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COMMERCIAL SOLVENTS CORPORATION

TERRE HAUTE, INDIANA

February 12, 1953
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Report No. Q-5
(Quarterly Summary)

SUBJECT: ONE Nitropolymer Research

CONTRACT: Nonr-397(00)

PERIOD COVERED: November 1, 1952 to
January 31, 1953

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Report No. Q-5

CONTRACT FULFILLMENT

This quarterly report is submitted in partial fulfillment of
Contract Nonr-397(00).

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I. SUMMARY

A. This quarterly summary report is the fifth under Contract Nonr-397(00) and covers the period from November 1, 1952 to January 31, 1953. The object of this contract is as follows: "Shall conduct research in the synthesis of polynitro compounds to include, but not necessarily be limited to, a review of the chemistry and the processes of preparation of the more useful products of research from the nitropolymer program and investigate the application of processes not now employed in the preparations."

B. The more important results and conclusions of the work reported are presented below.

1. 2,2-Dinitropropanol has been prepared from 1-chloro-1-nitroethane by the Ter Meer reaction in 51% yield with no isolation of pure intermediate compounds.

2. 2,2-Dinitropropanol has been prepared in 80% yield from pure nitroethane by the Shechter-Kaplan reaction with no isolation of intermediate compounds.

3. It has been concluded that for pilot plant preparation of 2,2-dinitropropanol the Shechter-Kaplan reaction offers less potential hazard, gives better quality product under the reported conditions, and compares favorably costwise with the Ter Meer reaction.

4. 2,2-Dinitropropyl acrylate has been prepared from glacial acrylic acid and dinitropropanol in 79% yield, using trifluoroacetic anhydride as the dehydrating agent. The procedure is characterized by low temperature and short reaction time, and it gives a clean product without distillation.

5. 2,2-Dinitropropyl acrylate has been prepared from acrylyl chloride and dinitropropanol in 79% yield, using anhydrous aluminum chloride as a catalyst. The reaction proceeds at a low temperature with a short reaction time and gives a slightly colored product without distillation.

6. If the dinitropropyl acrylate monomer is acceptable for polymerization without distillation, the process using acrylyl chloride is the most favorable costwise.

7. Methyl 4,4-dinitroheptanedioate has been prepared in a 0.15 lb-mol batch in the pilot plant in 39% yield based on nitromethane. The 4,4-dinitroheptanedioic acid was obtained from the ester in a 92.5% yield.

II. TECHNICAL PROGRESS

A. INTRODUCTION

The present program is directed towards the industrial development of nitropolymer starting materials and intermediates. The comparison of the Ter Meer reaction and the Shechter-Kaplan reaction for the preparation of 2,2-dinitropropanol has been made and a pilot plant process studied. The comparison of various methods for preparing 2,2-dinitropropyl acrylate has been made and a

pilot plant process studied. The laboratory procedure for the preparation of methyl 4,4-dinitroheptanedioate has been scaled to a pilot plant process and a run has been made.

B. THE PREPARATION OF 2,2-DINITROPROPANOL (DNPOH)

1. Discussion

The problem of comparing the preparation of 2,2-dinitropropanol from 1-chloro-1-nitroethane by the Ter Meer¹ reaction and from nitroethane by the Shechter-Kaplan reaction was undertaken. The various paths of procedure and the yields are as shown on Fig. 1. The problem of preparing DNPOH in a grade pure enough so that crosslinking in the polymerization of the acrylate prepared from it is at a minimum hinges somewhat on the purity of the starting nitro compound. If the chloronitroethane is prepared from commercial nitroethane, Table I illustrates the potential impurities.

TABLE I

Boiling Points at Atmospheric Pressure

2-Nitropropane	120.3°C.
Chloronitromethane	123°
1-Nitro-1,1-dichloroethane	124°
1-Nitro-1-chloroethane	125°
2-Nitro-2-methylpropane	126.5°
1-Nitropropane	131.6°

From the similarity of boiling points of the possible impurities, it seems impossible to obtain a pure grade of chloronitroethane and a good grade of DNPOH from commercial material by reaction B of Fig. 1. If the potassium dinitroethane is purified, or the dinitroethane is fractionated, a good grade of DNPOH can be obtained by the Ter Meer reaction from commercial grade chloronitroethane. These latter two purifications involve potential hazards which make the procedure undesirable for pilot plant study.

The nitroethane used in reaction A, Fig. 1, can be purified by fractionation without too much difficulty. The impurities are illustrated in Table II.

TABLE II

Boiling Points at Atmospheric Pressure

Nitromethane	101°C.
Nitroethane	114°
2-Nitropropane	120°
1-Nitropropane	132°

Using purified nitroethane in the Shechter-Kaplan reaction it has been possible to obtain DNPOH of good quality, m.p. 87-90°C., in yields up to 80%.

1. Ter Meer, Ann. 181, 1-22 (1876).

As the Ter Meer reaction gives a lower yield than the Shechter-Kaplan reaction and also involves more labor, there is very little difference in the estimated cost of production.

2. Ter Meer Experimental Procedure

A stainless steel beaker is set up in an ice bath and is provided with an efficient stirrer. To the beaker is added 110 g. (1.0 mole) purified 1-chloro-1-nitroethane and 300 ml. methanol, and the solution is chilled to 0°C. Add dropwise a solution of 73 g. (1.0 mole) sodium nitrite and 6.6 g. (1.0 mole) potassium hydroxide in 160 ml. water and stir the slurry formed for 2½ hrs., filter while cold. After the yellow potassium dinitroethane precipitate has been washed with cold methanol, it is returned to the reaction vessel in the ice bath and 500 ml. water, 75 ml. (1.0 mole) 37% formaldehyde solution, and 10 g. urea are added. With stirring, 1 mole of sulfuric acid is added as a 20% solution, dropwise. The reaction solution is then warmed to 45°C. and stirred for 1½ hrs., followed by cooling and extracting twice with 750 ml. portions of ethyl acetate. The ethyl acetate extract is stirred with 1% carbon for an hour, dried over anhydrous sodium sulfate and evaporated. On pulling the resulting oil dry at 60-70°C. with a vacuum pump, 77 g. (0.51 mole, 51%) of waxy DNPOH sets to a solid, m.p. 84-87°C. The product has a slight yellow color.

3. Shechter-Kaplan Experimental Procedure

To a vessel equipped with a stirrer is added 150 g. (2.0 mole) of fractionated nitroethane, 340 ml. water, 265 g. ice, and a solution of 88 g. (2.1 mole) sodium hydroxide in 176 ml. of water. The ice can be replaced with water and the solution cooled externally. By ½ hr. the nitroethane has gone into solution as the anion and the temperature is below 30°C. After placing the reaction vessel in an ice bath, 149 ml. (2.0 mole) of 37% formaldehyde solution is added slowly from an addition funnel, keeping the temperature below 15°C. This requires about 15 min. and the solution is stirred at 15° to 25°C. for 1 hr. Following this interval, add 150 g. (2.2 mole) of sodium nitrite as a solution in 200 ml. water and stir an additional 15 min.

The reaction is carried out by rapidly adding the prepared salt solution of nitropropanol to 680 g. (4.0 mole) of silver nitrate in a 2 Kg. water solution with the pH adjusted to 6.0±0.2. The temperature of the reaction is kept less than 25°C. by external cooling or by the addition of ice. The reaction is allowed to proceed for ½ hr., during which time the initial light precipitate turns black, then gray-green; filter. The precipitate is washed twice with 250 ml. of water and the filtrate, which is approximately 2 liters if no ice is added during the reaction, is extracted twice with 1.5 liters of ethyl acetate. The combined extracts are stirred with 1% carbon for 1 hr., filtered with the aid of filter aid, and concentrated at reduced pressure at 50°C. or less. When all the solvent is removed, pull at least 3 mm. of mercury vacuum and gradually raise the temperature of the concentrate. Hold the temperature in the pot at 70°-75°C. with high vacuum until solidification begins, at which point the product is poured from the flask into a crystallizing dish and dried under vacuum for at least 6 hrs. The dinitropropanol obtained will amount to 210-240 g. (70-80%), have a slight yellow color and a m.p. of 80-90°C.

The silver nitrate is recovered from the filter cake with nitric acid in 96 to 98% yield. From 0.5 to 3.0% more silver nitrate is obtained by

making the raffinate, from the ethyl acetate extraction, basic with caustic and adding the resulting precipitate to the original silver cake.

4. Preliminary Pilot Plant Material and Labor Cost Estimate

The estimate is for material and labor only on a pilot plant process using the Shechter-Kaplan reaction which is based on a 0.20 lb-mol batch, 70% yield, and a 3% loss of silver nitrate.

Total material lost and used	\$31.12
Labor estimated	104.16
Total	\$135.28

Yield -- 21.0 lb. DNPOH

Cost per pound \$6.44

C. 2,2-DINITROPROPYL ACRYLATE (DNPA)

1. Discussion

The object of this investigation is to develop a pilot plant process and preferably one in which the produced DNPA does not have to be distilled. The DNPA has been prepared from DNPOH and glacial acrylic acid¹, and from DNPOH and acrylyl chloride². With both acrylyl chloride and acrylic acid the reactions were carried out in refluxing benzene and the time required was 4 to 5 days. The product was very dark and had to be distilled. Dr. L. W. Kissinger³ has used trifluoroacetic anhydride, mole per mole acrylic acid, to remove the water of esterification in a low temperature, short reaction time procedure. This technique gives a very nice product without distillation but the anhydride is very expensive at the present time. The low boiling point of the anhydride and the acid, which can be recovered and reconverted with sulfur trioxide, makes recoveries low, 75-80%.

The Navy Ordnance Laboratory has studied and used metal halides⁴ as catalysts in the esterification of DNPOH with acid chlorides. This technique permitted low boiling solvents as chloroform, and short reaction times, 3 hrs. When this technique was used in the preparation of DNPA from acrylyl chloride, more than the theoretical amount of hydrogen chloride had been swept from the reaction flask in 3 hrs. Acrylyl chloride is quite expensive and difficult to prepare and handle; however, for large runs it can be bought at low enough price to make its use attractive. The product is clean enough to polymerize without distillation.

1. Aerojet Engineering Corp., Contract NOa(s) 10867, No. 475, p. 57.
2. Kissinger, L. W., Private Communication.
3. Private Communication.
4. NAVORD Report 2497, 1952.

2. Experimental Procedure Using Trifluoroacetic Anhydride (TFAA)

To a flask equipped with stirrer, reflux condenser, and addition funnel, held in an ice bath, is added 150 g. (1.0 mole) DNPOH and 75 g. (1.0 mole) of 96% acrylic acid containing inhibitor. After cooling to 0°C., 220 g. (1.05 mole) of TFAA (as obtained from Minnesota Mining and Manufacturing Company) is added slowly, keeping the temperature below 10°C. When the addition is complete, remove the ice bath and stir until the reaction is at room temperature. Let stand several hours or overnight.

Remove the excess TFAA and the trifluoroacetic acid that is formed at reduced pressure using a dry ice trap. The residue is then poured into an equal quantity of water and extracted with ether. The ether extracts are washed four times with 250 ml. of water, 2% sodium hydroxide solution until the inhibitor is removed, and four more times with water. Dry the washed solution with magnesium or sodium sulfate, stir with 1% carbon, and filter. After removing the ether and pulling dry by pump, there remains 161 g. (0.79 mole; 79%) of a very slightly colored dinitropropyl acrylate.

3. Experimental Procedure Using Acrylyl Chloride

The 78.0 g (0.52 mole) of DNPOH is dissolved in 400 ml. chloroform in a liter flask equipped with stirrer, dropping funnel, and reflux condenser. After 10.3 g. (0.08 mole) of anhydrous aluminum chloride is added, the solution is warmed to the reflux point and 45 g. (0.50 mole) of acrylyl chloride which is dissolved in 100 ml. chloroform is added slowly over an hour. The reaction is stirred and refluxed for three additional hours. The resulting solution is washed well, first with four portions of 200 ml. water, 2% sodium hydroxide solution, water, sodium bisulfite solution, and finally with four more portions of water. To improve the color of the monomer, the chloroform solution is stirred with 1% carbon for 1 hr., allowed to stand over anhydrous sodium sulfate and filtered. After evaporating the chloroform and pulling dry with the vacuum pump at room temperature, there remains 80 g. (0.39 mole; 79%) of DNPOH which contains only slight color.

4. Preliminary Pilot Plant Material and Labor Cost Estimate

The cost estimate includes only material and labor for a pilot plant run of 100 lbs. of DNPA produced, based on an 80% yield in both procedures. The DNPOH cost is that of the estimate in this report plus 10% for utilities and 25% for overhead.

<u>Procedure</u>	<u>TFAA</u>	<u>Acrylyl Chloride</u>
DNPOH	\$ 940.00	\$ 940.00
Acrylyl Chloride at \$10.00 per pound		648.00
TFAA at \$15.00 per pound		
Recovered acid at \$5.00 per pound	1,341.00	
Acrylic acid	65.00	
Catalyst and solvent		68.00
Total	\$2,346.00	\$1,656.00
Yield -- 100 lbs. DNPA		
Cost per pound	\$23.46	\$16.56

D. PREPARATION OF 4,4-DINITROHEPTANEDIOIC ACID1. Discussion

The method of Aerojet¹ was used to prepare methyl 4,4-dinitroheptanedioate on a 0.15 lb-mol scale in the pilot plant. The yield from nitromethane was 39% as compared to the reported 36.5%. The ester was hydrolyzed to 4,4-dinitroheptanedioic acid in 18% hydrochloric in 92.5% yield.

2. Experimental

A slurry of sodium 2-nitro-1,3-propanediol is prepared by adding 6.9 lb. (0.16 mole) sodium hydroxide in 15 lb. water to a solution of 9.2 lb. (0.15 mole) nitromethane, 24.3 lb (0.30 mole) 37% formaldehyde solution, 15 lb. water and 15 lb. ice. The reaction vessel should be cooled externally as well as the ice used and should be well stirred. The sodium hydroxide is added so as to keep the temperature below 30°C., and the slurry that forms when about half the base has been added is stirred an additional hour. To the slurry is then added a solution of 11.5 lb (0.16 mole) sodium nitrite in 27 lb. of water, and stirring continued 15 min. When the temperature of the salt slurry is down to 15°C., it is ready for the nitration reaction.

A vat equipped with efficient stirring and cooling is charged with 51.0 lb. (0.30 mole) of silver nitrate or equivalent recovered silver nitrate solution. The pH of the solution is adjusted to 6.0±0.2 and the weight of the solution brought to 135 lb. To the silver nitrate solution is then added as rapidly as possible the sodium nitropropanediol slurry and the temperature is held under 30°C. A precipitate of flocculent green-gray silver is formed and after $\frac{1}{2}$ hr. stirring, the dinitropropanediol produced is deformedylated with 5.7 lb. of sodium hydroxide dissolved in 10 lb. of water. The pH is adjusted to 8.0. The reaction mixture is then filtered to remove the silver for subsequent recovery.

The filtrate from the reaction is warmed to 45°C. and 33 lb. (an excess) of methyl acrylate is added. The addition must be adjusted, to keep the temperature from rising too high and vaporizing the acrylate, and will require about 6 hrs. After stirring for 16 hrs., the solution has returned to room temperature and is filtered. A second crop is obtained which amounts to about one-fourth of the yield if the filtrate is allowed to stand several days and is cooled and filtered again. The total yield is 16.25 lb (0.0585 mole; 39%) of dry, tan colored, methyl 4,4-dinitroheptanedioate, m.p. 42-44°C. The ester can be recrystallized from methanol in 95% recoveries.

Four pounds (0.0144 mole) of the crude ester is placed in 6 liters of 18% hydrochloric acid and refluxed for 12 hrs., after which the solution is concentrated to one-half its volume and cooled. There is obtained 3.34 lb. (0.0133 mole; 92.5%) of white crystalline 4,4-dinitroheptanedioic acid, m.p. 138-140°C.

3. Pilot Plant Material and Labor Cost

This cost is based on the 0.15 lb-mol run made in the pilot plant.

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The silver nitrate loss was estimated at 2%; the yield was 39% of crude ester, and the overall yield from nitromethane to the acid was 36%.

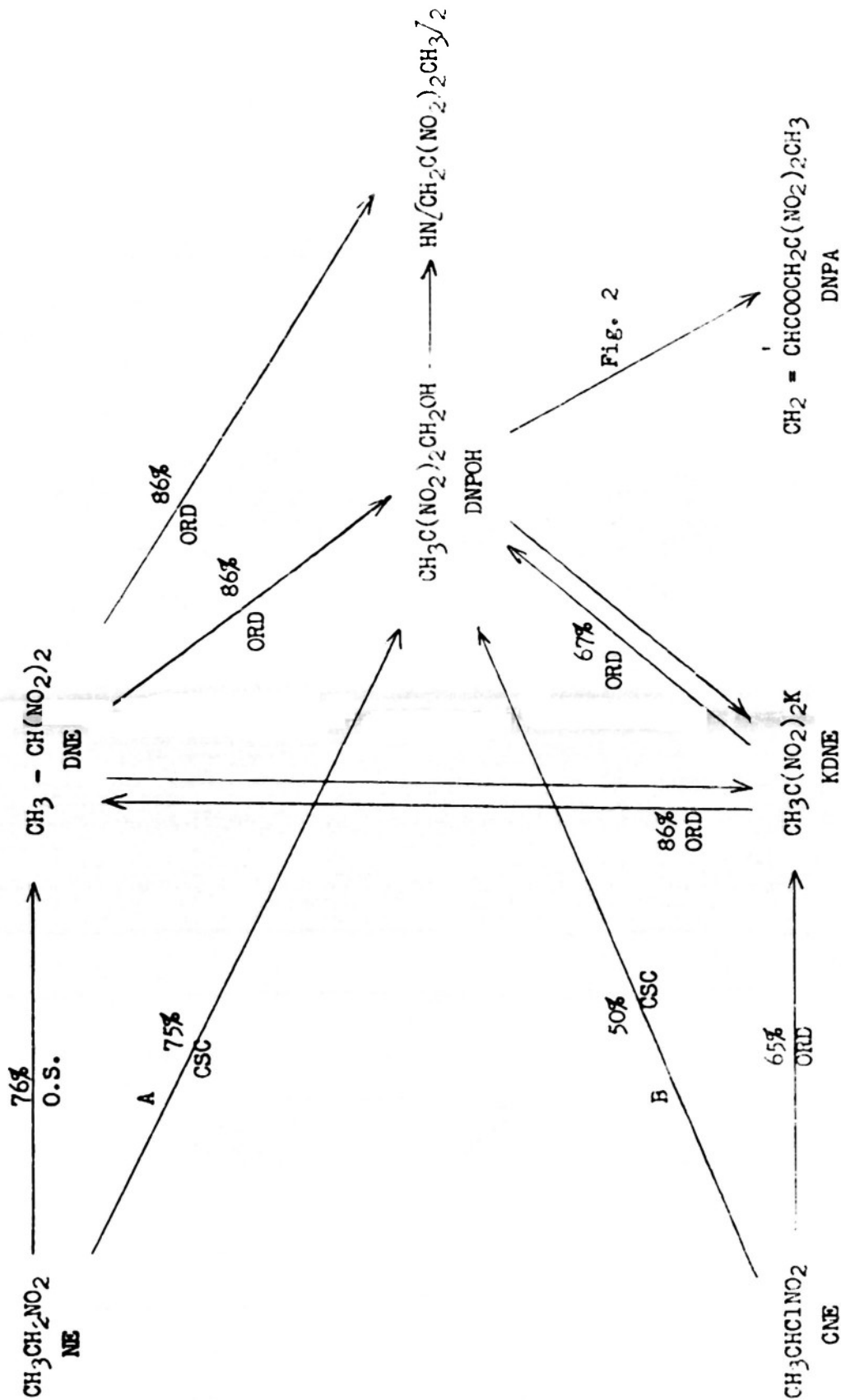
Total material	\$31.07
Total labor	<u>128.48</u>

Total	\$159.55
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Yield -- 13.5 lb. 4,4-Dinitroheptanedioic Acid

Cost per pound	\$11.80
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Fig. 1



CSC - Commercial Solvents Data

ORD - NAVORD REPORT 2448, June 1952

O.S.- Ohio State Report No. 1, May 1950

Fig. 2

2,2-Dinitropropanol (DNPOH)	+ acrylyl chloride	benzene solution Reflux 100 hrs. Distill product 53%	→	
	+ acrylyl chloride	chloroform solution AlCl ₃ catalyst Reflux 3 hrs. Undistilled product 79%	→	
	+ acrylic acid	benzene solution H ₂ SO ₄ catalyst Reflux 50-65 hrs. Distill product 65%	→	2,2-Dinitropropyl- acrylate (DNPA)
	+ acrylic acid	no solvent TFAA condensing agent Requires 6-8 hrs. re- action time Undistilled product 79%	→	

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