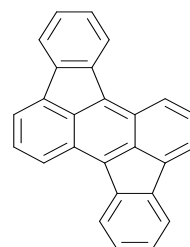
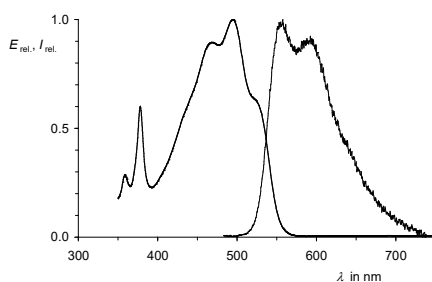
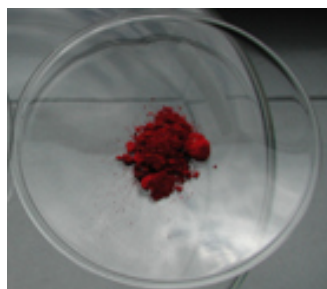


Rubicene



V. Sachweh, H. Langhals, 'Synthese von Reinst-Rubicen und Rubicen-Derivaten', *Chem. Ber.* **1990**, *123*, 1981-1987.

Synthesis of highly pure rubicene (RN197-61-5) and derivatives of rubicene.

Rubicene (RN197-61-5) from fluorenone and magnesium in solution of melt biphenyl:

Magnesium turnings (10.0 g, 411 mmol) under argon atmosphere were etched (activated) by means of a small amount of iodine with heating, treated with technical fluorenone (RN486-25-9, 93.7 %, 30.0 g, 156 mmol) and biphenyl (RN92-52-4, 100 g, 650 mmol), heated vigorously at 260-270°C for 51 h (vigorous refluxing of the biphenyl is essential because otherwise the formation of a by-product becomes dominating: $R_f = 0.65$, silica gel, toluene), allowed to cool, recrystallised from toluene (150 mL) with the application of a hot extractor (see H. Langhals, *Chem. Ber.* **1985**, *118*, 4641.) and washed with toluene for removing some exceeding biphenyl. Yield 5.60 g (22%) light red needles, m.p. 305°C (Ref. 305-307°C). R_f (silica gel, toluene) = 0.86. IR (KBr): $\tilde{\nu} = 3050 \text{ cm}^{-1}$, 1462, 1379, 766, 774, 744, 731, 724, 662, 615. UV/Vis (toluene): $\lambda_{\text{max}} (\lg \epsilon) = 296 \text{ nm} (4.6191)$, 312 sh, 359.5 (3.6550), 378.5 (3.9477), 470.5 (4.1021), 495 (4.1390), 514.5 sh. Fluorescence (chloroform, $\lambda_{\text{exit}} = 495 \text{ nm}$): $\lambda_{\text{max}} = 552 \text{ nm}$. $^1\text{H NMR}$ (CDCl_3 , 360 MHz): $\delta = 7.39$ (m, 2 H, 5-H, 12-H), 7.48 (m, 2 H, 6-H, 13-H), 7.76 (m, 2 H, 2-H, 9-H), 7.96 (d, $J_{3,2} = J_{10,9} = 6.9 \text{ Hz}$; 2 H, 3-H, 10-H), 8.00 (d, $J_{4,5} = J_{11,12} = 6.7 \text{ Hz}$; 2 H, 4-H, 11-H), 8.31 (d, $J_{7,6} = J_{14,13} = 7.5 \text{ Hz}$, 2 H, 7-H, 14-H), 8.57 (d, $J_{1,2} = J_{8,9} = 8.6 \text{ Hz}$, 2 H, 1-H, 8-H). $^{13}\text{C NMR}$ (CDCl_3 , 90 MHz): $\delta = 120.32$, 121.64, 123.73, 124.57, 125.54, 126.98, 128.13, 129.02, 129.28, 133.10, 133.54, 138.25, 139.43, 139.92. MS (70 eV): m/z (%) = 326 (100.0) [M^+], 163 (62.0), 149 (21.0), 92 (42.0), 91 (51.0), 59 (9.0), 58 (24.0), 43 (58.0), 41 (16.0), 39 (15.0), 32 (38.0). - X-ray powder pattern (Cu- K_α): $\theta (I_{\text{rel}}) = 9.8^\circ (1.000)$, 11.0 (0.116), 14.2 (0.022), 15.3 (0.276), (0.1 17), 15.5 (sh, 0.069), 18.4 (0.247), 20.4 (0.049), 23.1 (0.040), 23.9 (0.011), 25.1 (0.027), 26.5 (0.065), 30.6 (0.226), 31.4 (0.115), 37.3 (0.130), 38.7 (0.117). $\text{C}_{26}\text{H}_{14}$ (326.4): Calcd. C 95.68, H 4.32; found C 95.54, H 4.44.