of CuCl in 200 mL of TMEDA is heated to reflux (122 °C). For 1 h O_2 is bubbled through the solution. The TMEDA is removed at 0.1 Torr/40 °C and the residue worked up aqueous. Filtration over aluminum oxide with pentane/dichlormethane (80 : 20) yields 683 mg (63%) raw product. The

separation of **3a,b** is achieved by repetitive chromatography of the residue over aluminum oxide with pentane as eluent to give 109 mg (10.1%) (**3a**) as a first fraction followed by 48 mg (4.4%) **3b**. Compounds **4** and **5** were isolated as afterrun and preseparated by preparative GPC into **4** and **5** which were separated into their stereoisomers by preparative HPLC to yield **4a** (43 mg, 4.0%); as a second band a mixture of **4b,c** (192 mg, 17.8%) and as last fraction **4d** (51 mg, 4.7%); the pentamers **5** were isolated as a mixture of stereoisomers (130 mg, 12.0%).

Vögtle-Coupling of 1a: A solution of 470 mg (1.28 mmol) **1a** and 3.10 g (17.0 mmol) copper(II)acetate in 300 mL of acetonitrile is heated under nitrogen to reflux (80 °C) for 3 h. The acetonitrile is removed under reduced pressure. Aqueous workup with dichloromethane followed by filtration over aluminium oxide with pentane/dichloromethane (80 : 20) yields 409 mg (87%) raw product. The yield of the cycles and the partition into different stereoisomeres is determined by analytical GPC and HPLC. **3a** 78 mg (16.6%); **3b** 37 mg (7.9%); **4a** 31 mg (6.6%); **4b,c** 126 mg (26.6%); **4d** 35 mg (7.5%); **5** 61 mg (13.0%).

3a: mp > 230 °C dec.; IR (KBr) ν 3107, 2956, 2900, 2178, 2120, 1259, 1247, 841, 811 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 0.20 (s, 18 H), 0.21 (s, 36 H), 4.99 (s, 5 H), 5.03 (s, 10 H); ^{13}C NMR (CD_2Cl_2): δ 0.14 (q, 12 C), 0.17 (q, 6 C), 68.70, 68.87, 69.03, 73.68, 73.72, 74.04 (6 s, 12 C), 81.68 (d, 10 C), 81.79 (d, 5 C), 78.57, 78.70, 78.87, 80.91, 81.18, 81.26 (6 s, 12 C); UV (CH_2Cl_2) λ_{max} (ε) 264 (45050), 308 (29470), 391 (33280); MS (FD) m/z 1099.1; $C_{57}H_{69}Si_6Co_3$ (1099.5) Calcd C 62.27, H 6.33; found C 61.93 H 6.46.

3b: mp > 230 °C dec.; IR (KBr) ν 3107, 2955, 2900, 2176, 2126, 1259, 1247, 841, 812 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 0.22 (s, 54 H), 5.06 (s, 15 H); ^{13}C NMR (CD_2Cl_2): δ 1.56 (q, 18 C), 70.38, 75.17 (2 s, 12 C), 83.01 (d, 15 C), 80.31, 82.54 (2 s, 12 C); UV (CH_2Cl_2) λ_{max} (ε) 264 (49560), 392 (29890); MS (FD) m/z 1099.1; $C_{57}H_{69}Si_6Co_3$ (1099.5) Calcd C 62.27, H 6.33; found C 62.56 H 5.90.

4a: mp > 240 °C dec.; IR (KBr) ν 3108, 2954, 2925, 2904, 2366, 2179, 2124, 1259, 1247, 841, 812 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 0.21 (s, 72 H), 5.01 (s, 20 H); ^{13}C NMR (CD_2Cl_2): δ 0.19 (q, 24 C), 67.51, 75.24 (2 s, 16 C), 81.95 (d, 20 C), 77.91, 80.07 (2 s, 16 C); UV (CH_2Cl_2) λ_{max} (ε) 266 (75400), 360 (45750); MS (FD) m/z 1465.1; $C_{76}H_{92}Si_8Co_4$ (1466.1) Calcd C 62.27, H 6.33; found C 61.99 H 6.34.

4b: IR (KBr) ν 3107, 2955, 2925, 2898, 2364, 2178, 2129, 2125, 2050, 1259, 1247, 842, 812 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 0.21 (s, 54 H), 0.22 (s, 18 H), 4.99 (s, 5 H), 5.04 (s, 10 H), 5.06 (s, 5 H); ^{13}C NMR (CD_2Cl_2): δ 0.27 (q, 24 C), 67.68, 67.83, 75.05, 75.16, 75.28 (5 s, 16 C), 81.84, 81.94, 82.00 (3 d, 20 C), 78.02, 78.17, 80.10, 80.20 (4 s, 16 C); MS (FD) m/z 1465.1.

4c: ^1H NMR (CD_2Cl_2): δ 0.21 (s, 72 H), 5.02 (s, 20 H).

4d: mp > 240 °C dec.; IR (KBr) ν 3106, 2955, 2924, 2367, 2180, 2127, 1260, 1247, 842, 811 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 0.20 (s, 72 H), 5.06 (s, 20 H); ^{13}C NMR (CD_2Cl_2): δ 0.20 (q, 24 C), 67.69, 75.08 (2 s, 16 C), 81.81 (d, 20 C), 78.08, 80.16 (2 s, 16 C); UV (CH_2Cl_2) λ_{max} (ε) 266 (81480), 401 (36470); MS (FD) m/z 1465.1; $C_{76}H_{92}Si_8Co_4$ (1466.1) Calcd C 62.27, H 6.33; found C 61.91 H 6.50.

Desilylation of 3a: To a suspension of $Me_4N^+F^-$ (3.8 mg, 0.041 mmol) in DMSO (5 mL) 22 mg (0.020 mmol) of **3a**, dissolved in ethyl ether (5 mL), were added under N_2 and stirred for 16 h at 21 °C. Aqueous workup and chromatography (silica gel, pentane/dichloromethane 4 : 1) affords 12 mg (90%)

6: mp > 156 °C dec.; IR (KBr) ν 3104, 2178, 2117, 1004, 816 cm^{-1}; ^1H NMR (CD_2Cl_2): δ 4.19 (s, 6 H), 5.05 (s, 5 H), 5.09 (s, 10 H); ^{13}C NMR (CD_2Cl_2): δ 59.46, 59.73, 59.93 (3 s, 6 C), 61.77, 61.82, 62.05 (3 d, 6 C), 81.64 (d, 10 C), 81.70 (d, 5 C), 78.36, 78.47, 78.52, 80.23, 80.54, 80.59 (6 s, 12 C); UV (CH_2Cl_2) λ_{max} (ε) 261 (39570), 315 (30400), 383 (41210); MS (FD) m/z 666.3; $C_{39}H_{21}Co_3$ (666.5) Calcd C 70.28, H 3.16; found C 70.17, H 3.75.

Oxidative coupling of 2: A mixture of 5.41 g (60.8 mmol) CuCl and 10.1 g (81 mmol) $CuCl_2$ in 100 mL of pyridine was prepared in a 250 mL flask under nitrogen. Then 948 mg (4.05 mmol) of **2** (in 30 mL of pentane) were added over 14 h using a syringe pump. After aqous workup (dichloromethane) followed by chromatography over flash silica gel with pentane/dichloromethane (80 : 20) gave 26 mg (2.7%) of unreacted **2**, 19 mg (2.0%) open dimer, 124 mg (13.1%) **7a**, 31 mg (3.6%) **7b** and 34 mg (3.6%) of higher cycles were obtained.

7a: mp. 165 °C dec.; IR(KBr) ν 3097, 2925, 2215, 2152, 1413, 1108, 1003, 825 cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.34 (bs, 3 H), 4.35, 4.40 (2 s, 15 H), 4.60 (bs, 6 H); ^{13}C NMR (CD_2Cl_2): δ 68.63, 68.68, 68.83 (3 s, 6 C), 69.37 (d, 3 C) 71.64, 71.70 (2 d, 6 C), 72.26, 72.37 (2 d, 15 C), 74.15, 74.20 (2 s, 6 C), 77.86, 78.03, 78.07 (3 s, 6 C,); UV (CH_2Cl_2) λ_{max} (ε) 296 (449370), 319 (494230), 383 (103620), 464 (61910); MS (FD) m/z 696.1; $C_{42}H_{24}Fe_3$ (696.2.5) Calcd C 72.47, H 3.48; found C 72.38, H 3.54.

7b: mp. 176 °C dec.; IR(KBr) ν 3099, 2925, 2212, 2150, 1106, 1001, 825 cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.30 (t, $^3J_{HH}$ = 2.70 Hz, 3 H), 4.41 (s, 15 H), 4.56 (d, $^2J_{HH}$ = 2.70 Hz, 6 H); ^{13}C NMR (CD_2Cl_2). δ 69.71 (s, 6 C), 70.22 (d, 6 C), 72.37 (d, 6 C), 72.72 (d, 15 C), 74.93 (s, 6 C), 78.53 (s, 6 C); UV (CH_2Cl_2) λ_{max} (ε) 272 (382210) 296 (420510), 319 (475080), 382 (103620), 465 (61910); MS (FD) m/z 696.1; $C_{42}H_{24}Fe_3$ (696.2) Calcd C 72.47, H 3.48; found C 72.18, H 3.65.

References:

[1] (a) Fritch, J. R.; Vollhardt, K. P. C. *Organometallics* **1982**, *1*, 560. (b) Bunz, U. H. F. *J. Organomet. Chem.* **1995**, *494*, C8. (c) Bunz, U. H. F.; Enkelmann, V.; Beer, F. *Organometallics* **1995**, *14*, 2490. (d) Altmann, M.; Enkelmann, V.; Beer, F.; Bunz, U. H. F. *Organometallics* **1996**, *15*, 394.

Figure 1: Yields and isomeric distribution of **4**

Figure 2: ORTEP-plot of **3a**

Figure 3: UV-vis spectra of **3** - **5** and **7** in dichloromethane

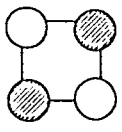
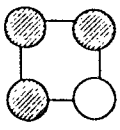
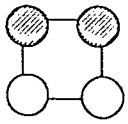
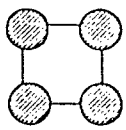
Isomer stereochemistry							
Isomer key	**4a**		**4b**		**4c**		**4d**
Expected isomeric ratio by statistics	1	:	4	:	2	:	1
isolated yields (%)	4.0		17.8				4.7

Figure 1 Yields and isomeric distribution of **4**

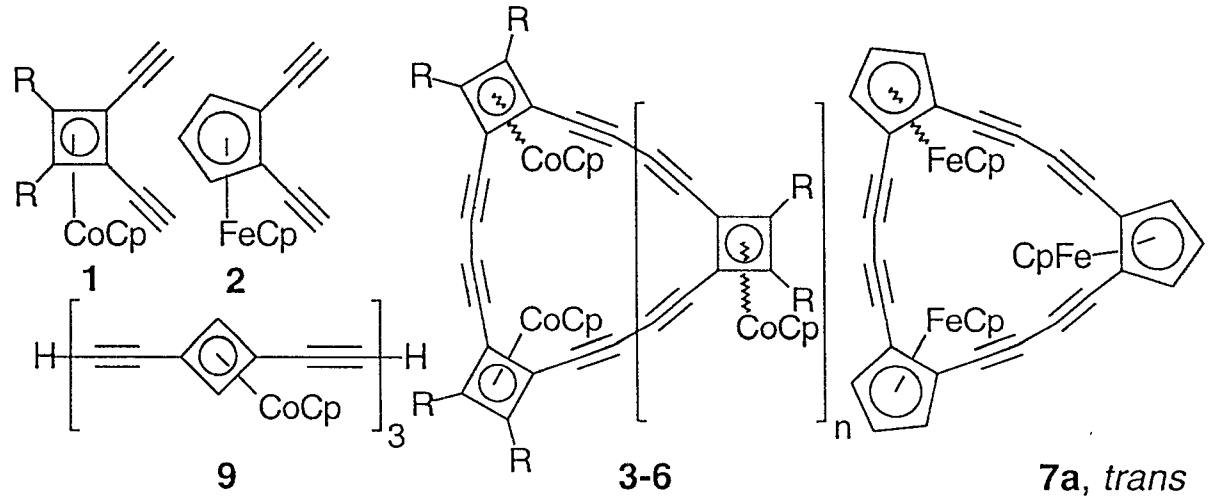
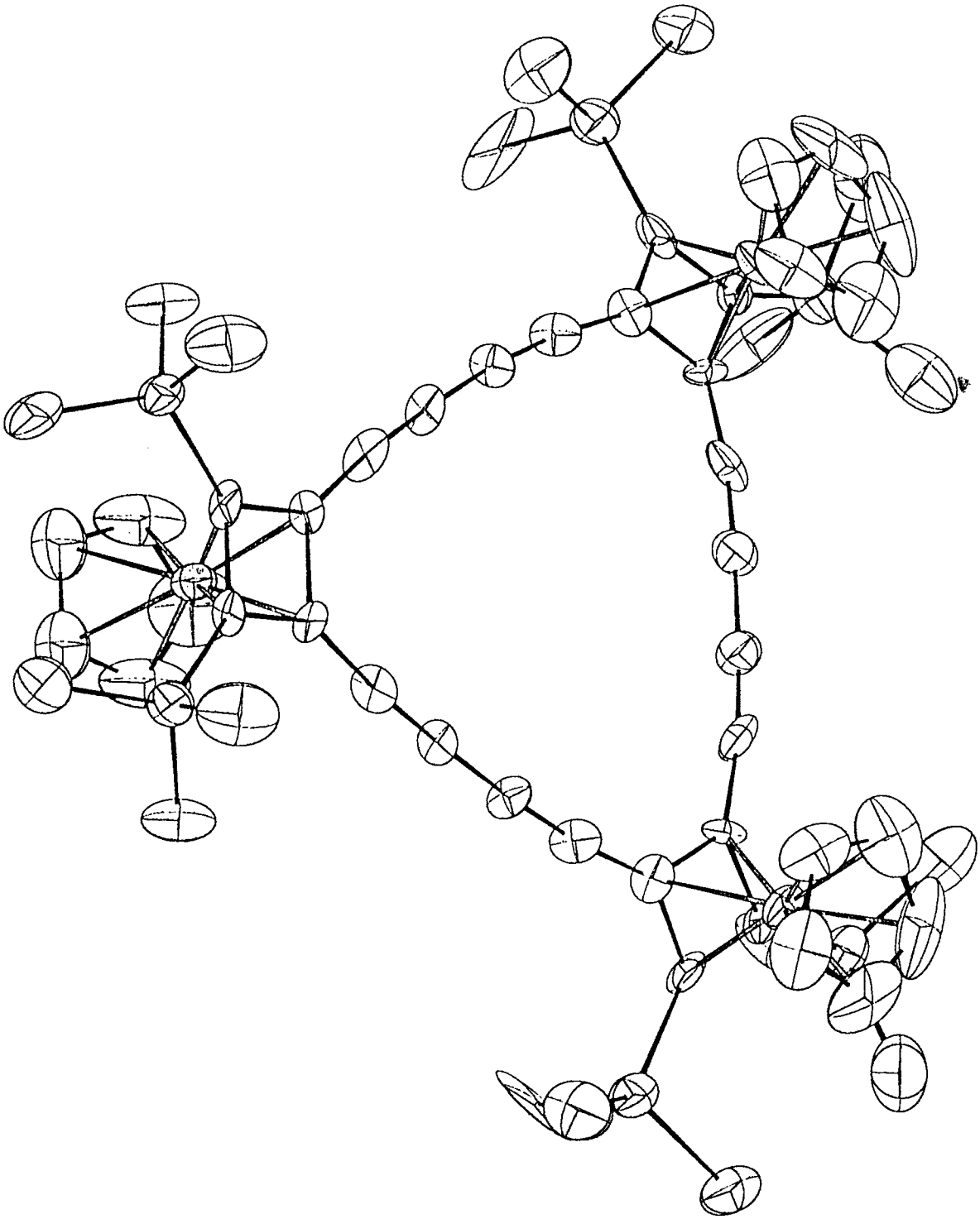


Figure 2



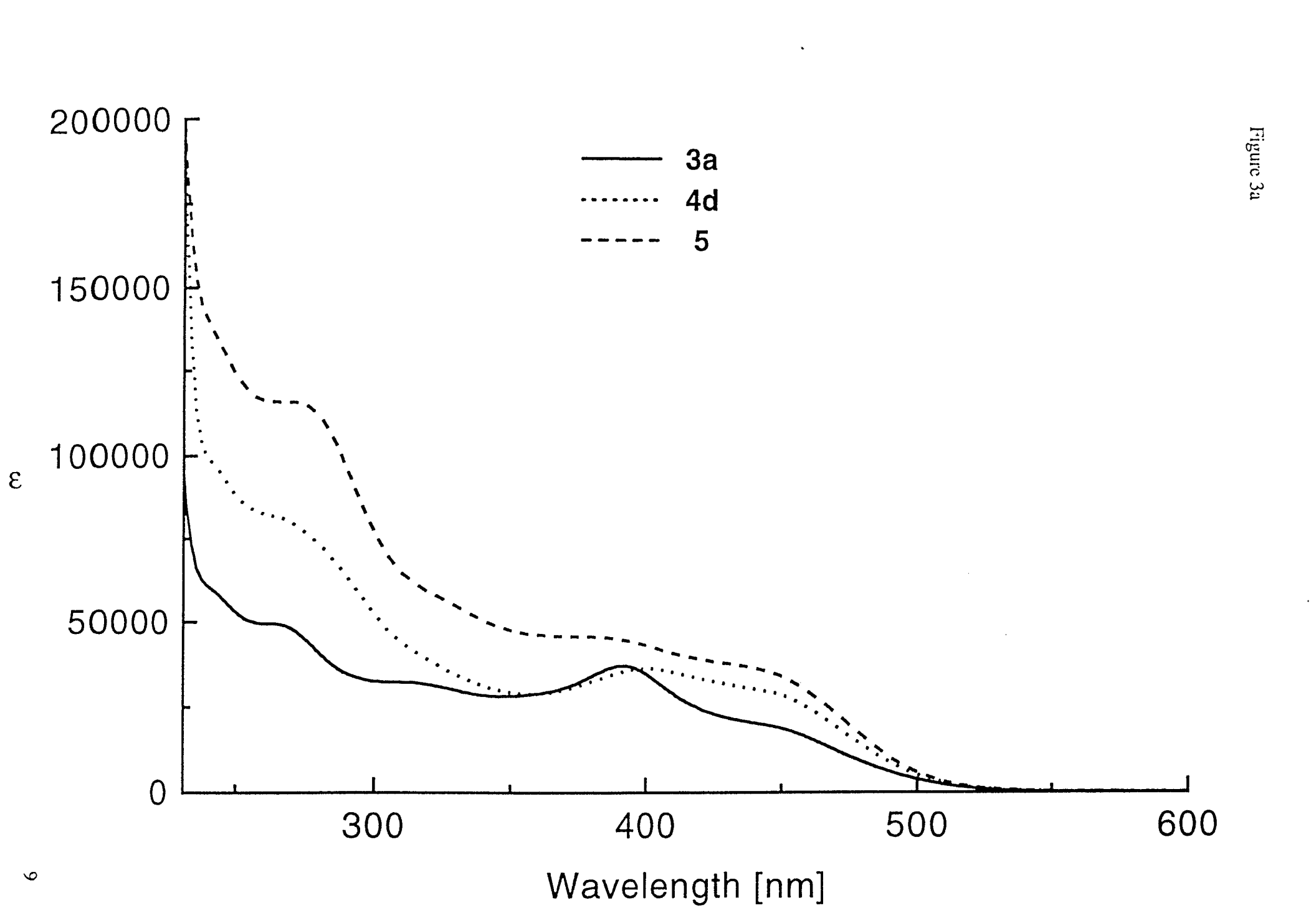

200000
150000
100000
50000
0
ε
3a
4d
5
300
400
500
600
Wavelength [nm]

Figure 3a

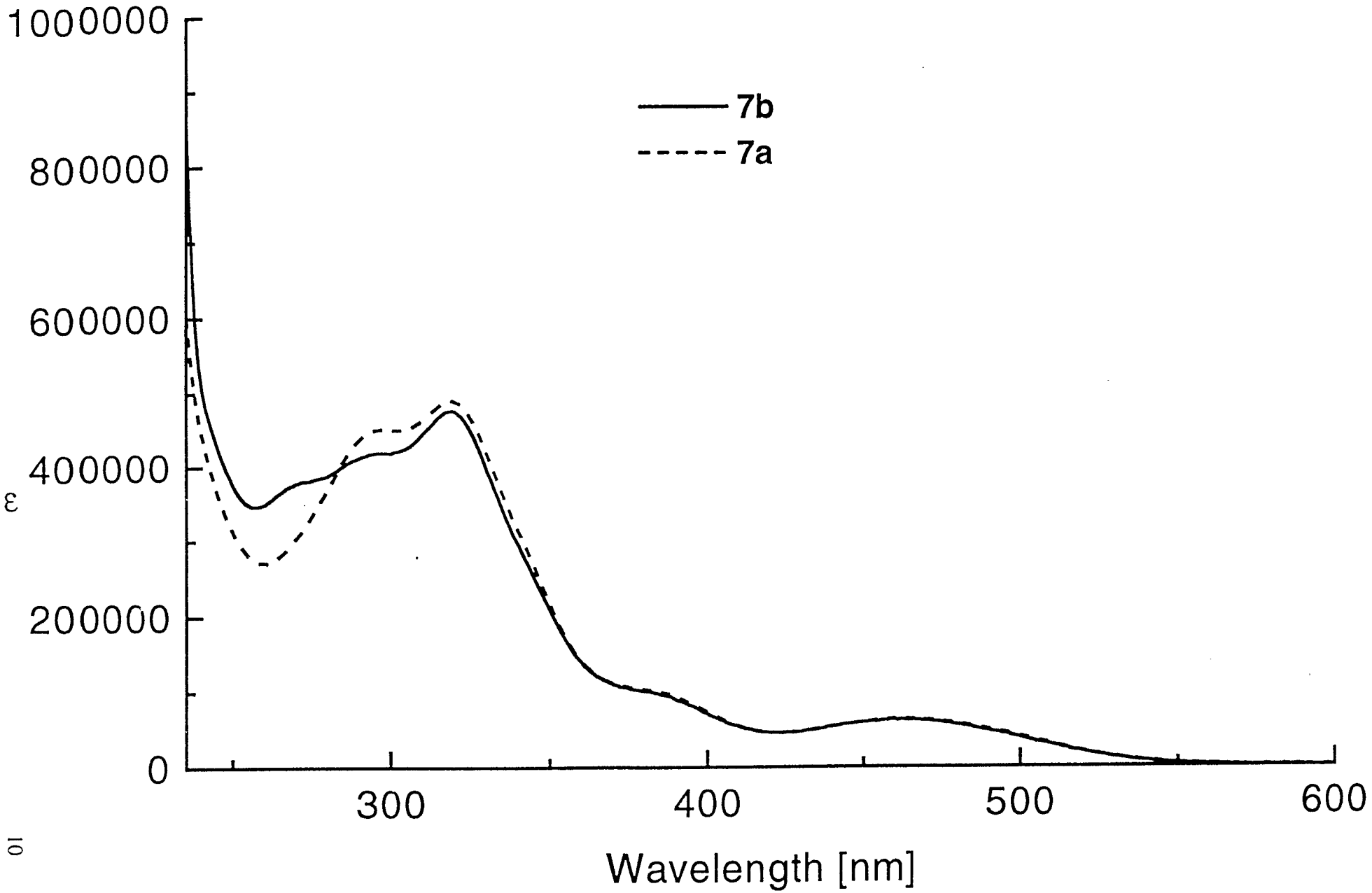

1000000
800000
600000
400000
200000
0
ε
10
300
400
500
600
Wavelength [nm]
7b
7a

Figure 3b

Bond Tables, Positional Parameters and U's

Atom	x/a	y/b	z/c	U(iso)	OCC
CO1	0.0455(3)	0.2500	0.1770(3)	0.0530	
C22	0.165(2)	0.2500	0.176(2)	0.1247	
C23	0.136(2)	0.2977(8)	0.137(2)	0.1209	
C24	0.091(2)	0.2799(7)	0.074(1)	0.1231	
CO2	0.1543(2)	0.4417(2)	0.6207(2)	0.0513	
C25	0.036(2)	0.424(2)	0.646(2)	0.0900	
C26	0.088(3)	0.409(2)	0.703(3)	0.1146	
C27	0.127(2)	0.459(3)	0.725(2)	0.1002	
C28	0.097(3)	0.502(1)	0.680(4)	0.0952	
C29	0.043(3)	0.480(3)	0.633(2)	0.1161	
H22	0.195(2)	0.2500	0.220(2)	0.1590	
H23	0.145(2)	0.3356(8)	0.150(2)	0.1650	
H24	0.067(2)	0.3034(7)	0.038(1)	0.1490	
H25	0.001(2)	0.399(2)	0.621(2)	0.1000	
H26	0.097(3)	0.372(2)	0.722(3)	0.1560	
H27	0.164(2)	0.463(3)	0.764(2)	0.1440	
H28	0.112(3)	0.541(1)	0.684(4)	0.1410	
H29	0.017(3)	0.501(3)	0.594(2)	0.1450	
SI1	-0.1113(4)	0.3409(3)	0.1560(4)	0.0614	
SI2	0.2373(5)	0.5384(3)	0.5004(5)	0.0727	
SI3	0.3557(4)	0.4444(3)	0.6666(4)	0.0695	
C1	-0.060(1)	0.2816(8)	0.203(1)	0.0353	
C2	-0.006(1)	0.2818(8)	0.265(1)	0.0213	
C3	0.029(1)	0.3185(9)	0.313(1)	0.0500	
C4	0.060(1)	0.3481(9)	0.360(1)	0.0274	
C5	0.097(1)	0.3779(9)	0.413(1)	0.0456	
C6	0.139(1)	0.400(1)	0.463(1)	0.0548	
C7	0.188(1)	0.418(1)	0.521(1)	0.0487	
C8	0.231(1)	0.4689(9)	0.544(1)	0.0484	
C9	0.271(1)	0.437(1)	0.603(1)	0.0426	
C10	0.228(1)	0.3864(9)	0.577(1)	0.0350	
C11	0.229(1)	0.330(1)	0.591(1)	0.0527	
C12	0.229(2)	0.279(1)	0.594(1)	0.0635	
C13	-0.047(1)	0.4029(8)	0.159(1)	0.1025	
H131	-0.073(1)	0.4335(8)	0.136(1)	0.1200	
H132	-0.034(1)	0.4123(8)	0.208(1)	0.1200	
H133	0.001(1)	0.3945(8)	0.133(1)	0.1200	
C14	-0.130(2)	0.329(1)	0.057(1)	0.0997	
H141	-0.156(2)	0.360(1)	0.036(1)	0.1280	
H142	-0.082(2)	0.321(1)	0.032(1)	0.1280	
H143	-0.164(2)	0.297(1)	0.054(1)	0.1280	
C15	-0.203(2)	0.359(1)	0.207(2)	0.1286	
H151	-0.229(2)	0.390(1)	0.184(2)	0.1550	
H152	-0.237(2)	0.328(1)	0.206(2)	0.1550	
H153	-0.190(2)	0.368(1)	0.257(2)	0.1550	
C16	0.140(2)	0.558(2)	0.457(3)	0.1051	0.593
H161	0.143(2)	0.594(2)	0.434(3)	0.1350	0.593
H162	0.128(2)	0.530(2)	0.421(3)	0.1350	0.593
H163	0.099(2)	0.559(2)	0.494(3)	0.1350	0.593
C17	0.257(4)	0.594(2)	0.572(3)	0.1358	0.593
H171	0.261(4)	0.630(2)	0.550(3)	0.1350	0.593

Atom	x/a	y/b	z/c	U(iso)	OCC
H172	0.215(4)	0.594(2)	0.608(3)	0.1350	0.593
H173	0.306(4)	0.584(2)	0.595(3)	0.1350	0.593
C18	0.313(3)	0.533(2)	0.424(2)	0.0707	0.593
H181	0.317(3)	0.569(2)	0.402(2)	0.1350	0.593
H182	0.364(3)	0.524(2)	0.445(2)	0.1350	0.593
H183	0.299(3)	0.506(2)	0.388(2)	0.1350	0.593
C162	0.345(3)	0.557(3)	0.487(4)	0.1246	0.407
H1621	0.349(3)	0.592(3)	0.464(4)	0.1350	0.407
H1622	0.374(3)	0.557(3)	0.533(4)	0.1350	0.407
H1623	0.367(3)	0.529(3)	0.456(4)	0.1350	0.407
C172	0.184(4)	0.541(3)	0.410(3)	0.1152	0.407
H1721	0.189(4)	0.577(3)	0.388(3)	0.1350	0.407
H1722	0.206(4)	0.513(3)	0.378(3)	0.1350	0.407
H1723	0.129(4)	0.533(3)	0.420(3)	0.1350	0.407
C182	0.188(4)	0.592(3)	0.566(3)	0.1943	0.407
H1821	0.191(4)	0.628(3)	0.545(3)	0.1350	0.407
H1822	0.134(4)	0.583(3)	0.578(3)	0.1350	0.407
H1823	0.219(4)	0.591(3)	0.610(3)	0.1350	0.407
C19	0.448(3)	0.429(3)	0.616(4)	0.0896	0.571
H191	0.447(3)	0.392(3)	0.596(4)	0.0940	0.571
H192	0.491(3)	0.432(3)	0.650(4)	0.0940	0.571
H193	0.455(3)	0.455(3)	0.577(4)	0.0940	0.571
C20	0.361(4)	0.515(2)	0.705(3)	0.1559	0.571
H201	0.405(4)	0.517(2)	0.738(3)	0.0940	0.571
H202	0.366(4)	0.543(2)	0.668(3)	0.0940	0.571
H203	0.313(4)	0.521(2)	0.732(3)	0.0940	0.571
C21	0.344(4)	0.394(2)	0.744(3)	0.1264	0.571
H211	0.386(4)	0.395(2)	0.779(3)	0.0940	0.571
H212	0.295(4)	0.400(2)	0.768(3)	0.0940	0.571
H213	0.343(4)	0.358(2)	0.721(3)	0.0940	0.571
C192	0.359(5)	0.379(3)	0.723(4)	0.0500	0.429
H1921	0.403(5)	0.383(3)	0.756(4)	0.0940	0.429
H1922	0.311(5)	0.373(3)	0.750(4)	0.0940	0.429
H1923	0.368(5)	0.348(3)	0.691(4)	0.0940	0.429
C202	0.342(4)	0.509(2)	0.726(3)	0.0500	0.429
H2021	0.386(4)	0.513(2)	0.759(3)	0.0940	0.429
H2022	0.339(4)	0.541(2)	0.696(3)	0.0940	0.429
H2023	0.294(4)	0.505(2)	0.753(3)	0.0940	0.429
C212	0.450(3)	0.451(3)	0.612(4)	0.0353	0.429
H2121	0.495(3)	0.454(3)	0.644(4)	0.0940	0.429
H2122	0.456(3)	0.419(3)	0.581(4)	0.0940	0.429
H2123	0.445(3)	0.483(3)	0.583(4)	0.0940	0.429
C71	0.054(7)	0.2500	0.893(5)	0.4570	0.25
CL71	0.125(5)	0.214(3)	0.843(5)	0.4570	0.33
CL72	-0.032(4)	0.2500	0.844(5)	0.4570	0.22
CL73	0.057(7)	0.2500	0.889(6)	0.4570	0.167
CL74	-0.014(7)	0.2500	0.744(6)	0.4570	0.167
C81	0.235(5)	0.2500	0.813(6)	0.4650	0.5
CL81	0.210(5)	0.215(2)	0.898(4)	0.4650	0.33
CL82	0.139(6)	0.220(7)	0.760(5)	0.4650	0.25
CL83	0.300(6)	0.184(3)	0.860(6)	0.4650	0.25
C91	0.330(6)	0.2500	1.046(5)	0.3860	0.5
C92	0.414(5)	0.2500	1.020(4)	0.3860	0.5
C93	0.410(5)	0.2500	0.943(4)	0.3860	0.5
C94	0.491(6)	0.2500	0.912(4)	0.3860	0.5
C95	0.543(5)	0.2500	0.976(6)	0.3860	0.5

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
CO1	0.063(4)	0.041(3)	0.063(4)	0.0000	-0.016(3)	0.0000
C22	0.05(3)	0.19(5)	0.24(5)	0.0000	-0.03(3)	0.0000
C23	0.12(3)	0.07(2)	0.25(5)	0.0000	0.08(3)	0.0000
C24	0.14(3)	0.15(3)	0.09(2)	0.0000	0.02(2)	0.0000
CO2	0.047(2)	0.062(2)	0.055(2)	-0.022(2)	0.011(2)	-0.006(2)
C25	0.06(2)	0.11(4)	0.15(4)	-0.04(3)	0.05(2)	-0.02(2)
C26	0.08(3)	0.21(5)	0.13(4)	0.02(4)	0.06(3)	0.02(4)
C27	0.07(3)	0.33(6)	0.08(3)	-0.08(4)	0.04(2)	-0.05(5)
C28	0.14(4)	0.13(3)	0.17(5)	-0.09(4)	0.08(4)	0.03(3)
C29	0.08(3)	0.18(5)	0.12(4)	-0.02(4)	-0.01(3)	0.02(3)
SI1	0.077(5)	0.056(5)	0.064(5)	0.005(4)	-0.022(4)	0.014(4)
SI2	0.093(6)	0.061(5)	0.074(5)	-0.007(5)	-0.018(5)	-0.012(5)
SI3	0.070(5)	0.082(6)	0.071(5)	-0.011(5)	-0.023(5)	-0.014(5)
C1	0.06(2)	0.07(2)	0.03(1)	0.01(1)	-0.03(1)	-0.01(1)
C2	0.05(1)	0.05(1)	0.04(1)	-0.01(1)	-0.04(1)	-0.01(1)
C3	0.03(1)	0.08(2)	0.06(1)	0.02(1)	-0.06(1)	-0.01(1)
C4	0.04(1)	0.07(2)	0.05(1)	0.01(1)	-0.04(1)	-0.02(1)
C5	0.04(2)	0.06(2)	0.06(2)	-0.01(1)	-0.02(1)	-0.00(1)
C6	0.04(2)	0.06(2)	0.08(2)	-0.00(1)	-0.01(2)	0.00(1)
C7	0.03(1)	0.07(2)	0.06(2)	-0.01(2)	-0.01(1)	0.01(1)
C8	0.07(2)	0.06(2)	0.05(2)	-0.03(2)	0.00(1)	-0.03(2)
C9	0.06(1)	0.05(2)	0.04(2)	-0.00(2)	-0.02(1)	-0.00(2)
C10	0.04(1)	0.02(1)	0.06(1)	0.01(1)	0.01(1)	-0.00(1)
C11	0.07(2)	0.07(2)	0.05(2)	-0.03(2)	0.01(1)	-0.02(2)
C12	0.07(2)	0.06(2)	0.06(2)	-0.01(1)	0.00(1)	-0.00(2)
C13	0.13(2)	0.05(1)	0.16(3)	0.01(2)	0.00(2)	0.01(2)
C14	0.21(4)	0.08(2)	0.11(2)	0.02(2)	-0.06(2)	0.05(2)
C15	0.11(3)	0.11(2)	0.20(4)	0.00(3)	0.01(3)	0.04(2)
C16	0.11(5)	0.12(6)	0.14(6)	0.02(5)	-0.07(4)	-0.03(5)
C17	0.41(9)	0.06(4)	0.11(6)	-0.02(4)	-0.06(7)	-0.00(5)
C18	0.28(8)	0.17(6)	0.17(5)	0.14(5)	0.05(5)	-0.06(5)
C162	0.18(6)	0.3(1)	0.13(6)	0.05(9)	0.00(6)	-0.17(8)
C172	0.13(9)	0.13(8)	0.2(1)	0.09(7)	0.04(8)	-0.04(6)

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
C182	0.17(7)	0.20(8)	0.3(1)	0.11(8)	-0.10(8)	-0.00(6)
C19	0.08(5)	0.14(9)	0.21(8)	0.11(7)	-0.08(6)	-0.03(5)
C20	0.24(8)	0.20(7)	0.09(5)	-0.00(5)	-0.00(5)	-0.09(6)
C21	0.11(5)	0.15(6)	0.15(6)	-0.06(5)	-0.01(5)	-0.00(5)
C192	0.10(8)	0.08(7)	0.06(6)	-0.02(6)	-0.10(6)	-0.01(6)
C202	0.07(4)	0.02(2)	0.05(4)	-0.02(2)	-0.06(4)	-0.04(3)
C212	0.04(6)	0.06(5)	0.12(8)	0.03(5)	0.06(6)	0.02(4)

CO1	- C22	1.99(3)
CO1	- C22	1.99(3)
CO1	- C23	2.03(2)
CO1	- C23	2.03(2)
CO1	- C1	1.97(2)
CO1	- C1	1.97(2)
CO1	- C2	1.97(2)
CO1	- C2	1.97(2)
CO2	- C25	2.08(2)
CO2	- C26	2.01(3)
CO2	- C27	1.98(3)
CO2	- C28	2.04(3)
CO2	- C29	2.09(3)
CO2	- C7	1.97(2)
CO2	- C8	2.00(2)
CO2	- C9	1.97(2)
CO2	- C10	1.97(2)
SI1	- C1	1.87(2)
SI1	- C13	1.84(2)
SI1	- C14	1.84(2)
SI1	- C15	1.83(2)
SI2	- C8	1.85(2)
SI2	- C16	1.86(4)
SI2	- C17	1.88(3)
SI2	- C18	1.88(3)
SI2	- C162	1.87(4)
SI2	- C172	1.86(4)
SI2	- C182	1.93(4)
SI3	- C9	1.84(2)
SI3	- C19	1.83(4)
SI3	- C20	1.84(4)
SI3	- C21	1.86(4)
SI3	- C192	1.87(4)
SI3	- C202	1.90(4)
SI3	- C212	1.86(4)
C22	- C23	1.43(2)
C22	- C23	1.43(2)
C23	- C24	1.43(3)
C24	- C24	1.43(4)
C25	- C26	1.40(3)
C25	- C29	1.39(3)
C26	- C27	1.43(3)
C27	- C28	1.40(3)
C28	- C29	1.36(3)
C1	- C1	1.51(4)
C1	- C2	1.44(2)
C2	- C2	1.52(4)
C2	- C3	1.36(3)
C3	- C4	1.23(2)
C4	- C5	1.36(3)
C5	- C6	1.25(3)
C6	- C7	1.40(3)
C7	- C8	1.48(3)
C7	- C10	1.45(3)
C8	- C9	1.47(3)
C9	- C10	1.47(3)
C10	- C11	1.37(3)
C11	- C12	1.21(3)
C12	- C12	1.41(5)

C23	- C22	- C23	106.5(23)
C24	- C23	- C22	109.3(17)
C24	- C23	- C22	109.3(17)
C24	- C24	- C23	107.4(10)
C29	- C25	- C26	108.7(23)
C27	- C26	- C25	105.9(23)
C28	- C27	- C26	107.5(23)
C29	- C28	- C27	108.5(24)
C28	- C29	- C25	109.3(24)
C2	- C1	- C1	90.2(12)
C2	- C2	- C1	89.8(12)
C3	- C2	- C1	139.6(19)
C3	- C2	- C2	130.4(11)
C4	- C3	- C2	174.5(21)
C5	- C4	- C3	176.3(23)
C6	- C5	- C4	172.4(25)
C7	- C6	- C5	172.7(25)
C8	- C7	- C6	139.0(23)
C10	- C7	- C6	130.1(22)
C10	- C7	- C8	90.1(17)
C9	- C8	- C7	89.8(17)
C10	- C9	- C8	89.4(16)
C9	- C10	- C7	90.7(17)
C11	- C10	- C7	130.2(22)
C11	- C10	- C9	138.7(23)
C12	- C11	- C10	172.6(27)
C12	- C12	- C11	177.3(17)