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1165

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T-416

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October 22, 1947

May 24, 1943

PREPARATION OF CATALYST 6434

A. Summary

The ready-to-use catalyst consists of terrana A treated with HF, and contains 10% WS_2.

B. Technical preparation.

1. Preparation of a mixture of hydrofluoric acid - treated terrana with the yellow salt.

2. Decomposition of the yellow salt and its conversion to WS_2.

3. The preparation of the material for pressing and the pressing of it.

4. The drying and after-treatment of the shapes made.

1). 300 kg of terrana A extra are combined in an indirect steam-heated covered pan with about 300 - 320 li. 8% HF, and mixed. After mixing for 25 - 30 minutes, 500 liters of an ammonium sulfide solution of ammonium sulfotungstate, containing about 7% WO_3 are added. The solution of the yellow salt is prepared from the mother liquor left after filtering off the yellow salt crystal meal. It contains 1 - 1 1/2 solids as WO_3, 11 - 12% NH_3 and 10% total H_2S. The solids content is increased to 7% WO_3 by adding yellow salt. The steam for heating is opened after the yellow salt solution has been added to terrana. The contents of the mixer are liquid at first, then become pasty, and finally form a dry granular mass, requiring a total drying time of about 8 hours. The ammonium sulfide and steam vapors escaping from the pan during the evaporation is lead into a closed absorber and the contents circulated through the ammonium sulfide solution provided with its own cooling coil.

When dry the products are sensitive to oxidation, and the pan filled with the dry material is cooled indirectly with circulating water before being emptied. The product obtained from the mixer pan is gray in color.

2) The yellow salt constituent is converted into the active WS_2 by heating the product in the mixer pan to about 21 - 22 mv (408 - 425°C). The granulated mass is for this purpose first coarsely crushed and then led through a closed horizontal system of electrically heated furnaces, using screw conveyors. N_2 and H_2S are introduced in appropriate places of the furnaces, and the decomposition of the yellow salt proceeds in a reducing atmosphere according to the equation.

$$(NH_4)_2WS_4 + H_2 = WS_2 + (NH_4)_2S + H_2S$$

The product is carried through a cooling arrangement into a set of containers protected against contact with air, and next ground in an impact mill to pass 3 mm mesh. The 6434 black powder obtained in this way is of a deep grey-black color.

3) Before pressing the mass into pills it must first be moistened with 30 parts by weight of water. This is done in an Eirich mixer with a built-in screw mixer (Kellergangläufer). When the terrana dried at 408 - 425° is mixed with water, the heat of hydration of the terrana becomes apparent. Because of the presence of danger of oxidizing the finely ground WS_2, the moistening of the product must be done in small portions of about 25 - 30 kg, and the moist product should be prevented from spontaneous heating by spreading it out in thin layers. Another precaution to take is to see that only well cooled mass be pressed in the pill press, as otherwise the hydration process may again be started by the heat of pressing and will cause a spontaneous heating of the pills and oxidation of WS_2. The pills are next smoothed out in a rotating drum, dried at about 7 mv (170°C), where they shrink somewhat and become stronger. The drying is done in vertical electrically heated tubular furnaces, with a nitrogen atmospheres passing through the pills. The dried pills are pigeon-gray.

The pills are next again treated at 21 - 22 mv (408 - 425°C) in a furnace of a similar construction as before, but in a stream of gas composed of hydrogen sulfide containing some hydrogen. The well cooled pills removed from that furnace are the splitting catalyst pills, ready to be introduced into the converters. The average filling weight of the 10 mm pills is 0.900 - 0.920 kg/li.

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