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IMPROVEMENT OF THE TECHNOLOGICAL PROCEDURE OF THE SEMI-INDUSTRIAL PRODUCTION OF 2,2-BIS (ETHYLFERROCENYL) PROPANE AS A CATALYST FOR COMPOSITE ROCKET FUELS

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Abstract:

2,2-bis (ethylferrocenyl) propane tecane is a derivative of ferrocene. It is obtained by a process that includes three reactions: a) acetylation of ferrocene, b) reduction of acetilylferocene to ethylferrocene and c) condensation of two molecules of ethyferrocene with acetone. In the course of work, significant improvements were made to the procedure, especially phase b), of the reduction of acetyferrocene to ethylferrocene. After detailed laborattory tests of the reduction of acethylferrocene according to Clemmensens method, we managed to define two variants of the reduction with an acceptable product yield. These improvements have created a lot of preconditions for the semi-industrial production of 2,2 – bis (ethylferrocenyl) propane. Key words: ferrocene, condensation, acetylation, acetylferrocene, ethylferrocene, reduction.

1. INTRODUCTION

Among the catalysts used in recent years to achieve high speeds in composite rocket fuels (2,2-bis(ethylferrocenyl) propane, EtFc-C(CH3)2FcEt, Fc= –[C5H4-Fe-C5H4-] occupies a very important place. By reviewing the literature, it can be seen that this compound has so far only been produced on a laboratory scale using a process that includes three reactions, namely: Acetylation of /1/ ferrocene (1), whereby acetylferrocene (2) is obtained. (1) HFcH →(CH3CO)2O (2) HFcCOCH3 (a) Reduction of /2/ acetylferrocene (2) to ethylferrocene (3). (2) HFcCOCH3 →reduction (3) HFcC2H5 (b) Condensation of /3/ ethylferrocene (3) with acetone (4), whereby 2,2-bis (ethylferrocenyl) propane (5) is obtained. (3) 2 HFcC2H5 + (4) CH3COCH3 → cat. (5) C2H5Fc-C(CH3)2-FcC2H5 (c) 1. SYNTHESIS OF 2,2-BIS (ETHYLFEROCENYL) PROPANE The reduction of acetylferrocene to ethylferrocene (phase b) represented the biggest problem in the realization and establishment of semi-industrial production, as well as the realization of semi-industrial conditions for the production of 2,2 bis(ethylferrocenyl) propane. It can be seen from the literature that eliferocene was synthesized in two ways: 1) by alkylating ferrocene and 2) by reducing acetylferrocene /5/. We did not accept the first method, alkylation of ferrocene, as a good and promising one, because alkylation usually results in several products whose separation is difficult and very complex. In addition, the low yield of alkylated products was another reason to abandon the alkylation of ferrocene. The second method, the reduction of acetylferrocene, seemed to us to be more acceptable from the point of view of the uniqueness of the reaction. Clemmensen's method of reduction with amalgamated zinc, catalytic hydrogenation and reduction using lithium aluminum hydride and aluminum chloride are mentioned in the literature for this reaction. Of these three methods, we chose reduction with amalgamated zinc because we estimated that it can be performed most easily in semi-industrial conditions. After detailed laboratory tests on the reduction of acetylferrocene according to Clemmennsen's method, we managed to define two variants of the reduction with an acceptable product yield. In the first variant, the two-phase system benzene-acetic acid was used, and the yield of ethylferrocene was 79%. In the second variant, the solvent ethanol   water-hydrochloric acid was used and the eliferocene yield was 72.5%. In both variants, the product is isolated by distillation under reduced pressure, and the complete synthesis and processing of the reaction mass (without distillation) can be completed in eight hours. The obtained product is of high purity, the structure of which was confirmed by the analysis of IR and NMR spectra. The last stage in the production process of 2,2-bis(ethylferrocenyl) propane (stage c) represents the condensation reaction of two molecules of ethylferrocene with acetone. The reaction is carried out in the presence of acid catalysts such as sulfuric acid, polyphosphoric acid, aluminum trichloride and others. We opted for the reaction in the presence of sulfuric acid. The reaction itself is performed in two parts. Preparation of sulfuric acid solution in methanol and reaction of ethylferrocene with acetone. The reaction takes place at a moderate temperature. The product obtained by the mentioned reaction is isolated by extraction with an organic solvent. By processing the solution and evaporating the solvent, a thick red oil was obtained in a yield of 71% based on ethylferrocene. The structure of the product was proven by the analysis of IR and NMR spectra.

2. CONCLUSION

2,2 bis-(ethylferrocenyl) propane is used as a high efficiency combustion catalyst for various composite solid propellants. It is characterized by excellent all-round performance and low mobility. As a dinuclear ferrocene derivative, it produces burning rate increasing and pressure lowering effects when added to a composite solid propellant using an AP oxidant. As already described, 2,2-bis(ethylferrocenyl) propane as a liquid derivative of ferrocene is obtained by a process that includes three reactions: a) acetylation of ferrocene, b) reduction of acetylferrocene to etiferocene and c) condensation of two molecules of ethylferrocene with acetone. In progress significant improvements to the laboratory procedure, especially phase b), were developed, i.e. reduction of acetylferrocene to ethylferrocene, which resulted in the replacement of the use of organic solvents and the reduced amount that was used earlier, and therefore had a negative impact on the environment and on the human health of the employees (participants) of the process. A new replacement solvent was used ethanol-water-hydrochloric acid, which did not have any negative effects and consequences. These improvements have created a lot of preconditions to carry out and realize semi-industrial production with an improved technological production process and a shortened reaction time of eight hours of work. This contributed to the increase in production and obtaining the desired quantity and quality of the product.

3. LITERATURE

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