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SEMI-PILOT SCALE PRODUCTION OF TETRANITROMETHANE

15 MAY 1955



U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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SEMI-PILOT SCALE PRODUCTION OF TETRANITROMETHANE

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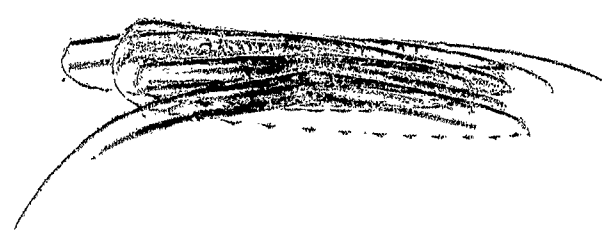
ABSTRACT: Tetranitromethane is at present an essential starting material for new experimental high explosives containing the trinitromethyl group. Several hundred pounds have been prepared by the reaction of 96-98% nitric acid with acetic anhydride on a semi-pilot scale. This somewhat hazardous reaction requires care and many precautions to conduct safely on an enlarged scale. The yields averaged 50% based on nitric acid and 35% based on acetic anhydride. The direct cost was \$2.40 for chemicals and \$9.85 for labor, largely skilled, per pound of 99.9% tetranitromethane.

EXPLOSIVES RESEARCH DEPARTMENT
U.S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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15 May 1955

This report is a description of the semi-pilot scale production of tetranitromethane performed under Task NOL-B2c-19-1-55. The reliability of the work and the validity of the conclusions are the responsibility of the author and the Chemistry Division of the U. S. Naval Ordnance Laboratory.

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SEMI-PILOT SCALE PRODUCTION OF TETRANITROMETHANE

INTRODUCTION

Tetranitromethane (TNM) is an essential raw material for the preparation of new explosive compounds containing the trinitromethyl group. Until February 1953 an adequate supply of TNM was available for the Navy's research and pilot plant new explosive program from the Nitroform Company, East Orange, New Jersey. In February 1953 the Nitroform Company was destroyed by an accidental explosion which killed all of the key personnel. The only other commercial supplier was Johnson and Scudder, Bloomfield, New Jersey, and their production was very limited and they were not interested in supplying TNM in large quantities.

This lack of a commercial source of TNM threatened to seriously hinder the experimental development of new high explosives. The Bureau of Ordnance (Re2c) recognized the immediate need and asked the Chemistry Division of the Explosives Research Department to start making TNM on a scale as large as was feasible with the equipment available. About this same time the Polynitro Chemical Company, College Park, Maryland started producing TNM on a limited scale.

Much work on the preparation of TNM on a laboratory scale has been reported since the beginning of the century. Only two of these reactions reported in the literature seem practical for a safe and convenient laboratory preparation of TNM, the reaction of acetic anhydride with nitric acid (1, 2), and that of acetylene with nitric acid (3,4,5), although both have serious drawbacks for large industrial scale use.

Until World War II TNM was only prepared on a laboratory scale. During the recent war both in Germany and this country TNM was prepared on a large scale. Schimmelschmidt, working at I. G. Farbenindustrie, developed a continuous process in an all glass apparatus for the nitration of acetylene to TNM on a scale of one kilogram per hour based on the work of Orton and McKie (5,6). In this country the Trojan Powder Company prepared a thousand pounds of TNM during the last war for the Air Force using the acetic anhydride-nitric acid procedure (7).

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It has also been known for many years that TNM is a by-product from the manufacture of trinitrotoluene(8), but very little work has been done toward separating and recovering the TNM. The Evans Research and Development Corporation (9) and this Laboratory have made some preliminary investigations of this source. We are planning to extend the work. A study of the original work of Orton and McKie has been made by the Naugatuck Chemical Division, United States Rubber Company (10), Evans Research and Development Corporation (11), and at present work is being done at this Laboratory (12).

Of the two practical methods available for the preparation of TNM in the pilot plant, we chose the acetic anhydride-nitric acid procedure. This was the most convenient to adapt to our equipment and thus start semi-pilot scale production with the minimum delay. Our equipment consisted of four Pfaudler Series "P" ten-gallon stainless steel reactors complete with agitators and fitted with a 3/4" aluminum cooling coil in addition to the full jacket around the kettle. All of the kettles were equipped with a solenoid dump valve as a safety feature. These were controlled from behind a concrete barricade. One kettle was used as a nitrator and the other three as aging kettles. The reactants were loaded into a stainless steel feed tank which was fitted with a delivery line to the nitrator. The acetic anhydride was transferred to the nitrator under nitrogen pressure. The nitric acid (96-98%) was added to the reaction mixture in the same manner but metered through a Brooks Full-View Rotameter.

The temperature was controlled during the addition of nitric acid with circulating chilled water. The flow was arranged so that one could circulate the cooling water through the reactor or by-pass it as the reaction required. The water was chilled to 3-5°C by a three horsepower Freon compressor.

After the nitric acid addition was completed the reaction mixture was transferred by gravity flow to another kettle for the five day aging cycle. The kettles used for the aging cycle were similar to the nitrator, but they were equipped for cooling by circulating tap water only. The temperature during the aging cycle was controlled at 25°C by automatically controlling this water flow to govern the heat of the reaction by two American Instrument Company "Quickset" bimetal thermoregulators mounted in wells and an Emil Greimer Company Electronic Relay

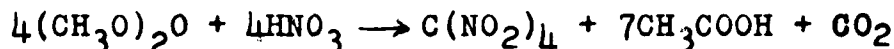
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Model G 24875 which controlled a normally open solenoid water valve. This arrangement was necessary especially for winter-time operation when our tap water got as low as 7°C during the period from November to April. The reaction mixture was drowned in thirteen-gallon glass carboys. Three liter three-neck flasks fitted with a bottom draining stop-cock, stainless steel agitator, and thermometer were used for the purification of the crude TNM. Figure 1 shows a schematic layout of our equipment. Figures 2 and 3 are photographs of our equipment.

EXPERIMENTAL

A. Developmental Experiments

Chattaway has suggested the following equation for the reaction (2):



Various methods have been suggested for carrying out the nitration. These involve differences in the reaction temperature during nitration, the use of a diluent, the concentration of nitric acid used as well as its N_2O_4 content, ratio of reactants, the presence of particles of glass, use of a catalyst, the length of time allowed for completion of the reaction and the temperature during the aging cycle. Numerous experiments were conducted in this Laboratory to determine the effects of some of the above variables on the yield of TNM.

Various concentrations of nitric acid were investigated. In general, it appeared that the more concentrated the acid the higher the yield of TNM based upon nitric acid. It is reported that 70% nitric acid yielded no TNM (13), 90% C.P. grade acid yielded 36% TNM, 95% C.P. grade acid yielded 40% TNM, 96-98% technical acid manufactured by the U. S. Naval Powder Factory, Indian Head, Maryland by the ammonia oxidation process, yielded on an average 50% TNM under comparable conditions. Red fuming nitric acids containing 6% and 20% nitrogen dioxide, were tried. Both mixtures gave about the same yields as obtained with 98% technical acid but the time could be reduced somewhat. White fuming nitric acid, 95%, decolorized with urea gave no TNM.

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The literature claims that the addition of small amounts of phosphorus pentoxide, sulfuryl chloride, and sulfuric acid will show an increased yield of TNM (7). All of these materials were tried but we were unable to substantiate these claims and in addition the reaction was difficult to control. Wyler also claimed that the use of quartz or pieces of chipped glass also increased the yield (7). We did not investigate this claim in our Laboratory. One reaction using a catalytic amount of boron trifluoride was tried which resulted in a "fume-off".

As to ratio of reactants, it was found that nitric acid in a 10% excess or 15% excess acetic anhydride gave equivalent and maximum yields. We chose excess acid for economical reasons. We purchased the acid for \$0.06/pound and 99% acetic anhydride was supplied in drums by Carbide and Carbon Chemicals Company at a cost of \$0.16/pound.

Table 1 gives a summary of our experimental data. No substantial improvement was obtained. However, comparable yields to present process could be obtained in a shorter aging cycle by the use of either red fuming nitric acid or sulfuric acid. The latter increased the hazard from self-heating and was also detrimental to the aluminum cooling coils. The addition of red fuming nitric acid to the 99.3% nitric acid helped. The yields given in the table are for crude TNM.

We have run the reaction by adding the nitric acid to the acetic anhydride and by the reverse procedure. The yields were the same by either order of addition. We feel that it is safer to add the acid to the anhydride rather than adding a potential fuel to a powerful oxidizing agent. If the anhydride is added to the acid, one passes through a composition during the addition of a good Sprengel-type explosive mixture which might be hard to control if the reaction temperature should accidentally become too high. Temperatures up to 40°C for a moderate time were not considered too hazardous during the aging cycle but during the initial mixing cycle such temperatures were usually associated with a rapid rate of increase which was dangerous and difficult to control. The incipient "fume-off" temperature was estimated at about 70°C as a result of a near accident by the Polynitro Chemical Company.

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All of our earlier nitrations were carried out at 10-15°C. We have noted recently that nitration temperatures of 15-20°C increased the yield of TNM. We also found that an aging temperature of 35°C offered some advantage over 25°C but was more hazardous and difficult to control. Figure 4 shows the effect of the aging temperature on the yield.

We have always agitated the reaction mixture during the aging cycle and experienced no difficulties. Aerojet General Corporation reported "fume-offs" during the aging cycle, but they do not agitate the reaction mixture during this period (14).

B. Preferred Pilot Scale Procedure

We modified the procedure reported in Organic Synthesis, which is essentially that of the Chattaway (2,15); and scaled it up to 190 moles for pilot plant production from the 0.5 moles described. A typical run in our pilot plant was as follows:

1. Preparation of TNM

a. Chemicals

Acetic Anhydride, 90%

Nitric Acid, 96-98% (oxides of nitrogen as HNO_2
.3 to .4%)

b. Procedure

The feed tank was charged with 20,700 ml of 99% acetic anhydride and then transferred to the nitrator under 20 psi of nitrogen gas. The acetic anhydride was agitated and chilled to 5°C by circulating chilled water through the coils inside the nitrator and through the jacket. The feed tank was next charged with 9,031 ml of 96-98% nitric acid. The acid was added to the anhydride by metering it through a rotometer at about 75 ml/minute. At this rate the reaction mixture self heated slowly and was controlled at 15-20°C by circulating the chilled water as needed. It usually required two hours to complete the addition.

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After the nitric acid addition, the reaction mixture was exothermic and the temperature was controlled at 25°C manually in the nitrator for several hours as a safety precaution before transferring by gravity flow to an aging kettle which was equipped for cooling by tap water only to control the temperature of the reaction mixture.

Standing instructions for emergency drowning of the nitration mixture called for the temperature to reach 35°C or to pass 30°C at a rate greater than 1° per minute. This had to be done only once.

The reaction mixture was transferred to one of three aging kettles by gravity flow. The temperature was controlled automatically at 25°C and the mixture agitated for a minimum of three days and preferably five days. After aging, the reaction was drowned with water by running both simultaneously into a thirteen-gallon glass carboy. The TNM separated as a layer on the bottom and the aqueous acid layer was siphoned off, diluted with capious quantities of water and discarded.

The crude TNM, 3225 ml, was transferred to a three-gallon glass bottle and washed and agitated with a continuous stream of water until acid free, allowing the wash water to overflow to the drain. When the TNM was acid free it was necessary to remove the water since we observed it to hydrolyze slowly. The batch was divided in half and each portion transferred to a three liter three-neck flask fitted with a thermometer, agitator, and bottom drain stopcock. 1050 Ml of 40% sulfuric acid was added to each flask and agitated for several minutes and then allowed to separate. The TNM was removed through the bottom stopcock and transferred to a similar flask where it was washed with three-five hundred-twenty-five ml portions of concentrated sulfuric acid. Occasional batches were found to evolve considerable heat and turn black if allowed to stand over the sulfuric acid for longer than 10-15 minutes. After the last washing the major portion of the acid was removed and the mixture allowed to stand overnight to insure complete separation. The yield was 3982 grams, a conversion of 43% based on nitric acid, m.p. 14.1°C (corr.), having a minimum purity of 99.9 mole percent.

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The purity of each lot of TNM prepared was checked by freezing point measured as follows: A 25 ml sample of TNM was placed in an unsilvered Dewar jacketed tube, fitted with an agitator and narrow range mercury thermometer which had been calibrated by the National Bureau of Standards, surrounded with an ice bath. The sample was agitated and a freezing point curve plotted. The temperature recorded was that obtained by extrapolation to the time of separation of the first solid material.

PHYSICAL PROPERTIES

The physical properties of TNM were measured on a sample having a purity of 99.996 mole percent. They are as follows (16,17):

Freezing Point	14.136°C
Boiling Point	126°C (760 mm)
Index of Refraction	n_D^{25} 1.4351
Density	1.65
Heat of Fusion	2250.6 $\pm$ cal/mole
Cryoscopic Constant	14.3°C mole solute/kg TNM

EXPLOSIVE PROPERTIES

Pure TNM is a relatively non-explosive liquid. The impact sensitivity on the ERL drop-weight impact machine with 2.50 kg weight is above 320 cm (TNT = 160 cm). Mixtures of TNM with hydrocarbons like toluene have been reported as being explosive and more sensitive to shock than nitroglycerine (18). TNM has been reported to form more sensitive mixtures with aromatic hydrocarbons than with aliphatic hydrocarbons.

We have made a preliminary study of the sensitivity of TNM when mixed with nitromethane and nitrobenzene. The impact sensitivities of these mixtures do not bear out the extreme sensitivity reported in the literature. Mixtures of TNM and nitrobenzene were found to be much more sensitive than mixtures with nitromethane. With both substances the maximum sensitivity occurred at the stoichiometric composition for explosion to CO₂

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and water. We don't feel that this method of measuring sensitivity is a good criterion. Gap-sensitivity measurements would probably be a better method. However, we do not feel that it warrants the effort at this time.

The impact sensitivities of TNM with nitromethane and nitrobenzene in various proportions are listed in Table 2 and Figure 5.

The impact sensitivity of the reaction mixture was measured immediately after the addition of nitric acid, one day later, five days later, and that of the crude product after drossing and separation. All of these materials appeared to be insensitive and gave no explosion on our drop weight machine with a 2.5 kg weight up to 320 cm.

HEALTH, SAFETY AND SANITATION

TNM, itself, is a violent poison (19). The preliminary effects of exposure to TNM vapors are characterized by a very penetrating odor and an irritating effect on the membranes of the eyes and nose. In the interest of health and safety, exposure to fumes of TNM above 5 ppm should be carefully avoided. Suitable gas masks should be used when it becomes necessary to work near any fumes. During our drossing operations concentrations of 10-15 ppm were encountered even with good ventilation (20). We have found the Mine Safety Appliance Co. all-service gas mask fitted with an all-service cannister model "S" very satisfactory. We tested the cannister by preparing a solution containing 53 ppm TNM and pulling the vapors from the TNM through the cannister for thirty minutes. At the end of this period 0.01 ppm TNM was detected in the filtered air, and this was deemed suitable protection for our needs. A detailed report on the toxic effects of TNM has been published by the U. S. Public Health Service (19).

TNM can be produced by the acetic anhydride-nitric acid procedure in pilot plant equipment safely. With a five day total reaction cycle with a 10 gallon nitrator and three aging kettles we could produce seventy pounds per month minimum and using a three day cycle a maximum of about one hundred-sixty pounds per month under ideal conditions. TNM can be produced on this scale at a labor and material cost of \$12.25/pound of which \$2.40 are for materials. This is not a cheap chemical,

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but it is a reasonable price for a research intermediate. On a larger scale it would be cheaper, but for production purposes the nitration of acetylene or recovery of TNM as a by-product from the manufacture of TNT would probably be more feasible. Both of these routes are under investigation at the present time.

ACKNOWLEDGMENTS

The helpful suggestions of Drs. D. V. Sickman and O. H. Johnson in the layout and design of the pilot plant are acknowledged. B. O. Wilkerson assisted in the production operations. Dr. O. H. Johnson and M. E. Hill performed a portion of the developmental work.

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TABLE 1
Summary of Developmental Experiments

Process	%HNO ₃	Moles HNO ₃	Moles AC ₂ O	Percent Yield 24 hrs.	Percent Yield 48 hrs.	Percent Yield 72 hrs.	Grams Crude TNM/ Liter of Reactants
Present	99.3	1.0	1.0	17	31	40	143/a
	95	1.0	1.19	-	-	-	122/a
	90	1.0	1.39	-	-	-	99/a
Red Fuming HNO ₃	20% NO ₂	1.0	1.34	34	44	44	155
HNO ₃ + Urea	95	1.0	1.0	-	-	-	None
HNO ₃ + Red Fuming HNO ₃	6% NO ₂	1.0	1.0	-	42	-	166
0.03m H ₂ SO ₄	99.3	1.0	0.89	-	42	42	160/b
0.03m BF ₃	99.3	0.1(est)	1.0	"Fume off" before addition of HNO ₃ -BF ₃ was completed			
0.06m Cu(NO ₃) ₂	99.3	1.0	1.0	13	22	-	-
0.06m Hg(NO ₃) ₂	99.3	1.0	1.0	13	22	-	-
0.25m P ₂ O ₅	99.3	1.0	1.0	51	35	-	113
0.06m SOCl ₂	99.3	1.2	1.0	-	43	-	150
0.2m H ₂ SO ₄	95	1.0	1.0	---	"Fume off" ---	-	157/c
0.03m H ₂ SO ₄	99.3	1.0	1.0	39	39	-	132/d
0.03m H ₂ SO ₄	99.3	1.0	1.0	17	37.2	-	-

/a Purified yields, all yield data based on HNO₃. /b 0.03m H₂SO₄ added after 4 hrs.
/c H₂SO₄ present initially. /d H₂SO₄ added after 24 hrs.

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TABLE 2
Impact Sensitivity of TNM Mixed With
Combustible Material

Diluent	Wt.% TNM	Impact Sensitivity (2.5 kg wt)	Oxygen Balance
Nitromethane	44.6	109 cm	CO ₂
"	21.2	196 cm	CO
"	65	180 cm	> CO ₂
"	85	210 cm	> CO ₂
Nitrobenzene	42.6	264 cm	< CO
"	61.7	44 cm	CO
"	76.3	22 cm	CO ₂
"	82.8	47 cm	> CO ₂
2,4-dinitrobutulene	95	300 cm	
Reference Sensitivities	{ Nitroglycerin 4-5 cm RDX 22 cm TNT 160 cm Pure TNM > 320 cm		

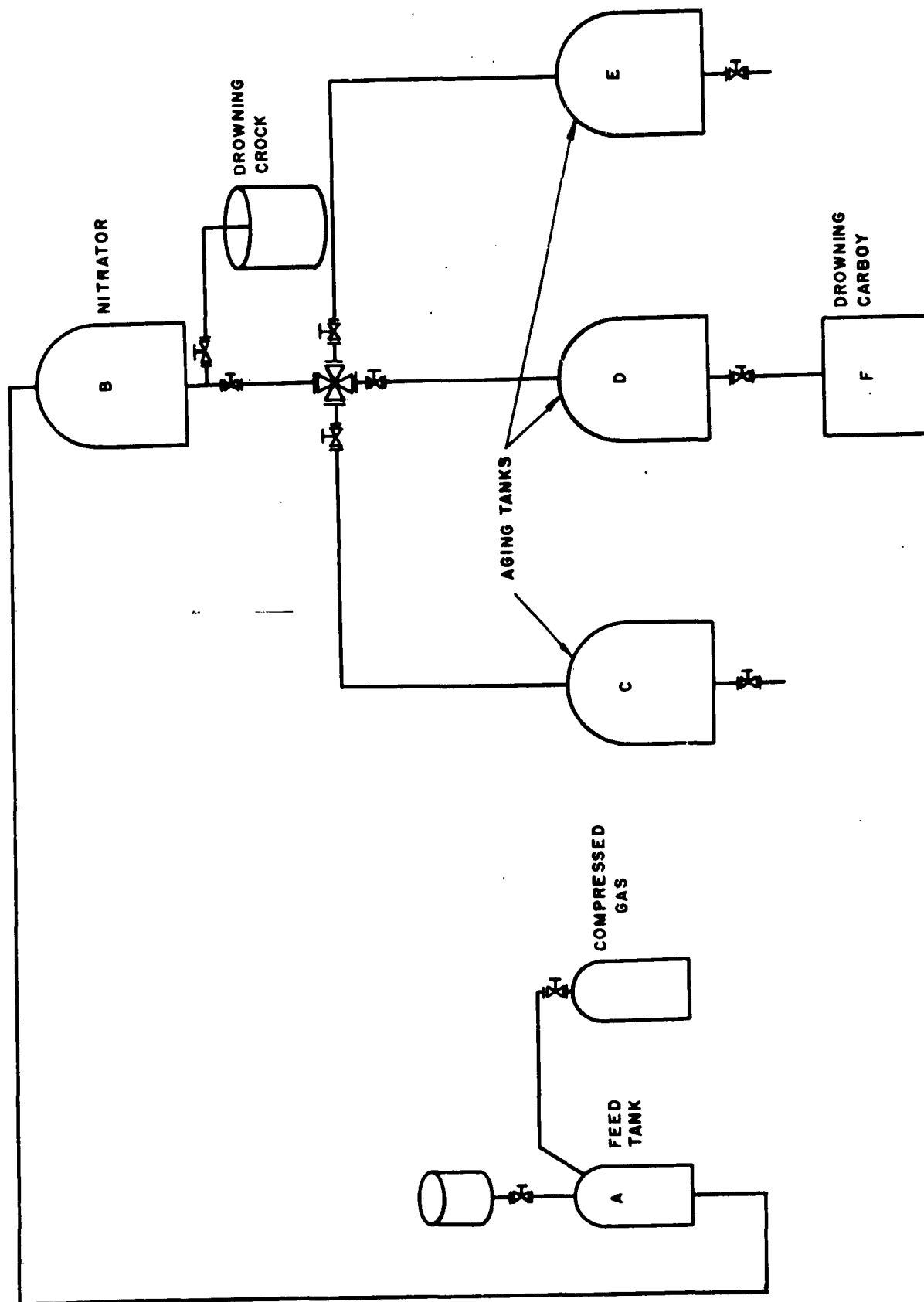


FIG.1 SCHEMATIC OF TNM PILOT PLANT

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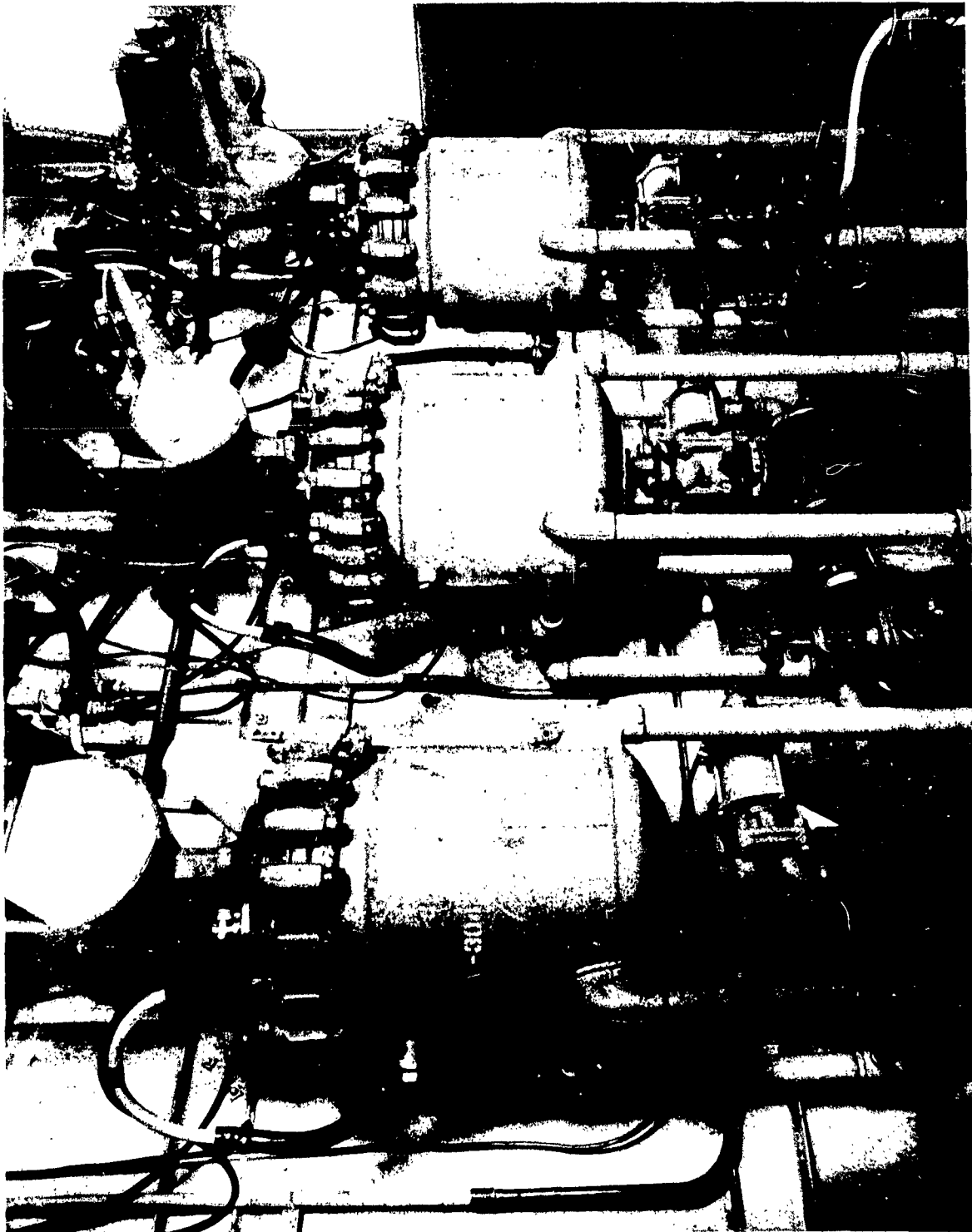
FIG. 2 NITRATOR



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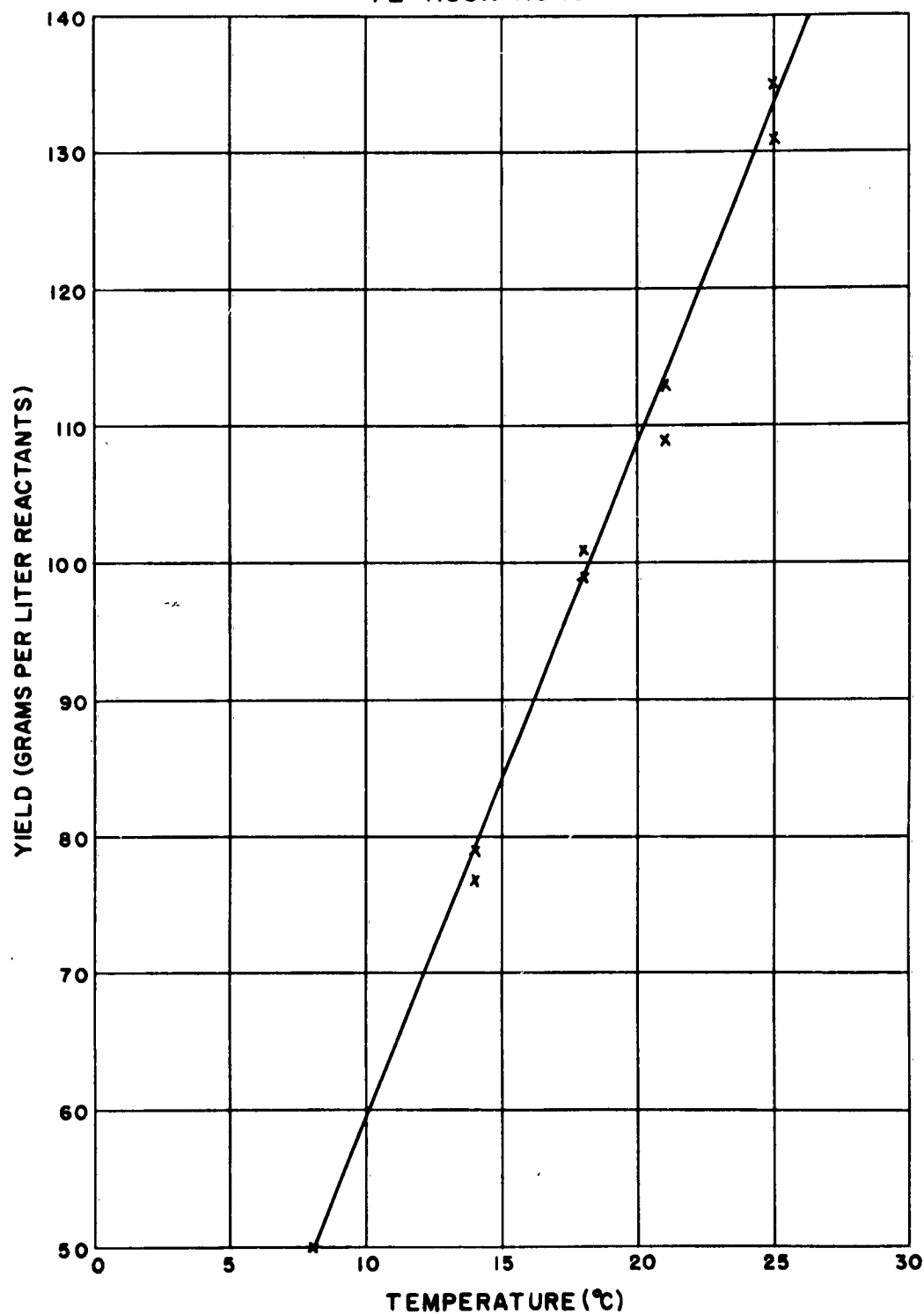
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FIG. 3 AGING KETTLES



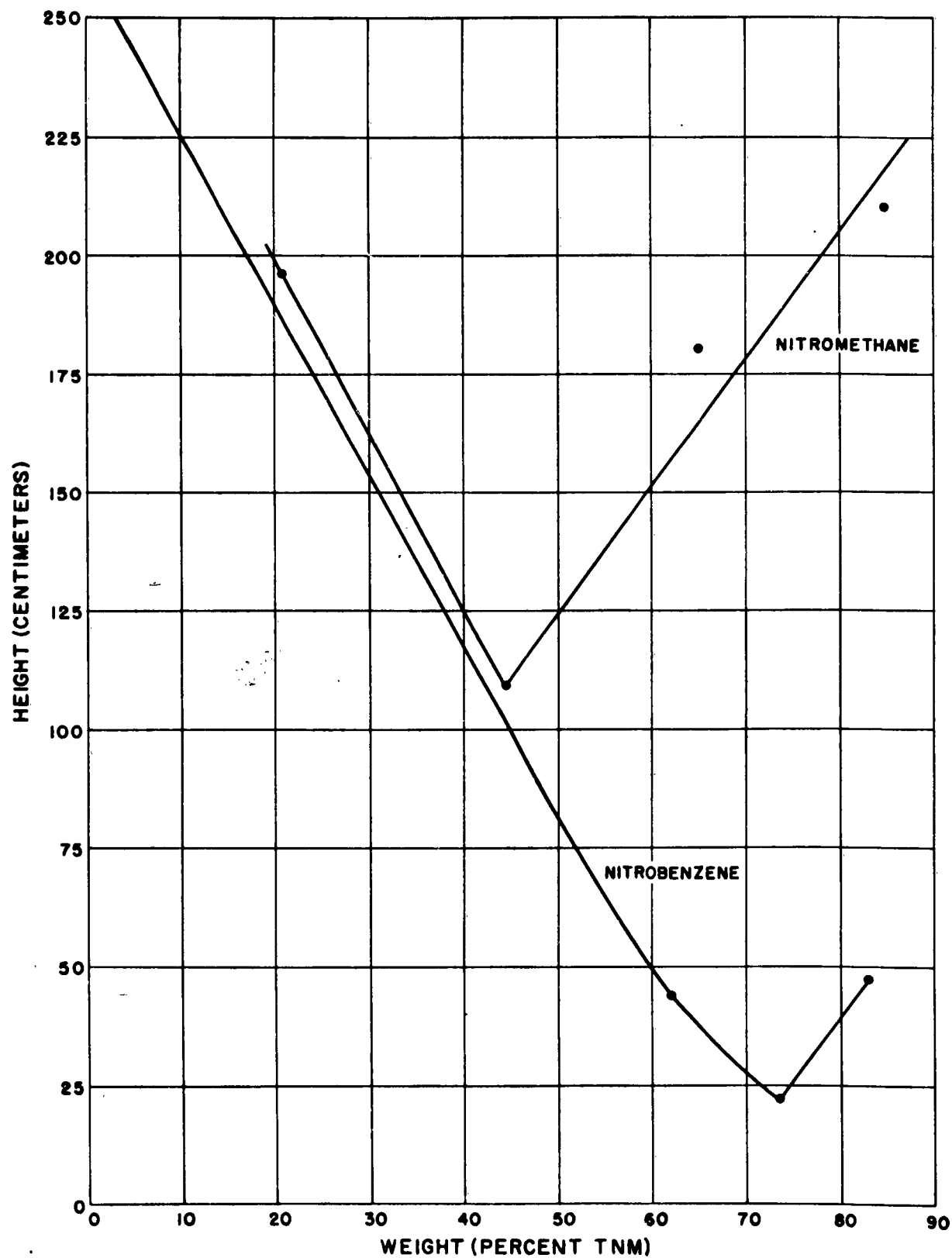
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FIG. 4 EFFECT OF TEMPERATURE ON YIELD
OF TNM
72-HOUR RUNS



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FIG.5 IMPACT SENSITIVITY OF TNM MIXED WITH HYDROCARBONS



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