ALIEN PROPERTY CUSTODIAN

PROCESS FOR CHEMICAL REACTIONS IN MELTS

Walter Beck, Hans Walter, Frankfurt A/Main, and Klaus Bonath, Kronberg I/Taunus, Germany; vested in the Alien Property Custodian

No Drawing. Application filed August 30, 1939

Object of our invention is a process for carrying out chemical reactions consisting in introducing gaseous or vaporous reactants into a melt. This melt serves as a heat carrier, acting itself catalytically or containing catalysts. The intro- 5 duction of the reactants into the melt is carried through in such a manner that the gases or vapors effect a permanent, thorough and uniform intermixture of the melt as well as of the introduced substances. The temperature in the melt 10 is well adjusted according to the conditions of the reaction. By these means it is possible to maintain the melt during the whole process at the desired temperature which proves to be of special advantage for the reaction. At the same time 15 it is possible to bring the reactants into intimate contact with each other and also with the melt and the catalysts. In consequence of the considerable capacity of heat of the salt melt with respect to the comparatively insignificant little 20 parts of the reaction participants which are present in the melt, all possible chemical reactions may be carried through isothermically, even if the reactions per se are qualified by marked absorption or evolution of heat. As a consequence of 25 the permanent intermixture of the melt, evolved by the gaseous or vaporous reactants, new heat transfer as well as catalytically effective contact surfaces between the melt and the reactants are constantly formed.

The introduction of the gaseous and/or vaporous reactants into the melt may be carried out with nozzles or the like. If necessary, the agitating effect may be enhanced by the introduction of a gaseous initial substance in excess or even in great excess. According to our invention it is also possible to introduce inert gases or vapors, as for instance, carbonic acid, water vapor and the like, besides the other gaseous or vaporous reactants, in order to increase the agitation in 40 melt.

Our invention is especially suitable for the carrying through of various reactions, such as for instance, the chemical addition of water, splitting off water, hydrogenation, dehrydrogenation, oxidation, dissociation, polymerisation and the like. At the same time organic compounds of different character, for example hydrocarbons, compounds containing oxygen, such as alcohols, aldehydes, carboxylic acids, ketones, phenols, amines and the like may be introduced into the melt and brought to conversion therein. Furthermore, reactions of an anorganic nature, for instance, the manufacture of hydrocyanic acid ex carbon monoxide and ammonia, or the manufacture of SO₃ and the like may be carried out according to our invention.

such as for instance, nitrates, nitrites, chromates, acetates, chlorides, cyanides, bromides, furthermore oxides, hydroxides, sulfates etc. of alkali metals, earth alkali metals, earth metals, rare earths, such as cerit chloride and so on. Furthermore carbonates, phosphates, borates, silicates, such as glasses or silica, and the like, cyanates, urea or other organic substances such as higher boiling or high melting hydrocarbons, for instance, paraffin, naphthalene, dekahydronaphthalene, halogenated naphthalene, diphenyloxide, nitrobenzene or the like may be used as initial substances for the bath.

The above mentioned compounds and substances may be used sometimes per se but usually we have found it advantageous to combine some of these compounds to form a mixture which is especially suitable for the necessary temperatures as well as for the desired reaction. Generally, the composition of the melt will be selected in such a manner that the temperature of the reaction will be counterbalanced between the solidification point and the evaporation point.

EXAMPLES

I. NITRATES

		Degree	es
		Centigr	
	30 KNO ₃ +70 Lino ₃		130
()	55 KNO ₃ +45 NaNO ₃		218
	50 KNO ₃ +50 NaNO ₃		
	50 NaNO ₃ +50 NaNO ₂		22 0
	II. CHLORIDES		
ŏ	(a)	_	
		Degree	
	01 Deci 10 deci 01 medi	Centigr	ade
	31 BaCl ₂ +48 CaCl ₂ +21 NaCl		430
0	50 BaCl ₂ +30 KCl +20 NaCl		34 0
	(b) Chlorides+carbonates		
	50 KCl+50 Na ₂ CO ₃		560
	(c) Chlorides+fluorides		
.5	85 CaCl ₂ +15 CaFi		645
	(d) $Chlorides + sulfates$		
	35 NaCl+65 Na ₂ SO ₄	-	620
0	(e) Chlorides+phosphates	5	
	35 K ₃ PO ₄ +67 KCl		72 0
	(f) Chlorides+borates		
5	88 BaCl ₂ +7 Na ₂ B ₄ O ₇ +5 MgO		980
,	(g) Chlorides $+$ carbonates $+$ su	lfates	
	40 NaCl+20Na ₂ CO ₃ +40 Na ₂ SO ₄		500
	III. CHROMATES		

IV. SULFATES				
25 K ₂ SO ₄ +75 Na ₂ SO ₄	830			
V. PHOSPHATES				
70 N84P2O7+30 K4P2O7	875			
44 K ₄ P ₂ O ₇ +56 KPO ₄	615			
VI. SILICATES				
45 BaSIO3+55 Na2SiO3	905]		
VII. BORATES				
50 NaBO ₂ +50 LiBO ₂	648			
VIII. BROMIDES				
50 KBr+50 NaBr	640]		
The regulation and maintainance of the guit				

The regulation and maintainance of the suitable temperature of the reaction may be carried into effect by external heating, but we prefer mostly to use internal heating. If the reactions 20 are of an exothermic nature the liberated heat may be used for internal heating, for instance in such a manner that the conversion is combined with an other exothermic reaction or combustion. We have found it advantageous to use 25 flues or dipping burners for internal heating, whereby the dipping burners may be arranged in a chamber adjacent to the reaction chamber in such a way that no mixture of the combustion gases with the reaction products takes place. 30 The heating may also be effected by means of an electric current, for instance, by resistance heating or by heating through electrodes whereby the salt melt acts as an electrolyte and the alternative current may be conducted through elec- 35 trodes in contact with the electrolytes. Furthermore, we have found it useful, to combine various heating means, for instance in such a manner that the main heat supply will be effected by means of dipping burners or flues whereas the fine adjustment will be carried out by resistance or electrode heating. Moreover, the use of internal heating has the advantage that the walls of the melting vessel may be made out of metallic as well as out of ceramic material and hence 45 be adapted perfectly to the thermic and chemical

If the heating is carried out with electrodes the latter may either act chemically or catalytically or influence the conversion in a favorable man- 50 ner. We have found it advantageous to use graphite electrodes with the manufacture of hydrocyanic acid ex carbon monoxide and ammonia, whereby the electrodes may deliver the carbon as well as remove the undesired oxygen. 55

By carrying out our invention we proceed sometimes in such a way that the substances to be introduced into the melt are subjected to a pre-heating and may then be used for internal heating or adjustment of temperature. The pre-heating of the substances to be introduced may be effected with the waste heat, for instance, coming from the flues. Excessive introduction of reaction participants or additional introduction of inert gases or vaporous substances will also lead to the formation of excessive heat.

Furthermore, the introduction of gaseous or vaporous substances into the melt may be used to blow liquid or finely divided solid substances into the melt.

As we have already mentioned, the melts may be used or their composition selected in such a 5 manner that they may act specifically or catalytically per se. Certain oxidising reactions may be improved by introducing of boric acid or borates or barium compounds into the melt. On the other hand, specifically acting catalysts, insoluble in the melt, may be used either alone or in combination, for instance, in such a manner that they are suspended in the melt in a finely or extremely finely powdered condition. Catalysts which tend to deposit in the melt or to rise therein, may be precipitated on suitable carriers which do not liquate and may be suspended in the melt.

As catalysts various elements and compounds such as for instance, metals, metal compounds, earth acids or salts may be used. For instance, hydrogenations may be carried into effect with the aid of the usual hydrogenation catalysts, such as nickel, iron, molybdenum, tungsten, zinc oxide. Oxidations may be carried into effect in the presence of oxidation catalysts, such as for instance. iron oxide, vanadic acid and so on; splitting off water will be carried out in the presence of aluminium oxide, titan dioxide, earth alkali phosphates and so on. Carriers for catalysts, which themselves tend to deposit, are aluminium oxide, voluminous or active silica, asbestos, magnesium oxide, beryllium oxide, charcoal, activated charcoal; carriers for ascending catalysts are, for instance, heavy metals, such as tungsten, copper and the like.

Our invention has been proved especially suitable for the carrying through of reactions which require high temperatures as well as for the maintainance of a constant temperature, such as for instance, the manufacture of acetylene or ethylene from methane. Furthermore, our invention is extraordinarily well suited for the carrying out of partial oxidation reactions as for instance, the manufacture of phthalic acid or maleinic acid by oxidation of aromatic hydrocarbons; or, for the manufacture of carboxylic acids, ketones, aldehydes or alcohols by partial oxidation of methane or its homologues.

Our invention may be carried out at usual pressure, under vacuo or at increased pressure. also at high and very high pressure. If it occurs that considerable quantities of the melt are carried over in a vapor- or mist like form together with the reaction products, these quantities may be collected in an absorption bulb and re-conducted into the melting vessel. The melts as well as the catalysts therein may be regenerated during the operation, for instance, in such a manner that one part of the melt will be passed over into a chamber adjacent to the reaction chamber, where it will be subjected to a regenerating treatment, for instance, by means of water vapor or oxidising gases and afterwards reconducted into the reaction vessel.

> WALTER BECK, HANS WALTER, KLAUS BONATH,