Org Lett

A Rotaxane-Like Complex with Controlled-Release Characteristics

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SUPPORTING INFORMATION

Experimental Section

Procedure for the preparation of [BMP25C8·3-H][PF_6]$_2$

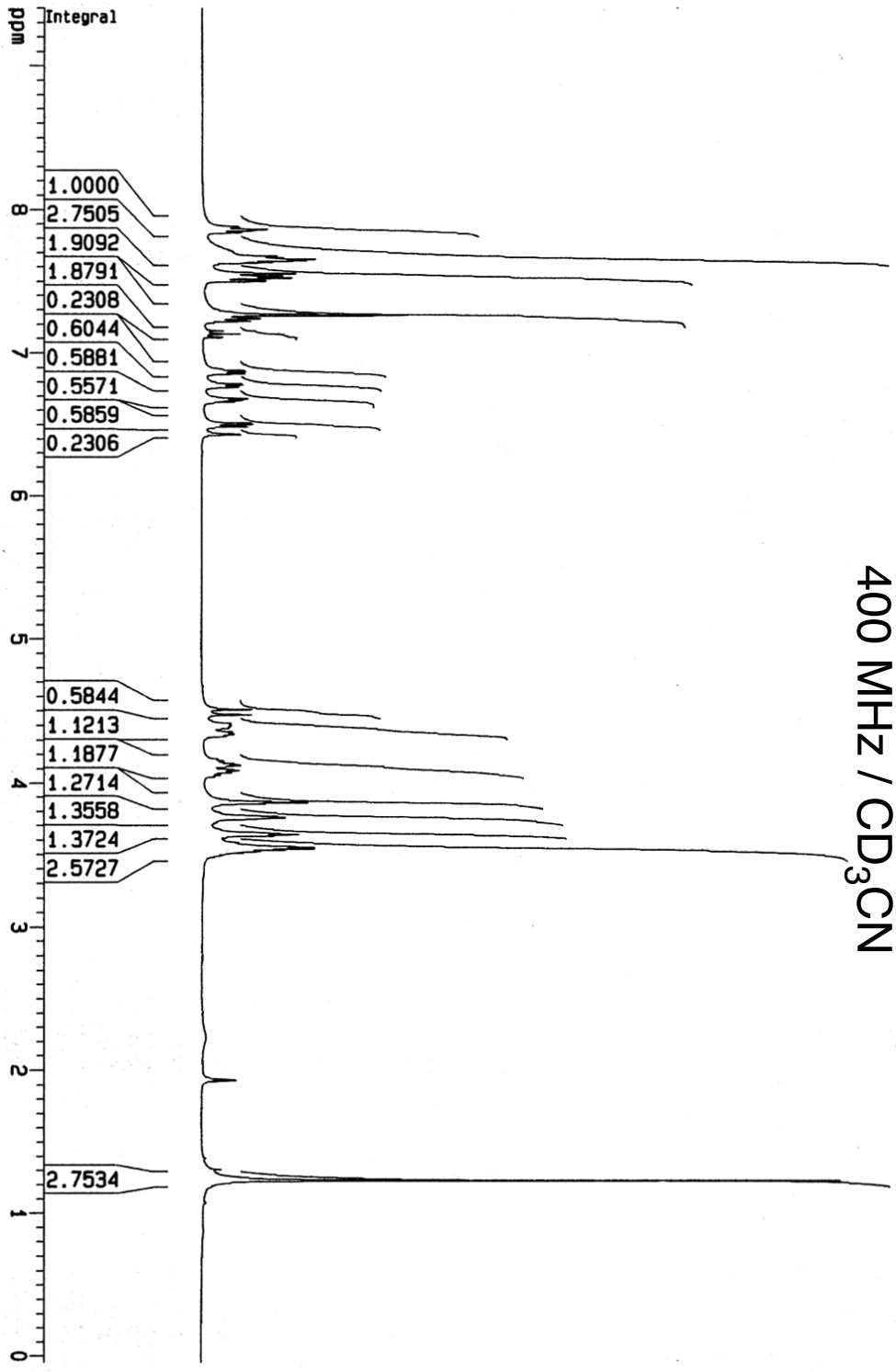
2-H·PF_6 (1.0 g, 2.0 mmol) and BMP25C8 (1.0 g, 2.2 mmol) were dissolved in $MeNO_2$ (10 mL). The solution was stirred at room temperature for 10 min and then PPh_3 (0.6 g, 2.3 mmol) was added. After stirring overnight at room temperature, the solvent was removed under reduced pressure, the solid residue was dissolved in MeCN (30 mL), NH_4PF_6 was added (2 g in 30 mL H_2O) and the organic solvent was removed under reduced pressure. CH_2Cl_2 (100 mL) was added to dissolve the precipitate and was washed by water (2 × 30 mL). The organic layer was dried ($MgSO_4$) and evaporated to dryness. The crude product was purified by column chromatography (SiO_2) using 10% MeCN in CH_2Cl_2 as eluent. The product was recovered as a white solid (1.1 g, 40%).

^{1}H NMR (400 MHz, CD_3CN): δ 1.23 (s, 9H), 3.40-3.90 (m, 20H), 4.0–4.2 (m, 4H), 4.3–4.5(m, 4H), 4.55 (d, 2H, J = 7.5 Hz), 6.42 (t, 1H, J = 2.2 Hz), 6.49 (dd, 2H, J = 4.1, 1.1 Hz), 6.67 (m, 2H), 6.77 (dd, 2H, J = 4.1, 1.1 Hz), 6.87 (m, 2H), 7.13 (t, 1H, J = 7.0 Hz), 7.2–7.3 (m, 6H), 7.49–7.58 (m, 6H), 7.61–7.80 (m, 8H), 7.86 (t, 3H, J = 7.0 Hz).

^{13}C NMR (100 MHz, CD_3CN): δ 30.3 (J_{PC} = 48.2 Hz), 31.4, 35.2, 52.4, 53.2, 68.60, 68.9, 70.2, 71.0, 71.2, 72.1, 104.4, 108.3, 113.1, 118.2 (J_{pc} = 85.7 Hz), 122.5, 126.7, 128.9, 129.3 (J_{PC} = 8.3 Hz), 130.4, 131.2 (J_{PC} = 12.6 Hz), 131.2 (J_{PC} = 3.1 Hz), 131.4, 132.2 (J_{PC} = 5.4 Hz), 132.8 (J_{PC} = 3.9 Hz), 135.1 (J_{PC} = 9.8 Hz), 136.4 (J_{PC} = 2.9 Hz), 147.2, 153.6, 160.8.

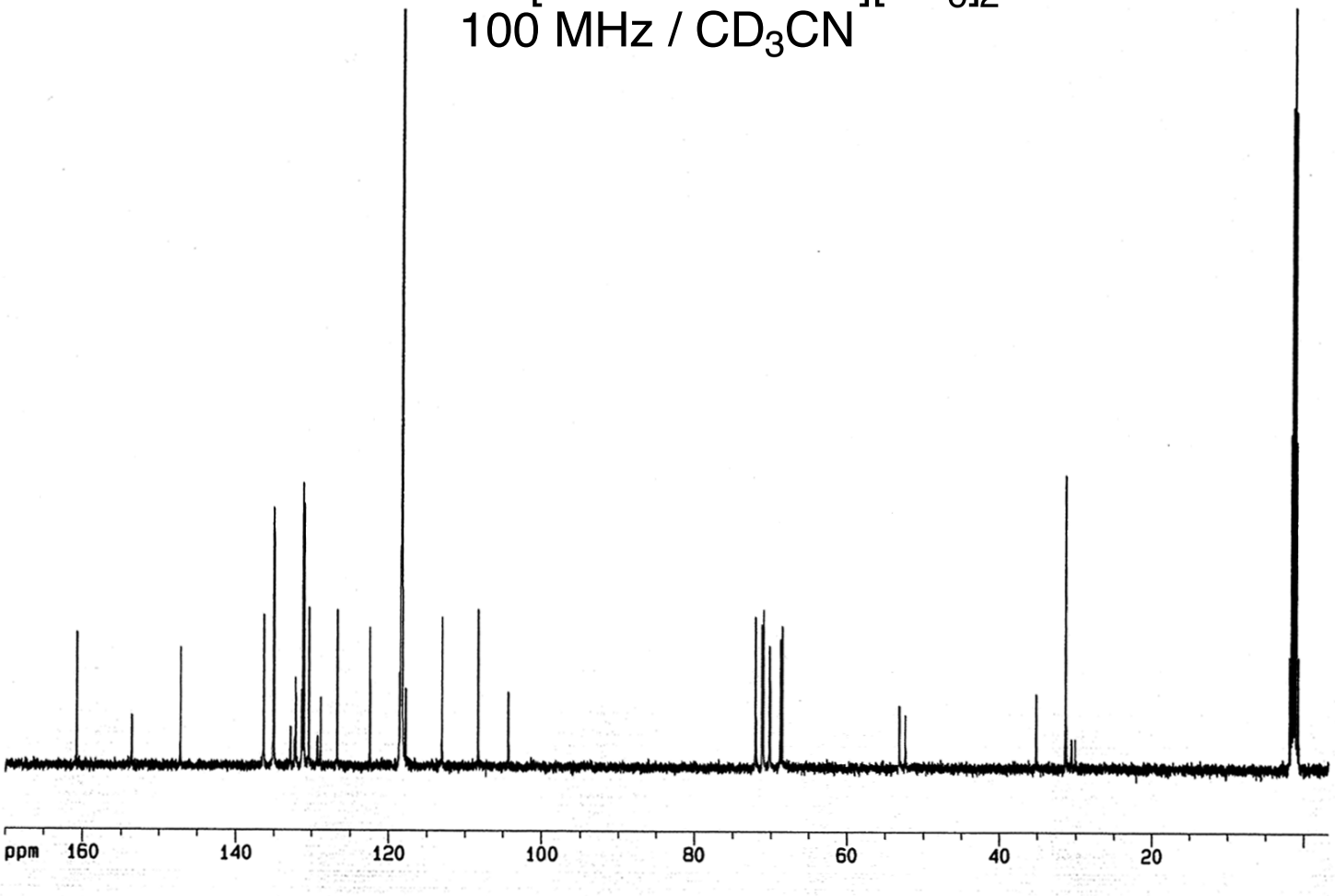
FAB-MS: *m/z* 1122 $[M–PF_6]^+$, 976 $[M–2PF_6]^+$.

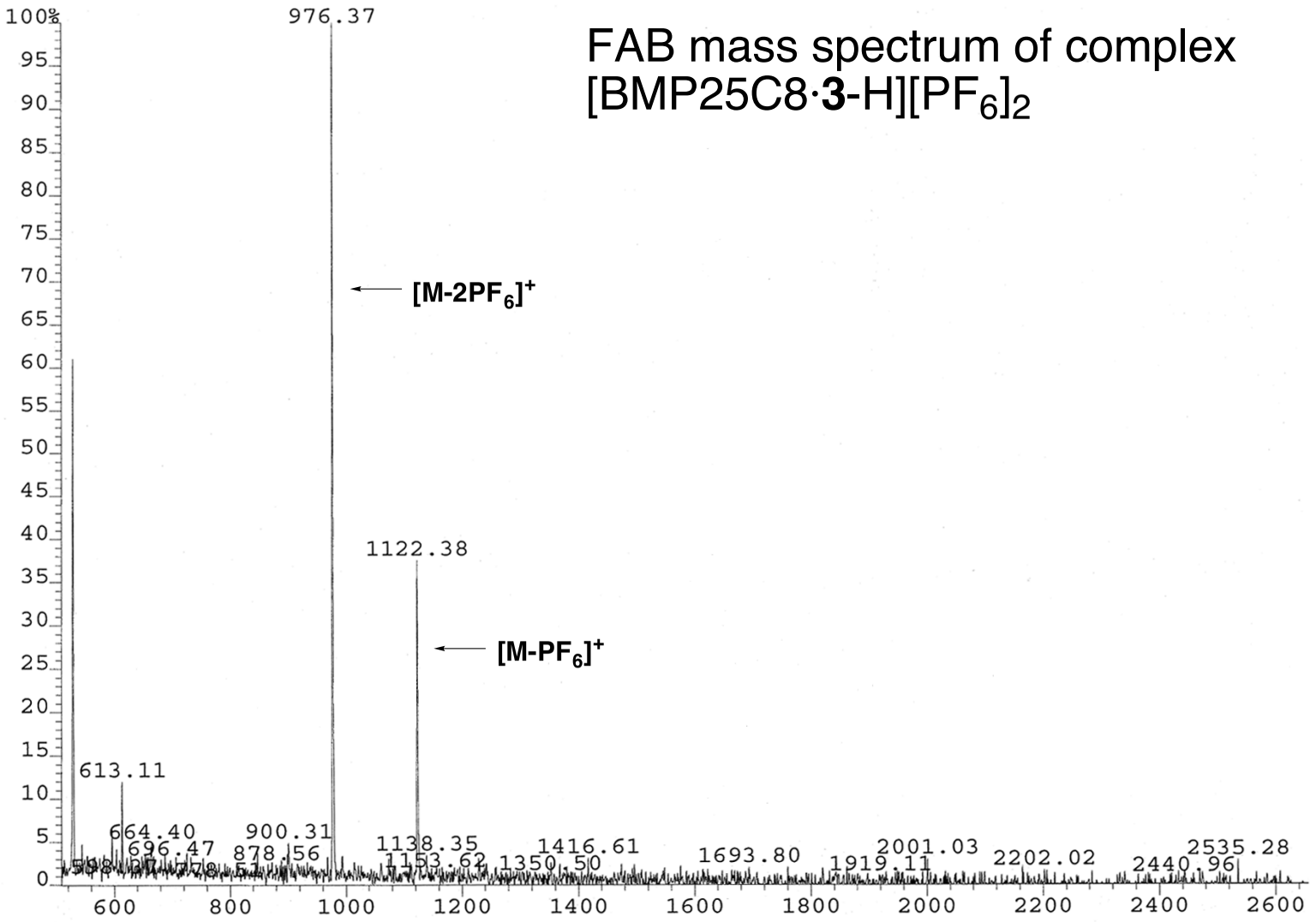
Anal: Calcd for $C_{61}H_{72}NO_8P_3F_{12}$: C, 57.77; H, 5.72; N, 1.10; Found C, 57.63; H, 5.70, N, 0.94.



^{1}H NMR of [BMP25C8·**3**-H][PF_6]2
400 MHz / CD_3CN

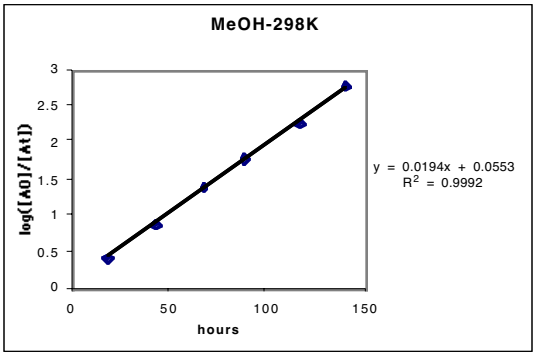
^{13}C NMR of [BMP25C8·**3**-H][PF_6]$_2$
100 MHz / CD_3CN

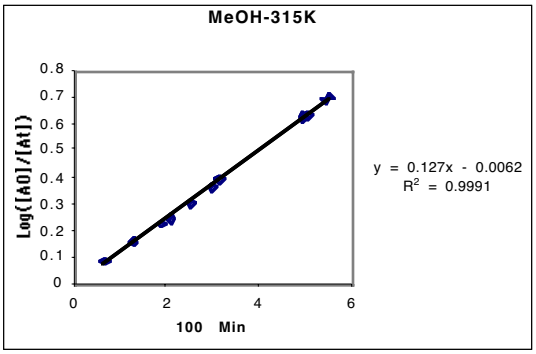


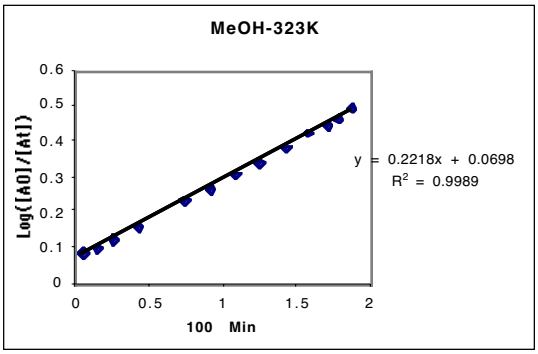


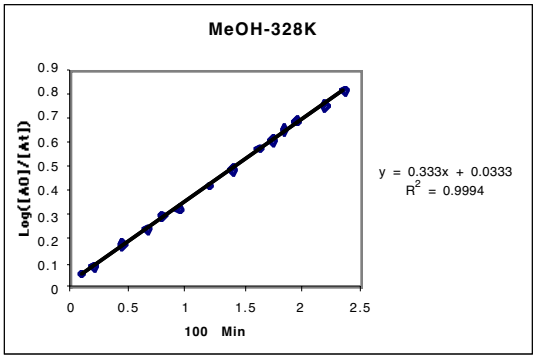
FAB mass spectrum of complex [BMP25C8·**3**-H][PF_6]$_2$

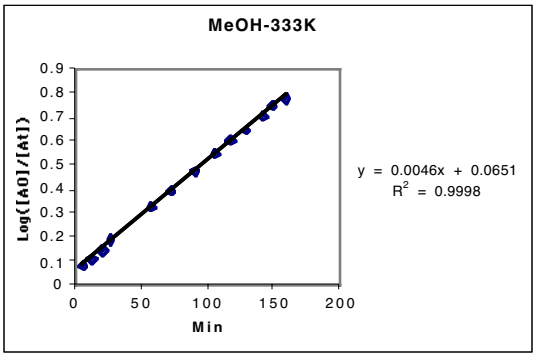
Complex [BMP25C8·**3**-H][PF_6]$_2$ dissociated in CD_3OD

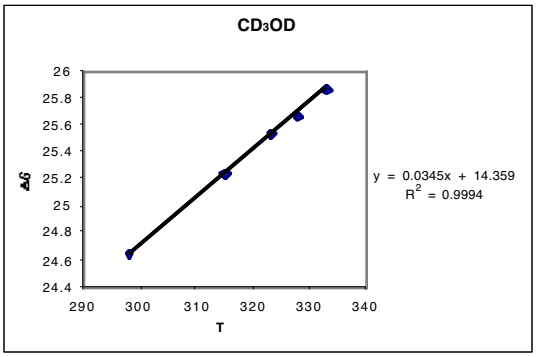




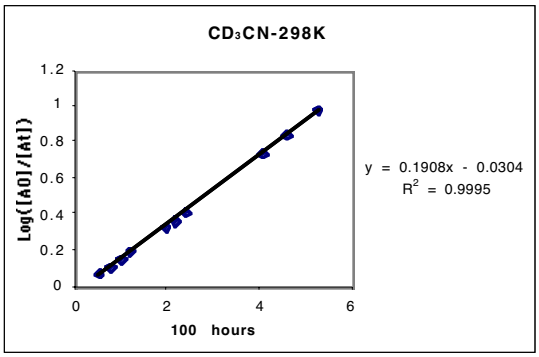


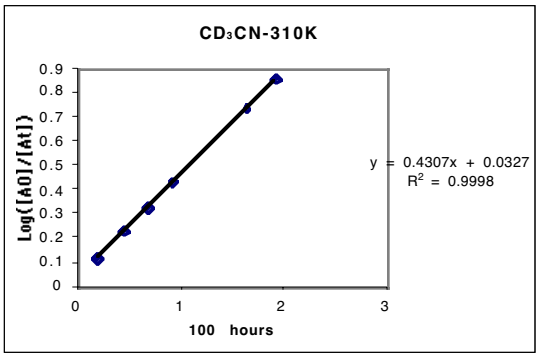


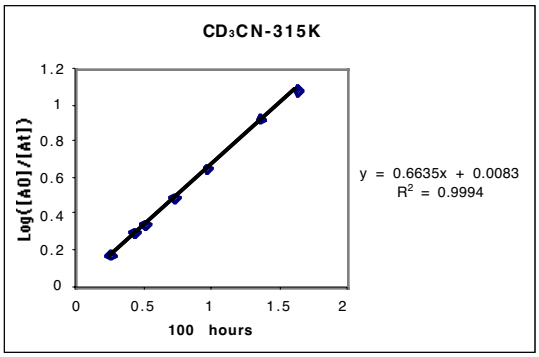


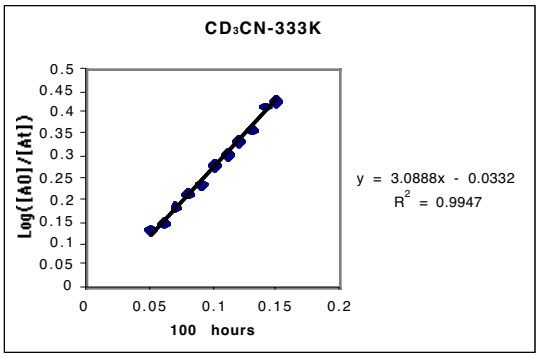


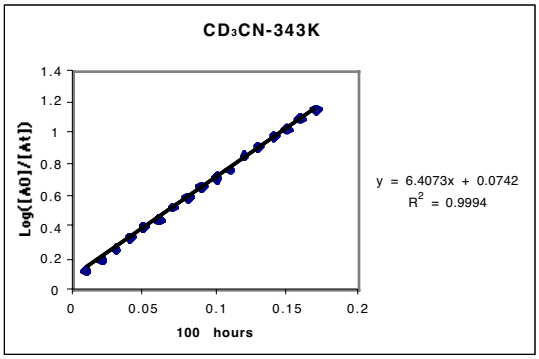
Complex [BMP25C8·**3**-H][PF_6]$_2$ dissociated in CD_3CN

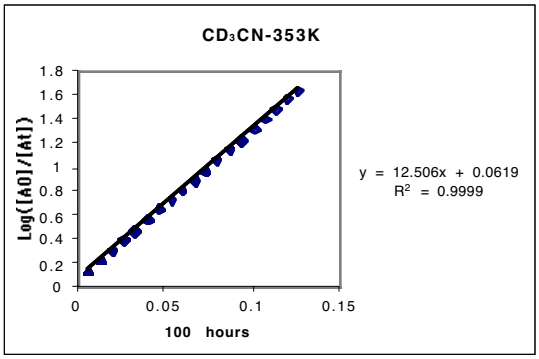


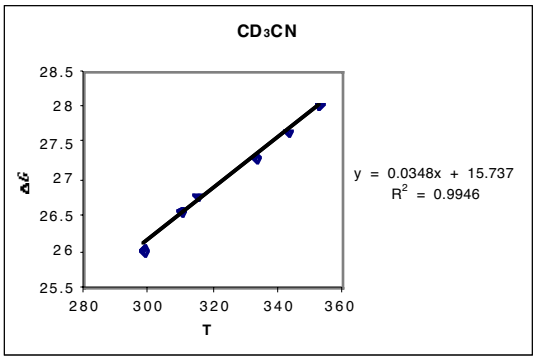




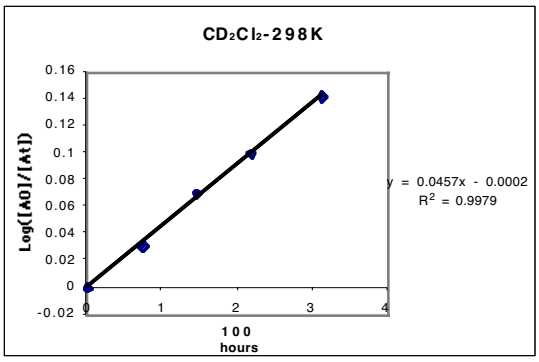


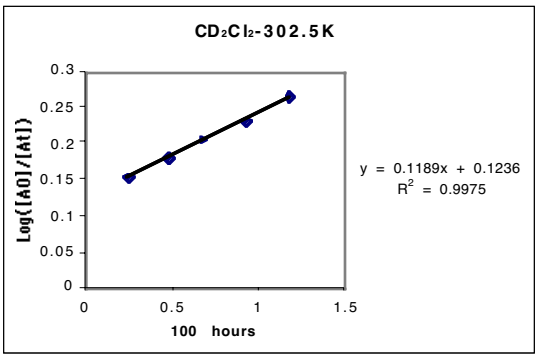


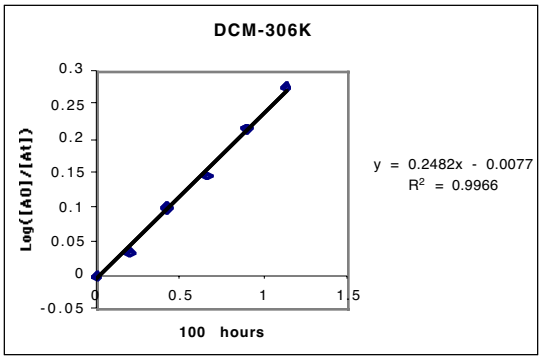


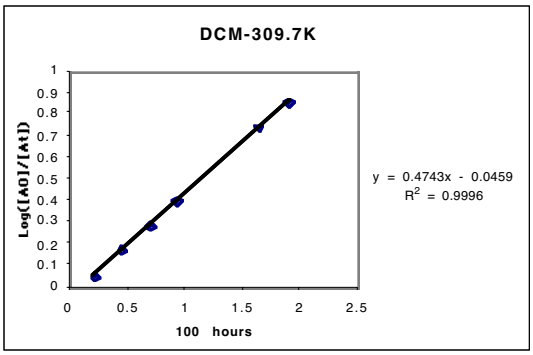


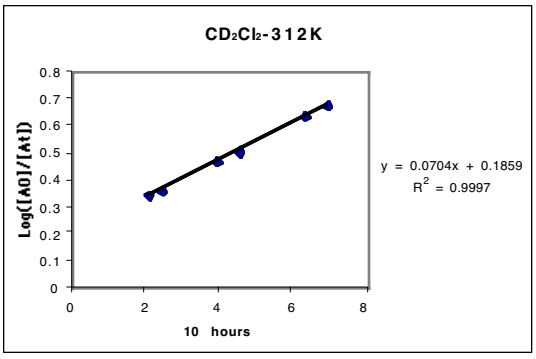
Complex [BMP25C8·**3**-H][PF_6]$_2$ dissociated in CD_2Cl_2

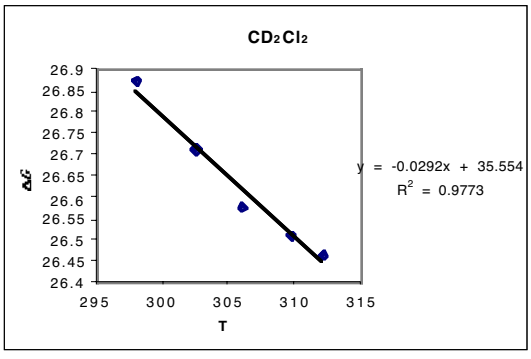












Complex [BMP25C8·**3**-H][PF_6]$_2$ dissociated in CD_2Cl_2/CD_3SOCD_3