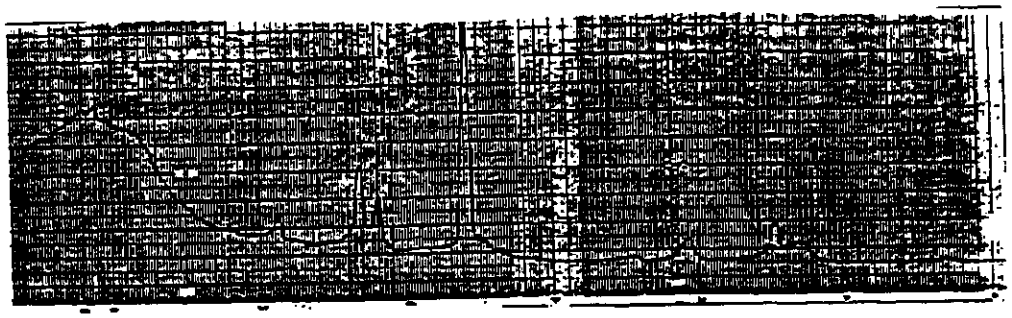
# APPENDIX E

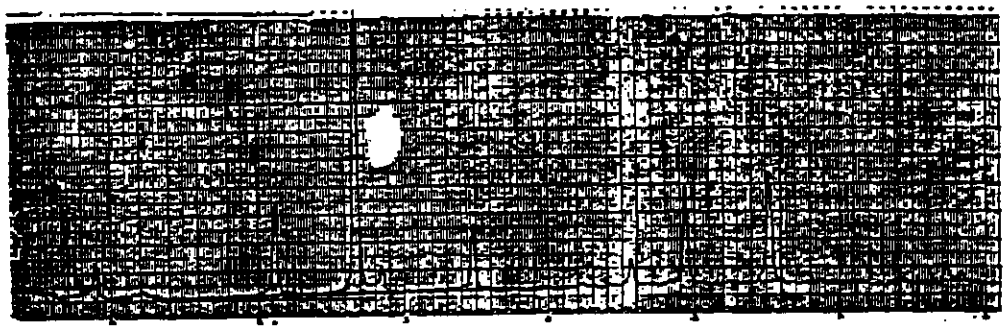
## SAMPLE XRD EXPERIMENT

## AND

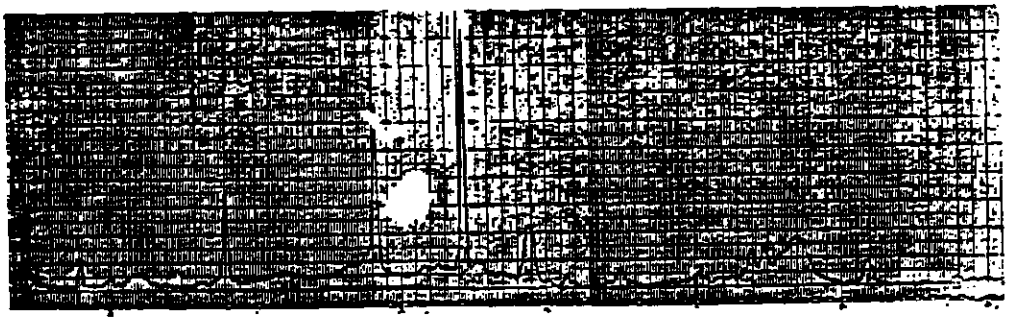
## LINE BROADENING CALCULATION



a. $Co_3O_4/Al_2O_3$ Ctalyst Used in This Study.



b. Baker Cobalt Oxide/MCB Activated Alumina.



c. ALCOA F-1 Activated Alumina (150 mesh).

Figure E.1. XRD Analysis Patterns of Catalyst Samples.

9-418

| d | 2.44 | 1.43 | 2.86 | 4.669 |
|---|---|---|---|---|
| I/I$_1$ | 100 | 45 | 40 | 20 |

$Co_3O_4$

Cobalt (II,III) Oxide

Rad. Cobalt λ 1.7889 Filter Fe Dia

Cut off I/I$_1$ Diffractometer

Ref. Nat. Bur. Standards Circ. 539 2 (1959)

Sys. Cubic S.G. $O_h^7$ - Fd3m

$a_o$ 8.084 $b_o$ $c_o$ A C

α β γ Z 8 Dx 6.054

Ref. Ibid.

ε α ω eV Sign

2V D mp Color Black

Ref

Sample prepared at NBS-by-heating cobalt fluoride to 950°C for 24 hours. Spectrographic analysis shows 1.0-0.1% Ca, Ni; 0.1-1.0% each of Al, Fe, Mg, Si; 0.001-0.01% each of Ba, Cu, Mn; 0.0001-0.0001% or Cr.

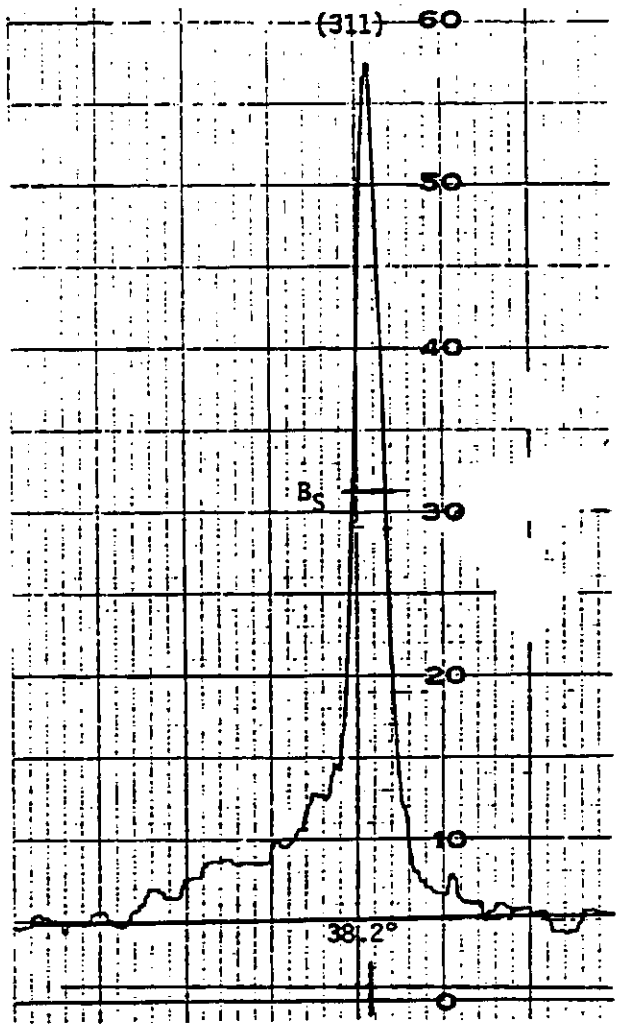
Pattern made at 24°C.

Spinel type structure.

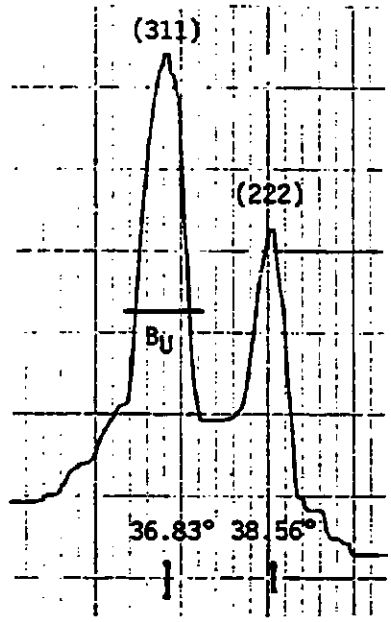
Replaces 1-1152

| d Å | I/I$_1$ | hkl | d Å | I/I$_1$ | hkl |
|---|---|---|---|---|---|
| 4.669 | 20 | 111 | | | |
| 2.860 | 40 | 220 | | | |
| 2.438 | 100 | 311 | | | |
| 2.333 | 11 | 222 | | | |
| 2.021 | 25 | 400 | | | |
| 1.650 | 11 | 422 | | | |
| 1.555 | 35 | 511 | | | |
| 1.429 | 45 | 440 | | | |
| 1.278 | 5 | 620 | | | |
| 1.233 | 11 | 533 | | | |
| 1.219 | 7 | 622 | | | |
| 1.167 | 3 | 444 | | | |
| 1.132 | 3 | 711 | | | |
| 1.080 | 7 | 642 | | | |
| 1.052 | 15 | 731 | | | |
| 1.010 | 7 | 800 | | | |
| 0.9529 | 5 | 822 | | | |
| .933 | 15 | 751 | | | |
| .927 | 5 | 662 | | | |

Figure E.2. JCPDS' Standard X-Ray Pattern (#9-418).




b. ALCOA F-1 Activated Alumina.




a. $Co_3O_4/Al_2O_3$ Catalyst.

Figure E.3. Portions of XRD Patterns Used in Line Broadening Calculation.

Average Crystallite Diameter of Catalyst Calculated by XRD Line Broadening.

Measured breadths, at half-maximum intensity, of the lines from both the standard and unknown are:

$B_S$ = 3/16 and $B_U$ = 5/16 inches.

For chart speed of 2 °/inch, $B_S = \frac{3}{8}°$ and $B_U = \frac{5}{8}°$.

According to Cullity [66], the crystallite broadening B is calculated as follows,

$$B = \sqrt{\left(\frac{5}{8}\right)^2 - \left(\frac{3}{8}\right)^2} = \frac{1}{2}^\circ,$$

which can be converted into B = 0.008727 radian.

The wavelength of copper $K\alpha_1$ is $\lambda$ = 1.54050 Å, and

Bragg angle for the catalyst sample is $\theta_B$ = 36.83°.

Now plug every thing into Scherrer formula, Eqn. (4.1),

$$t = \frac{(0.9)(1.54050\ \text{Å})}{(0.008727\ \text{radian})(\cos 36.83°)} = 198.491\ \text{Å}$$

this t is the averaged crystallite diameter of the cobalt/alumina catalyst.

Specific Surface Area of Catalyst Estimated by XRD Line Broadening.

Assume that the particle of catalyst crystallite is either cubic or spherical.

Apparent density of catalyst, $\rho$, is 1.6667 gm/ml or $1.6667 \times 10^6 \frac{gm}{m^3}$.

The specific surface area of the catalyst crystallite, A, can be calculated as follows [128],

$$A = \frac{6}{(1.6667 \times 10^6 \frac{gm}{m^3})(198.491 \text{ Å})(10^{-10} \frac{m}{Å})}$$

$$= 181.36 \frac{m^2}{gm}.$$