

AD732351

COPY NO. 51



TECHNICAL MEMORANDUM 2022

MOISTURE SORPTION
OF
LEAD BETA RESORCYLATE SALTS

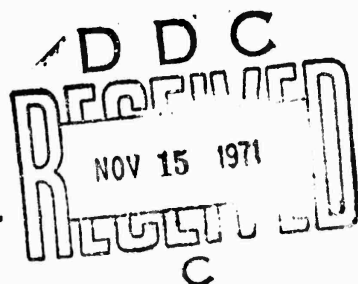
BY
DANIEL R. SATRIANA

OCTOBER 1971

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED.

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
Springfield, Va 22151

PICATINNY ARSENAL
DOVER, NEW JERSEY



51

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D		
<i>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</i>		
1. ORIGINATING ACTIVITY (Corporate author)		2a. REPORT SECURITY CLASSIFICATION
Picatinny Arsenal, Dover, New Jersey		Unclassified
		2b. GROUP
3. REPORT TITLE		
MOISTURE SORPTION OF LEAD BETA RESORCYLATE SALTS		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
5. AUTHOR(S) (First name, middle initial, last name)		
Daniel R. Satriana		
6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
October 1971	14	
8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)	
b. PROJECT NO. DA 1M2621302A211	Technical Memorandum 3881 2022	
c. AMCMS Code 5222.11.58500	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d.		
10. DISTRIBUTION STATEMENT		
Approved for public release: distribution unlimited.		
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY	
13. ABSTRACT		
Determination by differential thermal analysis and X-ray spectroscopy, of water sorption capabilities of lead beta resorcyate is described.		

DD FORM 1473
1 NOV 66

REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED

Security Classification

ACCESSION No.	
OPSTI	WHITE SECTION <input checked="" type="checkbox"/>
LOGC	BUFF SECTION <input type="checkbox"/>
UNANNOUNCED	<input type="checkbox"/>
JUSTIFICATION	
BY	
DISTRIBUTION/AVAILABILITY CODES	
DIST.	AVAIL. and/or SPECIAL
A	

The findings in this report are not to be construed as an official Department of the Army position.

DISPOSITION

Destroy this report when no longer needed. Do not return it to the originator.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Lead Beta Resorcyate Dibasic Lead Beta Resorcyate Monobasic Lead Beta Resorcyate Beta Resorcylic Acid						

Technical Memorandum 2022

MOISTURE SORPTION OF
LEAD BETA RESORCYLATE SALTS

By

Daniel R. Satriana

October 1971

Approved for public release: distribution unlimited.

DA Project: 1M262302A211
AMCMS Code: 5222.11.58500

Propellants Division
Feltman Research Laboratories
Picatinny Arsenal
Dover, New Jersey

The citation in this report of the trade names of commercially available products does not constitute official indorsement or approval of the use of such products.

ACKNOWLEDGEMENTS

Thanks are due to Mr. N. Gelber for the chemical determinations, Mr. J. Campisi for the X-ray diffraction data and Mr. E. Jones for the differential thermal analyses.

TABLE OF CONTENTS

	<u>Page No.</u>
Abstract	1
Conclusions	1
Introduction	2
Discussion of Results	2
Experimental	3
Figures 1 - 5	5-9
Distribution List	10

ABSTRACT

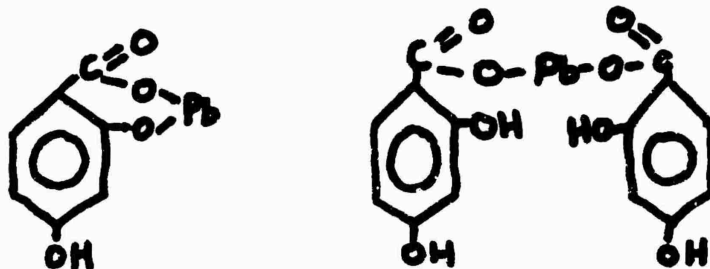
Determination by differential thermal analysis and X-ray spectroscopy, of water sorption capabilities of lead beta resorcyate is described.

CONCLUSIONS

Dibasic lead beta resorcyate (LBR) can be converted to a monohydrate on exposure to moisture. The hydrate has a unique crystal structure that can be distinguished from the anhydrous form by X-ray diffraction. Also, both forms are interconvertible without decomposition.

INTRODUCTION

Two LBR salts are known: the monobasic and the dibasic compounds. Their probable structural formulas are



Monobasic

Dibasic

The method used commercially in their preparation has not been divulged. The commercial material varies between batches, containing impure mixtures of the lead salts. Consequently, it was our objective to synthesize reproducibly pure monobasic and dibasic LBRs. As a result of this investigation, optimum conditions for the preparation of the pure salts were developed¹.

Infrared spectroscopy was one of the techniques used to characterize the pure LBR salts prepared in the laboratory. Spectra of the monobasic and the dibasic salts are shown in Figure 1. Better resolution of the OH and CH stretching frequencies is discernible in the spectrum of the monobasic salt, while the dibasic salt has a broad band at the same 3500-3000 cm^{-1} frequency range. These findings suggested that the difference was probably due to the presence of adsorbed or combined water. In the light of this observation, moisture sorption of the lead salts was studied.

DISCUSSION OF RESULTS

Through the use of differential thermal analysis (DTA) it was found that the thermal characteristics of the two salts are different. It is evident from Figure 2 that the thermogram of the monobasic salt

¹. Picatinny Arsenal Technical Memorandum 2021, dated October 1971.

has no endothermic transition, while the dibasic salt has two endothermic responses, one at 150°C., the other at 200°C. The endotherm appearing at 150°C. suggested either adsorbed moisture or a polymorphic transition.

By employing the following special technique, it was demonstrated that the endotherm at 150°C. was probably due to water of hydration: a sample of the dibasic salt was heated to 175°C. in the Differential Scanning Calorimeter (DSC), then cooled and placed in a humidity chamber for several hours. When the sample was heated again to 175°C. in the calorimeter, the endotherm reappeared. The sample was once more cooled to room temperature, and subsequently reheated to 175°C. This time the endotherm did not appear. However, when the sample was placed in the humidity chamber for several hours and again heated to 175°C., the endotherm reappeared. The results of this experiment are graphically shown in Figure 3.

Confirmation of the hydrate form of the dibasic lead salt was also obtained by X-ray diffraction. X-ray diffraction patterns were taken of a sample of the dibasic salt dried at 100°C. for 24 hours, and of the same sample after being in a humidity chamber at 90% RH for 72 hours. The spectra are shown in Figure 4. The changes in the major diffraction peaks signify differences in crystal structure. Evidently, these changes were due to the formation of the hydrate. The X-ray diffraction pattern of the monobasic lead salt is also included for purposes of comparison.

Thermogravimetric analysis (TGA) was used to determine the extent of hydration. The thermal decomposition of a sample of the dibasic salt is shown in Figure 5. The loss in weight was stepwise, the initial step corresponding to the endotherm at 150°C. of the DTA curve (Figure 2) and the second step to the endotherm at 200°C. It is evident that the loss in weight due to the decomposition of the hydrate is approximately 3.5%, which corresponds closely to the theoretical value (3.4%) for the monohydrate.

EXPERIMENTAL

X-Ray Diffraction

A Norelco wide angle diffractometer utilizing $\text{CuK}\alpha$ X-radiation was used to obtain X-ray diffraction patterns.

Differential Analysis

The 950 DuPont Gravimetric Analyzer and the Differential Scanning Cell for a 900 DuPont Differential Analyzer were used to measure thermal properties.

Infrared Analysis

Infrared spectra were obtained by means of the KBr pellet technique with a Perkin-Elmer, Model 621, Grating Infrared Spectrophotometer. The pellets, containing approximately 0.5% sample, were pressed to 0.7mm thickness.

Laboratory Preparation of Lead Beta Resorcyates

1. Monobasic Lead Beta Resorcyate

11.16g (0.05 moles) of lead monoxide (PbO) is added to 100 ml of absolute ethyl alcohol containing 16.96g (0.11 moles) of practical grade beta resorcylic acid. The mixture is heated at 60-70°C. for 1.5 hours with constant stirring. The product is filtered while still warm, washed twice with a small amount of warm (60°C.) absolute alcohol, and dried in the oven at 60°C. for several hours. The weight of the monobasic salt is 16.1g (theory 18.0g) or 89.5% yield based on the amount of PbO used in the reaction. Analysis: Found-Pb-57.29%, C-23.70%, H-0.18%; Calculated - Pb-57.66%, C-23.40%, H-1.12%.

2. Dibasic Lead Beta Resorcyate

11.16g (0.05 moles) of lead monoxide (PbO) is added to 175 ml of 50% (by volume) of ethyl alcohol containing 23.1g (0.15 moles) of practical grade beta resorcylic acid. The mixture is stirred at ambient temperature under a current of air for five hours. The solid is filtered, washed with ice cold 50% ethyl alcohol, and dried in the oven at 100°C. to constant weight. The yield of pure dibasic lead beta resorcyate is 23.8g (theory 25.7g) or 92.5% yield based on the amount of PbO used in the reaction. Analysis: Found - Pb-40.54%, C -32.85%, H-2.10%; Calculated - Pb-40.35%, C-32.75%, H-1.96%.

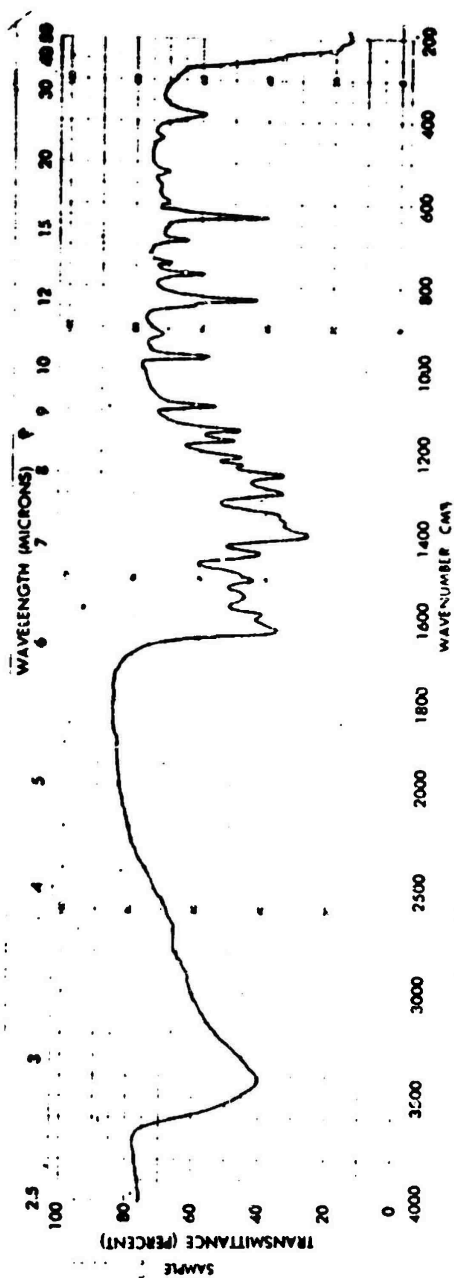
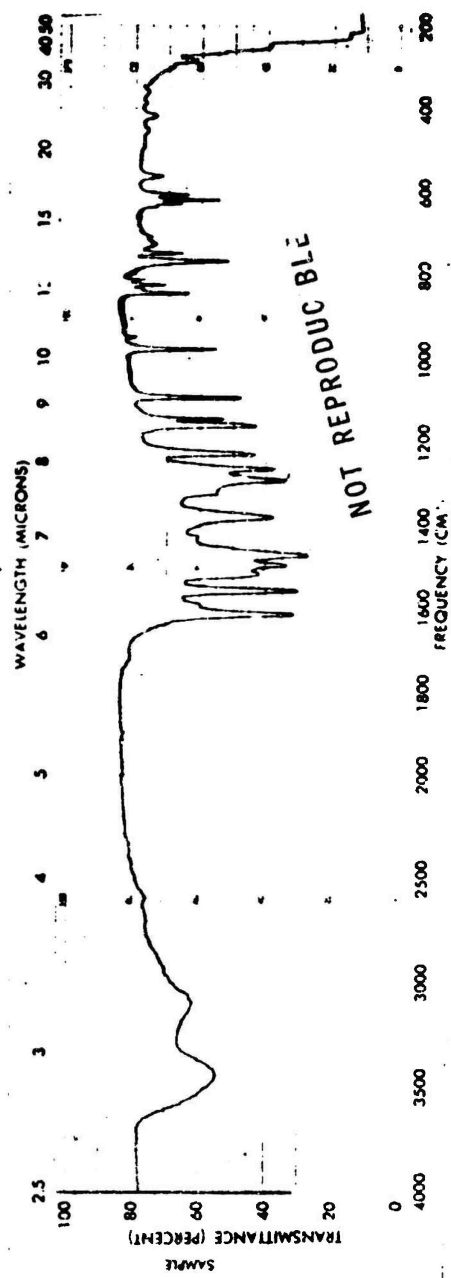


Fig. 1 Top - I.R. Spectra of Monobasic Lead Beta-Resorcylate
 Bottom - I.R. Spectra of Dibasic Lead Beta-Resorcylate

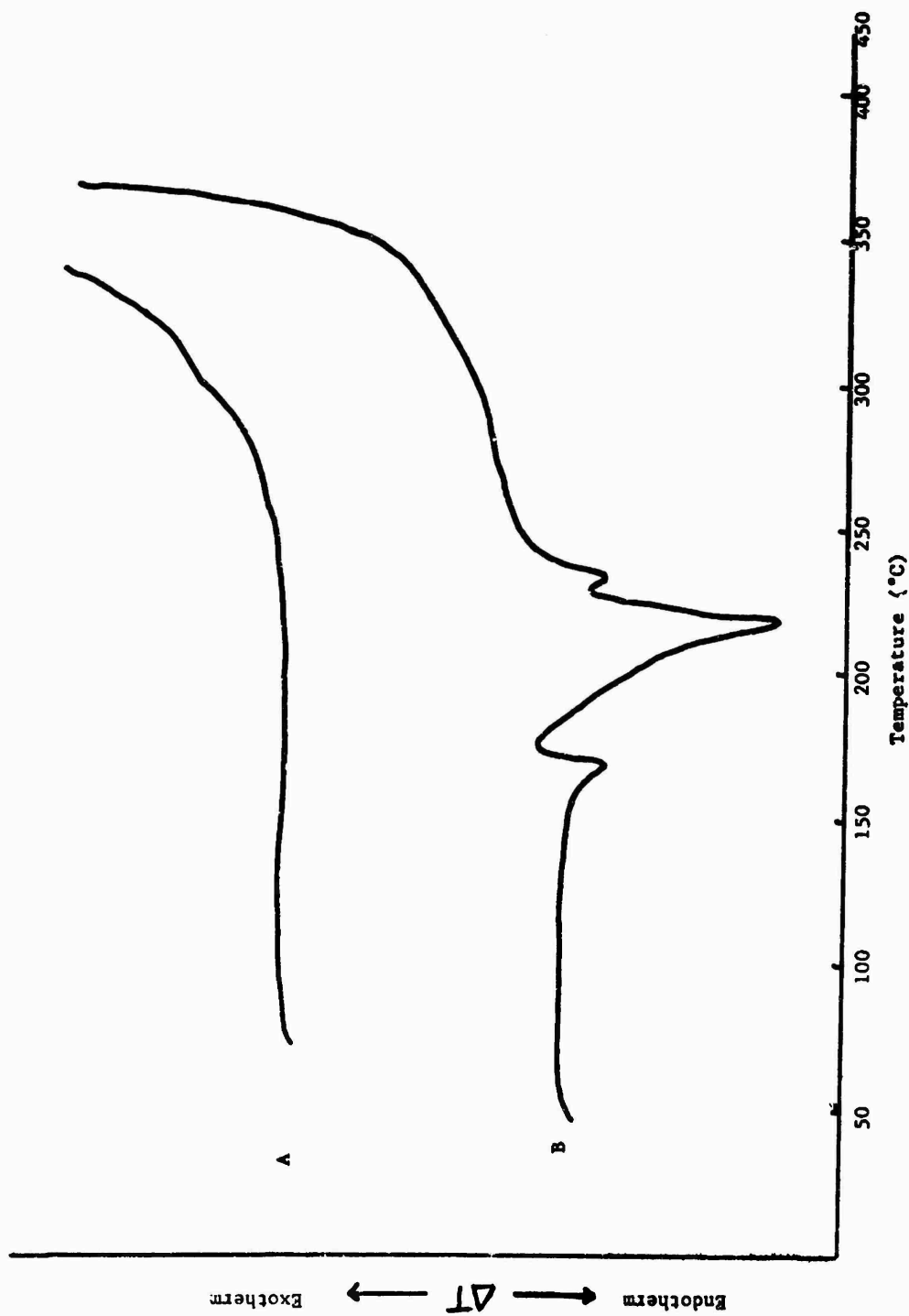
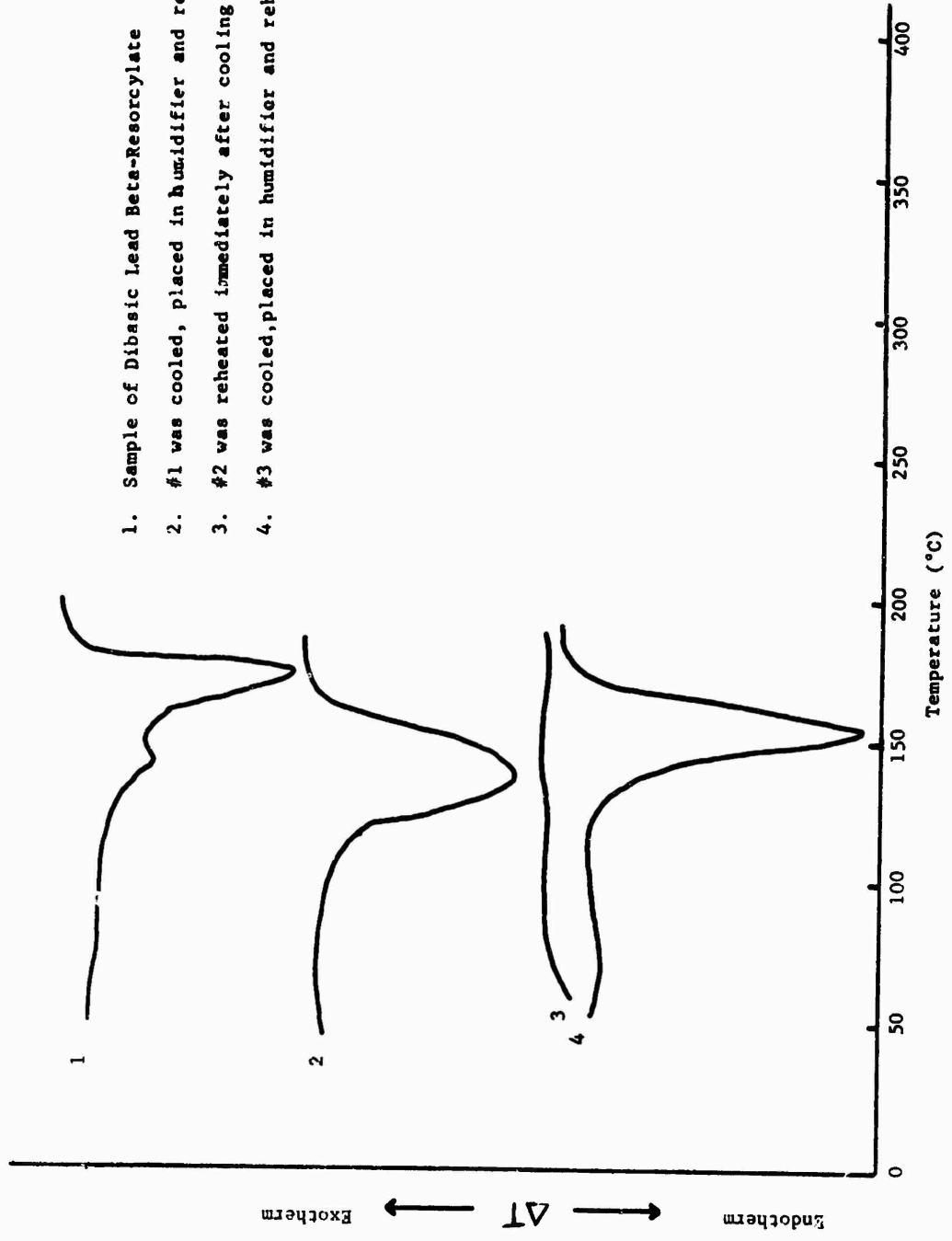


Fig. 2 A - Thermogram of Monobasic Lead Beta-Resorcylate
 B - Thermogram of Dibasic Lead Beta-Resorcylate



1. Sample of Dibasic Lead Beta-Resorcyate
2. #1 was cooled, placed in humidifier and reheated
3. #2 was reheated immediately after cooling
4. #3 was cooled, placed in humidifier and reheated

Fig. 3 Results of Experimental Treatment of Dibasic Lead Beta Resorcyate Salt In Differential Scanning Calorimeter

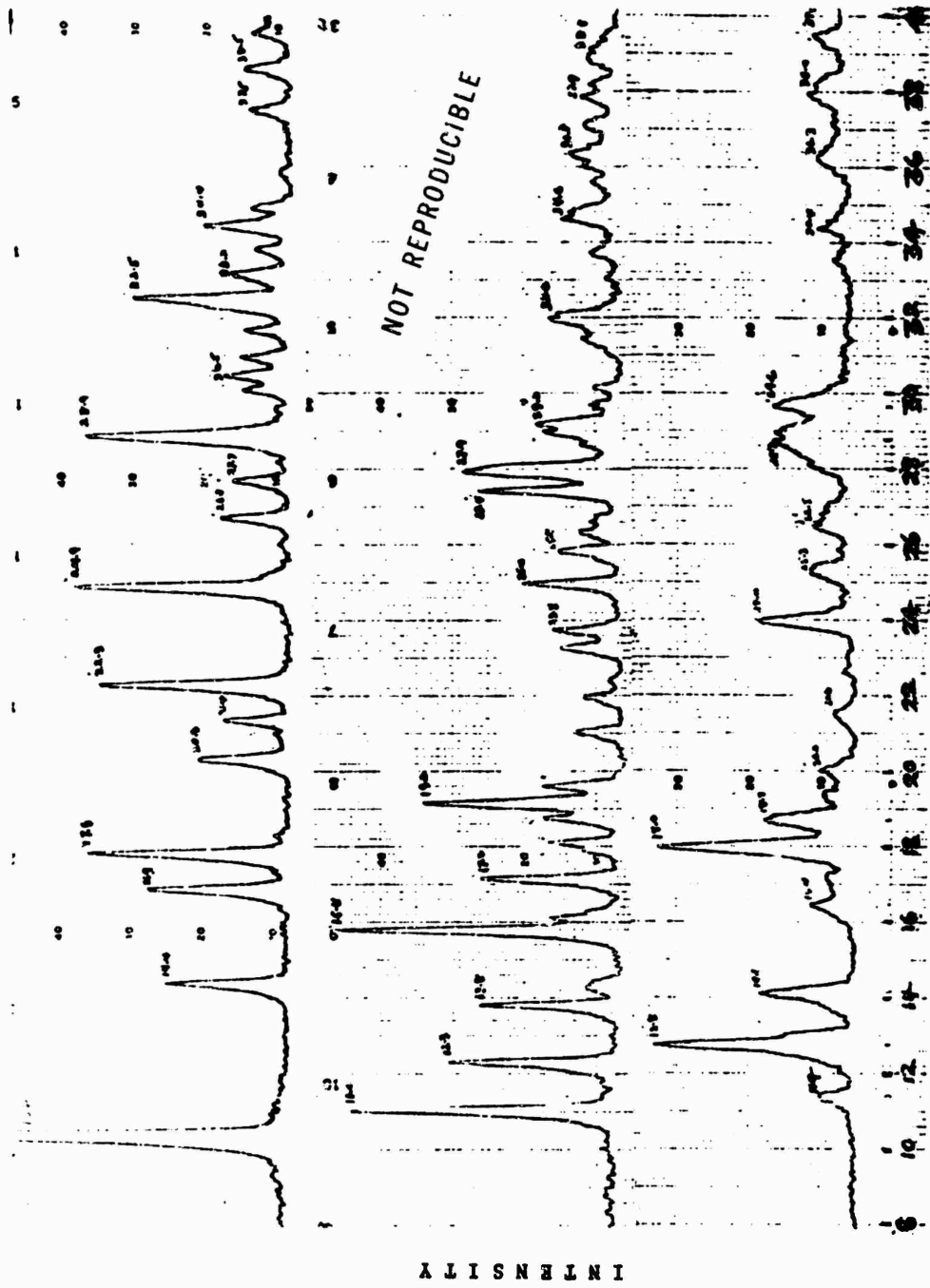


Fig. 4 Top - X-Ray diffraction pattern of monobasic lead beta-resorcylate
 Middle --X-Ray diffraction pattern of dibasic lead beta-resorcylate (72 hrs at 90°C RH)
 Bottom - X-Ray diffraction pattern of dibasic lead beta-resorcylate (dried at 100°C, 24 hrs)

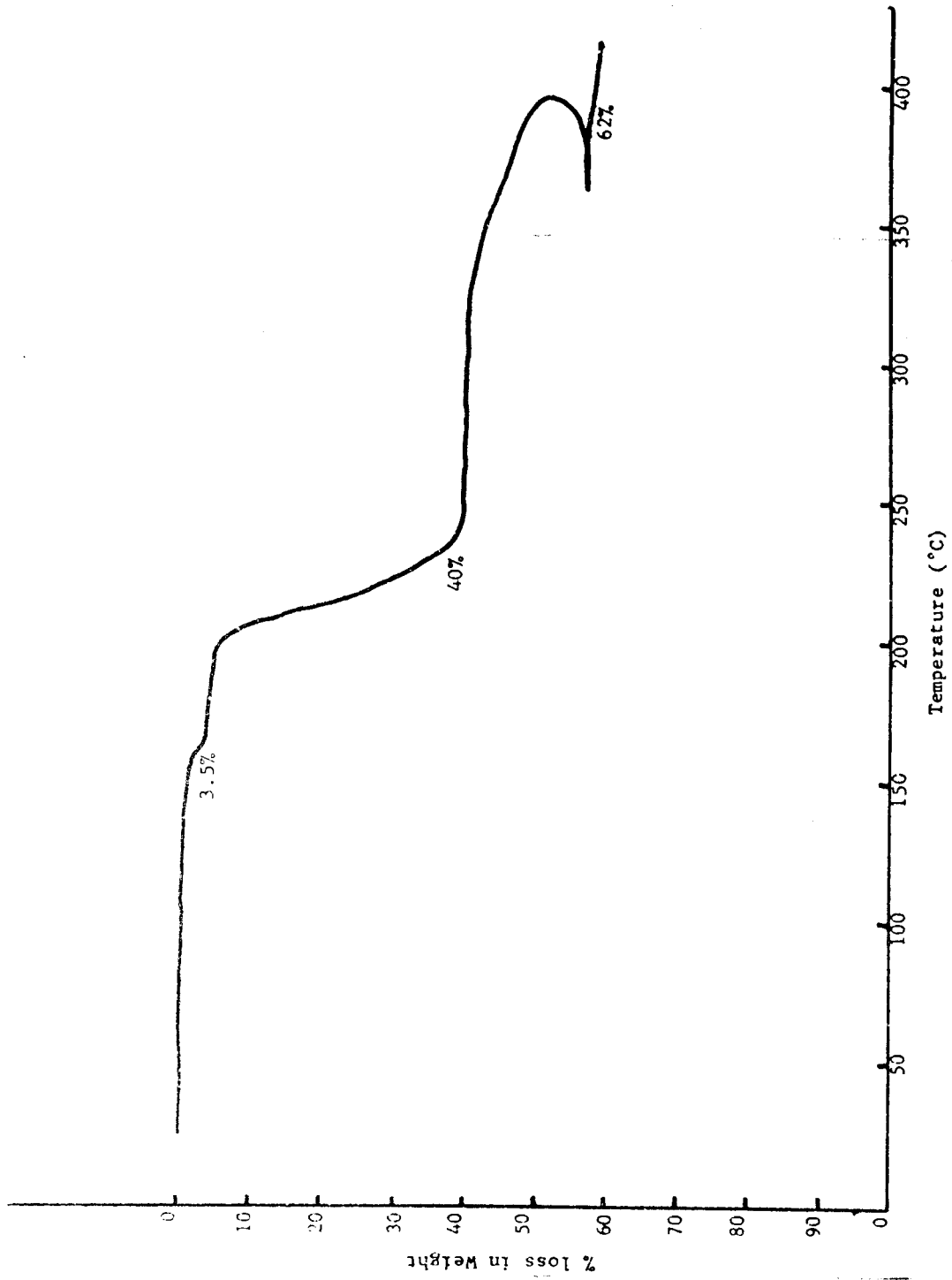


Fig. 5 Thermogravimetric Analysis of Dibasic Lead Beta-Resorcylate