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(12) Patent:

(54) MANUFACTURE OF OXYGENATED ORGANIC COMPOUNDS

(54) PRODUCTION DE COMPOSES ORGANIQUES OXYGENES

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(74) Agents:

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CLAIMS: [Show all claims](#)

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Specification. 251485

TO ALL WHOM IT MAY CONCERN :

Be it known that we, Mathias Pier, Martin Müller-Cunradi, Gustav Wietzel and Karl Winkler, Chemists, the first of Heidelberg and the others of Ludwigshafen-on-Rhine, Germany, having jointly invented a certain new and useful improvement in the MANUFACTURE OF OXYGENATED ORGANIC COMPOUNDS, do hereby declare that the following is a full, clear and exact description of the same.

It has been found that by the action of suitable catalysts on mixtures of oxides of carbon and hydrogen, or hydrocarbons rich in hydrogen, at an elevated pressure and temperature, methanol or other oxygenated organic compounds are produced with good yields.

When carrying out this process on an industrial scale, however, it has been found that certain difficulties are experienced, the catalysts soon losing efficiency, or giving rise to undesirable secondary reactions, resulting in the formation of hydrocarbons and the like.

A thorough investigation has shown that such irregularities are caused owing to the fact that the gases subjected to the catalytic action, even if carefully purified in the usual way, are not pure enough for the purpose in question and that it is indispensable to remove, besides organic compounds of sulfur, even traces of volatile compounds of iron, especially iron carbonyl compounds. Provided the gases are purified to such a degree that neither sulfur, nor iron, compounds can be found in them by an intimate analytic examination, the contact masses will work reliably and continuously, producing methanol and similar compounds.

According to the present invention it is rendered possible to employ a great variety of industrial gases. These should be suitably composed so as to contain hydrogen in an amount exceeding that of carbon oxide. For example, blue gas composed of, by volume, 40 per cent of carbon monoxide, 55 per cent of hydrogen,

4 per cent of nitrogen and 1 per cent of hydrocarbons, may be employed, or coal gas, coke oven gas, low temperature carbonisation gas or mixtures, and if so required, insufficient percentage of carbon monoxid, or hydrogen may be completed by adding proper gases, or removing an excess present until the proportion desired is attained, for instance 3 or 4 volumes of hydrogen to each one volume of carbon monoxid. Such corrections may be made either before or after purification of the industrial gases .

In order to effect the purification desired the bulk of the impurities is first removed in the usual way and unsaturated or aromatic hydrocarbons may also be removed and subsequently a purifying treatment, as efficient as possible, should be applied. Carbon oxysulfid and other organic sulfur compounds may be absorbed by active coal or decomposed with soda lime or potash lime, or absorbed as well as decomposed, either successively or simultaneously. Or the gas may be passed over a hot mass containing caustic alkali. A very efficient method of purification consists in mixing the gas with steam, conveying the mixture over a contact mass, suitable to convert part of the carbon monoxid into carbon dioxid, while liberating hydrogen, for example iron oxid, the organic sulfur compounds being thereby decomposed and sulfuretted hydrogen formed which can be removed without difficulty.

In order to completely remove iron carbonyl compounds from the gas which will not be retained by the ordinary means of removing contact poisons, special methods must be resorted to. To the said end, the gas may be led through a layer of active carbon, particularly selected for the retention of iron compounds or it may be conducted over a hot copper-containing mass, or a mass substantially similar to the methanol forming catalyst and the dust of iron particles, if any, produced retained by a filter.

All or part of these operations may be effected at increased pressure.

At the same time it is advisable to keep also the contact mass free from sulfur and other obnoxious admixtures, though small amounts of sulfur, chlorine, phosphorus, arsenic or the like are in most cases but little injurious especially if the contact mass contains a strong base, and in order to avoid contamination of the gases with volatile iron compounds on their way to the catalyst, the reaction chamber and metallic parts therein and such like should be lined with, or consist of, material resisting the action of carbon monoxid, such as for example copper.

We claim :

- 1) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxides with hydrogen, which is characterized by subjecting to the reduction such gases as have been freed from sulfur compounds and other usual contact poisons including organic sulfur compounds and from volatile iron compounds.
- 2) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxides with hydrogen, which is characterized by subjecting the gas mixture prior to the catalytic decomposition to the action of a hot copper-containing mass.
- 3) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxides with hydrogen, which is characterized by subjecting the gas mixture prior to the catalytic decomposition to the action of steam in the presence of a contact mass conducive to the formation of hydrogen and carbon dioxide, removing any sulfur compounds including organic sulfur compounds and then removing volatile iron compounds.
- 4) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxides with hydrogen, which is characterized by passing the gas mixture prior to the catalytic decomposition through a layer of active carbon sufficient to retain any volatile sulfur and iron compounds.
- 5) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxide with hydrogen, which is characterized by passing the gas mixture prior to the catalytic decomposition through several layers of active carbon until any volatile sulfur and iron compounds are retained.
- 6) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon

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oxids with hydrogen, which is characterized by passing the gas mixture prior to the catalytic decomposition through active carbon, then through a hot copper-containing mass, filtering the gas, all operations being effected under elevated pressure.

7) A process of manufacturing methanol or other oxygenated organic compounds by catalytically reducing, under pressure, carbon oxids with hydrogen, which is characterized by passing the gas mixture prior to the catalytic decomposition through active carbon then through a hot copper-containing mass, filtering the gas, all operations being effected under elevated pressure, then passing it over a contact mass causing the formation of methanol, while maintaining the pressure and while at an elevated temperature.

Signed at Ludwigshafen-on-Rhine, Germany, by the said Martin Müller-Conradi and Gustav Vietzel, this 9th October 1923.

Mathias Pier
Martin Müller-Conradi

Signed at Merseburg, Germany, by the said Mathias Pier and Karl Winkler this 12th October 1923.

Gustav Vietzel
Karl Winkler

Signed in the presence of :

Wilhelm Scherer
Gustav Adolph Reutlinger