

7-Amino-4,6-Dinitrobenzofuroxan, an Insensitive High Explosive

by
W. P. Norris
Research Department

JUNE 1984

NAVAL WEAPONS CENTER
CHINA LAKE, CALIFORNIA 93555



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FOREWORD

The performance of warheads in ordnance systems is maximized in terms of many variables including the properties of the explosive charge. Research on energetic materials at NWC seeks new explosives with superior properties for use in warheads. 7-Amino-4,6-dinitrobenzofuroxan is an easily prepared insensitive high explosive with calculated explosive power superior to TNT and equal to that of TATB. It is a valuable addition to the list of energetic materials which may be useful for warhead design.

This work was funded under Naval Weapons Center Independent Research funds, Program Element 61152N, Project Number ZR00001, Task Area ZR01301, and Work Unit Number 13805037. This report was reviewed for technical accuracy by Rodney L. Willer and Ronald A. Henry.

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30 March 1984

Under authority of
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Released for publication by
B. W. HAYS
Technical Director

NWC Technical Publication 6522

Published by. Technical Information Department
Collation Cover, 8 leaves
First printing. 180 copies

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NWC TP 6522	2. GOVT ACCESSION NO. ADA145665	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) 7-AMINO-4,6-DINITROBENZOFUROXAN, AN INSENSITIVE HIGH EXPLOSIVE		5. TYPE OF REPORT & PERIOD COVERED Final Report
7. AUTHOR(s) W. P. Norris		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Weapons Center China Lake, CA 93555		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Weapons Center China Lake, CA 93555		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS PE-61152N, Project-ZR00001, Task Area-ZR01301, Work Unit-13805037
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE June 1984
		13. NUMBER OF PAGES 14
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution is unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) 7-Amino-4,6-dinitrobenzofuroxan Insensitive Explosive High Density Explosive		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) See back of form.		

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(U) 7-Amino-4,6-dinitrobenzofuroxan, An Insensitive High Explosive, by W. P. Norris. China Lake, Calif., Naval Weapons Center, June 1984. 14 pp. (NWC TP 6522, publication UNCLASSIFIED.)

(U) 7-Amino-4,6-dinitrobenzofuroxan is an insensitive explosive (equal to TNT in impact sensitivity) with a calculated detonation velocity equal to that of TATB. A simple synthesis route is described. The position of the amino group is proved by reduction with triphenylphosphine to give 7-amino-4,6-dinitrobenzofurazan, a known compound. A by-product isolated from the reduction reaction is 2-(triphenylphosphinimido)-4,6-dinitroaniline.

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CONTENTS

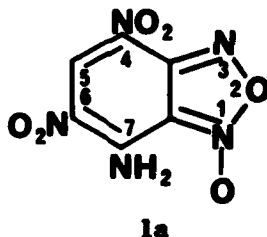
Introduction	3
Explosive and Physical Properties	3
Synthesis	4
Structure Proof	5
Conclusion	8
Experimental	8
Preparation of 7-Amino-4,6-dinitrobenzofuroxan (1a)	8
Reaction of 5-Chloro-4,6-dinitrobenzofuroxan (3) with Ammonia	10
Preparation of 7-Amino-4,6-dinitrobenzofurazan (4)	10
Reduction of 7-Amino-4,6-dinitrobenzofuroxan (1a) with Triphenylphosphine	10
References	12

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INTRODUCTION

This report describes the explosive and physical properties, synthesis, and structure proof of 7-amino-4,6-dinitrobenzofuroxan (1a).



EXPLOSIVE AND PHYSICAL PROPERTIES

7-Amino-4,6-dinitrobenzofuroxan is an insensitive, thermally stable explosive. It is a fairly dense, easily prepared compound with a calculated detonation velocity equivalent to that of 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) (see Table 1).

The introduction of an amino group into 4,6-dinitrobenzofuroxan (DNBF) has a remarkable effect upon physical and explosive properties. The melting point, density, and calculated detonation velocity all increase significantly and the impact sensitivity is reduced dramatically, as shown in Table 2.

TABLE 1. Properties of 7-Amino-4,6-dinitrobenzofuroxan.

Properties	Measurements
Molecular formula	$C_6H_3N_5O_6$
Molecular weight	241.12
Density ^a	$1.902 \pm 0.008 \text{ g/cm}^3$
Melting point (DSC, $10^\circ/\text{min}$) ^b	270° (decomposition)
Oxygen balance (CO)	-10
Percent nitrogen	29.1
Detonation velocity (calculated) ^c	$7.91 \text{ mm}/\mu\text{s}$
Detonation pressure (calculated) ^c	282 Kbar
Impact sensitivity (H_{50}) ^d	53 cm (TNT = 54 cm)
Heat of formation	$+36.79 \pm 0.72 \text{ Kcal/mol}$

^a Gas comparison pycnometer, type 6102-12, System Science and Software, LaJolla, Calif.

^b See Figure 1.

^c See Reference 1.

^d Bureau of Mines design instrument, type 12 tools, 2.5 Kg wt.

TABLE 2. Comparison of Properties of DNBF and 1a.

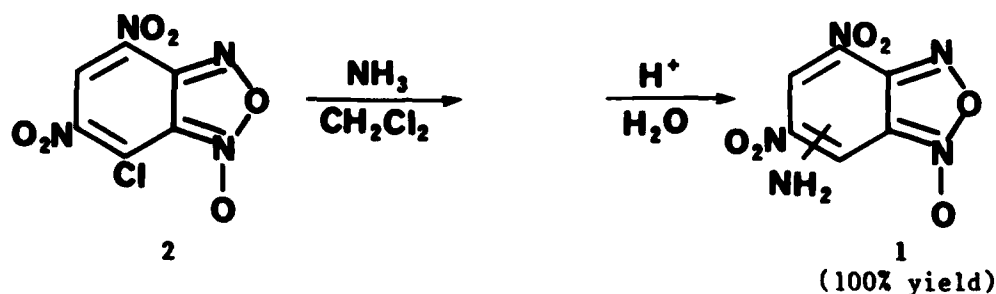
Properties	DNBF	1a
Melting point	$174-175^\circ$	270° (decomposition)
Density	$1.747 \pm 0.001 \text{ g/cm}^3$	$1.902 \pm 0.008 \text{ g/cm}^3$
Impact sensitivity	18 cm	53 cm
Detonation velocity (calc.)	$7.71 \text{ mm}/\mu\text{s}$	$7.91 \text{ mm}/\mu\text{s}$

SYNTHESIS

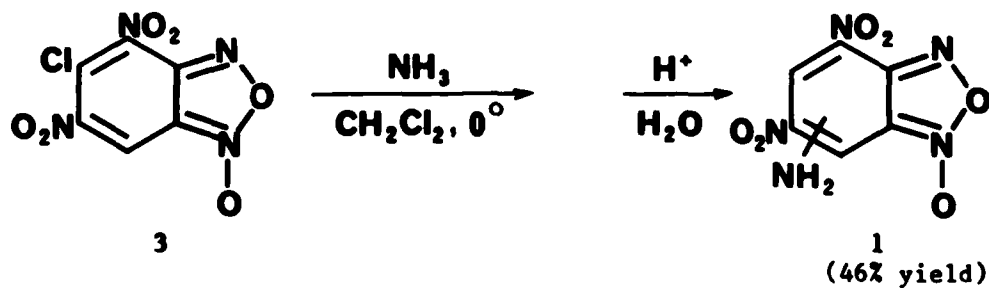
A synthesis of a monoamino-4,6-dinitrobenzofuroxan (1), by the thermal decomposition of 1-azido-3-amino-2,4,6-trinitrobenzene, was reported in 1968 (Reference 2). The compound was fully characterized, but the position of the amino group in the compound was not established.



A synthetic approach which would seemingly establish the correct structure of 1 would be the treatment of 7- or 5-chloro-4,6-dinitrobenzofuroxans (2 or 3) (Reference 3) with ammonia to give the 7- or

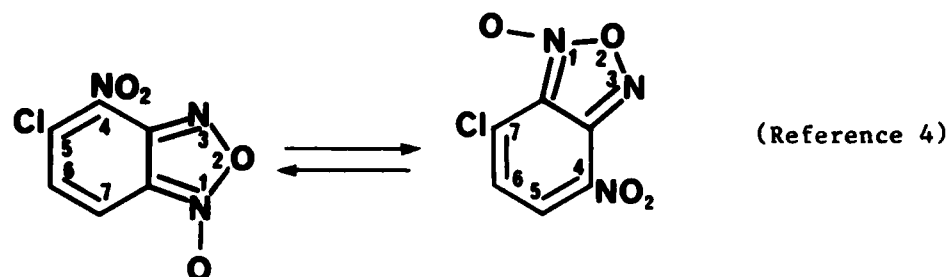


5-amino-4,6-dinitrobenzofuroxans (1a or 1b). However, treatment of either 2 or 3 with ammonia gives the same product.

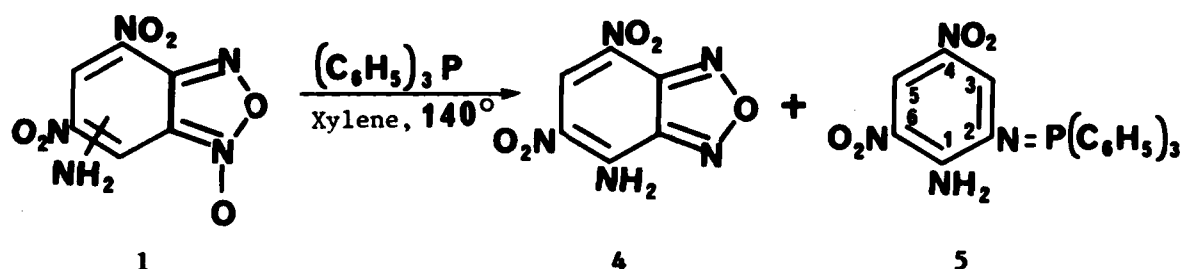


STRUCTURE PROOF

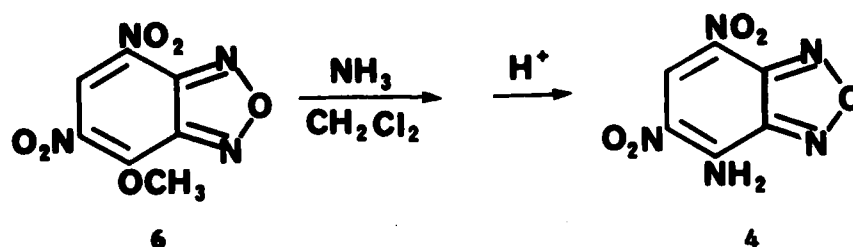
Molecular rearrangement may occur in compounds with a nitro group adjacent to a furoxan ring which interconverts the 5 and 7 positions (Reference 4). Thus, an approach other than simple substitution is



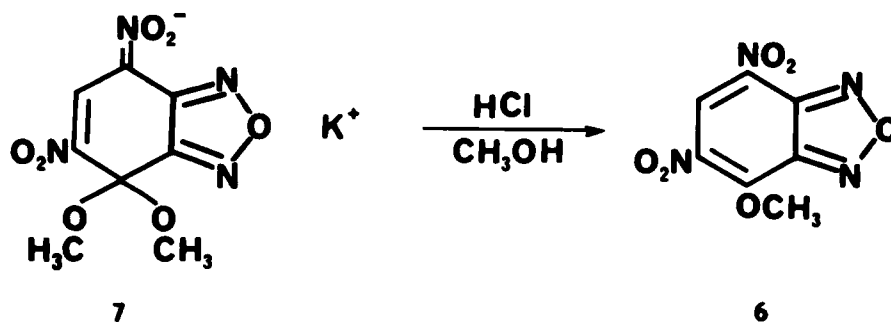
required to establish the position of the amino group in 1. Reduction of the furoxan ring to a furazan ring prevents this kind of isomerization. Treatment of 1 with triphenylphosphine, a reducing agent that converts 4,6-dinitrobenzofuroxan to the corresponding furazan derivative (Reference 5), gives 4. The reduction product, 4, was also synthesized



by an independent route from the known 7-methoxy-4,6-dinitrobenzofurazan (Reference 6), 6, so its structure is definitely established as 7-amino-4,6-dinitrobenzofurazan.



7-Methoxy-4,6-dinitrobenzofurazan, 6, is prepared by acidification of the Meisenheimer complex, potassium 7,7-dimethoxy-4,7-dihydro-4,6-dinitrobenzofurazanide, 7, the structure of which has been determined by X-ray crystallography (Reference 7).



An uncertainty remaining concerns a possible retro-Boulton-Katrutzky rearrangement (Reference 8)* of 1a at 140°C, the temperature of the reduction reaction, which conceivably could give the isomeric reduction product. However, a thermal analysis of 1a shows a perfectly straight heat flow versus temperature trace from 25 to 240°C which means, at least in the solid phase, there is no retro-Boulton-Katrutzky rearrangement occurring below 240°C and certainly not at 140°C. The aforementioned uncertainty is thus virtually eliminated.

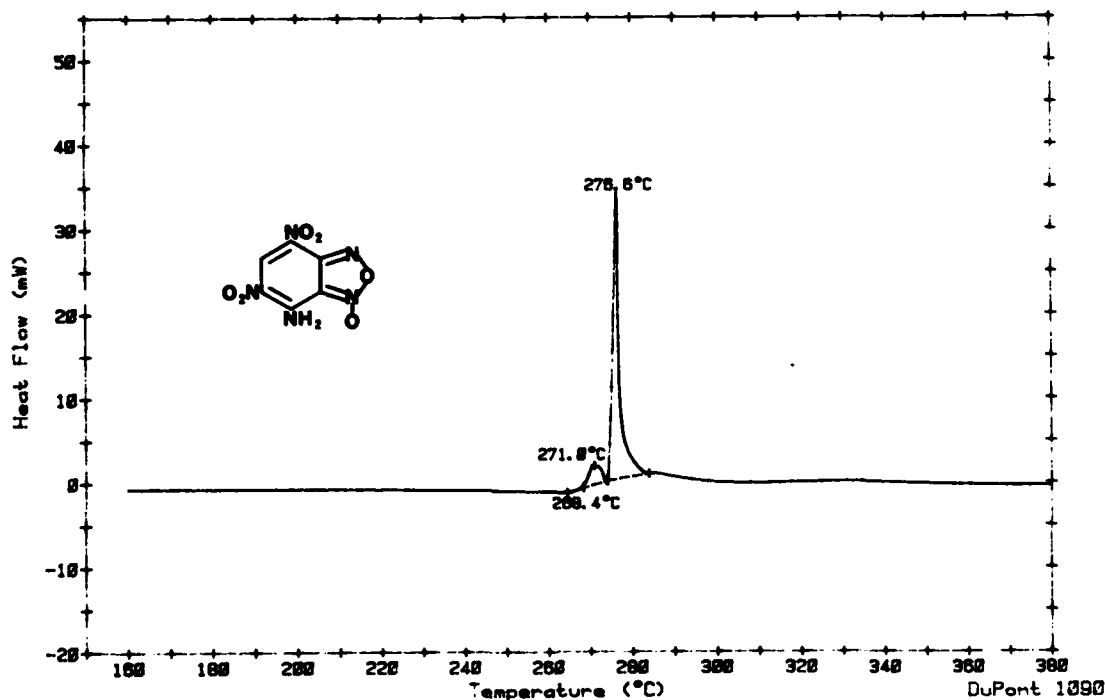


FIGURE 1. Thermal Analysis of 1a.

*Ghosh showed that 7-anilino-4-nitrobenzofuroxan changes to 5-anilino-4-nitrobenzofuroxan above 150° in the solid state. This is the retro-Boulton-Katrutzky rearrangement. The 5-anilino isomer in solution at 25° undergoes the normal Boulton-Katrutzky rearrangement to 7-anilino isomer.

The thermal analysis trace above 240°C, see Figure 1, possibly represents a retro-Boulton-Katritzky rearrangement process which, above 268°C, becomes a combination rearrangement-decomposition process. At 268°C, thermal contribution from exothermic decomposition exceeds endothermic rearrangement contribution causing the line to rise until at 271°C the endothermic process again prevails and the line drops. At about 274°C the decomposition process becomes predominant. The decomposition process may involve largely the rearranged isomer, 5-amino-4,6-dinitrobenzofuroxan.

There is another reduction product isolated. To this compound the structure, 2-(triphenylphosphinimido)-4,6-dinitroaniline, 5, is assigned. This structure is consistent with the elemental analysis, molecular weight, ^1H and ^{13}C NMR spectra, and the infrared spectrum of 5. The infrared absorption spectrum of 5 shows peaks at 3330 and 3210 cm^{-1} for the NH_2 group and after exchange with D_2O shows peaks at 2550 and 2400 cm^{-1} for the ND_2 group. Treatment of a chloroform solution of 5 (red in color) with gaseous hydrogen chloride forms gold colored plates, presumably the hydrochloride. Coupling of the two *meta* aromatic protons of 5 in the ^1H NMR spectrum gives doublets for H-5 and H-3, $J_{3,5} = 2.4$ Hz, and the H-3 doublet is split again by phosphorous in the adjacent triphenylphosphinimido group, $J_{\text{H,p}} = 1.1$ Hz. The ^{13}C NMR spectrum shows a C-2 doublet, $J_{\text{C,p}} = 2.3$ Hz, for ^{13}C coupling with phosphorous through nitrogen.

CONCLUSION

A new, easily synthesized, insensitive, high explosive, 7-amino-4,6-dinitrobenzofuroxan, is described. Its structure is proved by its reduction with triphenylphosphine to 7-amino-4,6-dinitrobenzofurazan, a compound of proven structure. Another reduction product, 2-(triphenylphosphinimido)-4,6-dinitroaniline is isolated and identified.

EXPERIMENTAL

PREPARATION OF 7-AMINO-4,6-DINITROBENZOFUROXAN (1a)

A solution of 5.00 g (0.0192 mol) of 7-chloro-4,6-dinitrobenzofuroxan³ in 150 ml of CH_2Cl_2 at 25°C is stirred under an ammonia atmosphere for 30 min. An orange colored solid begins separating immediately. At the end of the ammonia treatment, the orange solid is filtered off. Stirring the product in 100 ml of 3N HCl for 30 min and filtering gives 4.66 g (100% yield) of 1a, with a melting point of 270°C (decomposition) (see Figure 1). Recrystallization from CH_3CN gives 3.20 g of thin gold colored plates. Recrystallization from 70% nitric acid gives orange colored tabular crystals.

Analysis calculated for $C_6H_3N_5O_6$: C, 29.89; H, 1.25; N, 29.05.
 Found: C, 29.87; H, 1.28; N, 28.99.

Infrared spectrum: See Figure 2.

Mass spectrum: See Figure 3.

1H NMR ($DMSO-d_6$ + trace HCl , 30°) δ 10.10, 9.45 (NH_2 , s, nonequivalent); 9.00 (H-5, s). ^{13}C NMR ($DMSO-d_6$, TMS standard) δ 120.4 (C-4, s); 132.5 (C-5, s); 121.6 (C-6, s); 142.8 (C-7, s); 110.9 (C-8, s); 146.2 (C-9, s).

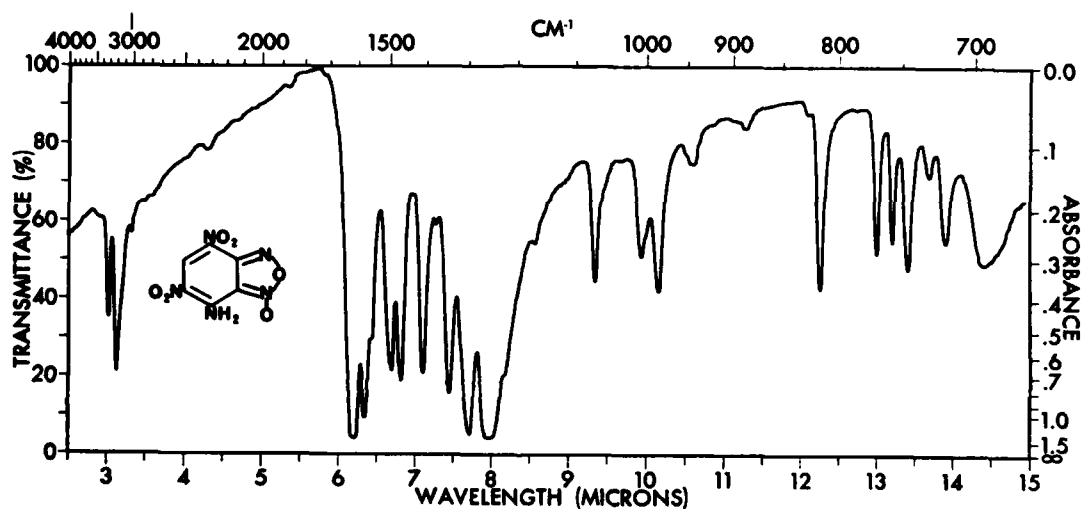


FIGURE 2. Infrared Spectrum of 7-Amino-4,6-dinitrobenzofuroxan (1a).

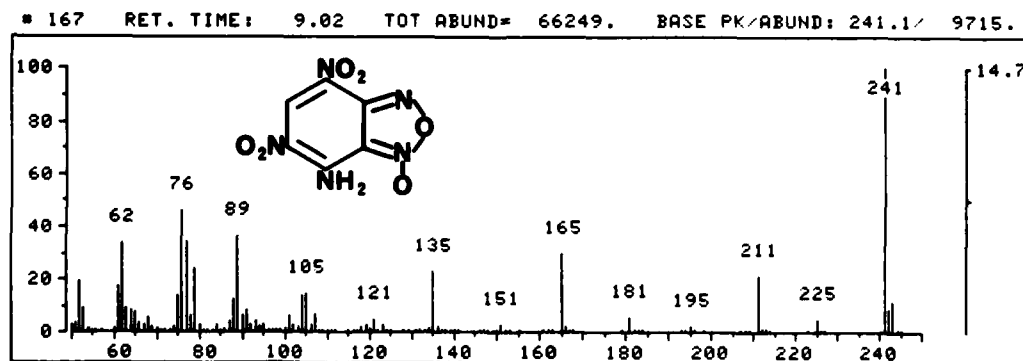


FIGURE 3. Mass Spectrum of 7-Amino-4,6-dinitrobenzofuroxan (1a).

REACTION OF 5-CHLORO-4,6-DINITROBENZOFUROXAN (3) WITH AMMONIA

A solution of 0.562 g (0.00216 mol) of 5-chloro-4,6-dinitrobenzofuroxan in 25 ml of CH_2Cl_2 cooled to -10°C , is treated with gaseous NH_3 , with stirring. An orange colored solid separates immediately. After 10 min, the suspended solid appears orange-red in color and the liquid phase is likewise orange-red in color. The solid product is filtered off and stirred in 50 ml of 3N HCl at 25°C to give 0.237 g (46% yield) of a tan powder with an infrared spectrum identical to that of 1a.

PREPARATION OF 7-AMINO-4,6-DINITROBENZOFURAZAN (4)

A solution of 0.595 g (0.00232 mol) of 7-methoxy-4,6-dinitrobenzofurazan (Reference 6) in 10 ml of CH_2Cl_2 at 25°C is stirred under an ammonia atmosphere for 10 min. An orange-yellow colored solid begins separating immediately. Volatiles are removed on a rotary evaporator to leave 0.565 g of the ammonium salt of 4. This is stirred for 10 min in 50 ml of 1N HCl, filtered, washed and dried to give 0.485 g (93% yield) of 4. Recrystallization from CH_3CN gives 0.353 g of 4, with a melting point of $249-253^\circ\text{C}$ (decomposition) [Lit. m.p. $215-245^\circ\text{C}$ (decomposition) (Reference 2)].

Analysis calculated for $\text{C}_6\text{H}_3\text{N}_5\text{O}_5$: C, 32.01; H, 1.34; N, 31.11.
Found: C, 32.02; H, 1.39; N, 31.04.

Infrared spectrum: See Figure 4.

^1H NMR ($\text{DMSO}-d_6$ + trace HCl, 40°) δ 10.91, 10.08 (NH_2 , s, nonequivalent); 9.08 (H-5, s).

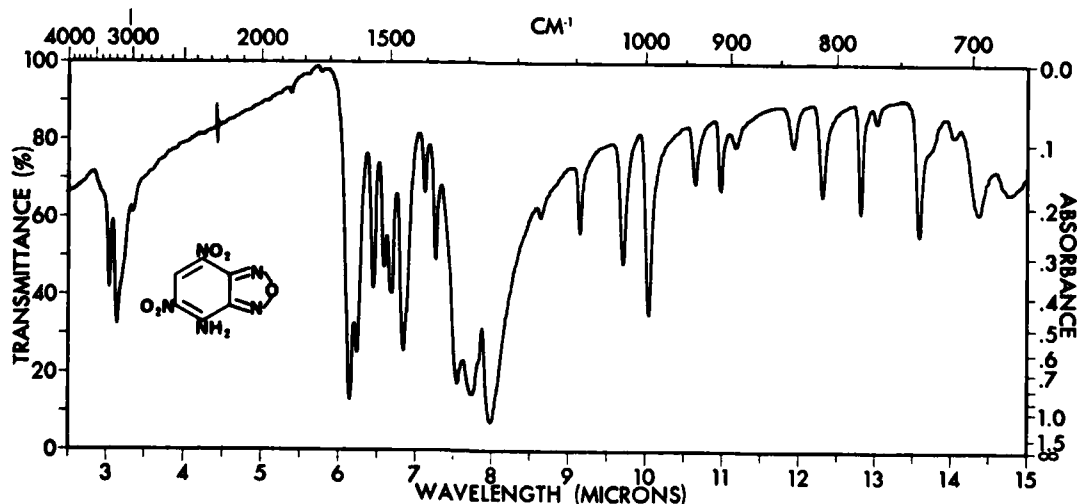


FIGURE 4. Infrared Spectrum of 7-Amino-4,6-dinitrobenzofurazan (4).

chromatography on a 35 x 210 mm silica gel column using CH_2Cl_2 as the developing solvent. Following unreacted triphenylphosphine there are three major bands.

The first band collected gives 0.20 g of a red solid, 5. Recrystallization from CCl_4 gives red needles of 2-(triphenylphosphinimido)-4,6-dinitroaniline (5) with a m.p. of 235-236°.

Analysis calculated for $\text{C}_{24}\text{H}_{19}\text{N}_4\text{O}_4\text{P}$: C, 62.88; H, 4.18; N, 12.22; P, 6.76. Found: C, 62.61; H, 4.25; N, 12.05; P, 7.04.

Infrared spectrum, 3330 and 3210 cm^{-1} ($-\text{NH}_2$); 2550 and 2400 cm^{-1} ($-\text{ND}_2$). Mass spectrum parent ion $m/e = 458$. ^1H NMR (CDCl_3 , 30°) δ 8.48 (H-5, d, $J_{3,5}$ 2.4 Hz); 7.8-7.5 (C_6H_5 m); 7.04 (H-3, dd, $J_{3,5}$ 2.4 Hz, $J_{\text{H,P}}$ 1.1 Hz). ^{13}C NMR (CDCl_3 , 30°), (nitroaromatic ring) δ 146.8 (C-4); 146.4 (C-6); 140.9 (C-1); 127.4 (C-5); 112.2 (C-2, d, $J_{\text{C,P}}$ 10.0 Hz); 112.0 (C-3); [$(\text{C}_6\text{H}_5)_3\text{P}$] δ 136.3 (C-P); 132.8 (para-C, d, $J_{\text{C,P}}$ 2.3 Hz); 132.5 (ortho-C, d, $J_{\text{C,P}}$ 10 Hz); 129.3 (meta-C, d, $J_{\text{C,P}}$ 12.3 Hz).

The second band collected gives 0.083 g of 7-amino-4,6-dinitrobenzofurazan (4). The infrared spectrum of this material is identical with the infrared spectrum, Figure 4, of the separately synthesized 7-amino-4,6-dinitrobenzofurazan (4).

The third band to be collected gives 0.10 g of triphenylphosphine oxide, with a m.p. of 156-157°. The infrared spectrum is identical to that of an authentic sample of triphenylphosphine oxide from Aldrich Chemical Co.

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- 2 Rockwell International Corporation, Canoga Park, CA
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