

AD-757 662

ISOLATION AND DISPOSAL OF CHEMICAL
INGREDIENTS UTILIZED IN ILLUMINATING
FLARES

Kenneth A. Musselman

Naval Ammunition Depot
Crane, Indiana

January 1973

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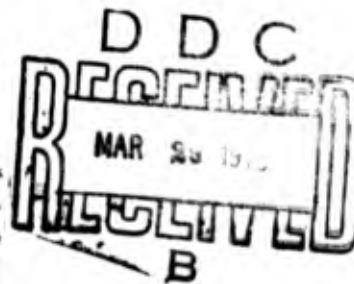
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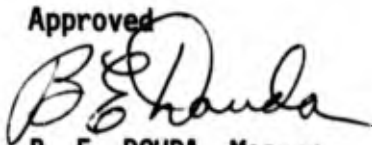
RDTR No. 217
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ISOLATION AND DISPOSAL OF CHEMICAL INGREDIENTS
UTILIZED IN ILLUMINATING FLARES

by

DR. KENNETH A. MUSSELMAN
Research Chemist

Approved



B. E. DOUDA, Manager
Chemical Sciences Division
Research and Development Department

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Security Classification

DOCUMENT CONTROL DATA - R & D

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1. ORIGINATING ACTIVITY (Corporate author)		2a. REPORT SECURITY CLASSIFICATION	
Naval Ammunition Depot Crane, Indiana 47522		UNCLASSIFIED	
		2b. GROUP	
3. REPORT TITLE			
Isolation and Disposal of Chemical Ingredients Utilized in Illuminating Flares			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (First name, middle initial, last name)			
Dr. Kenneth A. Musselman			
6. REPORT DATE		7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
January 1973		36 43	6
8a. CONTRACT OR GRANT NO		9a. ORIGINATOR'S REPORT NUMBER(S)	
ORDTASK ORD-332-003/060-1/UF 53-554-301		RDTR No. 217	
b. PROJECT NO		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
c.			
d.			
10. DISTRIBUTION STATEMENT			
Approved for Public Release; Distribution Unlimited			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY	
		Naval Ordnance Systems Command (ORD-0332) Washington, D. C. 20360	
13. ABSTRACT			
A common method of disposing of waste materials resulting from the production of illuminating flares is to burn them in a burning pit. This adds pollutants in the form of smoke and toxic or noxious fumes. To eliminate this source of pollution, a relatively facile scheme for isolating and reclaiming the magnesium and for the disposal of the sodium nitrate and binder residues from waste flare compositions has been developed. This scheme involves washing the flare compositions with water, and in some instances with acetone or other solvents, to remove sodium nitrate and most of the binder material. The remaining magnesium is dried and prepared for sale as scrap or for use in the production of new flares. Luminous intensity data, collected on prototype flares utilizing the reclaimed magnesium, suggest that reuse of the unadulterated material in illuminating composition production may not be feasible. Sodium nitrate, aqueous solution, is being evaluated for its nutrient value as a fertilizer and the intractable, infrangible binder is sent to land-fill.			

DD FORM 1473 (PAGE 1)

0102 014 6600

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- Security Classification

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Flares Illuminating Flares Pollution Magnesium Sodium Nitrate Binder Material Magnesium/Sodium Nitrate/Binder Waste Materials Reclaimed Magnesium Control Magnesium						

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FOREWORD

This paper was prepared for presentation at both the Military Pyrotechnics Section of the American Ordnance Association held on 25-26 October 1972 at Picatinny Arsenal, Dover, New Jersey, and the JANNAF Safety and Environmental Protection Working Group held on 29 November - 1 December 1972 at New Orleans, Louisiana.

ABSTRACT

A common method of disposing of waste materials resulting from the production of illuminating flares is to burn them in a burning pit. This adds pollutants in the form of smoke and toxic or noxious fumes. To eliminate this source of pollution, a relatively facile scheme for isolating and reclaiming the magnesium and for the disposal of the sodium nitrate and binder residues from waste flare compositions has been developed. This scheme involves washing the flare compositions with water, and in some instances with acetone or other solvents, to remove sodium nitrate and most of the binder material. The remaining magnesium is dried and prepared for sale as scrap or for use in the production of new flares. Luminous intensity data, collected on prototype flares utilizing the reclaimed magnesium, suggest that reuse of the unadulterated material in illuminating composition production may not be feasible. Sodium nitrate, aqueous solution, is being evaluated for its nutrient value as a fertilizer and the intractable, infrangible binder is sent to land-fill.

INTRODUCTION

It is necessary to dispose of waste illuminating flare compositions from pyrotechnic production at this Depot and at contractor's plants. These production wastes may either be bulk composition or defective illuminating flare candles. At NAD Crane alone, for example, during peak production periods, over 900 pounds of pyrotechnic bulk production wastes per operating day are accumulated. Even during low production schedules, this figure can amount to 500 pounds per day. A common method of disposing of these pyrotechnic wastes is to burn them in an open burning-pit in a sparsely populated area. In the past, when it was convenient, disposal by coastal Depots of unserviceable items was accomplished by dumping at sea. These methods tend to either induce water pollution or pollute the air with smoke and toxic or noxious fumes. With the increased interest in anti-pollution, it is now necessary to develop non-polluting methods for pyrotechnic waste disposal.

OBJECTIVE

The purpose of this work was to find an ecologically acceptable route to disposal of magnesium/sodium nitrate/binder waste materials which come from the manufacture of illuminating flares.

Major problems associated with this task were:

1. To develop methods to physically and/or chemically isolate the component materials utilized in these items.

2. To find methods for disposing of these materials in a safe and ecologically-permissible manner.

3. To develop the laboratory methods and techniques for accomplishing these objectives.

In addition, the possibility that the separated components could be reclaimed (i.e., salvaged) and reused or sold as scrap was also considered.

SUMMARY

During the tenure of this study the following were accomplished:

1. We developed an ecologically-permissible procedure for disposal of illuminating flare composition.

2. We developed techniques to reclaim and salvage the chemical ingredients utilized in illuminating flare compositions.

a. Magnesium: The reclaimed magnesium is recovered as a free-flowing material. Suggestions for possible disposition of this material are as a fuel, in other pyrotechnics in which visible light output is not paramount, or to offer it for direct sale.

b. Sodium Nitrate: The sodium nitrate is reclaimed as an aqueous solution. Suggestions for potential disposition of this material are to utilize this solution as a fertilizer or to reconstitute it and reuse it in pyrotechnics. In pursuing the former suggestion, we awarded a contract to Purdue University Agriculture Department to ascertain the nutrient value of aqueous solutions.

c. Binder: Unfortunately, the reclaimed polymerized binder has no beneficial value and, as such, is disposed in a land-fill operation. This low-volume (ca. 7% wt. of scrap), intractable, infrangible material is inert and will have no deleterious effect upon the environment.

3. The procedure is applicable to compositions utilizing both ellipsoidal¹ and atomized² magnesium powder.

DEFINITIONS

At this time I would like to define a few terms that will be utilized in this presentation:

Bulk Composition: Unconsolidated, agglomerated magnesium/sodium nitrate/binder (polymerized) illuminating flare composition.

Candle: The unit produced by high-pressure consolidation of magnesium/sodium nitrate/binder composition into some type of container.

Efficiency: A measure of flare performance defined as candela seconds per unit weight of flare candle composition, (cd-sec)/g.

Reclaimed Magnesium: Magnesium separated and isolated from bulk magnesium/sodium nitrate/binder (polymerized) illuminating flare production wastes according to the procedure developed and reported herein.

Control Magnesium: New, as purchased magnesium. Used as a control sample in preparing prototype flare candles.

DISCUSSION

The work presented here was conducted in two phases. Phase I was directed toward the separation and disposal of constituents from the bulk illuminating composition; Phase II was directed toward the disposal of candle wastes. This latter phase also included an engineering pilot study to ascertain those parameters necessary for designing a safe, efficient semi-works production unit.

We considered that family of illuminating composition which accounts for the largest production of scrap, viz., Mk 24, Mk 45, and MLU 32A illuminating flares^{3,4,5} and the Mk 11 Illuminating Projectile Load.⁶ Waste composition from these items is comprised of magnesium, sodium nitrate, and a polymeric binder, the latter being either epoxy or unsaturated polyester.

The approach to the solution of this problem was first to separate the three ingredients and then to dispose of them in an ecologically permissible and, possibly, useful manner. Consideration was given to reusing the magnesium in future flare production and its sale as scrap. Sodium nitrate was considered for use as a fertilizer. Unfortunately, the polymerized binders cannot easily be converted to any useful purpose and, therefore, were proposed for disposal in a landfill. However, these latter materials should not be a threat to our ecological system.

Two different polymeric binders, unsaturated polyester and epoxy, are used suggesting the need for different disposal treatments. While

this work was concerned initially only with scrap bulk material, it appeared that a relatively simple, facile wet-crushing technique could be employed to convert candle composition into a form that could be processed by the procedures reported below.

Two laboratory procedures were developed for disposing of scrap bulk composition, one for composition containing an epoxy binder and the other for composition containing a polyester binder. These procedures, with slight modification, were adapted for scale-up to the engineering study.

The procedures used in the breakdown of the flares were quite similar in both the Mk 24 and Mk 45 Aircraft Parachute Flares with the exception that the Mk 364 Fuze was removed from the Mk 45 Aircraft Parachute Flare prior to the start of the flare breakdown. The complete Mk 24 and Mods Flare (see Figures 1, 2, 3 and 4) and the Mk 45 and Mods Flare without fuze (see Figures 5, 6, 7 and 8) were placed in an air operated vise and a hydraulically operated ram was used to press the inner components from the outer tubes. The candle was separated from the parachute assembly by cutting the suspension cables from the suspension cup and/or block (see Figures 1 - 8). The suspension cups and/or blocks were removed from the candle paper tube by cutting through the tube with a remotely operated lathe (see Figures 9, 10 and 11). The paper tube was scored longitudinally in six positions with a remotely operated saw and the paper tube was

removed from the candle composition (see Figures 12, 13, 14 and 15).

The initial approach toward the candle composition breakdown was to follow the procedure developed in the laboratory. This procedure involved the placement of the composition in a solvent such as acetone, agitation for approximately 30 minutes, decantation of the mother liquor, followed by two water wash-agitation steps. Although this technique worked, the reaction rate was considerably slower than anticipated. In order to increase the rate of reaction, another solvent, methylene chloride, was employed. Laboratory tests indicated that this solvent substantially increased the breakdown reaction rate for both polyester and epoxy binders. The reaction of this solvent with these binders caused the hardened binder to soften and separate from the magnesium and sodium nitrate. That portion of the binder that is not removed by the solvent is washed away by either acetone or isopropyl alcohol, depending upon the binder system being handled.

The bare candle composition was placed in a trough on the mixing tank (see Figure 16) which contained a sufficient quantity of solvent and water to cover the candles during the crushing operation. The candles were crushed under an air cylinder operated crushing blade as the candle was advanced in steps by using a second cylinder and ram (see Figure 17). The crushed candle pieces were fed into the mixing tank and allowed to soak in the solvent solution or stirred using an air operated mixing motor. That binder which was not dissolved

(see Figure 18) was skimmed from the surface and placed into a drum for storage and the solvent was pumped out of the mixing tank into storage tanks. The mixing tank was filled with either acetone, isopropyl alcohol and/or water to remove the solvent and sodium nitrate from the magnesium. The wash/rinse solutions were pumped out of the mixing tank into storage tanks. A typical process is presented in Table I.

The remaining magnesium was removed from the mixing tank through a bottom drain valve and collected on a 0.093 inch mesh and a 0.011 inch mesh screen (see Figure 19). Undissolved lumps of composition were retained on the top (0.093 inch mesh) screen and the loose magnesium was retained on the bottom (0.011 inch mesh) screen. After the rinse solution had drained from the screens, the loose magnesium was placed in open pans (see Figure 20) and placed in a vented, 140°F oven to dry. The dried reclaimed magnesium was screened through a #20 screen (841 micron) and that portion which passed through was analyzed and used to press new candles. Laboratory analyses were run on each batch of dried magnesium and a representative analysis is shown in Table II.

About seven percent of the insoluble, agglomerate material remains after washing/decanting and sieving. This material is composed of polymerized binder and entrapped magnesium and, as such, will have to be sent to landfill disposal. This material is not hazardous, and

therefore, will not constitute a safety or health hazard. The filtered sodium nitrate aqueous solution (wash water) is retained for later use as a potential fertilizer; in fact, Purdue University is currently evaluating this latter material to determine its nutrient value.

Prototype candles, 35.66mm x 95.25mm, were prepared from both control and reclaimed magnesium and tested in our photometric tunnel. Each candle contained $58 \pm 2\%$ magnesium, $38 \pm 2\%$ sodium nitrate and $4 \pm 0.5\%$ epoxy binder. These small prototype flares were then evaluated in our photometric light tunnel according to those conditions described in specification MIL-C-18762. Burn time, luminous intensity and candle efficiency data are summarized in Figures 21, 22 and 23 and in Table III. From these data, the following observations can be made regarding the reclaimed magnesium:

1. Burn Time: A 10% increase, from 47 to 52 seconds, in burn time is achieved. This increase is not necessarily an asset.

2. Luminous Intensity: A 15% decrease, from 100,000 to 85,000 cd., is attained. This is a significant decrease in light output.

3. Efficiency: A 6% decrease, from 45,000 to 42,000 cd.-sec./g., in candle efficiency is observed.

It is believed that the increase in burn time and the consequent reduction in both luminous intensity and candle efficiency is brought about by the formation of a thin layer of hydroxide and/or oxide on the surface of the magnesium.

A property observed during the flare burning evaluation phase of this investigation was that the reclaimed magnesium gave more consistent light output and even burn than was observed during the burning of candles containing new magnesium. These latter candles, even though they yielded an apparent higher efficiency and luminous intensity, exhibited more variation and erraticism. This property, observed for the reclaimed magnesium, possibly can be explained by assuming that the surface of this metal has been "treated" in a uniform manner, and thus, does not expose greatly different surfaces during pyrolysis.

ACKNOWLEDGEMENTS

The author wishes to express his gratitude to the Explosives and Pyrotechnics Branch, Code 033, and the Utilization and Disposal Branch, Code 043, of Naval Ordnance Systems Command for their funding support for this study. Thanks also to Mr. J. E. Short, Jr., who directed and completed the engineering phase of this program.

REFERENCES

1. Magnesium powder, ellipsoidal, Type IV, granulation 30/50 meeting specification MIL-P-14067 B(MU) and Source Control Drawing NAVAIR Drawing 2151463.
2. Magnesium powder, atomized, Type III, granulation 18 meeting specification MIL-M-382 B.
3. Mk 24 Mod 4 Parachute Aircraft Flare, NAVAIR List of Drawings LD 615207, NAVAIR Drawing 2150922.
4. Mk 45 Mod 0 Parachute Aircraft Flare, NAVAIR Data List DL 214527, NAVAIR Drawing 2150992.
5. MLU 32A/B99 Aircraft Flare, NAVAIR Data List DL 67A82D100, NAVAIR Drawing 67A82B8014.
6. 5"/54 Projectile (I11.), Mk 48 Mod 1 with load, Mk 11 Mod 0 (I11.), MTF and ADF Mk 89 Mod 0 (Loaded and Fuzed Assembly) NAVORD List of Drawings LD 615133, NAVORD Drawing 2141490.

TABLE I

Typical Production Procedure for
Magnesium Reclamation

Eight Mk-45 Mod 0 Candles

30 min. water/methylene chloride (60/40) wash

15 min. water wash

15 min. isopropyl alcohol wash

15 min. water wash

Dump into screen

Dry at 140°F - 24 hrs.

NOTE: All wash steps are agitated @ 570 rpm.

TABLE II

Representative Analysis of Reclaimed
Magnesium (30/50) from Illumination Flares

Magnesium, ¹ %w	96.78
Sodium Nitrate, ² %w	0.14
Acid Insolubles, ³ %w	3.06
Magnesium Oxide, ⁴ %w	< 0.01
Magnesium Hydroxide, ⁴ %w	< 0.01

¹ EDTA Colorimetric Analysis

² Atomic Absorption Spectroscopy

³ Dilute HCl

⁴ X-ray Diffraction Analysis

TABLE III
Summary of Photometric Test Data

	Burn Time sec.			Luminous Intensity cd.			Efficiency cd.-sec./g.		
	No. Sxs.	Mean	Std. Dev.	No. Sxs.	Mean	Std. Dev.	No. Sxs.	Mean	Std. Dev.
Control Magnesium	120	47.3	2.0	120	100,272	7,752	120	45,019	2,309
Reclaimed Magnesium	99	52.1	1.3	99	85,231	4,722	99	42,303	2,120

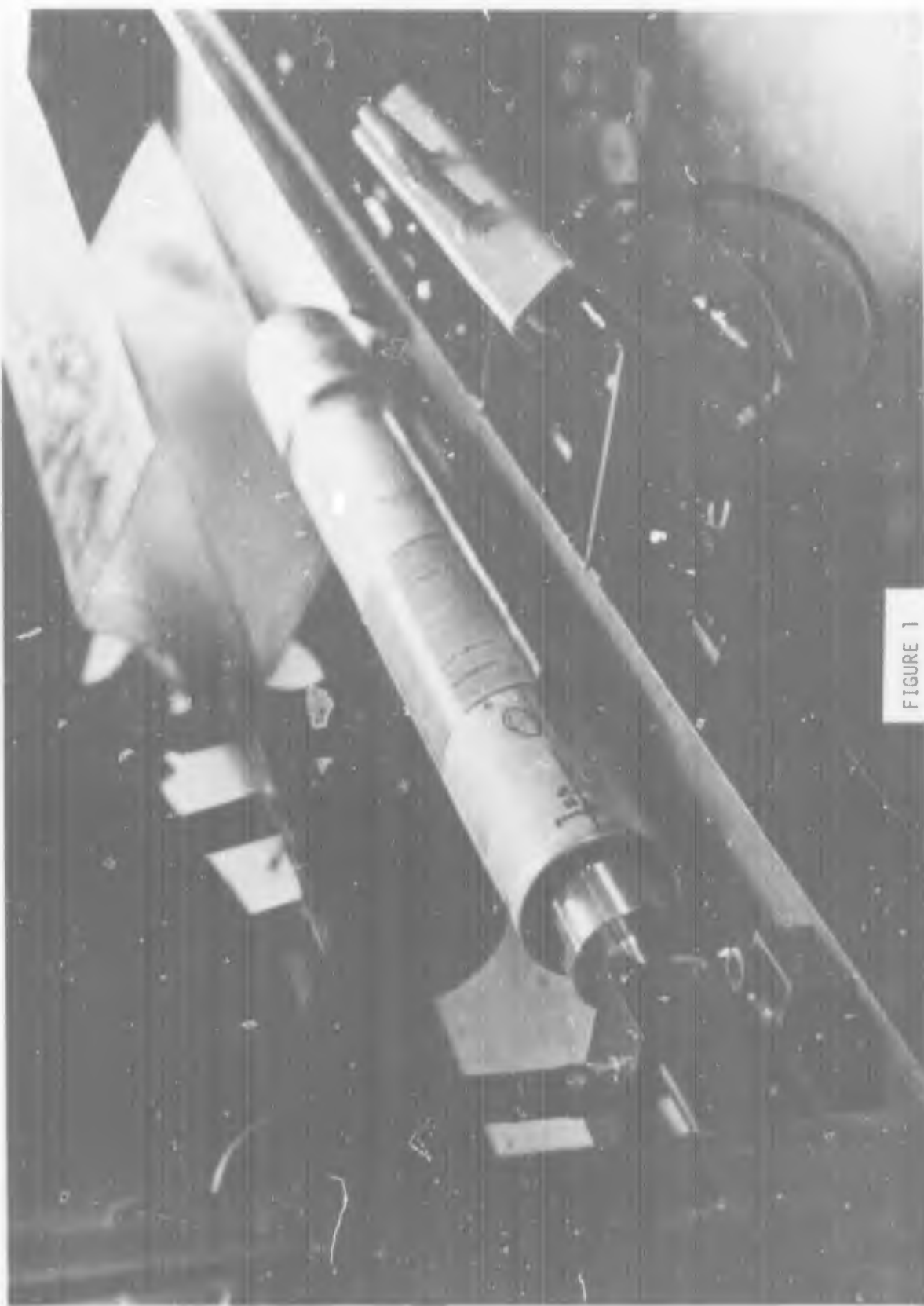


FIGURE 1



FIGURE 2



FIGURE 3



FIGURE 4



FIGURE 5

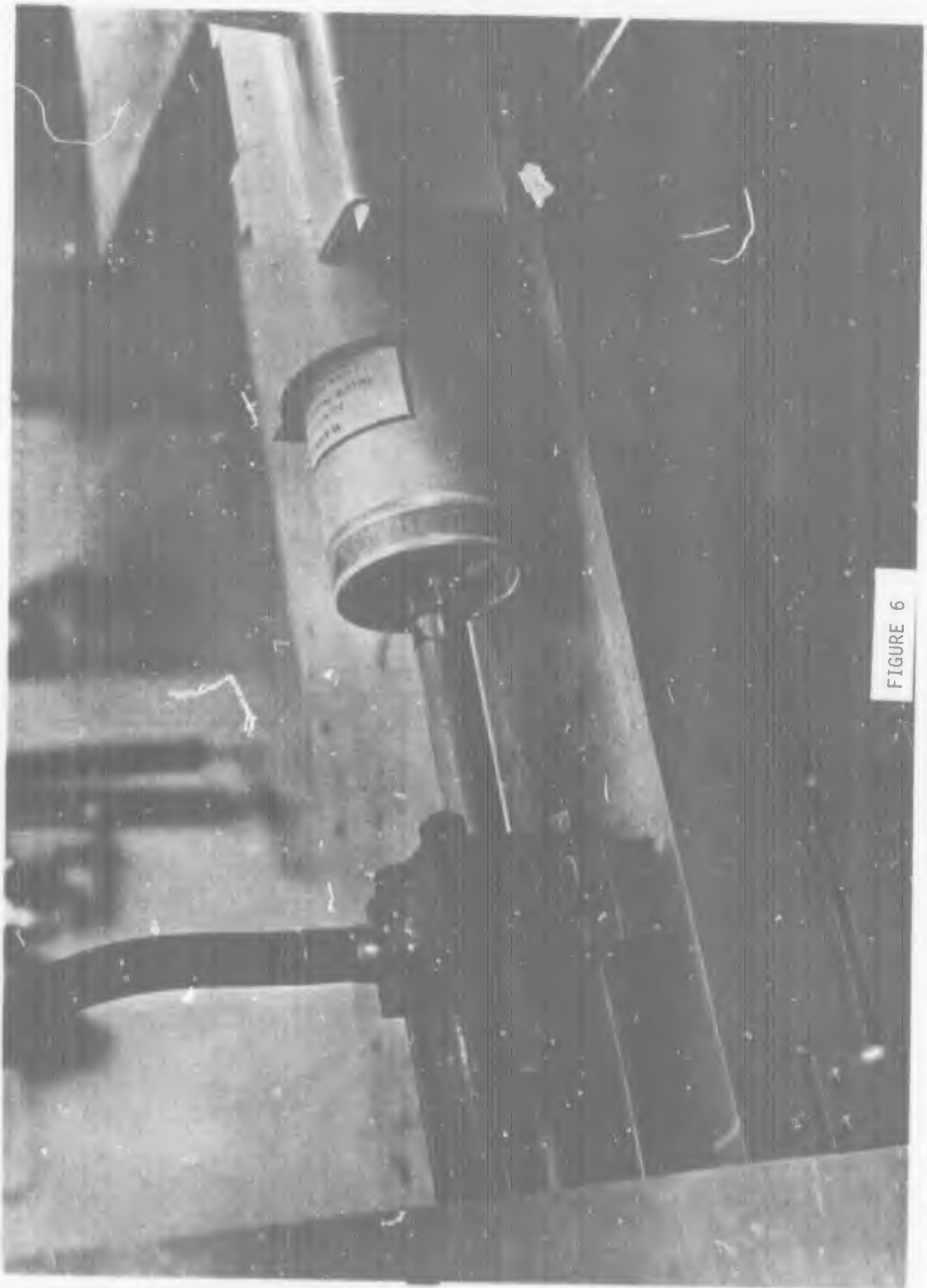


FIGURE 6

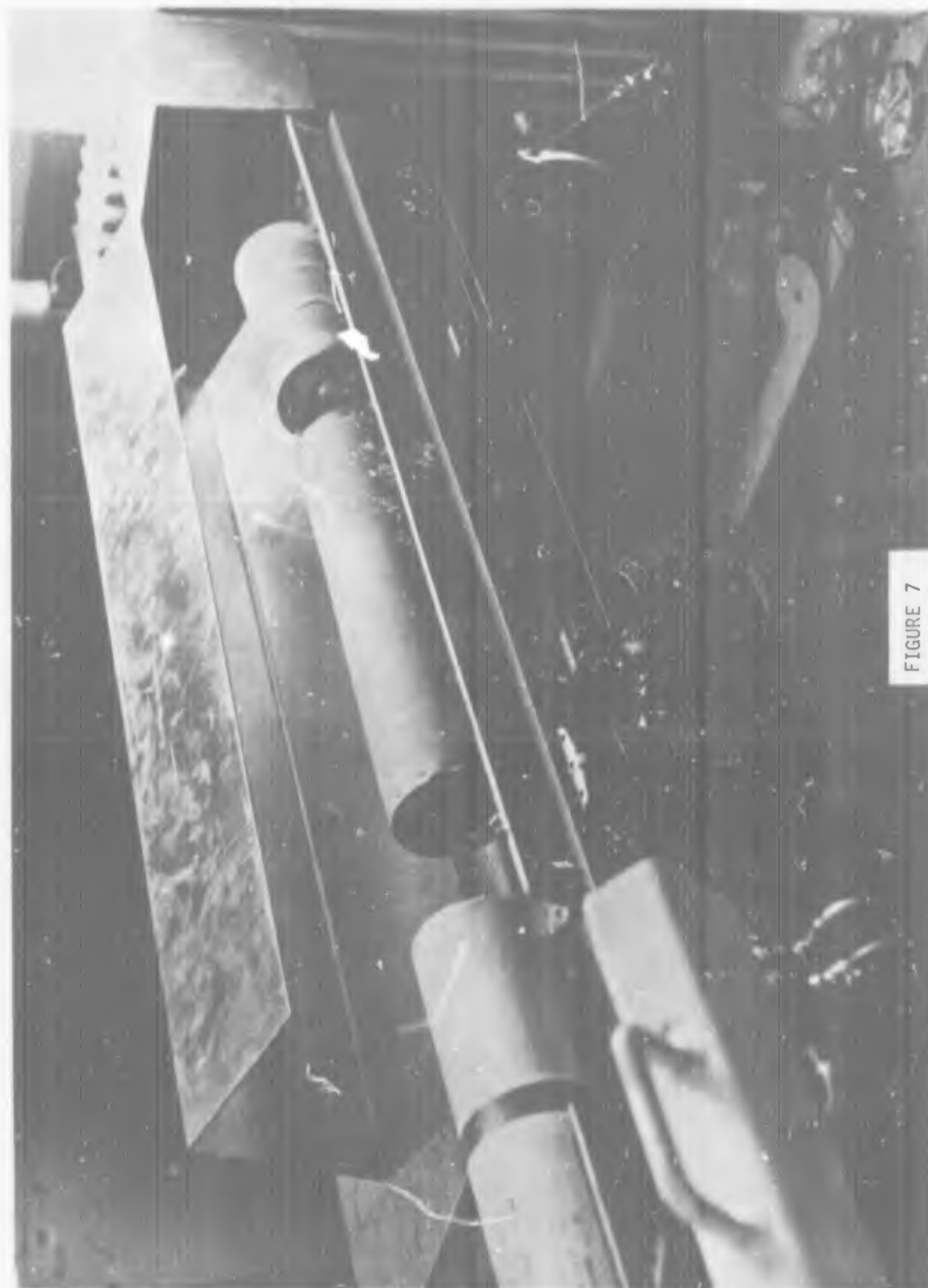


FIGURE 7

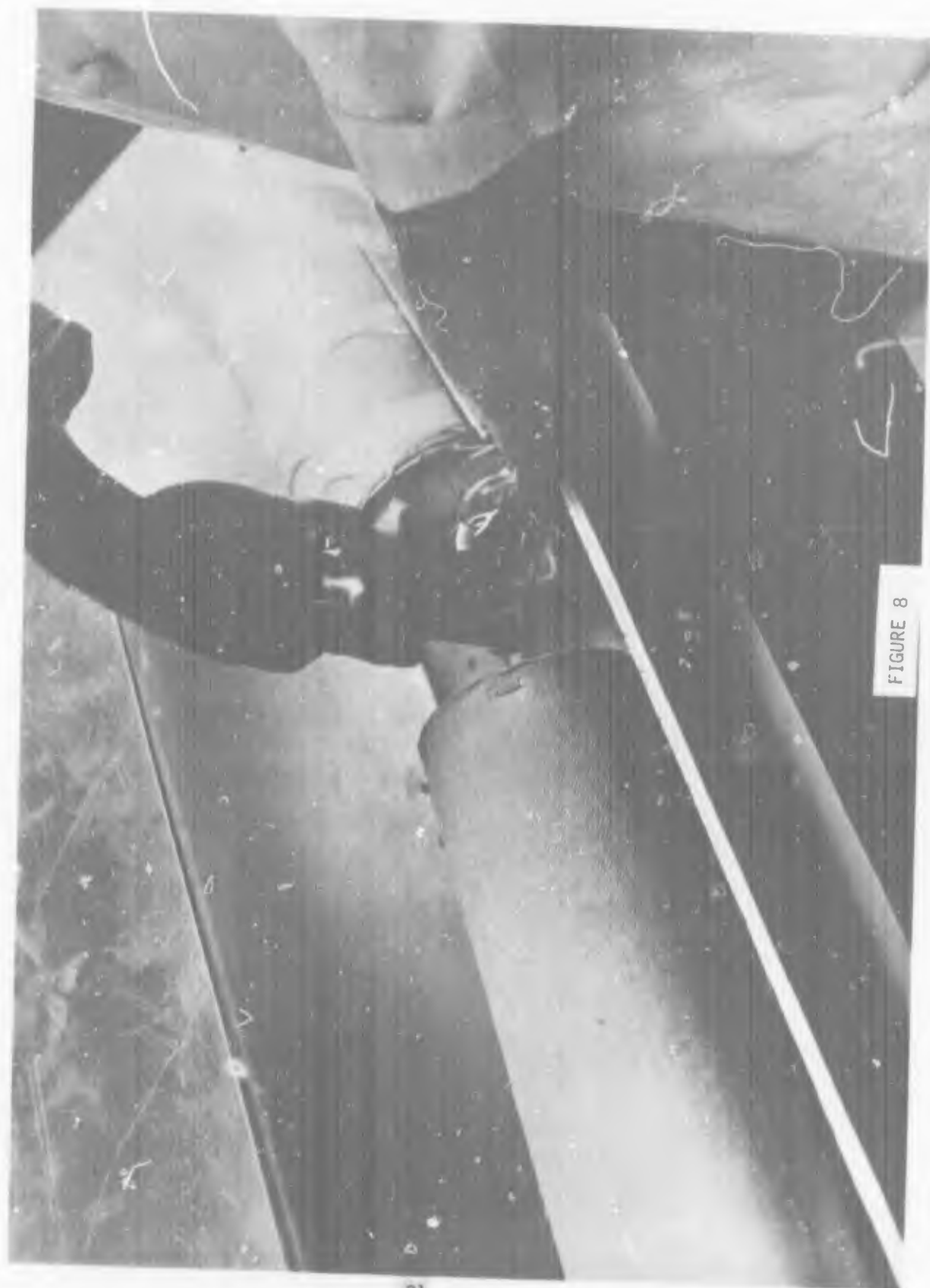


FIGURE 8







FIGURE 11



FIGURE 12



FIGURE 13



FIGURE 14



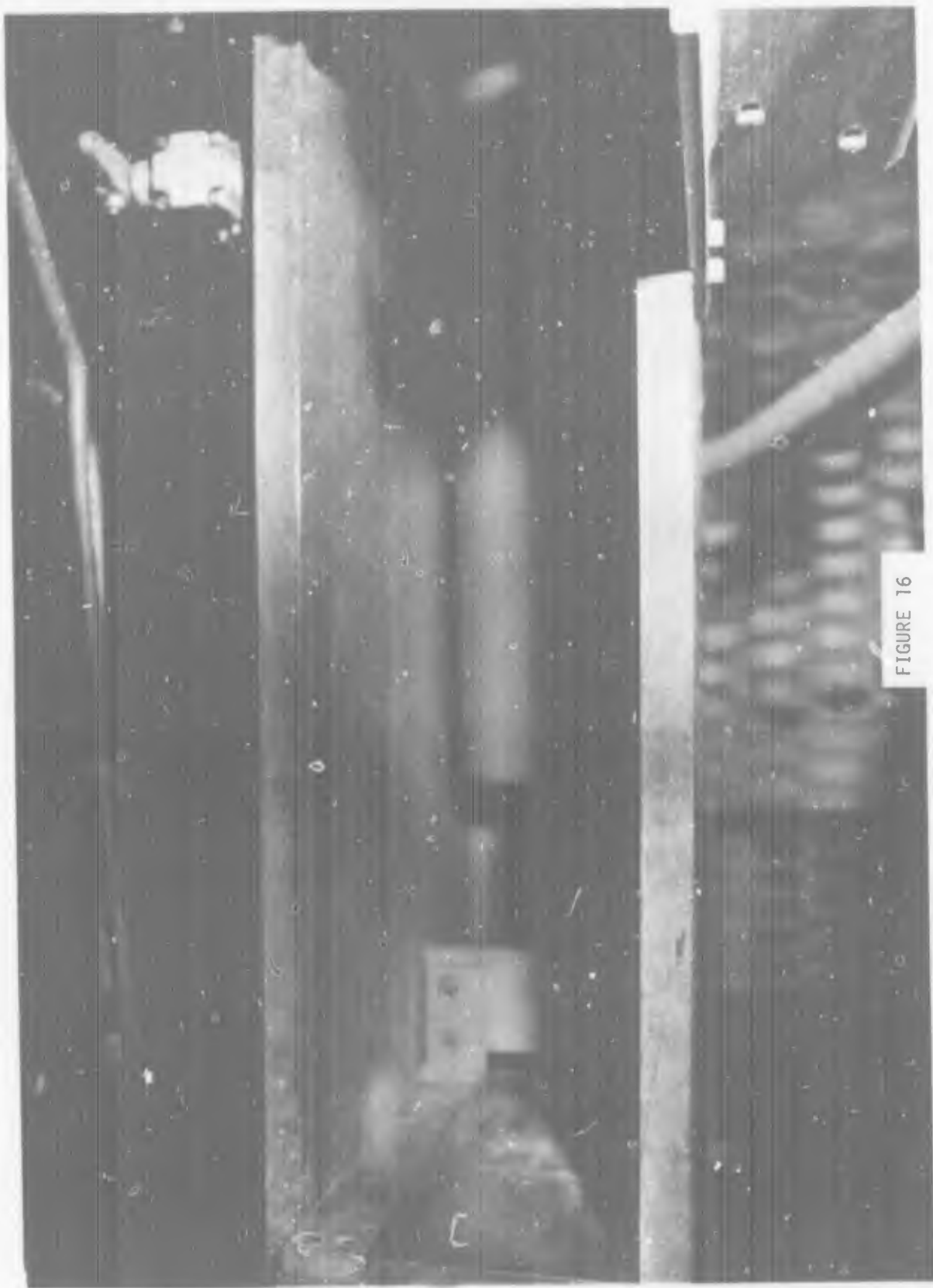


FIGURE 16

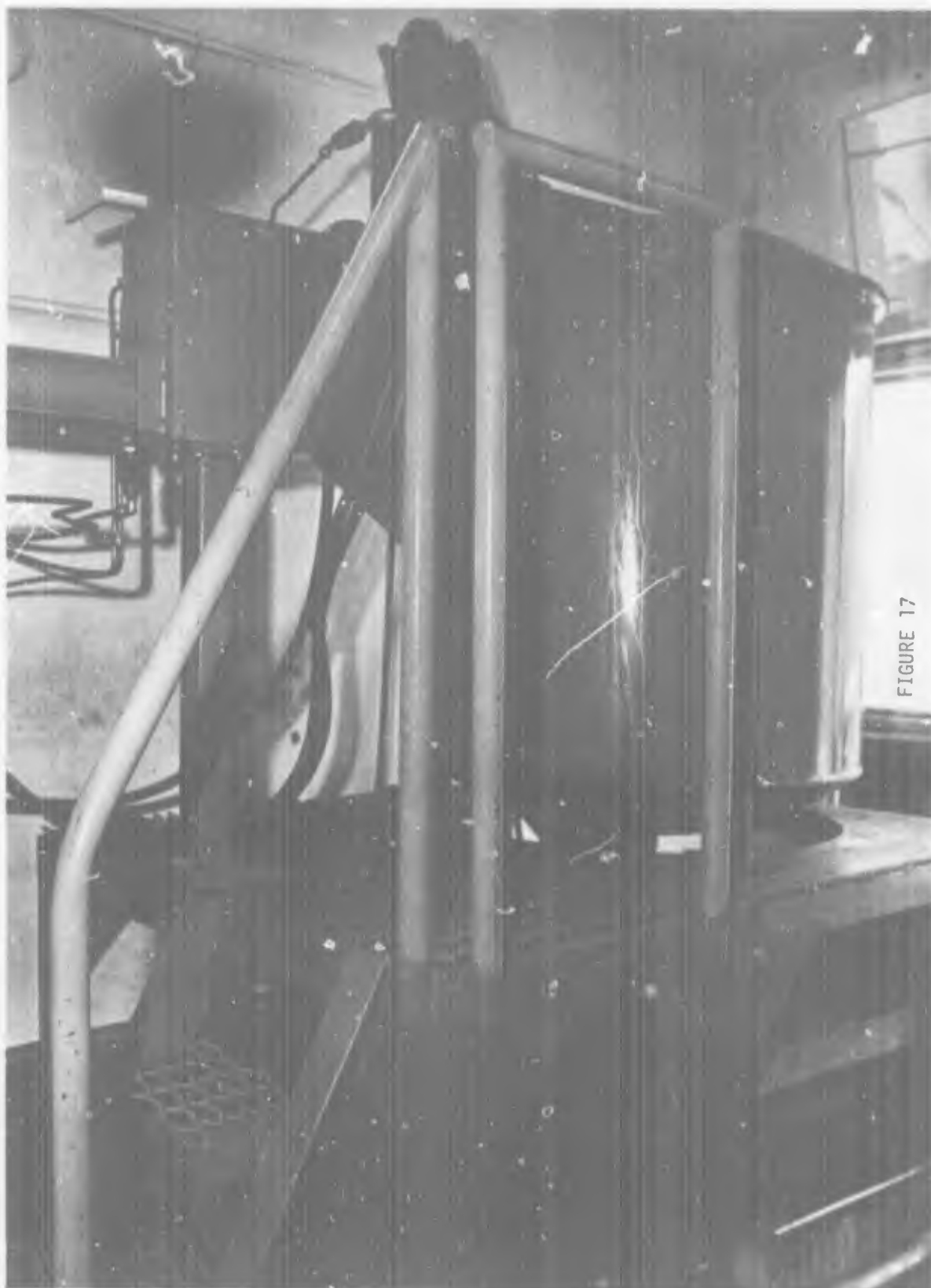


FIGURE 17



FIGURE 18

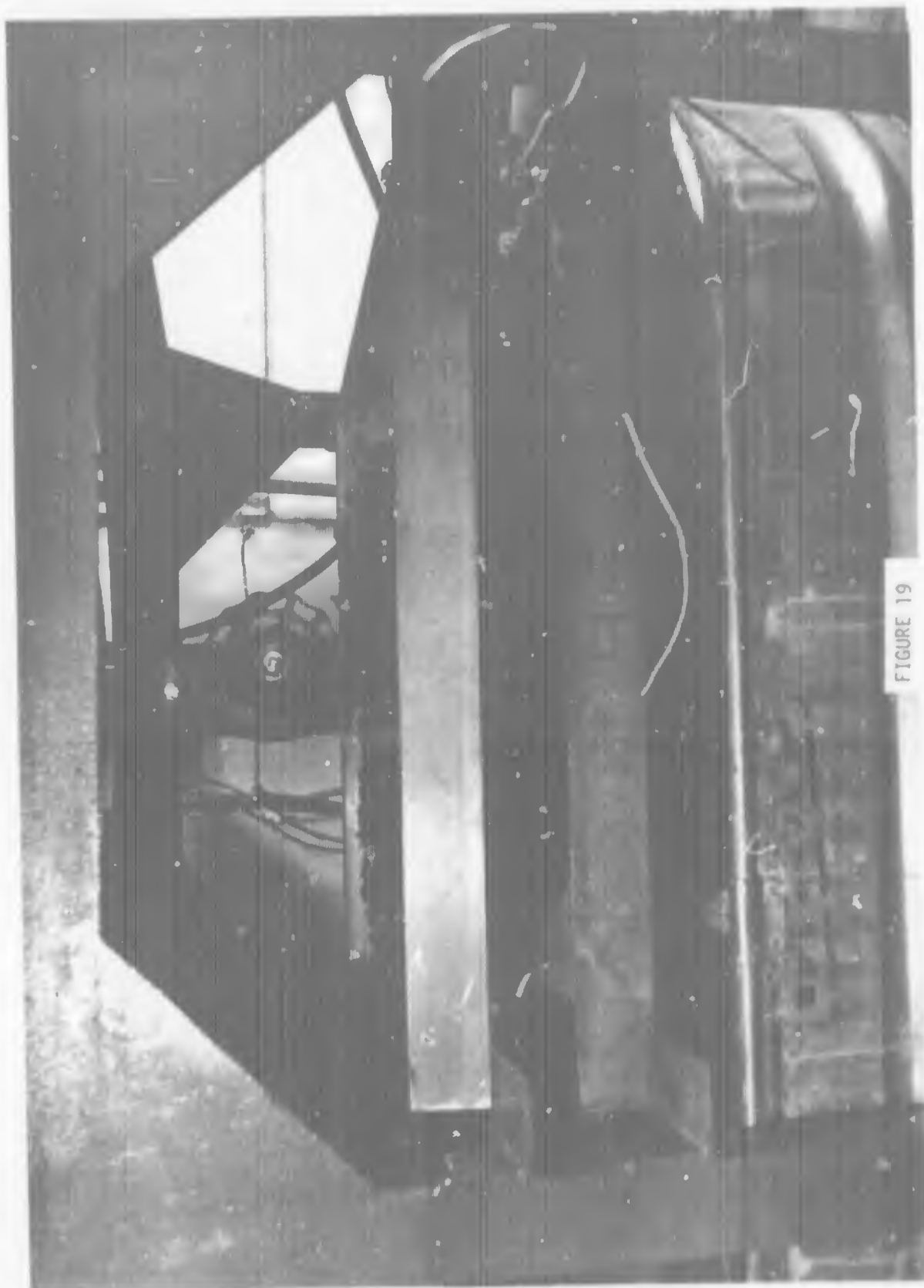
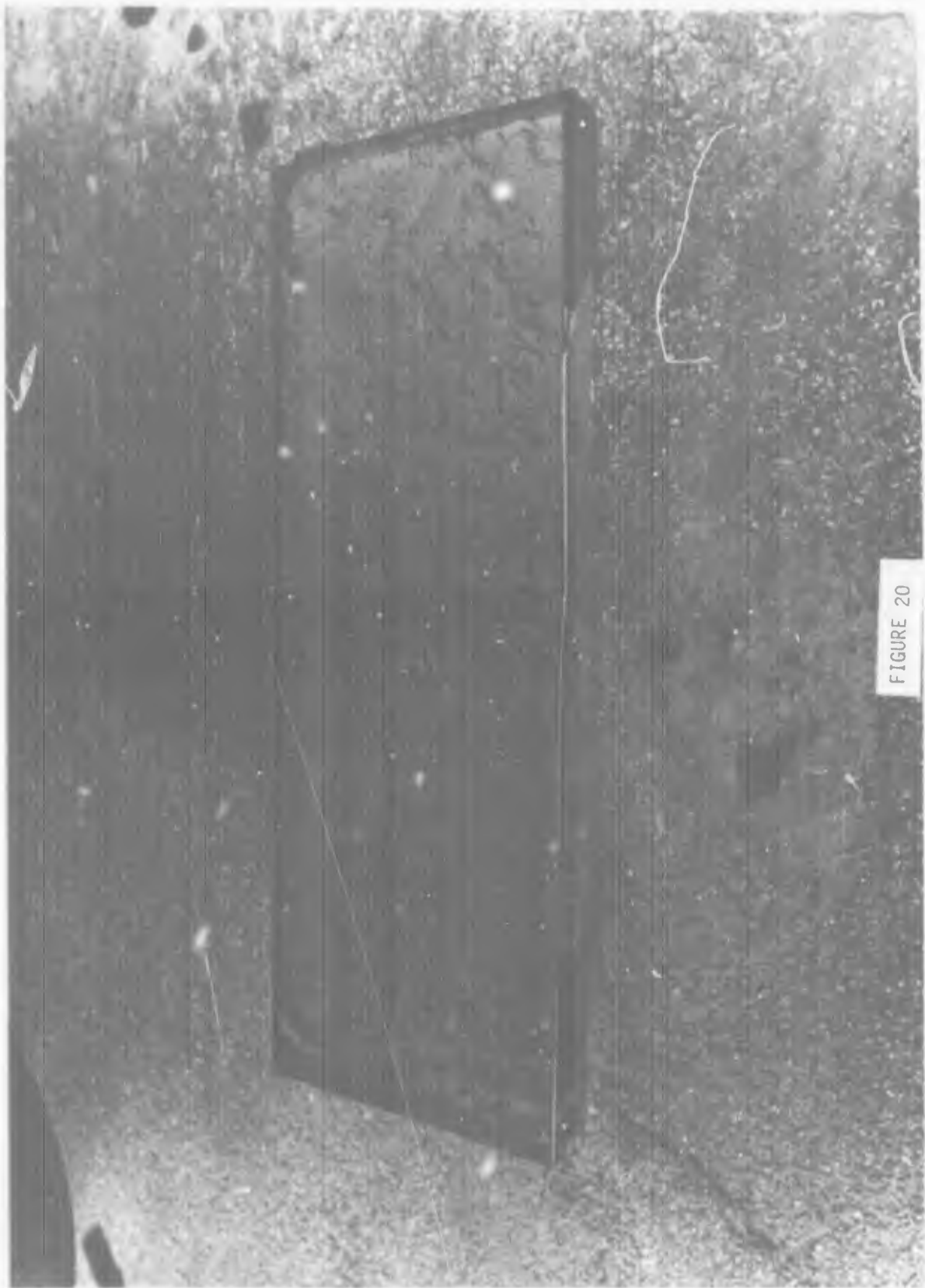


FIGURE 19



BURN TIME FOR CONTROL AND RECLAIMED MAGNESIUM

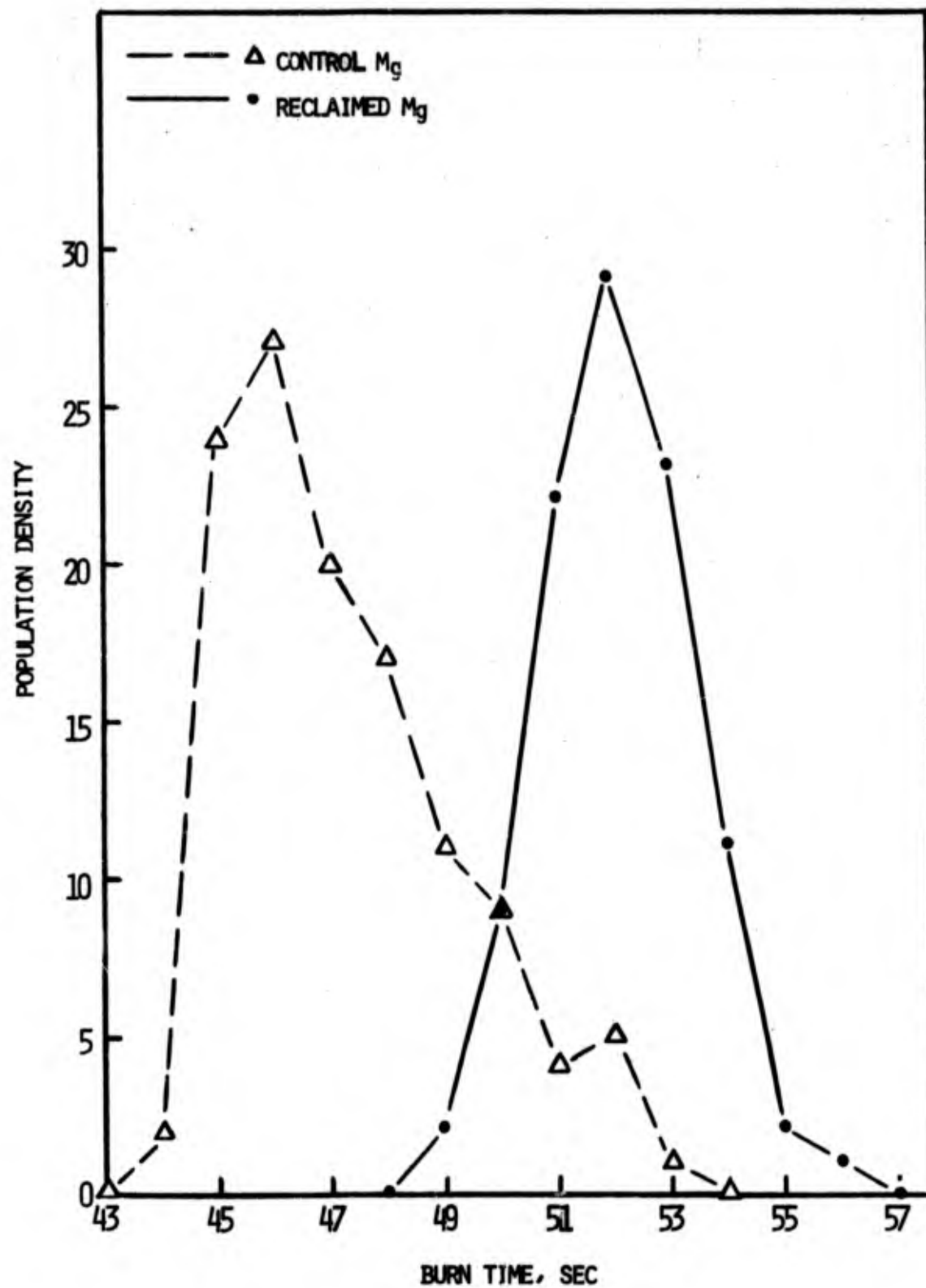


FIGURE 21

LUMINOUS INTENSITY FOR CONTROL AND RECLAIMED MAGNESIUM

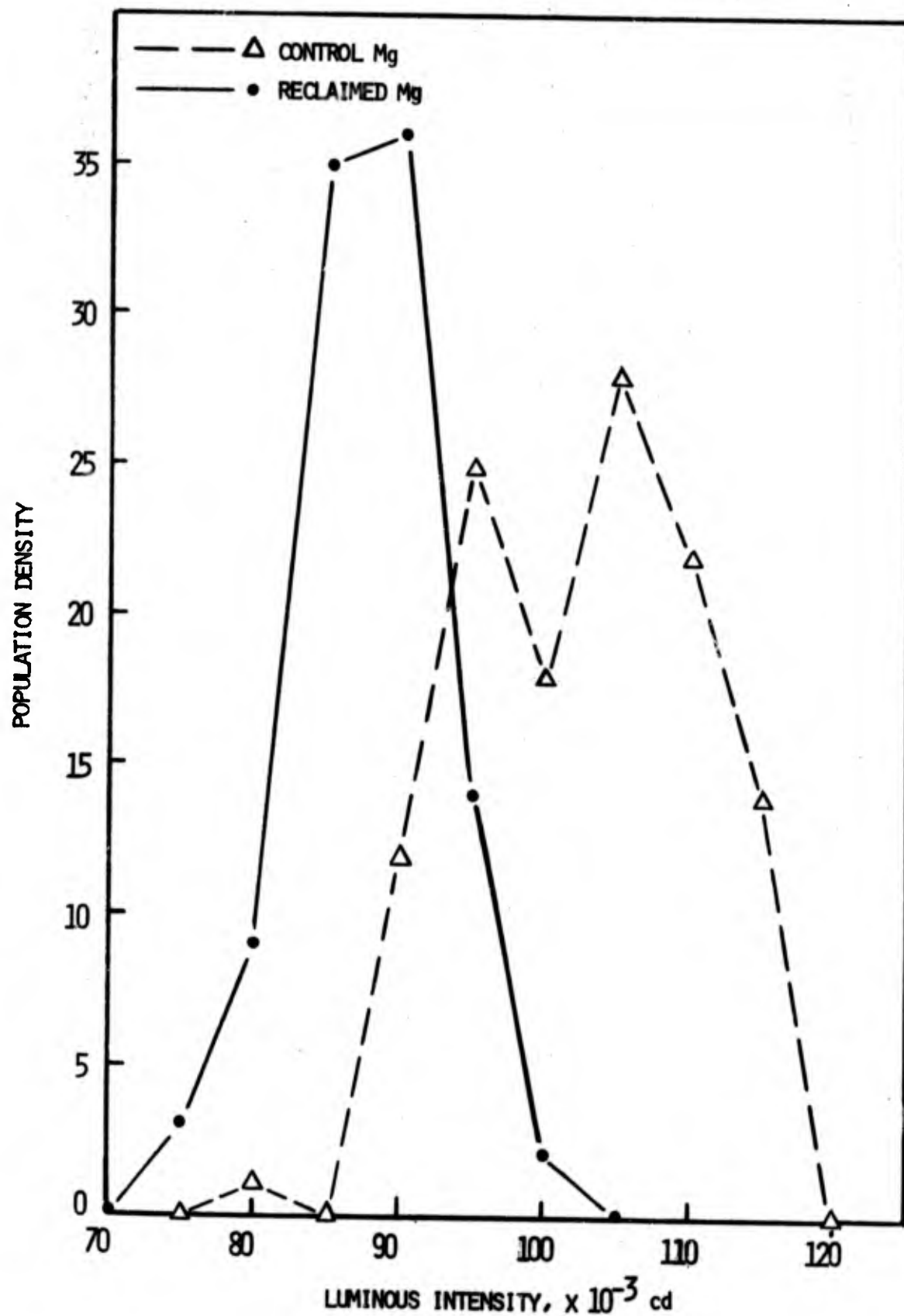


FIGURE 22

EFFICIENCY FOR CONTROL AND RECLAIMED MAGNESIUM

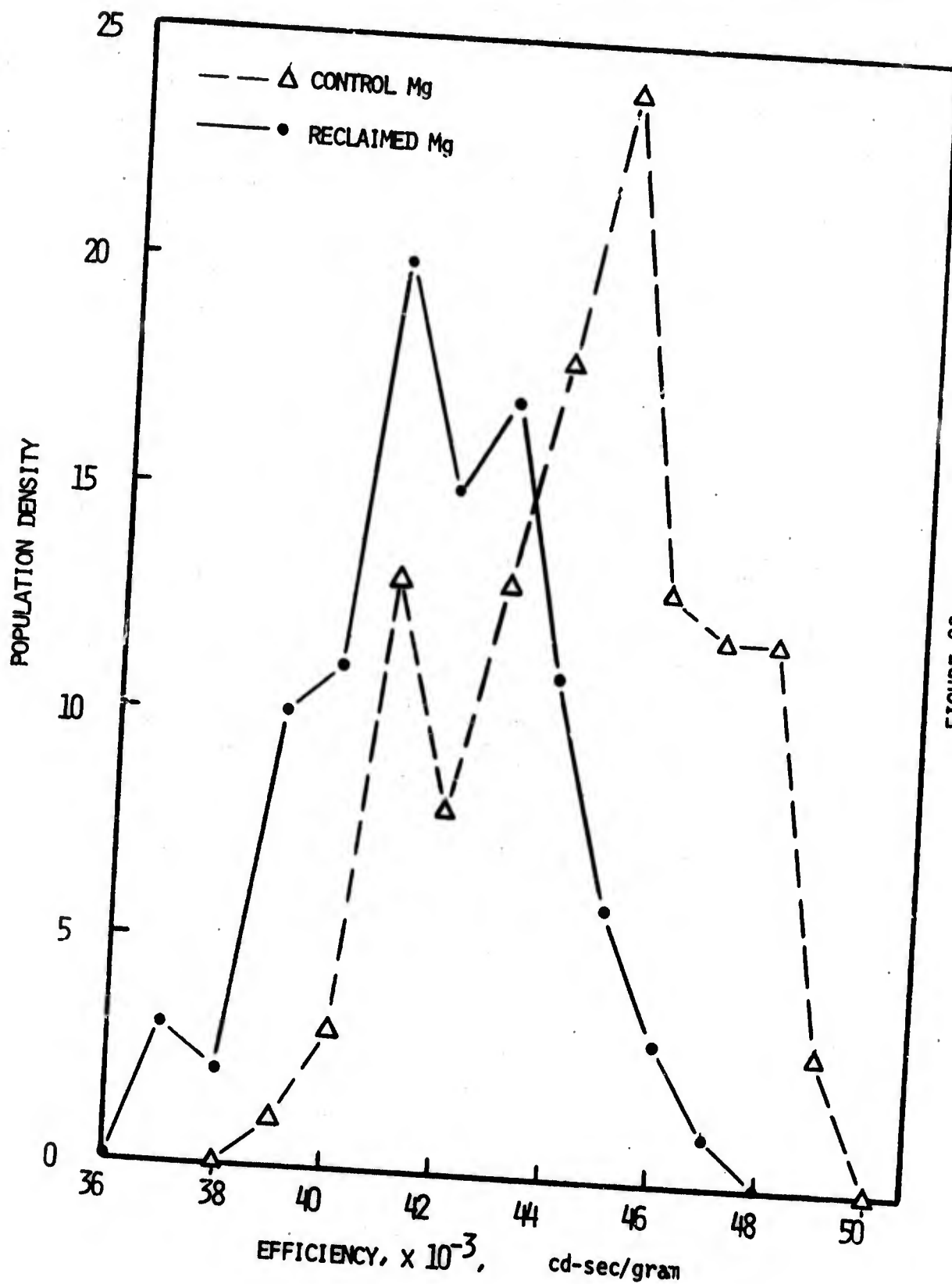


FIGURE 23