

Figure 1 - Panel a: Stacked CO<sub>2</sub> desorption curves after previous CO isothermal titration of surface oxygen using different Pt/C sample loading a) 20mg b) 5 mg c) 1 mg and d) 0,1 mg,. Panel b: Correlation plot of log CO<sub>2</sub> vs log Catalyst content (hollow triangle is result obtained from Quantachrome ChemStar TPx apparatus), Panel c: log CO vs log Catalyst content



Figure 2 - Panel a: The  $4\mu m$  Cu powder titration data is processed as following: online fragmentation ratio is directly obtained from stabilized (after 450 s) a) N<sub>2</sub>O (amu 44) and b) N<sub>2</sub> (amu 28) MS raw signals, the N<sub>2</sub> contribution from N<sub>2</sub>O fragmentation is thus calculated (curve a times the ratio obtained) c) and subtracted from N<sub>2</sub> raw signal and obtain N<sub>2</sub> net signal (curve b minus curve c) d). Panel b: Integration of the N<sub>2</sub> product net peak after each N<sub>2</sub>O titration process on  $4\mu m$ 

Cu metallic powder from -40 to 80 °C. The titrations are performed by exposing 20 sccm 0.5% N<sub>2</sub>O in Ar after the catalyst is reduced in 20 sccm hydrogen/Ar (1:9) at 100 °C for 10 min.



Figure 3 - Panel a): CO<sub>2</sub> product from 200°C pre-oxidized Au/TiO<sub>2</sub> in isothermal CO titration at: a) 130°C, b) 100°C, c) 80°C, and d) 40°C (Inset: Comparison of 80°C and 130°C decay curves; 1/CO<sub>2</sub>\* coverage vs time, Panel b): Tabulated results of total CO<sub>2</sub> desorbed versus experiment temperature (Inset: expanded view of plateau region)