APPENDIX D

SAMPLE CHEMISORPTION EXPERIMENT

Table D.1. Sample Chemisorption Experiment.

Run #: 08154.

Catalyst: 1.0001 gm of Co_3O_4/Al_2O_3 (9.3 wt.% of cobalt, 60-150 mesh).

Reduction: Start heating with hydrogen (Air Product ultra-pure carrier 99.999%) of 0.74 ft^3/hr (room conditions: 84 °F and 28.91 in-Hg @ 86.5 °F), to 750 °F in 2 hrs, stay at 750 °F for 20 hrs and finally cool down with hydrogen.

Outgassing: at 730 °F for 1 hr.

Manifold volume: 66.08 ml.

Dead volume: 46.28 ml.

Manifold vacuum: 6.60×10^{-7} torr after degassing ion gauge.

Adsorption temperature: 83 °F.

Amount of Hydrogen Uptaken

Let P_1 be the pressure of hydrogen in the manifold before expanding into the sample cell, in torr,

P_{eq} the equilibrium pressure of previous adsorption step, in torr,

and P_2 the equilibrium pressure of this adsorption step. Then, the number of moles of hydrogen, n_H, adsorbed in this step can be calculated as follows:

$$n_H = \frac{P_1 V_M + P_{eq} V_d - P_2 (V_M + V_d)}{RT} \qquad (D.1)$$

where V_M is the volume of manifold, in ml,

V_d is the dead volume, in ml,

R is the gas constant, 62400 $\frac{\text{torr-cm}^3}{\text{gm.mol-K}}$,

and T is the adsorption temperature, in °K.

P_1	P_{eq}	P_2	n_H (μmol)
1042.4	-	608.9	24.9
1005.7	608.9	840.1	13.0
870.9	840.1	857.2	6.1

Total hydrogen uptaken of the monolayer = 44.0

Number of Initial Active Sites

Assume that the number of initial active sites, N_S, is equal to the number of accessible surface atoms of cobalt in the fresh catalyst after reduction. For hydrogen adsorption stoichiometry on cobalt, X_H = 1.0 hydrogen atom per surface cobalt atom, then by Eqn. (4.2), N_S can be calculated:

$$N_S = (44.9 \frac{\mu mol}{gm})(\frac{1 \text{ mol}}{10^6 \ \mu mol})(6.023 \times 10^{23} \frac{\text{molecules}}{\text{mol}}) \times$$

$$(2.0 \frac{\text{H atoms}}{H_2 \text{ molecule}})(1.0 \frac{\text{active site}}{\text{H atom}})$$

$$= 5.29 \times 10^{19} \frac{\text{active sites}}{\text{gm-catalyst}}.$$

Cobalt Surface Area

With the site density, $n_S = 14.6 \frac{\text{active sites}}{(\text{nm})^2}$ [181], the surface area of cobalt, A_S, can be calculated by Eqn. (4.3):

$$A_S = \frac{5.29 \times 10^{19} \ \frac{\text{active sites}}{\text{gm-catalyst}}}{\left(14.6 \ \frac{\text{active sites}}{\text{nm}^2}\right)\left(\frac{1 \ \text{nm}^2}{10^{-18} \ \text{m}^2}\right)}$$

$$= 3.62 \ \frac{\text{m}^2}{\text{gm-catalyst}}.$$