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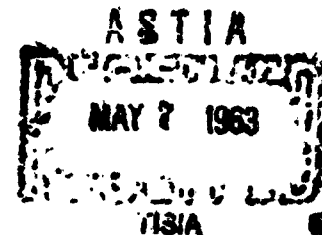
**RESEARCH ON A SUITABLE DISPERSING
OR ANALYZING CRYSTAL FOR X-RAY
SPECTROSCOPY**

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-63-311

April 1963

CATALOGED BY ASTIA
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Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio



Project No. 7364, Task No. 736405

(Prepared under Contract No. AF 33(657)-7756 by the
Isomet Corporation, Palisades Park, New Jersey; Warren
Ruderman, Edward Losin and Dudley Uphoff, authors.)

Aeronautical Systems Division, Dir/Materials
and Processes, Physics Lab, Wright-Patterson
AFB, Ohio
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X-RAY SPECTROSCOPY. Final report, Apr 63,
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Unclassified Report

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to 90A were developed and show exceptional
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FOREWORD

Work reported here was performed at Isomet Corporation, Palisades Park, New Jersey, from 15 December 1961 through 14 December 1962 by Dr. Edward T. Losin, Mr. Dudley Uphoff, Mr. George Ball, Mr Michael Zylberberg and Miss Rosanne Saul, under Contract No. AF33 (657)-7756 with the Directorate of Materials and Processes, Aeronautical Systems Division. The work was in support of Project No. 7364, Task No. 736405. Mr. David Fischer, Physics Laboratory, Analytical Branch, served as Contract Monitor.

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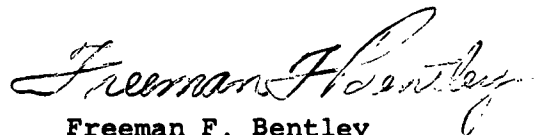
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ABSTRACT

A large number of potential compounds were investigated for application as X-ray analyzer crystals with $2d$ spacings from 25 to 100A. Four crystals with $2d$ values from 31 to 90A were developed and show exceptional promise. Although large crystals of these compounds were not grown, X-ray analysis of small crystals indicated excellent reflectivities.

This technical documentary report has been reviewed and is approved.



Freeman F. Bentley
Chief, Analytical Branch
Physics Laboratory
Materials Central

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1. INTRODUCTION

X-ray fluorescence spectroscopy has become a powerful analytical tool for the detection of trace quantities of the elements. In this technique primary X-rays from a target strike the sample and generate the characteristic X-rays of the elements present. The sample may be a solid or a liquid. A collimator selects a parallel bundle of secondary X-rays and directs them to an analyzer crystal. For any setting of this crystal only X-rays of wavelength will satisfy Bragg's law [$n\lambda = 2d \sin \theta$] and will be diffracted so as to reach the detector. Here n is the order of the diffraction, d is the interplanar spacing of the crystal in Angstroms, and λ is the angle between the radiation incident upon the analyzer crystal and the plane of the crystal. As the analyzer crystal is slowly rotated, the various lines of the elements are detected and recorded by the detector. The wavelengths of the lines determine the elements present in the specimen, and the intensities are a measure of the quantity of each element.

The analysis for light elements by the X-ray fluorescence technique has been limited by means for analyzing the ultrasoft secondary radiation. Potassium acid phthalate which was recently developed by Isomet Corp., has the largest $2d$ spacing - 26.4A - of any commercially available analyzer crystal. It is desirable to develop crystals with $2d$ spacings from 25 to 100A. These crystals would be useful in detecting the K-spectra of light elements such as boron, carbon, nitrogen, oxygen and fluorine, and the L and M spectra of the heavier elements such as iron, manganese, chromium, vanadium and titanium. The L and M spectra would provide useful information on the bonding and electronic structure of the atoms.

Three different techniques have been used for analyzing soft X-radiation. Low incidence, ruled gratings have been used successfully to disperse ultrasoft X-rays, but good signal-to-noise ratios are not achieved for wavelengths below approximately 100A. Conventional single crystals such as gypsum, EDDT, have been used for the detection of X-rays up to about 15A. Two dimensional soap film analyzers have been used with some success for wavelengths greater than 15A. These film analyzers are prepared (1,2) by depositing successive monolayers of soap onto the surface of a plate. The typical soap that has been used is barium stearate.

Manuscript released by the authors February 1963 for publication as an ASD Technical Documentary Report.

1. T.C. Furnas, Jr., and E. W. White, WADD Technical Report 61-168, May 1961.
2. ASD-TDR-62-519, August 1962.

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Single crystals are believed to offer the best possibility for high resolution analysis of ultrasoft X-rays in the region of 25 to 100Å.

A good analyzer crystal should have the following characteristics:

1. Stable to the atmosphere
2. Very low vapor pressure, so that it will be stable in a vacuum tunnel
3. Melting point greater than 50°
4. 2d spacing from 25 to 100Å
5. Good reflectivity for ultrasoft X-rays
6. Good mechanical strength
7. Good cleavage properties in a plane perpendicular to the direction of maximum 2d spacing. This is not essential but is a very useful property
8. Ease of preparation
9. Good crystal growth characteristics
10. Low absorption of the incident X-rays

The development of new analyzer crystals was carried out in the following stages:

1. Literature Survey. The literature was searched for compounds having large lattice spacings. Such references as Chemical Abstracts, Wyckoff and Landolt-Bornstein were reviewed for possible compounds. Only if the space group and lattice constants were known could the maximum 2d spacing be determined. In many cases this information was not available, and the other properties of the compound under consideration were examined to determine if it appeared worthwhile to purify or synthesize the compound and obtain X-ray data. Since this involved considerable work, only very promising compounds were so treated.

2. Preparation of Materials. In some instances the compound could be obtained commercially, and it was then purified and small crystals prepared by slow evaporation from a saturated solution. More often, particularly after the initial group of materials from the literature had been examined, new compounds were postulated that were not commercially available, or had not been synthesized before. In these cases the compounds were synthesized, purified and small crystals prepared from solution.

3. X-Ray Evaluations. Powder diffraction patterns were obtained for promising compounds using $\text{CuK}\alpha$ radiation. It was thus possible to determine the largest 2d spacing and to get some information about the relative intensity of the reflection. Admittedly,

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it would be better to use ultrasoft radiation to make the measurements, but the necessary equipment was not available. The powder technique enabled us to make many measurements in a relatively short time.

4. Growth of Large Crystals. Most of the effort went into finding crystals having suitable properties. There was neither enough time nor funds to grow large crystals of the compounds that were found to have exceptional promise.

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2. CHOICE OF COMPOUNDS

Tables 1, 2 and 3 list the compounds that were studied. Table 1 gives the compounds that proved to be unsuitable or not particularly promising. The principal reasons for discontinuance were:

1. A shorter 2d spacing than anticipated was found. This may have been the result of an absence of a [100] reflection or, in the case of new compounds with no known literature data, a shorter 2d spacing than hoped for.
2. Poor X-ray reflectivity.
3. Poor crystallinity. Contributing factors were absence of good solvents; tendency to form powdery or tiny crystals; formation of multiple crystals; formation of soft crystals which curled or bent easily; or formation of an oil which could not be crystallized.
4. Instability. Some compounds were found to be unstable in the atmosphere.

The fatty acids, while having satisfactorily long 2d spacings, were found to be very difficult to grow. Small flat thin plates could be prepared, but no success was obtained in growing larger crystals. The salts of the fatty acids could not be dissolved in solvents, and even small crystals could not be obtained.

The major effort was concentrated on:

1. Compounds analogous to potassium acid phthalate.
2. Amides of long-chain hydrocarbon acids.
3. Half potassium salts of dibasic acids.
4. Diesters of dibasic acids.

Large crystals of various acid salts of phthalic acid were prepared for use by the Contract Monitor in studying the effect of the cation on the X-ray reflectivity. The ammonium, potassium, sodium, rubidium and cesium acid phthalates were prepared. The 2d spacing is virtually the same ($\sim 26\text{\AA}$) for all of these compounds.

TABLE 1

LIST OF CRYSTALS ON WHICH WORK WAS DISCO

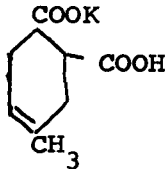
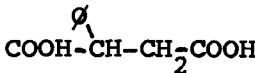
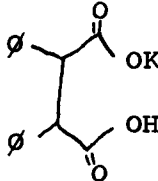
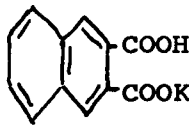
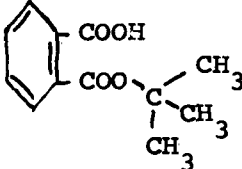
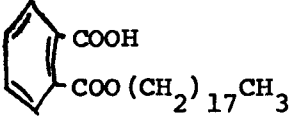
<u>Compound</u>	<u>Formula</u>	<u>M. P.</u>	<u>Maximum 2θ Spacing, Å</u>
Half K Salt of 4-Methyl-4-Cyclohexene-1,2-Dicarboxylic Acid		-	28.4
Hexadecanedioic Acid	$\text{HOOC}(\text{CH}_2)_{14}\text{COOH}$	-	35
α -Phenylsuccinic Acid		-	20
Half K Salt of α, β -Diphenylsuccinic Acid		-	48 Also 23.6
Potassium Acid Adipate	$\text{HOOC}(\text{CH}_2)_4\text{COOK}$	-	21
Potassium Acid Glutarate	$\text{HOOC}(\text{CH}_2)_3\text{COOK}$	-	18
Potassium Acid Sebacate	$\text{HOOC}(\text{CH}_2)_8\text{COOK}$	-	22.4
Half K Salt of Naphthalene-2,3-Dicarboxylic Acid		-	26
Tert-Butyl Hydrogen Phthalate		-	-
Octadecyl Hydrogen Phthalate		70	-
dl-Methionine	$\text{CH}_3\text{S}(\text{CH}_2)_2\text{CH}(\text{NH}_2)\text{COOH}$	283	31.6

TABLE 1

OF CRYSTALS ON WHICH WORK WAS DISCONTINUED

	<u>M. P.</u>	<u>Maximum 2d Spacing, A</u>	<u>Reflectivity</u>	<u>Crystallinity</u>	<u>Remarks</u>
COOH	-	28.4	Medium	Poor	
COOH	-	35	Medium	Poor	
COOH	-	20	Low	Fair	
	-	48 Also 23.6	Very Low Strong	Fair	
OOK	-	21	Fair	Good	
OOK	-	18	Medium	Excellent	
OOK	-	22.4	Good	Fair	
OOH	-	26	Very Low	Good	
OOK	-	-	-	Very Poor	Only oil obtained
CH ₃ H ₃	70	-	-	Poor	
) 17 ^{CH₃}					
(NH ₂) COOH	283	31.6	Strong	Poor	Strong tendency for multiplicity



TABLE 1

(CONTINUED)

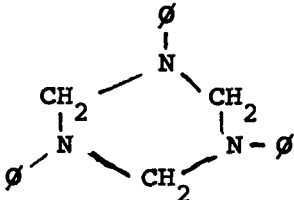
<u>Compound</u>	<u>Formula</u>	<u>M. P.</u>	<u>Maximum 2d Spacing, A</u>
Hexatriacontane	$C_{36}H_{74}$	95	95 (Literature)
S-Triphenyltriazine		140	71.7 (Literature)
Eicosanoic Acid	$CH_3(CH_2)_{18}COOH$	76	90
Docosanoic Acid	$CH_3(CH_2)_{20}COOH$	80	105
Docosanoamide	$CH_3(CH_2)_{20}CONH_2$	113.5	90
Stearamide	$CH_3(CH_2)_{16}CONH_2$	105	72
Half K Salt α -Dodecylsuccinic Acid	$KOOC-CH-CH_2-COOH$ $C_{12}H_{26}$	-	64.8
Potassium Acid Terephthalate		-	19

TABLE 1
(CONTINUED)

<u>la</u>	<u>M. P.</u>	<u>Maximum 2d Spacing, A</u>	<u>Reflectivity</u>	<u>Crystallinity</u>	<u>Remarks</u>
	95	95 (Literature)	-	-	Unable to find suitable solvent
	140	71.7 (Literature)	-	-	Unstable in air
CH ₂ N-Ø					
OH	76	90	Medium	Very Good	{ Crystals are soft, difficult to grow, thick sections
OH	80	105	Medium	Good	
NH ₂	113.5	90	Medium	Fair	
NH ₂	105	72	Strong	Fair	
OH	-	64.8	Low-Medium	Poor	
	-	19	Medium	Good	



TABLE 2

LIST OF CRYSTALS SHOWING PROMISE

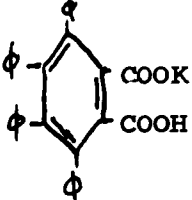
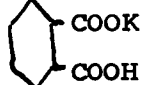
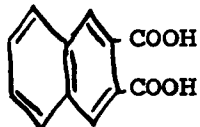
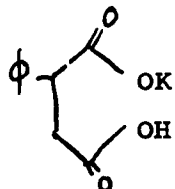
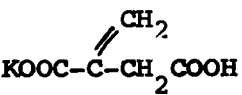
<u>Compound</u>	<u>Formula</u>	<u>M.P.</u>	<u>Maximum 2d Spacing, Å</u>
Half K Salt of Tetraphenyl Phthalic Acid		-	44
Half K Salt of Cis-Cyclohexane-1,2- Dicarboxylic Acid		-	28
Naphthalene-2,3-Dicarboxylic Acid		-	41
Half K Salt of α-phenyl Succinic Acid		-	30
Barium Itaconate		-	21.6

TABLE 2

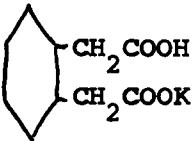
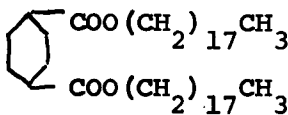
LIST OF CRYSTALS SHOWING PROMISE

<u>M.P.</u>	<u>Maximum 2d Spacing, A</u>	<u>Reflectivity</u>	<u>Crystallinity</u>
-	44	Medium	Fair
-	28	Medium	Excellent
-	41	Low-Medium	Good
-	30	Strong	Good
-	21.6	Strong	Good



TABLE 3

LIST OF CRYSTALS SHOWING EXCEPTIONAL PROM

<u>Compound</u>	<u>Formula</u>	<u>M.P.</u>	<u>Maximum 2d Spacing, A</u>
Half K Salt Cyclohexane -1,2- Diacetic Acid		-	31.2
Diocetyl Adipate	$\text{CH}_3(\text{CH}_2)_{17}\text{OOC}(\text{CH}_2)_4\text{COO}(\text{CH}_2)_{17}\text{CH}_3$	64	90
Diocetyl Terephthalate		85	84
Tetradecanoamide	$\text{CH}_3(\text{CH}_2)_{12}\text{CONH}_2$	103.4	54

1

TABLE 3

LIST OF CRYSTALS SHOWING EXCEPTIONAL PROMISE

	<u>M.P.</u>	Maximum 2d <u>Spacing, A</u>	<u>Reflectivity</u>	<u>Crystallinity</u>
	-	31.2	Very Strong	Good
$\text{PO}(\text{CH}_2)_{17}\text{CH}_3$	64	90	Extremely Strong	Excellent
CH_3 CH_3	85	84	Strong	Excellent
ONH_2	103.4	54	Extremely Strong	Good



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3. PREPARATION AND PURIFICATION OF SELECTED COMPOUNDS

Commercially available materials were purchased and purified by repeated crystallization. Certain promising materials, however, were not available and had to be synthesized. Details of the synthesis of the more important compounds utilized for this program are as follows.

Diocetyl adipate and diocetyl terphthalate were prepared by refluxing and stirring two moles of octadecanol with one mole of the acyl chloride and two moles of dry pyridine in anhydrous benzene for 16-20 hours. The pyridinium chloride formed in the course of the reaction was filtered off while the solution was hot. The filtrate was then washed with water and the diester permitted to crystallize out of solution. The products were readily purified by multiple recrystallizations from benzene.

The long-chain acid amides were prepared by heating the corresponding acid and thionyl chloride until the mixture became homogeneous. The resulting acid-chloride was then poured into cold concentrated ammonium hydroxide. After washing the crude amide with water and drying, the material was repeatedly recrystallized from isopropyl alcohol.

The other half-acid salts were prepared in the usual fashion utilizing purified reagents.

Octadecyl hydrogen phthalate was prepared by refluxing an equimolar mixture of octadecanol and purified phthalic anhydride in dry benzene for ten hours. Concentration of the reaction mixture afforded crude octadecyl hydrogen phthalate, melting point 68.8 - 69.6°. Repeated recrystallization of this material gave a poorly crystalline product, melting point 69.2 - 69.6°C.

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4. CRYSTAL GROWTH

4.1 Methods

4.1.1 Preparation of Seeds

None of the compounds that were considered appeared suitable for growth from the melt. The potassium salts do not melt without decomposition. The organic crystals, because of their low melting points, appeared very difficult to grow from the melt. Consequently, all the crystal growing efforts were concentrated on techniques involving growth from solution.

In the conventional solution growth of crystals, a seed is rotated in a saturated solution whose temperature is gradually lowered. The first requirement, therefore, is to obtain high quality seed crystals. Seeds were prepared by slow evaporation at constant temperature of a saturated solution of the compound. Selected crystals that were produced in this fashion were removed from the nutrient solution and re-inserted in another saturated solution, which was then allowed to evaporate slowly and isothermally. In this way seed crystals were prepared suitable for rotator growths and also for X-ray tests. The choice of a solvent often played a pronounced role in the quality of the seeds that were produced. The importance of obtaining seeds of the highest possible quality must be emphasized. The defects in poor seeds are propagated during crystal growth in a rotator, producing flaws and multiplicities. In some crystal species there was little tendency to produce good single crystals. Instead, clusters of tiny crystals were produced and efforts to produce individual crystals were unsuccessful. In a few instances, a new solvent cured the problem.

When good seeds were prepared the next step was to mount them in such a fashion that they could be rotated in a solution growth apparatus such as shown in Figures 1 and 2. The conventional method of doing this involves drilling a hole in the seed and mounting the seed on a wire which is then attached to a shaft which rotates. The organic crystals being developed were much too fragile to be handled in this fashion. Another method of mounting seeds involves tying the seed with a fine wire which is then mounted on the shaft. Again the fragility of the seeds precluded this technique. The principal technique for attaching the seeds involved cementing them to a flat plate, or clamping them between the two jaws of a tiny spring clamp.

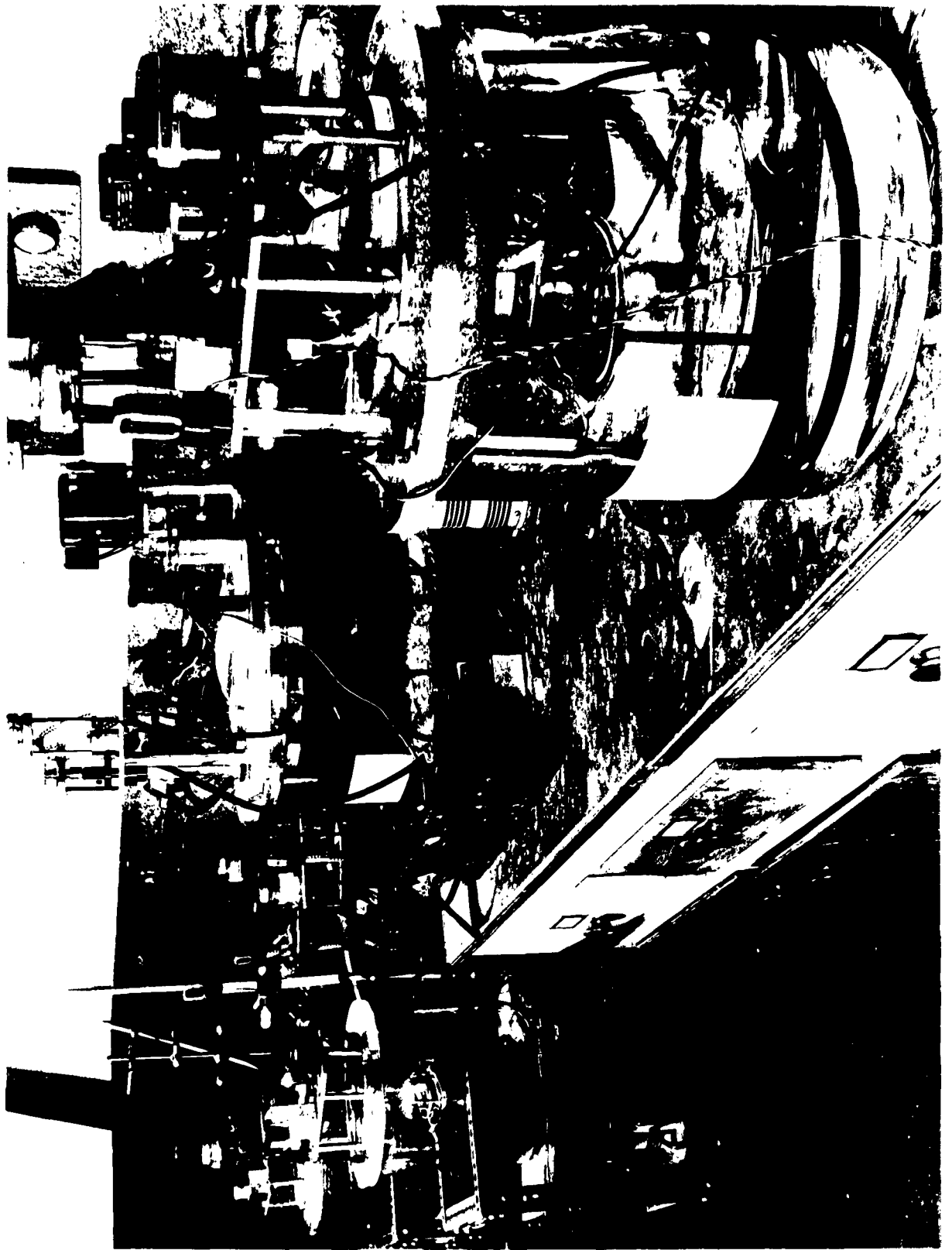


FIGURE 1. Battery of Solution Crystal Growers

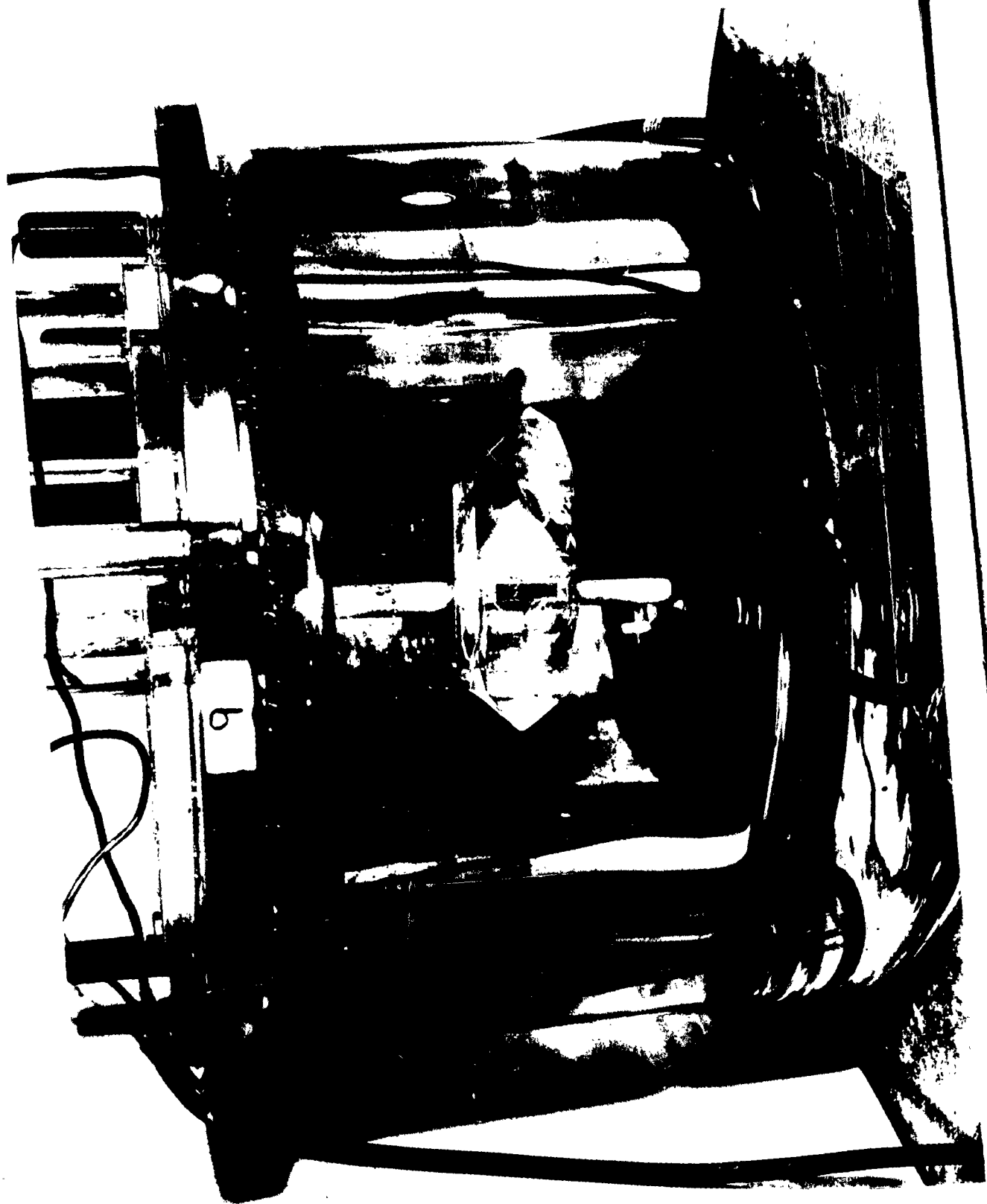


FIGURE 2. Typical Solution Crystal Growth Apparatus

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4.1.2 Rotator Apparatus

As shown in Figures 1 and 2 the crystal rotator consists of two concentric battery jars. The inner jar contains a saturated solution of the compound from which crystals are to be grown. The outer battery jar contains water heated by an immersion heater and maintained at a constant temperature by means of a mercury thermoregulator. The inner jar has a tight fitting lid which prevents evaporation of the solution. A shaft comes through the center of the lid by means of a rotating seal. The shaft is motor driven and alternates direction every three rotations. Spokes are inserted in the shaft so that seeds can be mounted on the ends of the spokes. Depending upon the solvent used, all the constructional materials are either glass or stainless steel or inert plastic materials such as lucite or teflon.

A solution of the compound is saturated at approximately $50-60^{\circ}$ and filtered into the rotator solution jar. By observing the concentration gradients in the solution near a suspended seed crystal, while varying the temperature over a range of 0.5° , the temperature of the apparatus can be brought to the exact saturation point of the nutrient solution. When the solution is thus thermally stabilized, the rotator assembly and the seeds are lowered into the solution and the solution cover is bolted in place. The temperature is raised slightly to dissolve the surface layer of the seed, after which the temperature is returned to the saturation temperature and growth is started.

The set point of the thermoregulator is driven down at the rate of approximately $1/4^{\circ}\text{C}$ per day by means of a small synchronous motor. The drive rate is actually varied to match the solubility changes with temperature and as the growing crystal surface area increases.

Considerable difficulty was experienced with highly volatile organic solvents because of evaporation losses through the lid of the inner battery jar. Tighter seals were used to minimize this effect.

Several sizes of crystal rotators were available for this work. For some materials only a small amount of compound was available and in this case we used a small rotator with a capacity of one liter of nutrient solution, rather than the three liter capacity generally used for experimental work.

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A typical crystal growth run with a rotator requires about 30 to 40 days. Since a period of about one to two weeks is required between runs to make the modifications suggested by the previous run, it can be seen that the growth of crystals by this technique involves a lengthy period of time.

4.2 Experimental Results

4.2.1 Acid Phthalates

Crystals of the following acid salts of phthalic acid were prepared and submitted to the Contractor Monitor: ammonium, potassium, sodium, rubidium, cesium. The crystals were 1/2" x 1/2" in cross section and 1/8" thick and were mounted on aluminum holders to fit the X-ray equipment in the Analytical Branch at ASD.

4.2.2 Other Materials

The following compounds, whose properties are listed in Table 3, were supplied to the Project Monitor in the form of small seeds for evaluation. There was inadequate time or funds to prepare larger crystals of these very promising crystals.

Half K Salt of Cyclohexane-1,2-Diacetic Acid
Dioctadecyl Adipate
Dioctadecyl Terephthalate
Tetradecanoamide

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5. X-RAY STUDIES

5.1 Diffractometer Powder Technique

In almost all cases the diffractometer powder technique was used. The powder camera was used whenever very weak lines were observed in the diffractometer and it was desired to verify that these reflections occurred.

The diffractometer used was a Siemens unit with a Krypton geiger tube detector. All samples were powdered to plus 325 mesh screen and were mounted in an aluminum holder. Care was taken to achieve coplanarity of sample surface with diffractometer focussing arc to assure the accuracy of the measured 2θ angle. The diffractometer was periodically aligned by the Cornu reversal method to assure accuracy of the measured angle. Scanning speeds were $1/4^\circ/\text{min.}$, and $1/2^\circ/\text{min.}$, with time constants to give angular accuracy of 0.05° or better.

Using this technique we were able to see (with $\text{CuK}\alpha$ radiation) $2d$ spacings of up to $120\text{\AA}$. Where necessary the entrance slits were closed down to less than 0.8 mm to achieve best results at these low angles.

5.2 Powder Camera Technique

The Debye-Scherrer camera used was a Siemens unit with a diameter of 57.3 mm . The beam collimator was 0.4 mm dia. and designed for measurements down to $2\theta = 4^\circ$. All samples were mounted in a 0.5 mm capillary tube of Lindemann-Glass. $\text{CuK}\alpha$ radiation was used throughout, with exposure times of one to four hours.

Films were mounted using the Straumanis technique to compensate for film shrinkage during processing. Thus if S = distance of line from $2\theta = 0^\circ$ mark, L = distance of film from 0° to 180° mark, then

$$\frac{S}{L} = \frac{2\theta^\circ}{180^\circ}$$

Where we were unable to see a first order line, the spacing of the first order was inferred from the measured high orders.

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6. CONCLUSIONS AND RECOMMENDATIONS

6.1 Discussion of Results

Table 1 lists the crystals on which work was discontinued for a variety of reasons. In some cases the results did not warrant further effort, while in other instances the results had some promise but there were other compounds that looked more promising. Some of the compounds in Table 1 could conceivably lead to good results.

Table 2 lists a number of crystals which show promise, but were not pursued beyond the preparation of seeds because other compounds looked more attractive. This Table has a number of compounds which could yield useful crystals.

Table 3 lists four compounds having 2d spacings from 31 to 90A. These materials look extremely promising and have not only good crystalline properties but excellent reflectivity for X-rays. The work already carried out indicates that large useful crystals could be prepared from all of these compounds.

6.2 Recommendations

It is recommended that work on the following compounds be continued to progress from the stage of seed crystals to large single crystals suitable for X-ray spectrometers:

Half K Salt of Cyclohexane-1,2-Diacetic Acid
Diocetyl Adipate
Diocetyl Terephthalate
Tetradecanoamide

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Rpt Nr ASD-TDR-69-311. RESEARCH ON A SUIT-
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X-RAY SPECTROSCOPY. Final report. Apr 69.
10p. incl illus., tables, 2 refs.

Unclassified Report

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- I. AFSC Project 7964.
Task 796405
- II. Contract AF 33
(657)-7756
- III. Inmet Corporation
Palisades Park,
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- IV. W. Ruderman, et al.
- V. Aval fr OTS
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