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COMPLETE SPECIFICATION.

2611

Process of Regeneration of Contact Masses for the Catalytic Hydrogenation of Carbon Oxides.

I, GEORGES PATART, Civil Engineer, 50, rue Spontini, Paris, France, a citizen of the French Republic, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

For synthesizing methyl alcohol and other organic compounds containing oxygen by the reduction of oxides of carbon, use can be made of contact masses consisting of one or more definite basic salts resulting from the combination of zinc oxide with metal oxides of an acid nature (such as chromates, tungstates, vanadates, manganates of zinc) each salt containing at least two equivalents of zinc oxide for one equivalent of the acid oxide. With such catalyzers of a marked basic character and under the form of definite salts, the reacting gases need not be specially purified and freed from sulphur and iron compounds, and the contact masses will preserve for a long period their normal catalytic activity, for instance for 200 hours' operation, until they have fixed a very considerable amount of sulphur.

This invention has for its object to restore the initial activity of the contact masses when they have fixed such a substance which is well known to be most prejudicial to catalytic reactions, and this only by a simple and inexpensive treatment.

For this purpose, the contact masses are regenerated by submitting the same to an oxidising calcination until the impurities which can be volatilised are eliminated, the zinc oxide being again combined with the acid oxides which have thus been brought to their higher degree of oxidation.

In carrying out my improved process, use is made of a basic chromate of zinc for instance, corresponding to the combination of 3 molecules of zinc oxide with one molecule of chromic acid, the contact mass being agglomerated in the form of grains. Such a basic chromate of zinc may be mixed with relatively large quantities of alkaline sulphates (as far as 15—20 per cent.) without having any prejudicial action upon the hydrogenating catalysis, whereby use may be made of such chromates of zinc which are prepared industrially for the manufacture of pigments and in which the alkaline sulphates have not been eliminated.

Such compounds are formed into grains by the known processes. By the use of such contact masses it is possible to employ gas mixtures which have not been specially purified in view of a complete elimination of impurities (and chiefly of the sulphur compounds); in this manner the apparatus can be operated for over 200 hours on a closed circuit without any marked decrease in its activity.

When however the sulphur compounds have accumulated on the contact mass in such quantities as to obstruct the pores, these impurities, and chiefly the sulphur compounds, are eliminated from the contact mass, according to the invention, by a mere oxidising calcination of the spent catalytic mass, such calcination being obtained for instance by heating the contact mass to red heat in an open crucible or by slow circulation through an oxidising flame. In these conditions, the volatile impurities, and chiefly the sulphur compounds, are expelled even to the last traces, both by sublimation and by oxida-

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tion, and further the oxides which are contained in the contact mass, and which have been more or less reduced to their lower degree of oxidation (i.e. to sesquioxide of chromium, tungsten, vanadium or manganese) now return almost entirely to the state of higher oxides having an acid nature (i.e. chromic, vanadic, tungstic or manganic acids), and will again form definite combinations with the zinc oxide, as may be observed both by the change in the colour of the substance and by chemical analysis.

The aforesaid method of renegerating by calcination is both easy and simple; the texture of the grains forming the contact mass is not changed, and it is simply necessary to remove the very small amount of dust which always results from the manipulations, in order that the contact mass thus treated, when placed in the catalyzing chamber will operate with the same efficacy and for the same length of time as when it consists of fresh material.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process of regeneration of the contact masses of the type referred to, which consists in submitting the same to an oxidising calcination until the impurities which can be volatilised are eliminated, the zinc oxide being again combined with the acid oxides which have thus been brought to their higher degree of oxidation.

2. A process of regeneration of the contact masses of the type referred to, substantially as described.

Dated this 12th day of May, 1926.

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