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TERRE HAUTE, INDIANA

May 9, 1952
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Report No. Q-2
(Quarterly Summary)

SUBJECT: ONR Nitropolymer Research

CONTRACT: Nonr-397(00)

PERIOD COVERED: February 1, 1952 to April 30, 1952

SUBMITTED BY: John T. Minor

APPROVED BY: *Jerome Martin*
Jerome Martin
Director of Research

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Report No. Q-2

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 second under Contract Nonr-397(00) and covers the period from February 1, 1952 to April 30, 1952. The object of this contract is as follows: Shall conduct research in the synthesis of polynitr. 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. Butanol is the best solvent tested for the extraction of 2-nitro-1,3-propanediol from its aqueous solution.

2. Material and labor costs for 2-nitro-1,3-propanediol amount to \$1.10 per pound based on a production rate of 200 lb. per day and a yield of 45%. These costs may be lowered to \$0.76 per pound when the production rate is increased to 2000 lb. per day.

3. In the liquid phase nitration of cyclohexene, the solid formed is cyclohexene nitrosite.

4. In the liquid phase nitration of cyclohexene, some of the lower boiling oil isolated is 1-nitrocyclohexene.

5. By the use of the Ter Meer reaction, 1,1-dinitroethane was prepared in 54% yield from 1-chloro-1-nitroethane.

6. The 1,1-dinitropropane was prepared by the Shechter-Kaplan reaction in 89% yield.

7. The 2,2-dinitropropane was prepared by the Shechter-Kaplan reaction in 97% yield.

8. A preliminary cost estimate for the materials and labor for 2,2-dinitropropane amounts to \$1.65 per pound based on 114 lb. per day production, 95% yield and 1.0% loss of silver nitrate.

9. 2,2-Dinitrobutane was prepared in 86% yield by the Shechter-Kaplan reaction.

10. 1,1-Dinitrobutane was prepared in 79% yield by the Shechter-Kaplan reaction.

II. TECHNICAL PROGRESS

A. INTRODUCTION

The present program is directed towards the industrial development of nitropolymer starting materials and intermediates.

With process development as the main object, the study of the chemistry of 2-nitro-1,3-propanediol has been continued. The liquid phase nitration of cyclohexene has been studied and some products identified. The use of the Ter Meer and Shechter-Kaplan reactions have been studied in the preparation of gem dinitroalkanes. The dinitroalkanes prepared were 1,1-dinitroethane, 1,1-dinitropropane, 1,1-dinitrobutane, 2,2-dinitropropane, and 2,2-dinitrobutane.

B. THE 2-NITRO-1,3-PROPANEDIOL (NPD) PROCESS

In the process for the preparation of 2-nitro-1,3-propanediol, the product must be extracted from an aqueous solution. As a means of finding the optimum solvent for the extraction step, the distribution coefficients of NPD between several solvents and water were determined. The solvents investigated were ethyl ether, butyl acetate, nitromethane, ethyl acetate, 1-butanol at 25°C., and 1-butanol at 50°C. The coefficients expressed as $\frac{\text{concentration in solvent layer}}{\text{concentration in water layer}}$ for a 0.55 molar solution were 0.058, 0.16, 0.20, 0.41, 0.66, and 0.74, respectively. Thus butanol seems to be the solvent of choice. Furthermore, the evaporation of the butanol solution removes dissolved water as the azeotrope. The dry product can be recrystallized from butanol, water, or ethyl ether. The best quality of product is obtained when ethyl ether is used as the recrystallization solvent.

As the extraction coefficient is not very favorable, the extraction is carried out best by the use of a countercurrent extraction column. Column data are given in the Appendix A.

A preliminary cost estimate for the production of NPD which includes only material, energy, and labor costs amounts to \$1.10 per pound. This is based on small-scale production, 200 lb. per day. On a 2000 lb. per day production, these costs might drop to \$0.76 per pound. See Appendix B.

C. THE LIQUID PHASE NITRATION OF CYCLOHEXENE

In a study of high pressure reactions of nitric acid, cyclohexene was reacted with 10, 20, 30, and 67% nitric acid at 1000 lb. per square inch pressure, and temperatures from 60 to 130°C. Also, cyclohexene was reacted with 20% nitric acid in acetic anhydride at 1000 psi and 60°C. The mole ratio of nitric acid to cyclohexene was 4-1, and the contact time in the heated zone was 20 min. With 10% acid below 100°C. there was no reaction and above 30°C with 67% acid complete oxidation predominated. In all intermediate cases oil was formed, solid in some.

At 70°C., 30% nitric acid, conditions were best for the formation of solids, being 8% by weight of the reacted cyclohexene. Oil was formed in 33% by weight at the same time. With 20% nitric acid in acetic anhydride at 60°C., 48 g. of oil were obtained from 47 g. of cyclohexene fed.

In a series of studies of dinitrogen tetroxide and olefins, Baldock, Levy and Scaife¹ made a rather complete study of the reaction products when dinitrogen tetroxide and cyclohexene were reacted with and without solvent. A solid was

1. Baldock, Levy, and Scaife, J. Chem. Soc., 152, 2627 (1949).

formed in 3% yield which had been known by Baeyer² as the cyclohexene nitrosite (m.p. 150°); by Wieland and Blumlick³ as the nitrosite (m.p. 145°); and by Bloomfield and Jeffrey⁴ as the cyclohexene pseudo-nitrosite (m.p. 153°). Wieland suggested that the solid material was a dimer and the work of Bloomfield and Jeffrey proves the point. Bloomfield, working with nitric oxide in solvents, obtained the pseudo-nitrosite, nitrocyclohexenes, and unstable viscous oil. Baldock was able to separate the oil into 2-nitrocyclohexyl-nitrate, 1,2-dinitrocyclohexane, and 2-nitrohexylanol. The latter arising from the hydrolysis of 2-nitrocyclohexyl-nitrite.

The solid obtained from the liquid phase nitration of cyclohexene in our work has a decomposition point of 150-151°C. after recrystallization from glacial acetic acid. After recrystallization from ethyl acetate, the melting point (dec.) was 149-150°C.

Analysis required for nitronitrosohexane dimer, $(C_6H_{10}N_2O_3)_2$: C, 45.6; H, 6.3; N, 17.6.

Found: C, 45.1; H, 6.3; N, 17.3.

A fractionation curve (Appendix C) of the oil obtained from cyclohexene and aqueous 20% nitric acid at 100°C. indicates a complex mixture. A cut at 35-45°C. at a pressure less than 1 mm. had the analysis: C, 55.0; H, 7.1; N, 10.7. Required for isomeric nitrocyclohexenes, $C_6H_9NO_2$: C, 56.7; H, 7.1; N, 11.0. A sample of the oil was oxidized with alkaline permanganate and a solid material obtained. After the solid was recrystallized from nitric acid, the melting point, 151-152°C. (recorded for adipic acid, 151-153°C.), and solubility indicate adipic acid. This confirms the suggestion that some of the light oil was 1-nitrocyclohexene.

One cut of the oil at 48-50° (less than 1 mm.) gave the analysis: C, 59.1; H, 7.8; N, 9.5, which corresponds to $C_6H_9.4N_0.8O_2.6$. Another cut at 100-101°C. (less than 1 mm) analyzed: C, 48.0; H, 6.3; N, 10.2, corresponding to $C_6H_9.3N_1.1O_3.3$. Obviously these oils were not pure compounds.

D. 1,1-DINITROETHANE

1. Discussion

The Ter Meer reaction was run to obtain 1,1-dinitroethane from 1-chloro-1-nitroethane as described by Jacobson⁵ with modification of the acidification step. The acidification of the sodium salt of 1,1-dinitroethane was carried out at 0°C. with hydroxylammonium chloride and the pH brought to 3 with hydrochloric

-
2. Baeyer, Annalen, 278, 110 (1894).
 3. Wieland and Blumlick, Annalen, 424, 77 (1921).
 4. Bloomfield and Jeffrey, J. Chem. Soc., 147, 120 (1944).
 5. Jacobson, H. W., Ph.D. Thesis, Purdue Univ., 1942.

acid. A yield of 54% of distilled 1,1-dinitroethane was obtained. The yield reported by Jacobson was 51%.

2. Experimental

To 55 g. (0.50 mole) of 1-chloro-1-nitroethane dissolved in 100 ml. of methanol at 0°C. was added a solution of 50 g. (0.50 mole) of potassium nitrite in 80 ml. of water. To this vigorously stirred solution at 0°C., 33 g. (0.50 mole) of potassium hydroxide in 100 ml. of methanol were added dropwise. A yellow-green precipitate appeared and was filtered off after one-half hour additional stirring. The precipitate was returned to the flask and the ice bath and slurred with water. The slurry was acidified while stirring with 70 g. (1.0 mole) of hydroxylammonium chloride in 20% solution, then the pH was brought to 3 with 6 normal hydrochloric acid. After extraction with ether and drying, the oil was distilled at 66° (6 mm.) to obtain 28.7 g. (0.27 mole, 54%) of colorless 1,1-dinitroethane.

E. 1,1-DINITROPROPANE

1. Discussion

The preparation of 1,1-dinitropropane was carried out as described by Aerojet⁶ where the 1,1-dinitropropane was removed from the silver by forming the sodium salt of the former and filtering off the silver. On acidification and extraction of the filtrate with ether, a 97% yield of crude, slightly yellow oil was obtained. Steam distillation of this crude material produced an 89% yield of dry, practically colorless 1,1-dinitropropane.

The preparation was repeated and the product isolated by steam distillation of the total reaction mixture. By this modification, only 60% of undistilled dry 1,1-dinitropropane was obtained.

2. Experimental

To 250 ml. of water the following were dissolved in order: 22.5 g. (0.55 mole) of sodium hydroxide, 45 g. (0.50 mole) of 1-nitropropane, and 37.5 g. (0.55 mole) of sodium nitrite. This solution was then added rapidly with stirring to 170 g. (1.0 mole) of silver nitrate dissolved in 500 ml. of water with 150 g. of ice present.

After stirring for one-half hour, 38 g. (0.5 mole) of 48% sodium hydroxide were added slowly. After filtration, the filtrate was cooled to 0° and acidified to a pH of 3 with 20% sulfuric acid, 35 g. of hydroxylammonium chloride being present. The oil that separated was removed, then combined with two ether extracts of the aqueous layer. The 58.6 g. (0.48 mole, 97%) of oil that remained after removal of the ether was steam distilled to obtain 53.5 g. (0.445 mole, 89%) of dry, undistilled 1,1-dinitropropane.

6. Aerojet, 590, 55 (1952).

F. 2,2-DINITROPROPANE1. Discussion

The 2,2-dinitropropane was prepared by the Shechter-Kaplan reaction so that some comparison could be made of that procedure with the liquid phase nitration of propane and 2-nitropropane. A simple method of isolation of the product was found to be steam distillation of the reaction mixture before filtering off the silver. Good quality 2,2-dinitropropane was obtained in 97% yield even when production grade reagents and recovered silver were used.

A preliminary cost estimate for the materials used and labor for small-quantity production, 100 lb. per day, indicates that this process might be favored over the pressure nitration of 2-nitropropane, but not in large quantities over the pressure nitration of propane. The cost of material and labor is \$1.65 per pound (Appendix D) based on 95% yield and 1.0% loss of silver nitrate.

2. Experimental

After dissolving 45 g. (0.50 mole) of 2-nitropropane in a solution of 41.5 g. (0.55 mole) of 48% caustic in 200 ml. of water at 30°C. or less, 37.5 g. (0.55 mole) of sodium nitrite were dissolved. The solution was then cooled to tap water temperature. After the pH of 1.0 mole of recovered silver nitrate in 500 ml. had been adjusted to about 5, the prepared solution of 2-nitropropane was added rapidly with vigorous stirring. About 150 g. of ice were added to keep the temperature during the addition below 25°C.

After stirring for one-half hour, the pH was raised to 9 with sodium hydroxide and the mixture steam distilled. The distillate was filtered and the white precipitate was warmed above 50°C. to separate the water. There were obtained 58.2 g. (0.485 mole, 97%) of solid 2,2-dinitropropane.

G. 2,2-DINITROBUTANE1. Discussion

The preparation of 2,2-dinitrobutane was carried out by the Shechter-Kaplan reaction in the usual manner. The product was isolated by steam distillation of the reaction mixture before filtering off the precipitated silver metal. On a 0.50 mole run, an 80% yield of dried 2,2-dinitrobutane was obtained. The reaction was repeated on a 4.0 mole basis and no difficulties were encountered in the procedure, obtaining an 86% yield of good colored, undistilled, dry 2,2-dinitrobutane.

2. Experimental

Using 412 g. (4.0 moles) of 2-nitrobutane, 180 g. (4.4 moles) of sodium hydroxide and 300 g. (4.4 moles) of sodium nitrite in 1500 ml. of water, the reaction was carried out in the same manner as was the preparation of 2,2-dinitropropane. The addition to 8 moles of silver nitrate, pH about 6, was carried out in a 12-liter flask from which the product was later steam distilled to obtain 512 g. (3.46 mole, 86.6%) of dried 2,2-dinitrobutane.

H. 1,1-DINITROBUTANE1. Discussion

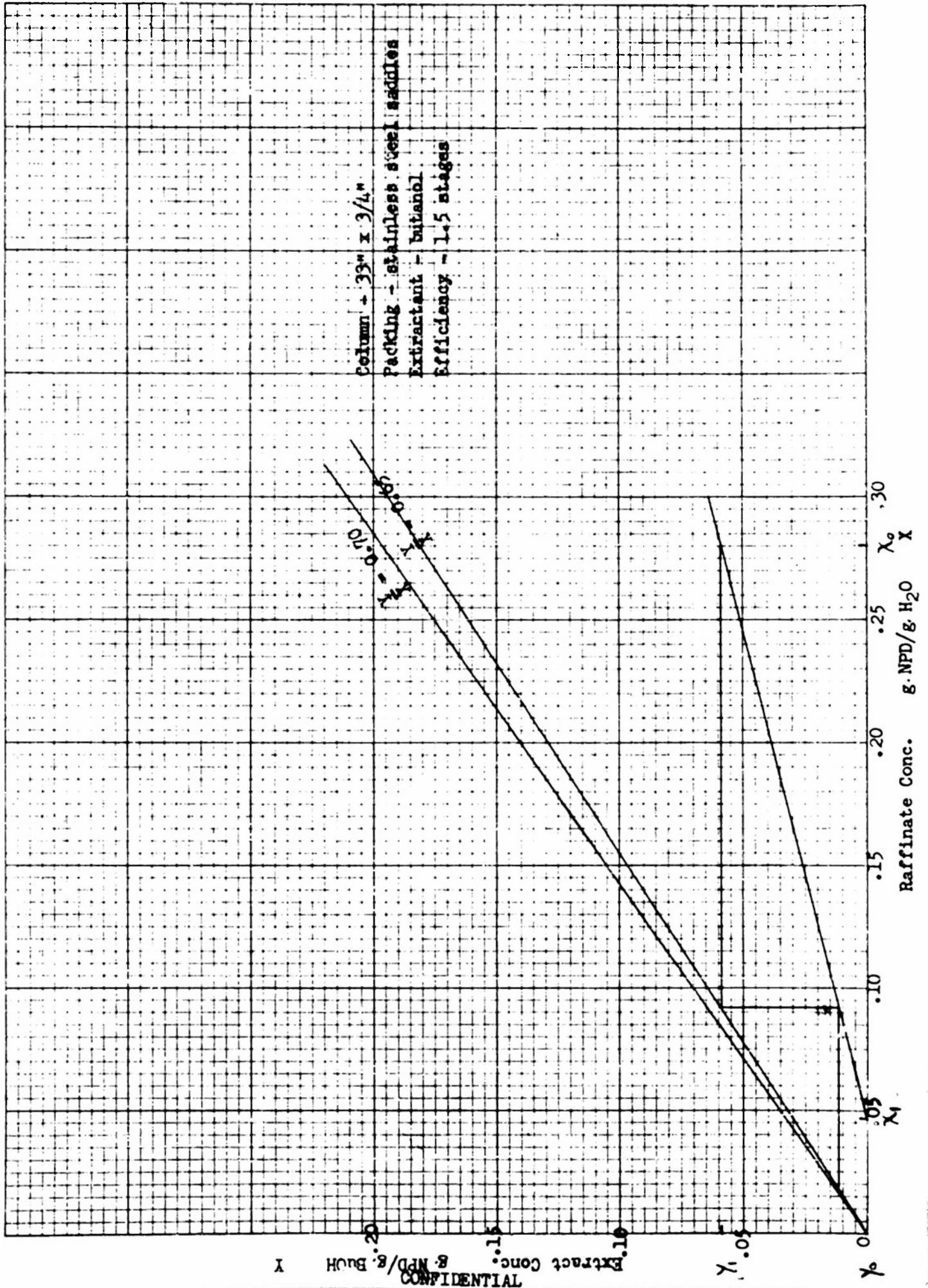
The preparation of 1,1-dinitrobutane was achieved through the Shechter-Kaplan reaction. On a 0.5 mole run, no trouble was encountered in the steam distillation before the silver had been filtered and a 90% yield was obtained. When the reaction was carried out on a 4.0 mole basis, there was much difficulty with "foaming" silver metal during the steam distillation. On subsequent runs the 1,1-dinitrobutane was dissolved as the sodium salt, the silver filtered off, and the 1,1-dinitrobutane regenerated with dilute sulfuric acid. In this manner a yield of 79% of undistilled 1,1-dinitrobutane was obtained.

2. Experimental

There were dissolved in 1500 ml. of water 180 g. (4.4 moles) of sodium hydroxide, 412 g. (4.0 moles) of 1-nitrobutane, and 300 g. (4.4 moles) of sodium nitrite, and the solution was cooled to tap water temperature. To 8.0 moles of recovered silver nitrate in 3-1/2 liters, pH about 6, was added 1 kg. of ice followed with vigorous agitation by the prepared nitrobutane solution.

After stirring for one hour, 170 g. (4.0 moles) of sodium hydroxide as a 50% solution was added and stirring continued for one-half hour. After filtration, the filtrate was acidified at 10°C. with 20% sulfuric acid. The oil was separated and dried to yield 471 g. (3.18 moles, 79.6%) of 1,1-dinitrobutane.

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APPENDIX B

Preliminary Cost Estimate for 2-Nitro-1,3-propanediol

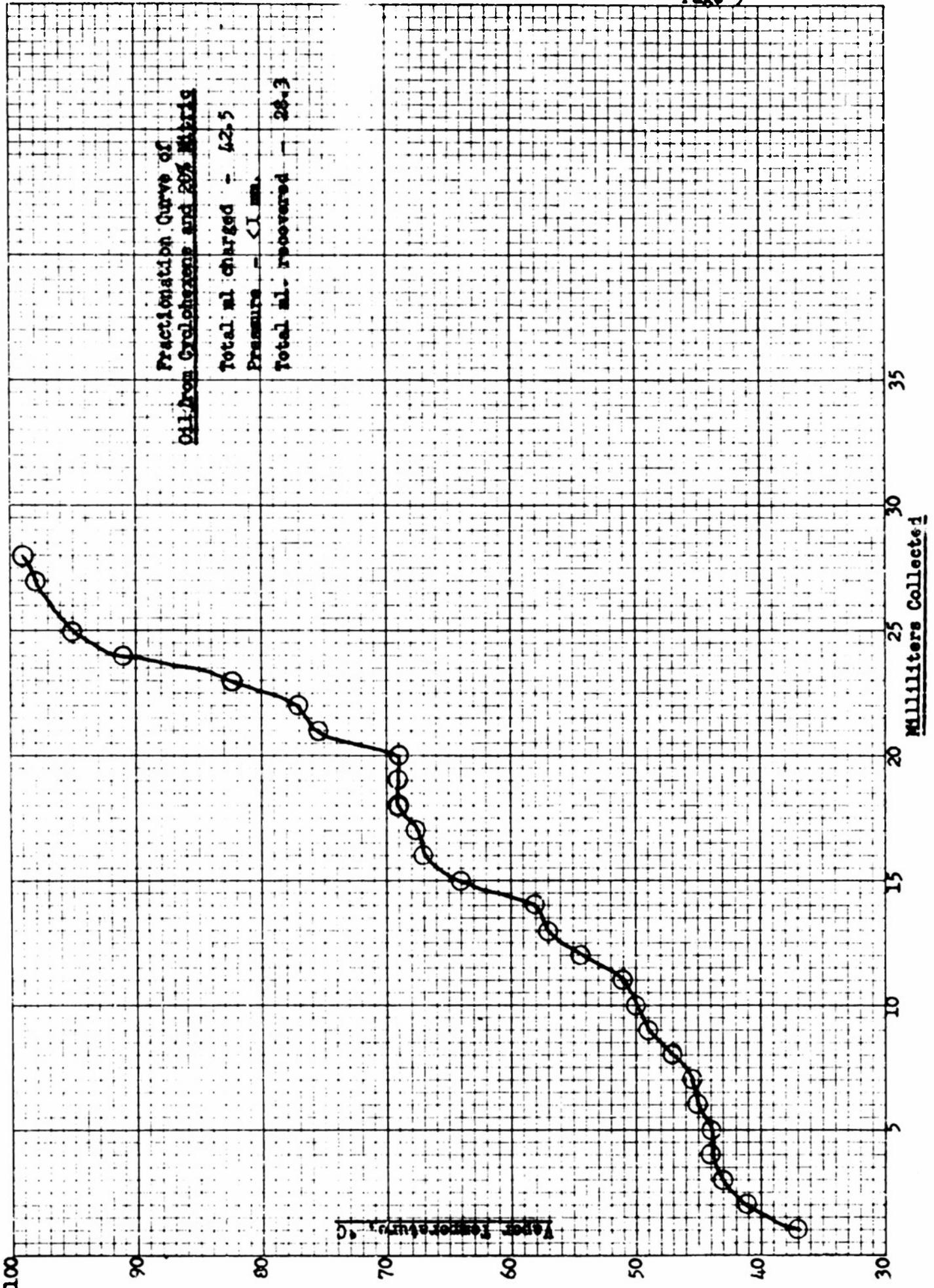
Basis: 200 lb. per 20 hr., 45% yield

<u>Material</u>	<u>Moles</u>	<u>Weight</u> (lb.)	<u>Weight of</u> <u>Solutions</u> (lb.)	<u>Price</u> <u>Per</u> <u>Pound</u>	<u>Total Cost</u>
Formaldehyde (37%)	8	240	650	.07	16.80
Nitromethane	4	244	240	.25	61.00
Sodium hydroxide	4	160	480	.0335	5.36
Sulfuric acid	2	200	1330	.03	6.00
Urea	1	60	60	.06	3.60
Butanol (loss)		150		.15	22.50
Water			<u>1000</u>		
Total Material			3760 lb.		<u>\$115.26</u>

Labor for two operators and supervision at \$4.26/hr. for 20 hr.	85.20
Heating, cooling, and power costs	<u>20.00</u>
Total cost for material, labor, heating, cooling, and power	\$220.46
Cost per pound	\$1.10

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APPENDIX D

Preliminary Cost Estimate for 2,2-Dinitropropane
by the Shechter-Kaplan Reaction

Basis: 95% yield - 114 lb. per 24 hr. - 1.0% loss of silver nitrate

<u>Material</u>	<u>Moles</u>	<u>Weight</u> (lb.)	<u>Weight of</u> <u>Solutions</u> (lb.)	<u>Price</u> <u>Per</u> <u>Pound</u>	<u>Total Cost</u>
2-Nitropropane	1.0	89	89	.25	22.20
Sodium hydroxide	1.1	44	440	.0335	1.47
Sodium nitrite	1.1	76	76	.0875	6.65
Silver nitrate	2.0	340	990		
1% loss	0.02	3.4	_____	9.00	30.60
Total per mole run			1595		
Gallons per mole run			180		
Nitric acid	3.0	189		.05	<u>9.45</u>
Total					\$70.37

Labor for two operators and supervision
at \$4.26/hr. for 24 hr. 102.24

Heating, cooling, and power costs 15.00

Total cost for materials, labor, heating,
cooling, and power \$187.61

Cost per pound \$1.65

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