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AFNOL PREPARATION

W. T. Quinlin

DEVELOPMENT DIVISION

JULY - SEPTEMBER 1971
SANL 900-004

For
Lawrence Livermore Laboratory
Livermore, California

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DISCLAIMER

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Section D

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ABSRRACT

A nominal 15-pound batch of FEFO plasticized AFNOL was in preparation. Both the first and second esterifications were completed. (An equipment malfunction resulted in a loss of the product and the reaction vessel).

DISCUSSION

The starting 2,2-dinitropimelicacid chloride (DNPCl) was that which was reclaimed by an extraction method(*1*) from the crude commercial product. The 2,2,8,8-tetranitro-4,6-dioxononanediol (DINOl) used in the first esterification was an Aerojet product(*2*) which was used without further purification. The reaction was run in a 5-liter, 5-neck, round bottom flask fitted with mechanical stirring, a nitrogen purge and a reflux condenser. A water trap was attached to the condenser to monitor the evolution of hydrogen chloride. The heating was accomplished by a constant temperature ethylene glycol bath.

DINOl was melted in the reaction flask under a flow of dry nitrogen to remove the water of hydration. The DNPCl was added to the melt in small portions over a two-hour period under the nitrogen purge. After each addition the reaction was stirred until the excessive foaming subsided. The completeness of the reaction was followed by trapping the evolved hydrogen chloride gas in the water trap and titrating with sodium hydroxide. When the reaction rate dropped to less than 2% per hour, ethylene chloride was added and the solution brought to reflux in order to rinse the vessel walls.

The second esterification was a solution reaction in ethylene dichloride involving the reaction product from above, 1,1,1-trimethylolpropane (TMP) and the plasticizer bis 2,2-dinitro-2-fluoroethylformal (FEFO). The reaction was run in a 50-liter glass reaction

Second Esterification	
Prepolymer	∿ 1240 g
1,1,1-Trimethylolpropane	131 g
FEFO	5850 g
Ethylene Dichloride	7500 g

vessel fitted with a reflux condenser, nitrogen purge, electrical heating mantle, mechanical stirrer and vacuum take-off. The FEFO used was a Rocketdyne product consisting of 5.85 kg of FEFO dissolved in ethyl acetate. The ethyl acetate was

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(1) NPD Endeavor No. 101, June 1971
(2) Aerojet General Corp.

removed by vacuum distillation in the 50-liter vessel. The residual FEFO was allowed to remain in the vessel. The product from the previous esterification was added to the 50-liter vessel and two liters of ethylene dichloride were used to wash out the 5-liter flask from the first esterification. The wash was also added to the 50-liter vessel TMP (131 g) and the remainder of the 7.50 kg of ethylene dichloride were then added. The reaction was brought to reflux (∿ 80 C) for 20 hours.

The removal of the ethylene dichloride was then started by vacuum distillation. During this process a malfunction occurred with the reaction vessel resulting in loss of the product and the vessel.