

Leuna - October 25, 1941

THE DETERMINATION OF CHROMIUM AND ZINC IN CATALYSTS

A rapid method has been developed for the determination of chromium and zinc in the isobutyl alcohol catalyst. The method avoids the time-consuming precipitation of chromium as lead chromate and the removal of the excess of lead by digestion with sulphuric acid.

~~A simple reduction of the chromate-zinc solution was successful and it could be shown that zinc can be titrated besides trivalent chromium salt with potassium ferrocyanate using diphenylamine as internal indicator. In order to reduce the chromate the sulphuric acid solution is boiled with alcohol for $\frac{1}{2}$ hour.~~

A sample of the catalyst is fused with a 10-fold amount of sodium and potassium carbonate. The fused mass containing all the chromium in the form of chromate is dissolved in HNO_3 (1 : 1), concentrated carefully on a sand bath, evaporated to dryness and dissolved in dilute sulphuric acid.

Chromium and zinc are determined by titration in aliquot portions of this solution.

For the determination of chromium, the sulphuric acid solution is treated with 5 - 10 drops of a solution of diphenylamine in sulphuric acid (0.2% diphenylamine solution) and is subsequently titrated with ferrous ammonium sulfate to a color change from violet to green. (1 cc. of 0.1 N $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 = 2.532$ mg. Cr_2O_3) The results obtained in this way agree very well with the results of the iodometric titration.

Zinc is determined by titration with potassium ferrocyanate with diphenylamine as indicator after reduction by boiling for $\frac{1}{2}$ hour with alcohol in the presence of chromium in the form of trivalent chromium salt.

The titration is carried out at 60 - 70°C. in 1 - 1.5 N H_2SO_4 solution (10 cc. H_2SO_4 (1 : 1) in 100 cc.) in the presence of 1% of ammonium sulphate. 4 - 5 drops of a solution of diphenylamine in sulphuric acid are used as indicator and 2 drops of a 10% solution of potassium ferricyanate solution are added since comparatively small amounts of zinc are to be determined. The color changes from blue to green-yellow. In order to obtain satisfactory results, the titer of the potassium ferrocyanate solution must be determined under exactly the same conditions as obtain for the actual titration.

The following solutions are to be used:

1. Potassium ferrocyanate solution
10.558 grams of potassium ferrocyanate per liter
0.15 grams of potassium ferricyanate per liter
0.2 grams of potassium carbonate per liter
2. 1% solution of diphenylamine in concentrated sulphuric acid
3. 10% solution of potassium ferricyanate

The average of a large number of determinations showed that 1 cc. of the ferrocyanate solution corresponds to 3.18 mg. of ZnO . The titer is purely empirical and is constant for several days but should preferentially be checked every day. Experiments were carried out to study the effect of chromium salts and it was found that they did not interfere with the determination when it was carried out according to the above-described procedure.