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BULK SYNTHESIS OF FLUOROEXPLOSIVES (U)

Dale A. Warner
Peninsular ChemResearch, Inc.

TECHNICAL REPORT AFATL-TR-67-154

OCTOBER 1967

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BULK SYNTHESIS OF FLUOROEXP.

Dale A. Warner

In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Armament Laboratory (ATWT), Eglin AFB, Florida 32542.

FOREWORD

This technical report was prepared under Air Force Contract AF 08(635)-4827, "Bulk Synthesis of Fluoroexplosives."

The work was administered under the direction of Air Force Armament Laboratory (AFATL), Air Force Systems Command, Eglin Air Force Base, Florida, 32542. Carl Kyselka (ATWT) was program monitor for the Air Force.

The research program was conducted by Peninsular ChemResearch, Inc., Gainesville, Florida under the technical supervision of W. Lamar Miller and Dale A. Warner.

This document, except the title and abstract, is classified "CONFIDENTIAL" in its entirety in accordance with AFR-205-1, Paragraph 11b, because of the nature and potential military application of the research and data described herein.

This report covers work conducted from 19 January 1965 through 31 August 1967.

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This technical report has been reviewed and is approved.

Noble E. Brown
for GEORGE P. BRENNER, Colonel, USAF
Chief, Weapons Division

UNCLASSIFIED ABSTRACT

The synthesis of twenty pounds of each of three and forty-five pounds of a fourth fluoroexplosive has been carried out following general procedures supplied in R & D Exhibit No. ATW 64-84. Modifications to some of these procedures were made to improve yields of products or handling procedures at the scale being run. Laboratory equipment used in this work is described.

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Armament Laboratory (ATWT), Eglin Air Force Base, Florida 32542.

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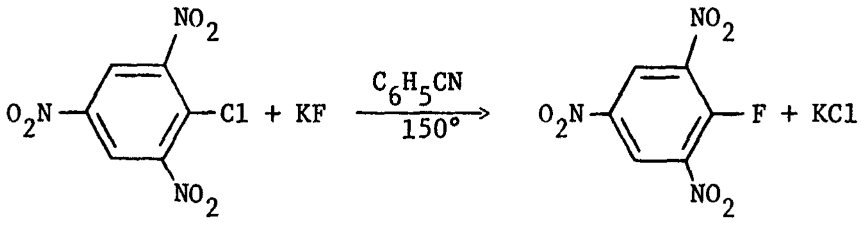
SECTION I

DISCUSSION

A. Picryl Fluoride (PF)



(PF)

Previously published procedures[1] for the replacement of the chlorine atom in picryl chloride to give PF have specified addition of anhydrous potassium fluoride (KF) to a heated solution of picryl chloride. Strong evolution of heat and nitric fumes, and the formation of a dark gummy precipitate on this addition gave strong evidence for the pick-up of destructive amounts of water by the KF on even very short exposure to local air. No refinement of drying and handling procedures for batch operation could prevent water absorption by the KF.

The problem was overcome by reversing the order of addition of reactants to the benzonitrile solvent. Potassium fluoride which had been dried at 150°C for three days was placed in the reaction vessel and covered with one-half the required quantity of benzonitrile along with about 200 ml. of benzene. The benzene-water azeotrope was distilled and water separated until dryness of the stirred slurry was assured.

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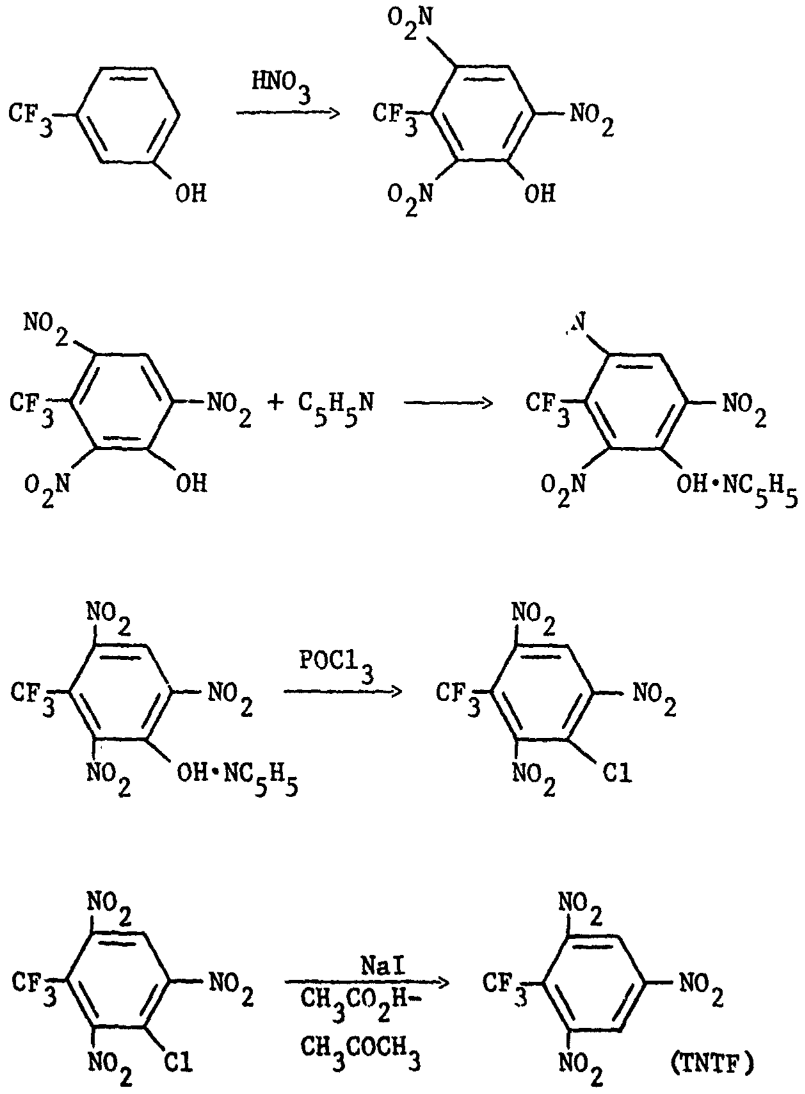
(1) Technical Documentary Report ATL-TDR-64-19, Contract AF 08(635)-2965, E. I. duPont de Nemours & Co., April, 1964.

Remaining benzene was distilled off to a vapor temperature of 140°C. Then a solution of picryl chloride in the remaining quantity of benzonitrile was added dropwise from a closed circuit dropping funnel. In contrast to the "normal" mode of addition, it was found necessary to supply heat to the mixture to maintain the desired temperature. Yields ranged from 27-75%, as compared with 50% reported by DuPont.

B. 2,4,6-Trinitrobenzotrifluoride (TNTF)



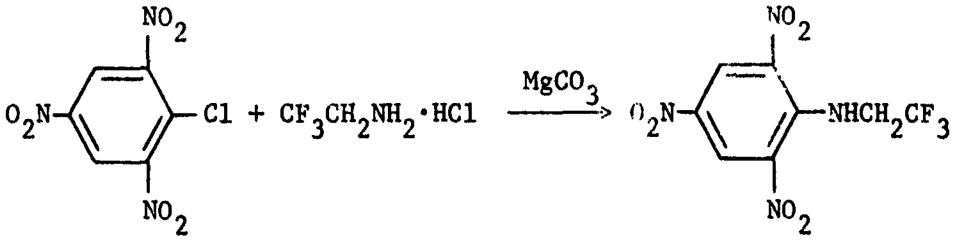
The successful production of TNTF required two modifications in the purification procedure described by DuPont. The reference procedure called for taking up the crude TNTF, which separates as an oil on pouring the glacial acetic acid-acetone solution into aqueous sodium sulfite, in hot trichloroethylene and cooling to obtain a solid material. On the other hand, subjecting the separated oil to a sharp spray of water - as from a common shower head - leached out sufficient acetic acid to permit the crude TNTF to solidify without further treatment.

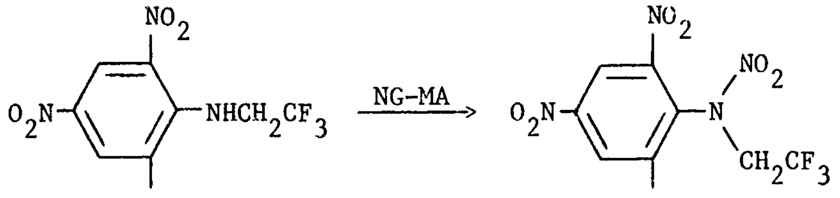
The major modification was elimination of the inefficient and time-consuming column chromatography over neutral alumina to remove colored impurities from the TNTF. On one occasion a 10-lb. batch of TNTF was lost on a column from which it could not be eluted.

A single recrystallization of the crude, water-washed product from benzene removed a gum. The crystals were then exhaustively extracted with boiling heptane. At the boiling point of heptane the crude product was liquid, which would make this process suitable for continuous operation in a counter-current liquid-liquid extractor. The residue from heptane extractions was a highly colored mixture, which was completely removed only by two or more passages through the alumina column. Cooling the heptane gave pale yellow crystals of TNTF, which was up-graded to >98% purity by one recrystallization from benzene.

The other procedural points in this four-step synthesis were followed with little or no deviation from reported procedures.

C. β,β,β-Trifluoroethyl Tetryl (TFET)

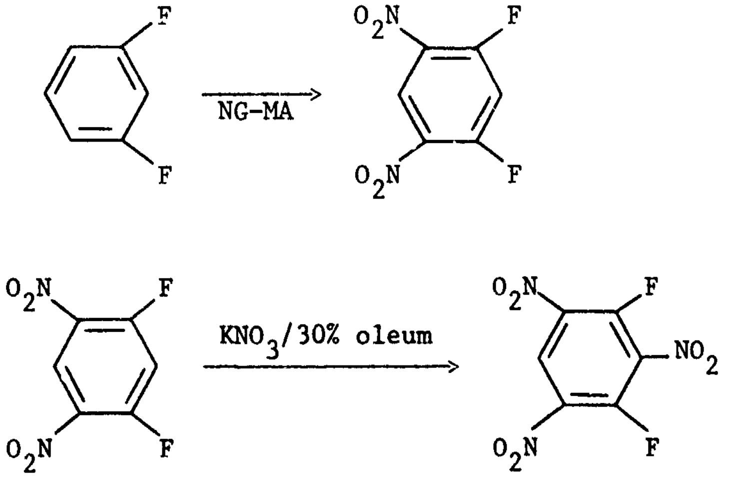




(TFET)

The synthesis of TFET was carried out as already described[1] with no major problem.

D. 1,3-Difluoro-2,4,6-trinitrobenzene (DFTNB)



(DFTNB)

The first of these two steps was carried out as described by DuPont, giving yields in excess of 90%. The second step presented extraordinary difficulties, as a consequence of the extremely facile hydrolysis of DFTNB. The crude or purified product was found to hydrolyze in vacuo at 25° during efforts to dry it; in solution in benzene, trichloroethylene, acetone, carbon tetrachloride, or methylene chloride above 30° during attempts to recrystallize it; in contact with liquid water - especially in the presence of mineral acid or metal salts - during the air-drying process; or in contact with ice while filtering the quenched reaction mixture. In addition, conversion and yield in the reaction was decreased by stirring the reaction mixture.

Ultimately these difficulties were overcome by attention to some procedural points not stressed by DuPont.

1. During the nitration reaction, agitation was stopped when the desired temperature was reached: about 20 hours. The reaction period was extended 60 hours beyond this point.

2. Utmost speed in pouring the reaction mixture over ice, filtering, and washing free of all traces of acid. Runs wherein this sequence required longer than 15-20 minutes suffered severe yield loss.

3. Immediate and quantitative removal of water from the crude product either by slurrying in carbon tetrachloride and decanting water from the top, or dissolving the wet solids in a sufficiently large amount of methylene chloride at 25° and decanting the water from the top.

4. Strict avoidance of contact with trichloroethylene, which caused darkening of the product; or polar solvents, which caused hydrolysis.

Before the methylene chloride-carbon tetrachloride system described in the Experimental Section was discovered, several recrystallization solvents were studied with varying negative results. The results are tabulated below.

Recrystallization of Crude DFTNB

Solvent	Cycle No.	Insolubles[(a)]	Crystals[(b)]	Remarks
Triclene	1	Filtered off while hot	60-70% DFTNB	Product and liquor green to brown
	2	About 1/3 of DFTNB	85-90% DFTNB	Crystals convert to insolubles while vacuum drying
	3	1/3 to 1/2 of DFTNB	90-95% DFTNB	Still hydrolyzing

Notes: (a) Insolubles are taken to be the hydrolysis product of DFTNB; probably either 3-fluoropicric acid or 2,4,6-trinitroresorcinol.

(b) DFTNB and DFDNB assays based on glc over 15% SE-30 on Gas-Chrom P at 215°, iso thermal. Hydrolysis product is not eluted under any conditions.

Recrystallization of Crude DFTNB
(Continued)

Solvent	Cycle No.	Insolubles[a]	Crystals[b]	Remarks
Benzene	1	Most of DFTNB	Largely DFDNB Small crop	Hydrolysis occurs on heating to dissolve
Benzene/ Triclene	1	--	Quantitative hydrolysis of DFTNB	
Benzene/ CCl_4	1	Easily removed	DFDNB too insoluble; only first 10% of crystals >90% DFTNB. Fire hazard, since CCl_4 causes static sparks. Hydrolysis still occurs on drying.	
CCl_4	1	Quant. removal	Variable 10-80% DFTNB	Large volume of solvent required
Heptane	1	Quant.	Pure DFDNB	DFTNB either soluble or quant. destroyed
Chloro- benzene	1	Formed in all operations	60-65% DFTNB	M.p. 116-132°
	2	Formed in all operations	All hydrolysis product	Hydrolysis evidently complete; DFDNB/DFTNB in liquor >95/<5

Notes: (a) Insolubles are taken to be the hydrolysis product of DFTNB; probably either 3-fluoropicric acid or 2,4,6-trinitroresorcinol.

(b) DFTNB and DFDNB assays based on glc over 15% SE-30 on Gas-Chrom P at 215°, iso thermal. Hydrolysis product is not eluted under any conditions.

SECTION II

EXPERIMENTAL

A. Equipment

1. Five-gallon nitrator

The nitrator was a type 304 stainless steel open kettle of five-gallon working capacity. It was fitted with a jacket through which could be circulated 120° steam, brine, or well water at about 25°C. The agitator was of the flat-paddle type which cleared the inner wall by about 1/4". It was driven by an air motor. Temperature measurement was by a Weston dial thermometer viewed by mirrors from behind a 1/4" steel barricade. A one-inch plug valve in the bottom of the reactor permitted rapid dumping. The nitrator was raised four feet from the floor on an angle iron stand, to permit positioning dump vessels beneath it on a dolly.

2. 22-Liter glass reactor

A 22-liter Morton flask was specially fabricated by Scientific Glass Apparatus Co., Inc., Bloomfield, New Jersey, for this project. The center neck was 100 mm. "O" ring joint to which the air-driven agitator was secured by means of a flanged steel tubular support with a gland-type bearing for the agitator shaft. The side necks were fitted with 30-mm. "O" ring joints; a bottom drain of one-inch I.D. was fitted with a 35/45 ball joint, to which was attached a similarly equipped one-inch bore glass stopcock. Temperature control was accomplished with a remotely located Brown Pyro-Vane controlling pyrometer. The reactor was positioned on an angle iron stand, permitting the location of dump vessels under it on dollies.

3. Dump tank

A 304 stainless steel dump tank, fitted with a 1 1/4" Contromatics 316 stainless ball valve with Teflon seals at the bottom served both the five-gallon and 22-liter reactors. The agitator was driven by a one h.p. electric motor geared to give paddle speeds of 30 rpm., or by a geared-down air motor.

4. Filter

A porous "Teflon" filter mat on a bench type porcelain Buchner funnel served for filtration. Vacuum could be supplied by steam jet or water aspirator.

B. Picryl Fluoride

Four five-liter, three-neck flasks equipped with still heads, closed-circuit dropping funnels, and paddle-type agitators were used simultaneously in this preparation. The procedure is described below for one such set-up.

To the reaction flask were charged 750 ml. of benzonitrile (Matheson, Coleman & Bell), 1100 g. of anhydrous potassium fluoride, and 1000 ml. of benzene or toluene. The mixture was stirred and distilled to a vapor temperature of 150°. Now a hot (60°C) solution of 1000 g. of picryl chloride (Pierce Chemical Company) in 550 ml. of benzonitrile was added via the dropping funnel. It was necessary to supply heat to the flask from the heating mantle to maintain 140°C. When the addition was complete, the temperature was raised to 155° ±5°C and maintained at that point for 1 1/2 to 2 hours. Upon cooling to about 70°C, the mixture was filtered, and the cake extracted with 750 ml. of hot benzene. To the combined filtrates was added 4.0 liters of heptane (Phillips Petroleum), and the crude PF crystals collected by filtration. Evaporation of the filtrate gave an additional quantity of crude PF. Final product clean-up was by recrystallization from benzene-cyclohexane.

The impetus to develop this alternate procedure was supplied by an explosion and fire caused by a runaway large scale reaction in which the KF had been added to the hot picryl chloride. Lumping, fuming, precipitate formation, and a strong exotherm slowed the stirrer and reduced the efficiency of heat removal. No problems were encountered on adding the picryl chloride to the KF.

C. β,β,β-Trifluoroethyl Tetryl

1. 2,4,6-Trinitrophenyl β,β,β-trifluoroethylamine

The 22-liter glass reactor was supplied with a well-water cooled reflux condenser and a dropping funnel. To a slurry of 741 g. of picryl chloride (Pierce Chemical Company) and 282 g. of magnesium carbonate in 9.0 liters of methanol there was added a solution of 503 g. of trifluoroethylamine hydrochloride in 450 ml. of water. Upon completion of the addition the mixture was stirred under reflux for 1-1 1/2 hours, and treated with 4.5 liters of water. The slurry was cooled to near ambient temperature and filtered. The solids were water washed and air dried. The drying process took several days at 28-35°C and 50-65% relative humidity. In common with all other materials produced under this contract, this aromatic nitro compound may retain up to its own weight of water and still exhibit a "dry crystal" appearance. Typical yield, 99%.

2. β,β,β-Trifluoroethyl tetryl

With refrigerated brine in the jacket of the five-gallon steel nitrator, 630 g. of 2,4,6-trinitrophenyl β,β,β-trifluoroethylamine was added in 10-20 minutes to 39 lb. of stirred NG-MA (nitroglycerin mixed acid, which is a commercially available 50:50 mixture of nitric and sulfuric acids) at 5°C. The brine in the jacket was replaced with well water, and the nitration mixture allowed to come to ambient temperature over about one hour. The reaction mixture was poured over about 100 lb. of ice, with agitation. The crystals were filtered, water washed, and air dried. The product was obtained in >98% purity by dissolving in a minimum of acetone at 25°, filtering to remove black particles, and recovering by chilling to -78°C. Typical yield, 94.6%.

D. 2,4,6-Trinitrobenzotrifluoride

1. 3-Hydroxy-2,4,6-trinitrobenzotrifluoride

With brine circulating through the jacket of the five-gallon nitrator, 26.5 lb. of white fuming nitric acid was cooled to 0-5°C. Using a sigmamotor feed pump 2756 g. of 3-hydroxybenzotrifluoride was added in 1 1/2-2 1/2 hours, while keeping the temperature below 10°C. Some frothing and fuming, starting when the addition was about one-third complete, caused no problems.

When the addition of 3-hydroxybenzotrifluoride was complete, the reaction mixture was warmed to 20°C with well water and then to 35° with steam. The addition of a mixture of 1.8 liters of 94% H_2SO_4 and 450 ml. of fuming HNO_3 during 20-30 minutes was permitted to raise the temperature to about 55°. After this addition stirring was continued for 15 minutes before dumping the reaction mixture onto 50 lb. of ice. The resulting slurry stood overnight, the crystals were filtered off, water washed, and air dried. Yields: 4140-4440 g., 81.5%-87.1%.

2. Pyridine salt of 3-hydroxy-2,4,6-trinitrobenzotrifluoride

In a 20-gallon stainless steel open kettle with bottom drain, 11.33 Kg. of 3-hydroxy-2,4,6-trinitrobenzotrifluoride was dissolved in 70 lb. of methanol. To this solution 3.25 Kg. of pyridine was added in portions, with stirring, over 1 1/4 hours. The slurry was then stirred for an additional hour and filtered. After air drying, the product weighed 12.5 Kg. (87% of theory).

3. 3-Chloro-2,4,6-trinitrobenzotrifluoride

The reaction was carried out in a 5-liter three-neck flask. Phosphorus oxychloride (2.31 Kg.) was poured over 1.135 Kg. of solid pyridine salt (from 2. above). As the salt went into solution stirring was begun. The mixture was heated to 75-80°C, and the heat turned off. The temperature rose to 85-90°C; the mixture was stirred at this temperature for 15 minutes and poured slowly into 15 gal. of water.

Another run was started immediately. The pyridine salt (12.5 Kg.) available required eleven runs to react fully. All runs were dumped into the same tank of water, ice being added as necessary to keep the temperature between 30-35°. The crude product was filtered, washed with fresh water, and allowed to stand in the dump tank under water overnight. Upon filtering and air drying for 30 hours, the chloride retained 12% its weight of water. It was stored in this condition until used in the next step, when it was taken up in benzene, dried by decantation, and recovered by evaporation. Approximate overall yield, 9.86 Kg., 90.7%.

4. 2,4,6-Trinitrobenzotrifluoride

For each run the following were prepared:

a) solution of 4.0 Kg 3-chloro-2,4,6-trinitrobenzotrifluoride in 8.0 liters of acetone.

b) solution of 6.0 Kg of sodium iodide in 16 liters of acetone and 1.6 liters of glacial acetic acid.

c) solution of 2.10 Kg of sodium sulfite in 25 gal. water.

Solution b) was placed in a 50-liter three-neck flask equipped with a glass stirrer and "Teflon" paddle. While stirring slowly, solution a) was added in one liter increments allowing about 15 minutes stirring between increments. Stirring was continued overnight, and the reaction mixture was then poured into solution c). The aqueous layer was drawn off and the oily lower layer sprayed with high velocity water until it solidified. The solids were collected on a filter and air dried for two days. Crude yield, 2.65 Kg., 79%.

The combined crude from two runs (5.30 Kg.) was taken up in 6.36 liters of hot benzene and allowed to crystallize overnight. The 3.81 Kg. of first crop material was extracted with equal portions of boiling heptane totaling 24 liters. Cooling the heptane yielded 2.35 Kg. of 95-97% pure TNTF, 300 g. of second crop material, and 600 g. of residue. Crops 1 and 2 were recrystallized from benzene to give 2.4 Kg. of >98% pure material, yield ca. 71%. Additional small quantities of useful product could be recovered from the residue fraction by repeated heptane extraction and benzene recrystallization.

E. 1,3-Difluoro-2,4,6-Trinitrobenzene

1. 1,3-Difluoro-4,6-dinitrobenzene

With well water flowing through the jacket of the five-gallon nitrator, 1.63 Kg. of m-difluorobenzene was added from a glass dropping funnel to 55 lb. of NG-MA at such a rate that the temperature did not exceed 35°. Agitation of the viscous mixture was provided by two air driven paddle stirrers operating at full pressure. When the addition was complete, the cooling water flow was stopped and the water remaining in the jacket served to hold the temperature at 30-35° for an additional 15-minute period.

The mixture was poured, with agitation, onto 50 lb. of ice, and allowed to stand overnight. The crystals were then collected by filtration, washed free of acid with water, air dried, and finally vacuum dried. Typical yield, 90%.

2. 1,3-Difluoro-2,4,6-trinitrobenzene

The 22-liter glass reactor previously described was fitted with interchangeable thermocouple well and acid fill-tube for one side neck, and a 1-meter air condenser protected by a 3-liter "sweep-over" trap for the other side neck. The flask was loaded with approximately 11 liters (40 lb.) of 30% oleum through the 3/8" 316 stainless steel fill-tube. The fill-tube was replaced by the thermocouple well and a large powder funnel placed on the other side neck. While stirring at 150-200 rpm., 1.8 Kg. of KNO_3 was added in portions. The temperature rose to 90-105°C and copious fumes of SO_3 and NO_2 were evolved. Within 15 minutes the addition of 2.0 Kg. of 1,3-difluoro-4,6-dinitrobenzene (DFDNB) was begun. During this addition, which required 10-20 minutes, considerable foaming occurred while the nitro compound was dissolving, but it subsided immediately as each portion dissolved. The temperature dropped as much as 10°C during the addition of virgin DFDNB, and rose 10-15° during addition of recovered material.

Upon completion of the addition, the air condenser was returned to its place, the agitator speed was decreased to 25-30 rpm., and heating begun. Unless SO_3 evolution was appreciable, 175° was reached in 1 1/2-2 hours. Release of large volumes of SO_3 necessitated heating to 125° and holding for 20-30 minutes, then heating to 150° and holding for 20-30 minutes, and finally heating to 175°. After reaction temperature was reached, agitation was continued for 12-24 hours, then discontinued for the remainder of the reaction period. Heating for 60 hours from the time 175° was reached gave optimum yields. At the end of this time, the mixture was allowed to cool to 90-120° and poured onto ice with vigorous stirring. The rate of addition to the ice must be as rapid as possible while still keeping the temperature below 0°C. The success of this operation required simultaneous replenishment of the ice supply, draining the dump liquor into the filter, and regulation of the dump rate from the reaction vessel. Very large amounts of nitric fumes were evolved, requiring efficient ventilation of the area.

The solid on the filter was washed 2-3 times with ice water, as quickly as possible, and dissolved in about two gallons of methylene chloride at 25°C. With a little care, all the residual water could be removed by decantation. The liquid was then filtered through a small Buchner funnel and placed in a 5-gallon container. While stirring, one-half volume of carbon tetrachloride was added slowly. The mixture was then allowed to stand for 20-30 minutes and filtered. This crop was 85-95% pure DFTNB. It was redissolved in methylene chloride and reprecipitated with carbon tetrachloride, giving 98.5+% product. The liquor from the first crystallization was evaporated to dryness, and assayed for DFTNB. If less than 10% was found the solids were renitrated; if more than 10% DFTNB was found the recrystallization was repeated. Solids from other liquors were recovered and inserted into appropriate stages of subsequent runs. Typical yield from 2.0 Kg. DFDNB, 1.9 Kg. total crude, 1.0 Kg. recovered DFDNB, 800 g. 98.5+% DFTNB (68.6% yield based on 50% conversion).

APPENDIX

CHARACTERIZATION OF FLUOROEXPLOSIVES

A. Elemental Analysis

Compounds PF, TFET, and TNTF were analyzed by Galbraith Laboratories, Knoxville, Tennessee; DFTNB was analyzed at Peninsular ChemResearch. Samples were obtained from retained samples of lots sent to AFATL. Representative examples of the analytical results are given below.

	%C		%H		%N		%F	
Compound	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.
PF, high		32.12		2.33		18.36		8.55
low		31.97		2.00		17.88		8.24
avg.	31.18	32.04	0.87	2.14	18.18	18.18	8.22	8.44
TFET								
blended retainer	27.05	27.70	1.14	1.85	19.72	19.37	16.1	17.6
TNTF								
blended retainer	29.90	30.95	0.72	2.38	14.9	14.31	20.3	20.2
DFTNB, high		29.49		0.93		17.96		
low		28.57		0.86		15.93		
avg.	28.93	29.03	0.41	0.89	16.87	16.95	15.26	*

*Peninsular ChemResearch, Inc. does not have facilities for fluorine analysis.

B. Melting Points

The melting points of PF, TFET, and TNTF were determined by the conventional capillary tube method; the melting point of DFTNB was determined using a differential scanning calorimeter programmed at 20°C/min. Samples were obtained from retainers withdrawn from lots shipped to AFATL.

Compound	m.p.	
	Obs.	Reported by DuPont
PF	127-128.5°C	127.5°C
TFET	118-120	115
TNTF	89-90	88
DFTNB	138, dec.*	149

*This low melting point and subsequent strongly exothermic decomposition may have been caused by reaction with the aluminum sample pan.

C. Infrared Spectra

The infrared spectra attached were obtained by mulling retained samples with Nujol.

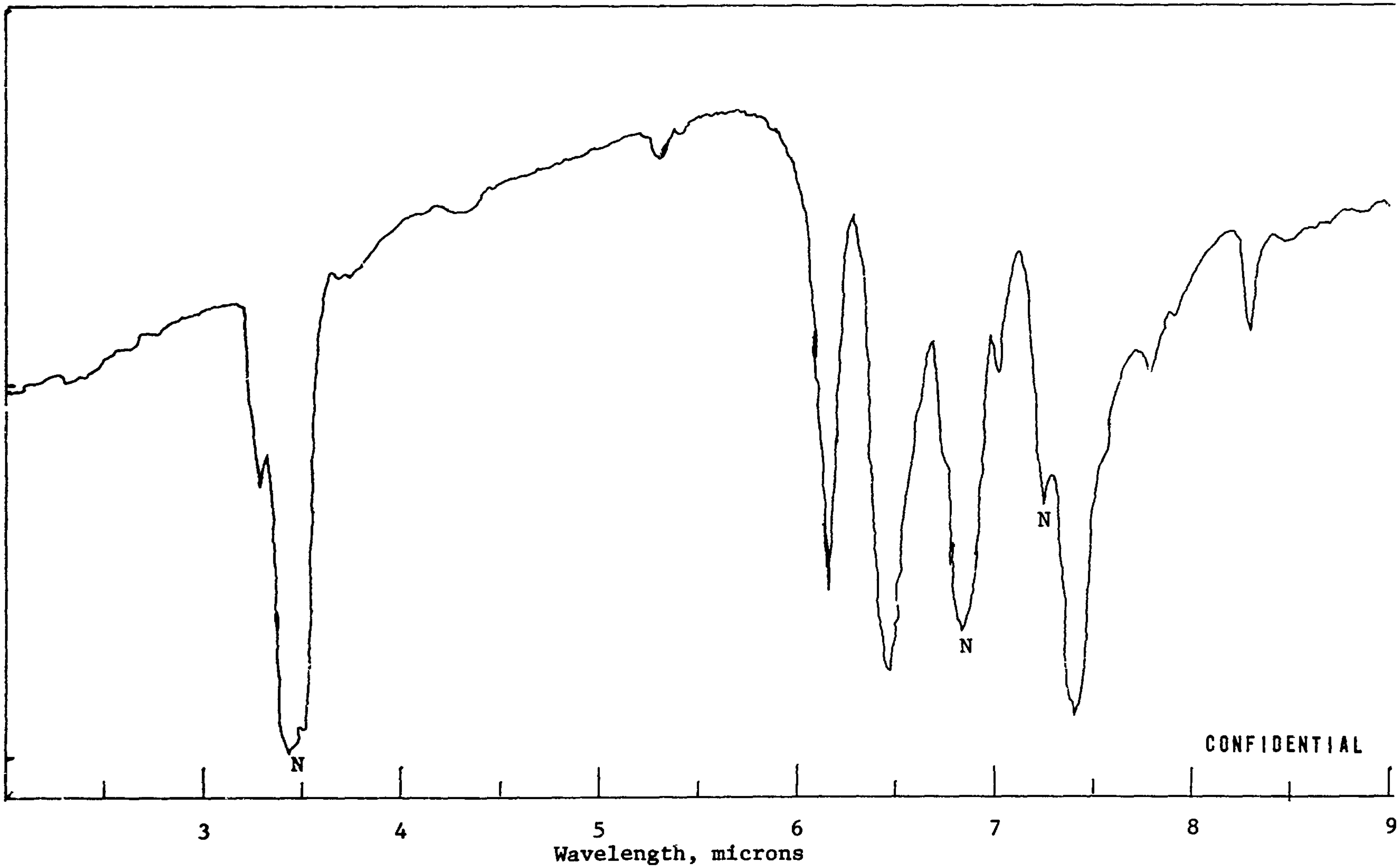
D. Gas Chromatography

Only TNTF and DFTNB were amenable to gas chromatographic examination. The latter was assayed in this way, and all material shipped was first shown to be at least 98.5% DFTNB prior to release from Peninsular ChemResearch.

Gas Chromatographic
Instrumental Parameters

Column	TNTF	DFTNB		
Length	8 ft.	8 ft.	or	6 ft.
Liquid phase	GE-SE-30	GE-SE-30	or	UC W-98
Loading	20%	20%		10%
Solid support	HMDS-Gas Chrom P	HMDS-Gas Chrom P		Diatoport S
Injector temperature	250°	250°		
Oven temperature	205°	220°		
Detector temperature	250°	250°		
Carrier gas	He, 40 ml/min.	He, 30 ml/min.		
Sample preparation				
Solvent	Benzene	Benzene		
Sample size	2-4μl	10 μl		

Quantitation was by normalization of the products of peak height and peak width at 1/2 height. Typical chromatograms are shown following.



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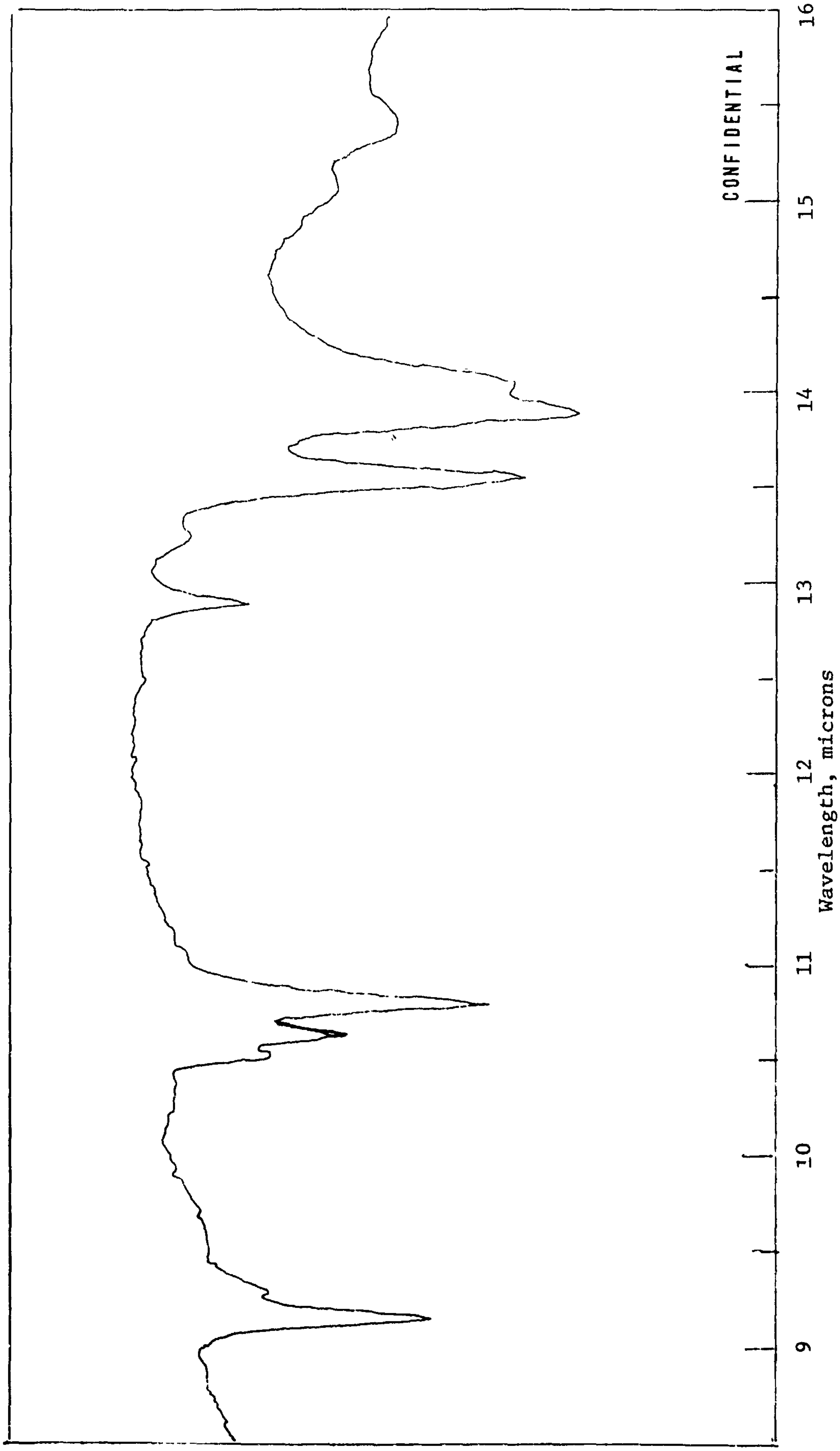
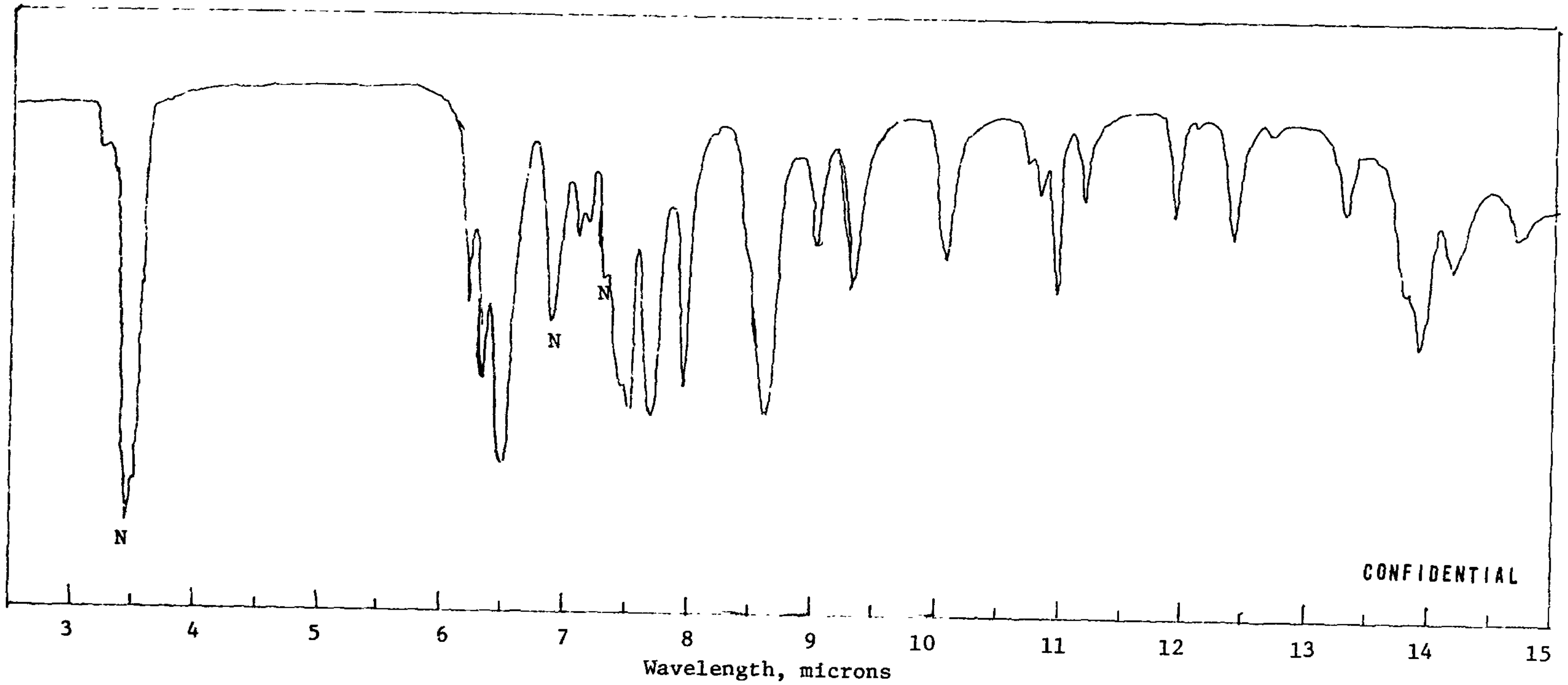
Infrared Spectrum of PF as the Nujol mull
Figure 1

N = Nujol absorption

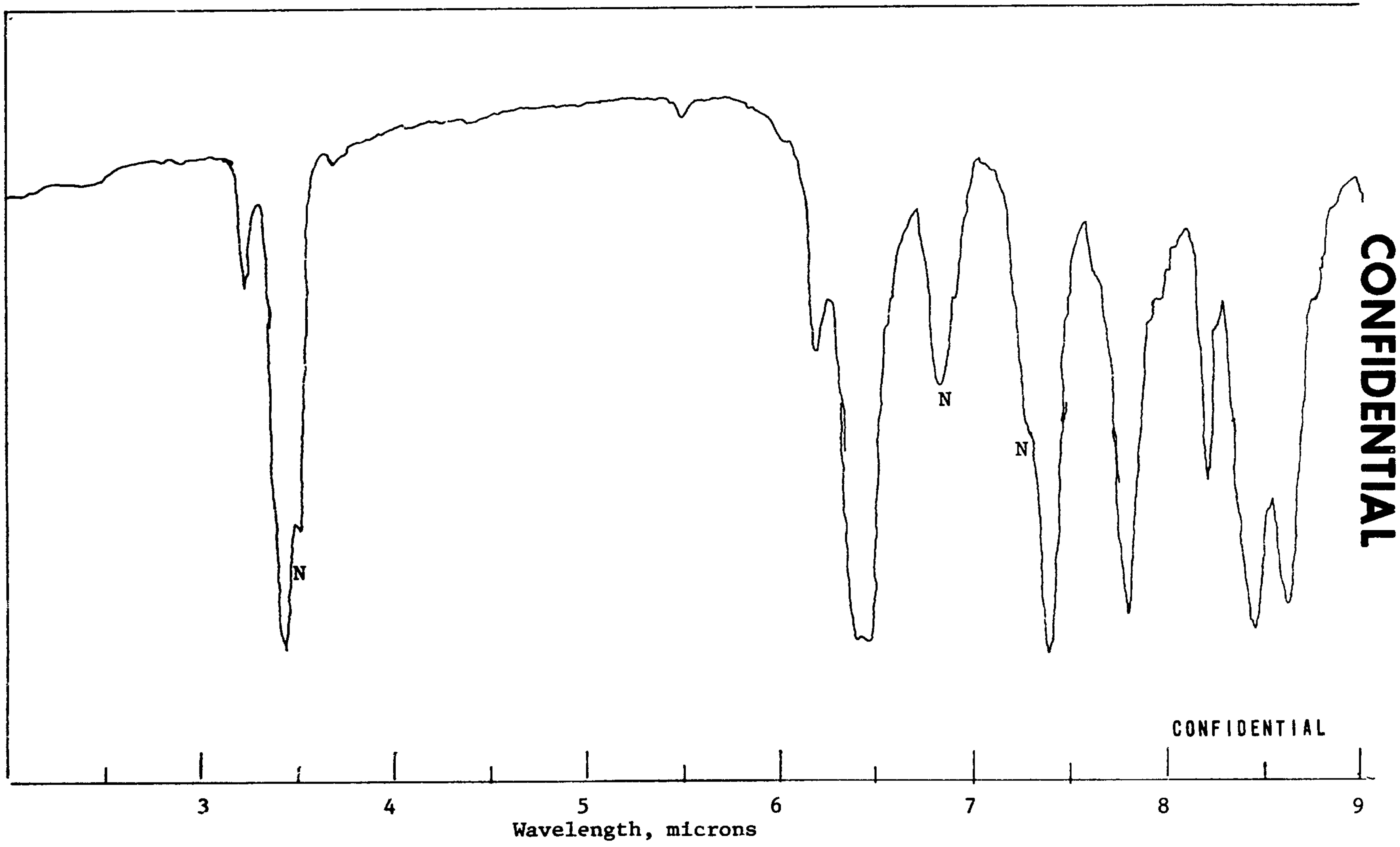


Infrared Spectrum of PF as the Nujol mull (Concluded)
Figure 1



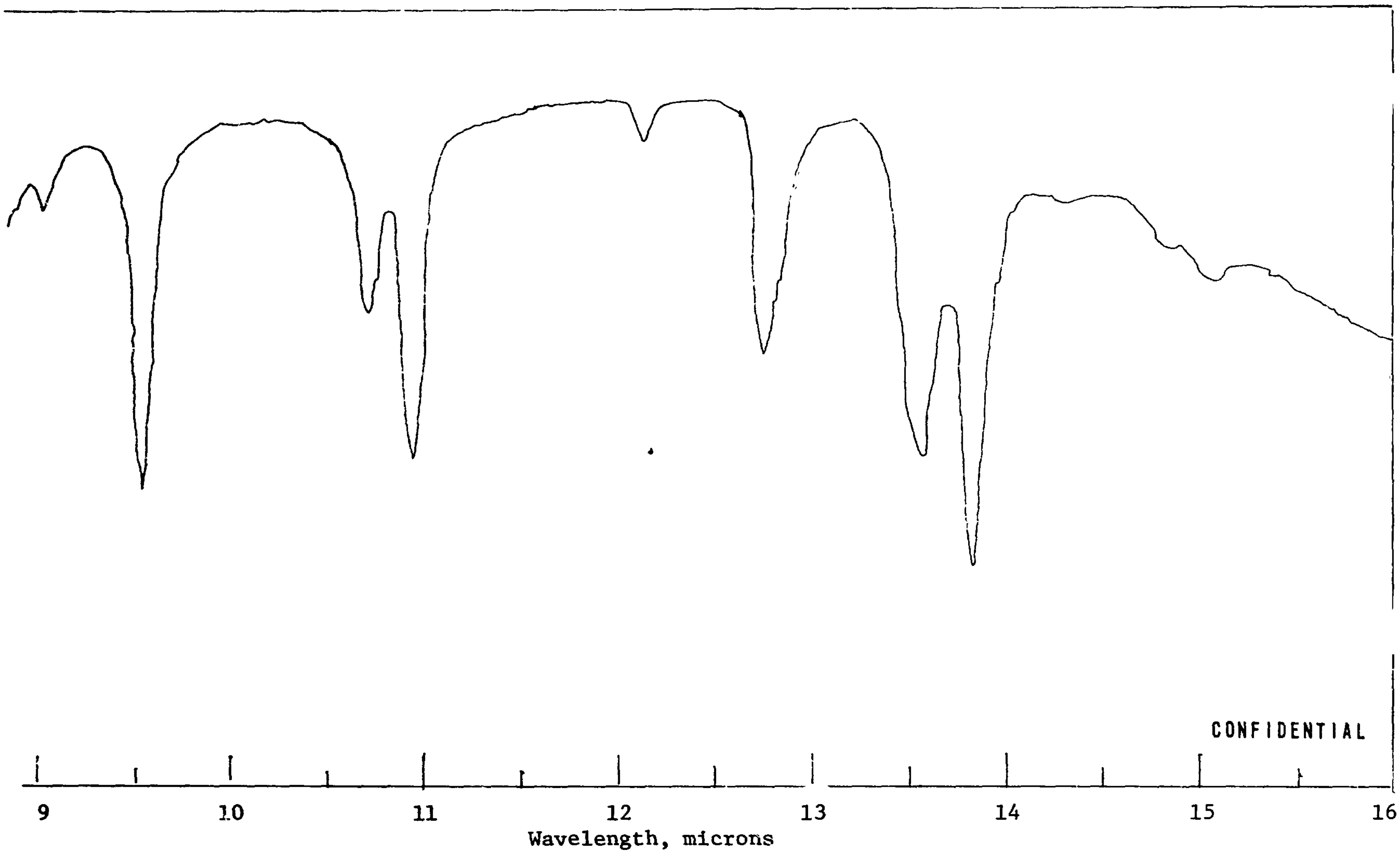
N = Nujol absorption

Infrared Spectrum of TFET as the Nujol mull
Figure 2

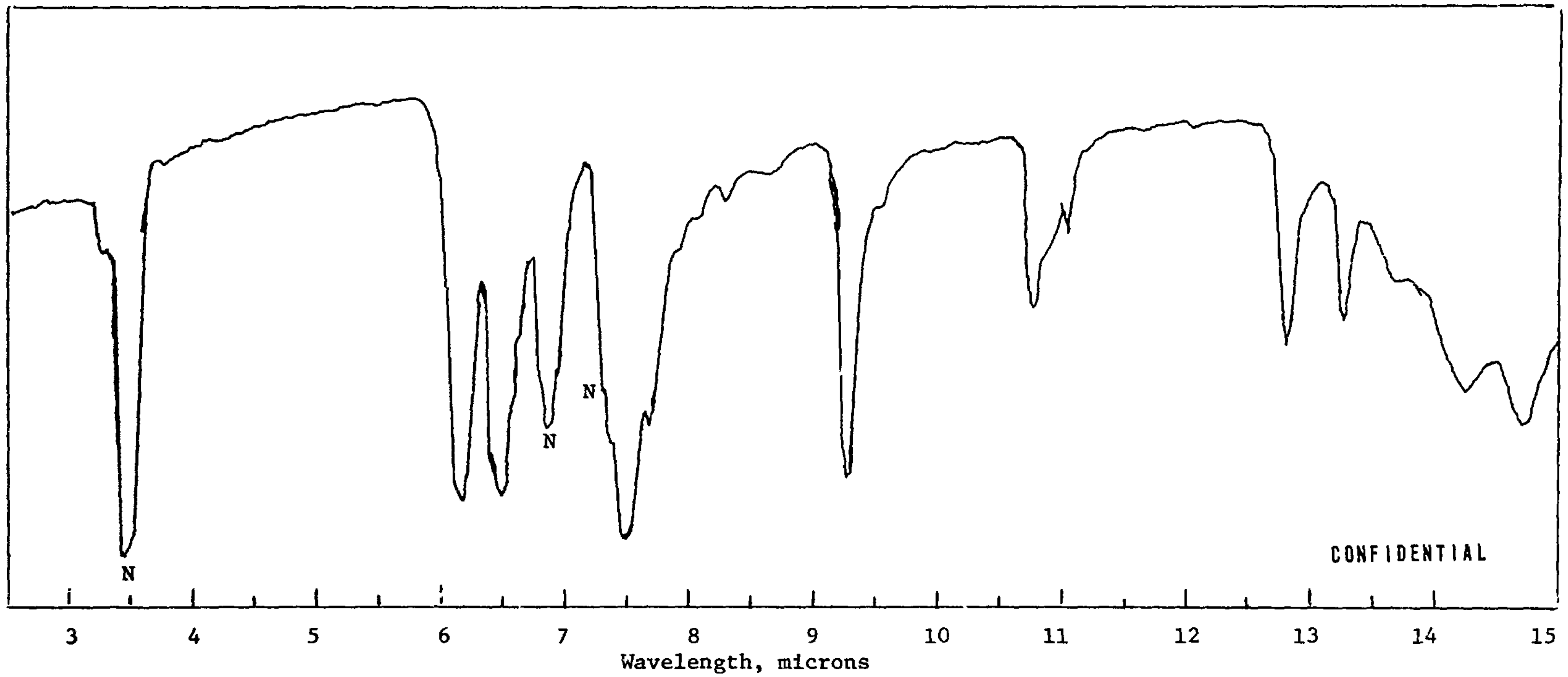


Infrared Spectrum of TNTF as the Nujol mull
Figure 3

N = Nujol absorption



Infrared Spectrum of TNTF as the Nujol mull (Concluded)
Figure 3



N = Nujol absorption

Infrared Spectrum of DFTNB as the Nujol mull
Figure 4

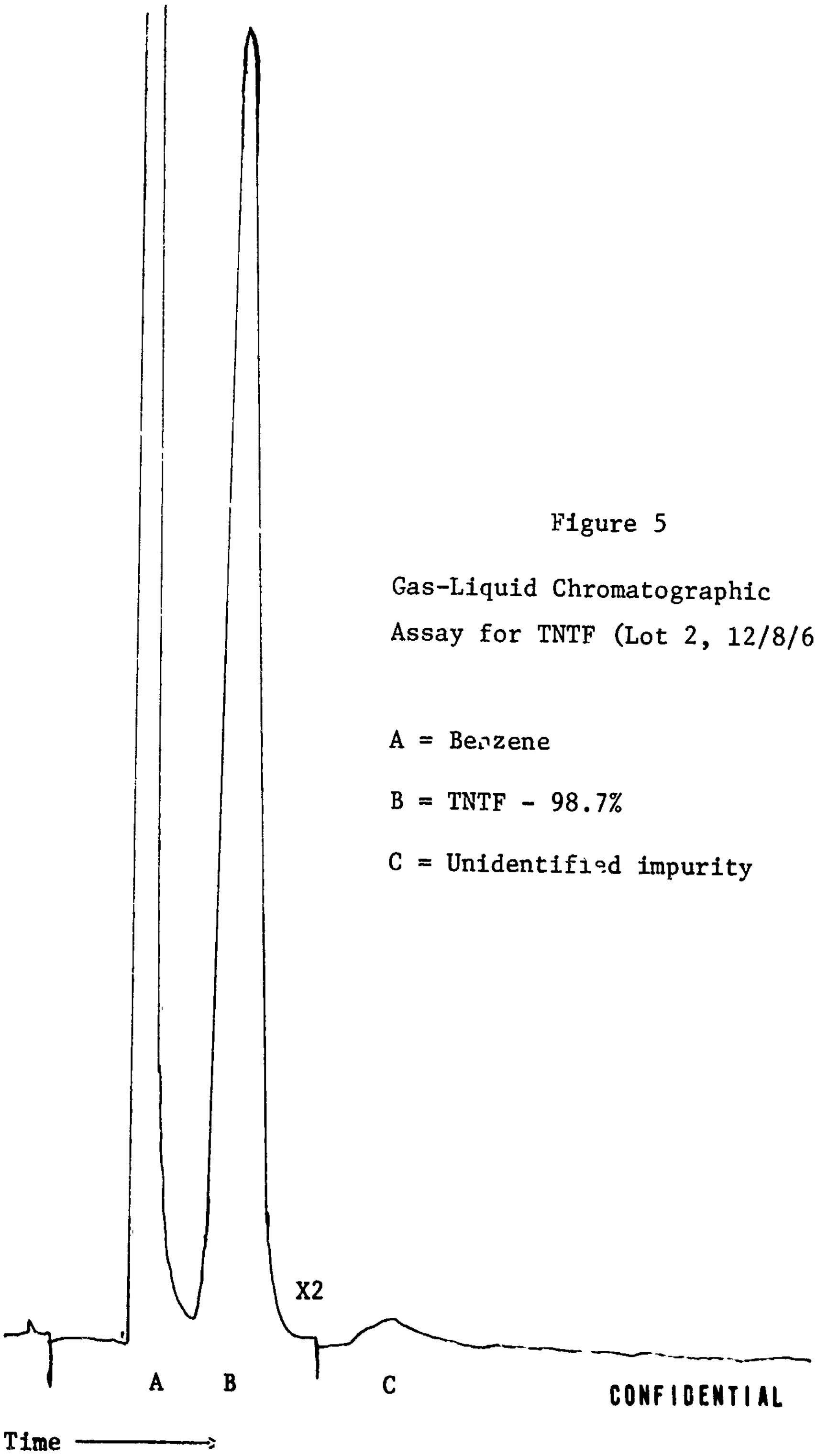


Figure 5

Gas-Liquid Chromatographic Assay for TNTF (Lot 2, 12/8/65)

A = Benzene

B = TNTF - 98.7%

C = Unidentified impurity

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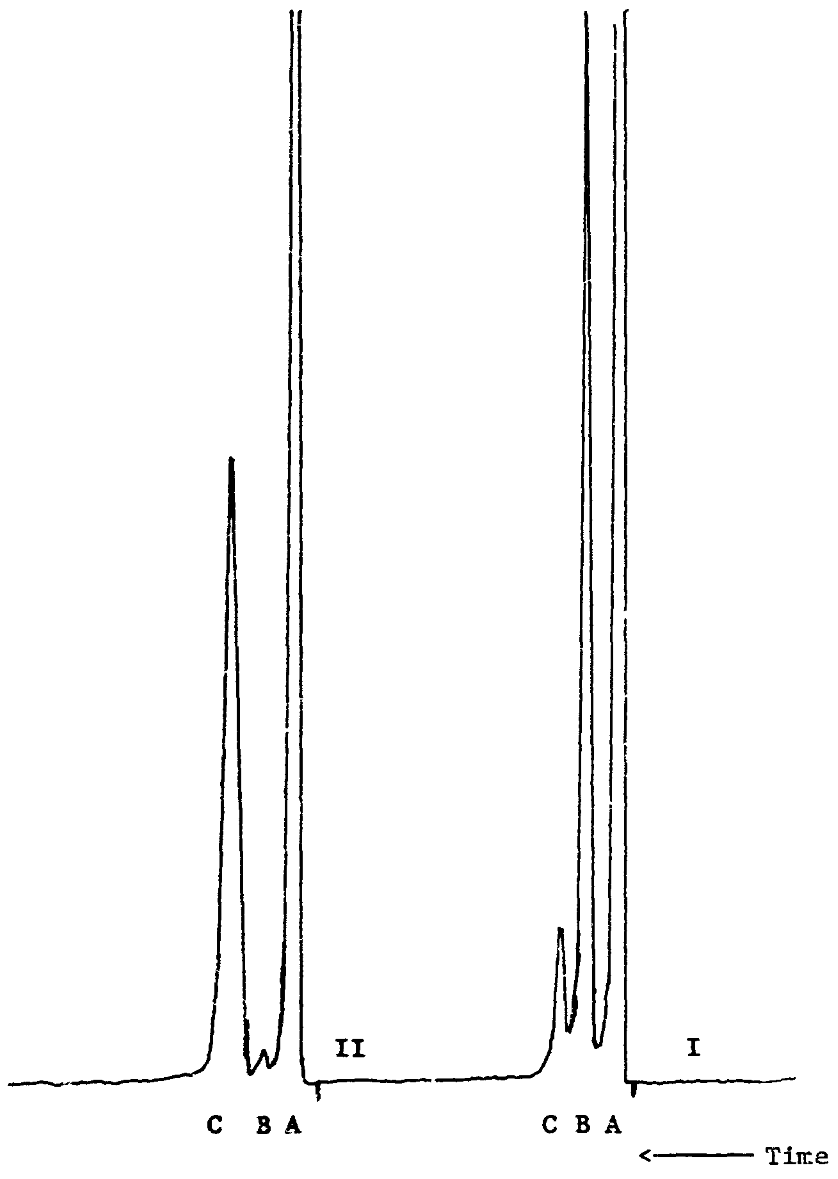

Figure 6

Gas-Liquid Chromatographic
Determination of DFTNB

A = Benzene

B = DFDNB

C = DFTNB

I = Crude Product
16.2% DFTNB

II = Recrystallized Material
98.6% DFTNB

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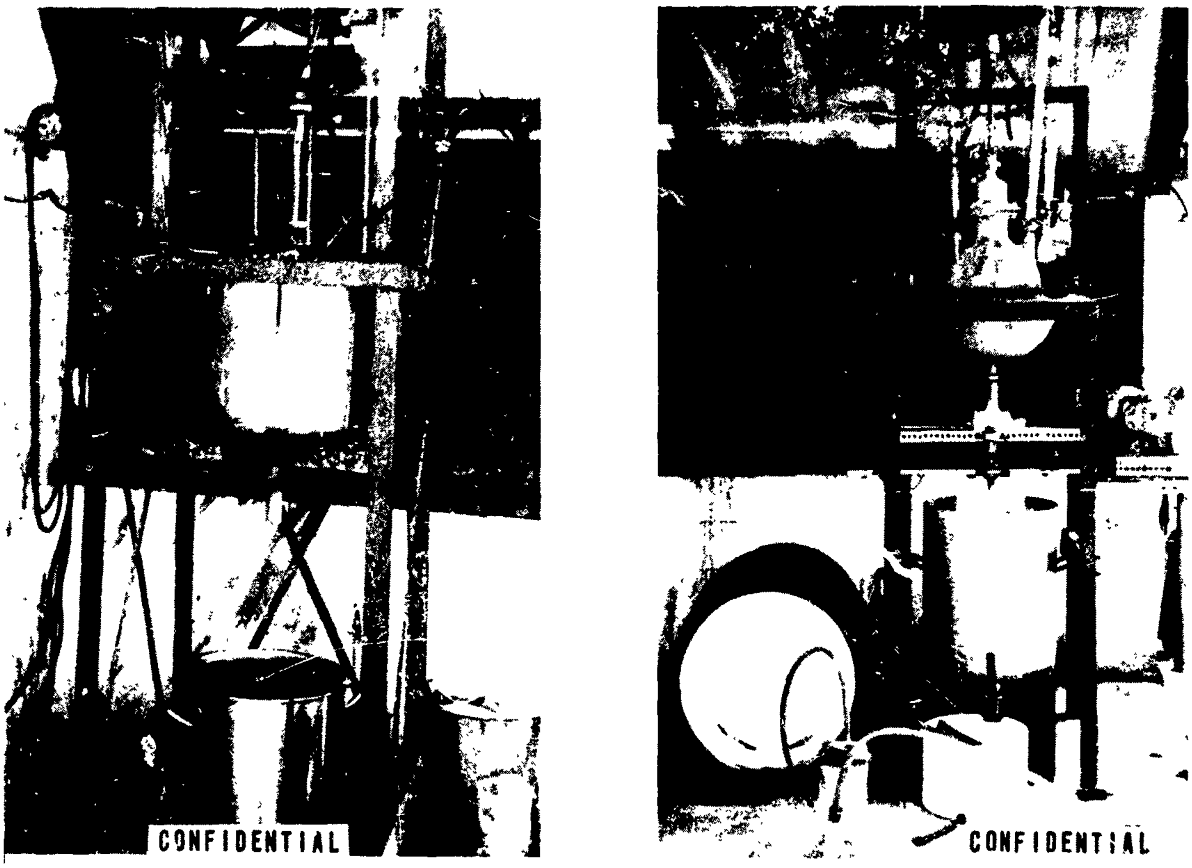
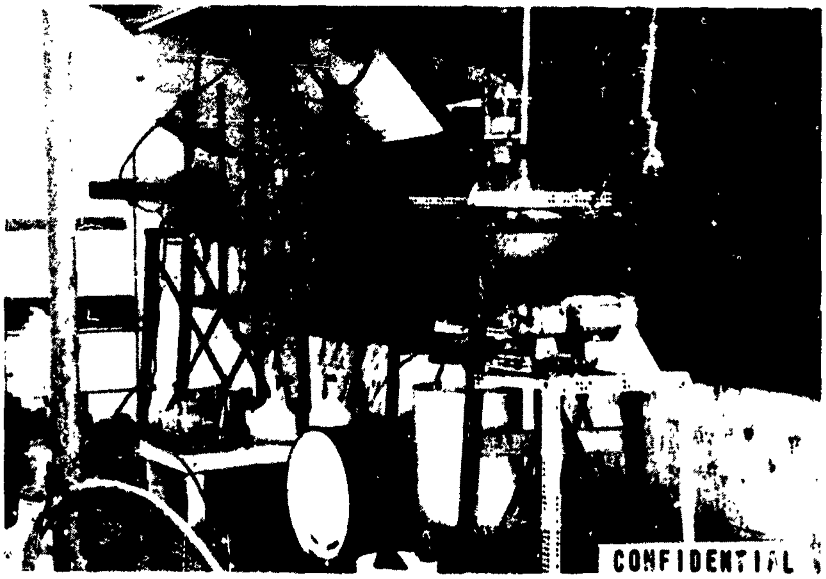


Five-Gallon Nitrator

22-Liter Glass Reactor



Overall View

Manufacturing Facilities for Fluoroexplosives
Figure 7

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Air Force Armament Laboratory
Air Force Systems Command
Eglin Air Force Base, Florida

13 ABSTRACT

The synthesis of twenty pounds of each of three and forty-five pounds of a fourth fluoroexplosive has been carried out following general procedures supplied in R & D Exhibit No. ATW 64-84. Modifications to some of these procedures were made to improve yields of products or handling procedures at the scale being run. Laboratory equipment used in this work is described.

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	ROLE	WT	ROLE	WT	ROLE	WT
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