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TECHNICAL REPORT 4766

IDENTIFICATION OF EXPLOSIVES
BY X-RAY DIFFRACTION



J. E. ABEL
P. J. KEMMEY

MAY 1975

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PICATINNY ARSENAL
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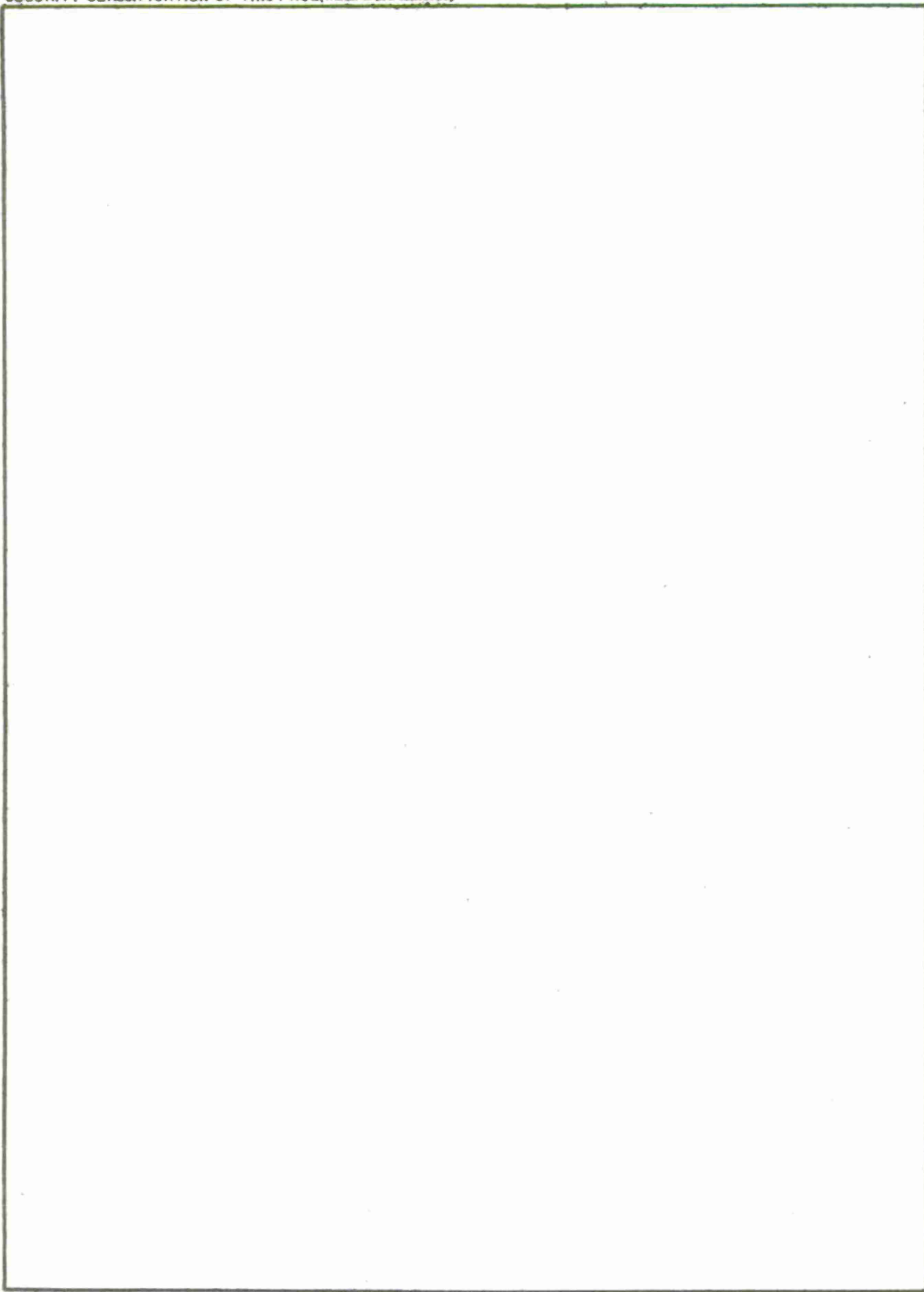
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<p>This report presents a computer program for producing a data bank of X-ray diffraction patterns on explosives and explosive mixtures. The data to be used in this data bank is obtained from a diffractometer, Debye-Scherer films, and a Gandolfi camera. The computer program allows the rapid retrieval of X-ray data on any explosive in the file, and also the identification of unknown explosives with ones in the file, using statistical matching techniques.</p>		

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FOREWORD

The identification of diffraction patterns of explosives has long been a time consuming task for scientists in the explosives field. Our search for methods to eliminate the difficulties has yielded the method described in this report.

The authors wish to express their appreciation to Dr. H. Fair and Mr. T. C. Castorina for their encouragement and technical advice.

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INTRODUCTION

The established method for the identification of crystalline materials is X-ray powder diffraction, and the primary source of this data is the ASTM Powder Diffraction File (Ref 1), published by the Joint Committee on Powder Diffraction Standards. This file consists of 21,500 diffraction patterns of crystalline materials, including explosives and related materials. Scientists in the explosive field routinely use this source for the identification of explosives, although the task is time consuming unless done by specialists. Such card files are normally located quite a distance from the laboratory. In addition, the latest data, which is of special interest to explosives researchers, is not in the file but has to be extracted from technical papers. A centrally located, up-to-date file would expedite identification efforts. We believe that X-ray data should be compiled in one master file so that a computer can be used to search the file for information on known substances, and identify unknown substances by comparing them with the substances already on file.

Forensic chemists require identification of explosives in small amounts, often in the form of the fragments of a single crystal. A Debye-Scherrer camera produces diffraction patterns of samples weighing 10-15 milligrams; however, the recently available Gandolfi camera allows the production of diffraction patterns from a single crystal as small as 30 microns (Ref 2). The diffraction patterns available through the use of the Gandolfi camera and the other X-ray equipment are included in the master file.

Forensic chemistry has also created a demand for methods for identifying explosive mixtures, but, the Powder Diffraction File is restricted to the identification of single compounds. Our recent work at Picatinny Arsenal indicates that some of the ingredients in a mixture or the mixture itself can be identified by X-ray techniques. Thus, X-ray patterns may serve as "fingerprints" for the identification of explosive mixtures. We believe that this data on explosive mixtures should also be included in the master file.

EXPERIMENTAL PROCEDURE

The identification data to be used in the computer program is obtained by the use of the following X-ray equipment:

- 1) X-ray diffractometer
- 2) Debye-Scherer film camera
- 3) Gandolfi camera

When the X-ray diffractometer is used, samples are prepared as recommended by the National Bureau of Standards (Ref 3). A silicon external standard is used to test the alignment of the diffractometer. A 25 percent by weight silver internal standard is used whenever possible, and linear interpolation corrections are applied to the reflections over the length of the diffractograms. The diffractogram is obtained using nickel filtered Cu K α radiation. A scanning speed of $1^\circ/2\theta$ per minute is normally used.

A 114.6-mm-diameter Debye-Scherer camera is used on small samples. The sample preparation methods and interpreting procedures recommended by Azaroff are followed (Ref 4).

When the Gandolfi camera is used, a single crystal or fragment is mounted on a glass capillary. Appropriate adhesives are used to mount the crystal.

The final data, obtained through the use of the equipment mentioned above, is tabulated in the format used in the ASTM Powder Diffraction File. Likewise, the format and conventions of the International Tables for X-ray Crystallography (Ref 5) are also adhered to.

COMPUTER PROGRAM FOR CATALOGING X-RAY SPECTRA

Our computer program is capable of creating a catalogue of label and spectral information for any number of materials and mixtures of materials. The ability to create a catalogue is supplemented by the ability to update or correct previous entries, and to store new information on explosives as it becomes available.

The information on each particular explosive is identified by a unique alphanumeric name of up to ten characters. When a user requires information from the file, he enters a coded request and the alphanumeric name. The coded request is typed into the first ten columns of an IBM card: The first column is "1" if spectral data should be read from the

data on file. The second column is "1" if a listing of the spectrum in a numerical form is required. If the third column is "1", the program will produce a bar graph of the spectral data. The production of the bar graph will be described in the Production of Bar Graphs paragraph. Columns four and five provide choices for the abscissa and the ordinate of the spectrum: the values 1, 2, and 3 are used. For example, in the fourth column, "1" specifies the angle 2θ , and "3" specifies d values. In column five, "2" specifies peak intensities, while "3" specifies Debye-Scherrer intensities. If the sixth column is "1", the computer searches the master file in order to find a match with the input data. The method by which the computer searches for matches will be described later. Finally, if the seventh column is "1", the spectrum will be added to the catalogue. Otherwise, no change is made to the existing catalogue. Columns eight and nine are blank. The value "1" must always be entered in column ten. An outline of the option card appears in Appendix 2.

COMPARISON BETWEEN SPECTRA

The program is designed to determine whether the similarities between two spectrum peak positions and peak intensity ratios are statistically significant or coincidental. In the first step, the program has the computer compare the positions of all the peaks in the submitted spectrum with the peaks in all other spectra. The computer compiles a list of coincidences where the differences in peak positions is less than .5 percent in all cases.

The relative ratios between the various peaks of the spectra must be approximately preserved if two samples are to match. In X-ray work, we assume that if two samples contain the same material, then peak ratios should not vary by more than 50 percent. On the basis of this assumption, all coincidences in the first list are divided into classes which consist of members whose peak ratios do not differ from each other by more than 50 percent. The class that most nearly coincides with the spectrum being identified forms the second, refined list. The second list is refined further according to peak position. To allow for the possibility that there is a systematic shift in peak position between the two samples, average peak displacements are calculated for all the coincidences in the second list. Then, the third list is produced by discarding all sets of coincidences where the peak shift differs from the calculated average shift by more than .0025 of the average peak d -spacing.

Finally, a figure of merit is calculated to measure the likelihood that parts of the spectra match. This figure of merit is calculated through the use of the following formula:

$$F \propto \frac{N}{N \cdot L + \sqrt{M \cdot L}}$$

where N is the number of peak coincidences in the final, third list, and L and M are the number of peaks in the original spectrum. The value of this figure of merit is determined by trial and error. A spectrum is considered to match the spectrum being identified if the figure of merit is greater than 100.

PRODUCTION OF BAR GRAPHS

It is useful to be able to have a rough representation of the spectrum for visual comparison. The program is designed to produce bar graphs where the vertical resolution in intensity is of the order of ten percent (peaks of less than three percent are ignored), and the horizontal axis consists of peak positions specified with a resolution of about one percent. The vertical axis is labeled "intensity," and the horizontal axis is labeled in both angular (2θ) and d value.

CALCULATED DIFFRACTOMETER PATTERNS

The determination of atomic positions by a complete structural analysis makes the calculation of both diffractometer and Debye-Scherrer patterns possible. The D. K. Smith computer program (Ref 6) makes these calculations and generates a data deck that can be used in the proposed master file. This data is of the highest reliability and is preferred to other data.

RESULTS

The data in the file is contained on five separate tapes to facilitate selective searches (see Appendix 1). Additions may be made to the file at any time. The tape number, explosive class, and present number of entries on each tape are given here:

<u>Tape</u>	<u>Explosive class</u>	<u>Number of entries</u>
1	explosive compounds	28
2	explosive mixtures	9
3	dynamites	26
4	Gandolfi data	3
5	all explosives	66

An investigator may want to confirm his identification of an explosive, such as ammonium nitrate, by comparing the data he has obtained on the explosive sample with data in the master file. His sample might consist of a powder weighing as little as ten milligrams or as much as one hundred milligrams. In the former case, the investigator would use a Debye-Scherrer camera to obtain the X-ray data; in the latter case, he would use a diffractometer. To confirm his identification, the investigator extracts the data for ammonium nitrate from Tape 1. Figure 1 shows the calculated diffractometer pattern and the data print-out for ammonium nitrate. The investigator compares the data on the sample explosive with the data from the master file, and the identification is confirmed. Similarly, anyone can request data print-outs from the master file on any explosive in the file.

Dynamites obtained from the United Kingdom consist of nitroglycerin and other constituents. Although nitroglycerin cannot be identified using X-ray techniques, the other constituents and explosives can be. X-ray data was obtained for twenty-five of these dynamites, and the data was listed on tape 3. A test case of Quarex was submitted for identification: Figure 2 shows the matching bar graphs, the input data, and the matching data in the file. The reliability of the match is shown under the bar graph; the figure of merit is 254.68, and 24 peaks were matched with a peak shift of .00.

Explosive mixtures, such as black powder and HMX/KEL-F, were entered into the master file to provide additional "fingerprints." When the color, particle size, and components of mixtures are contained in the file, the identification of an unknown mixture is highly reliable. For example, black powder, which consists of potassium nitrate, charcoal, and sulfur, is readily identified by the potassium nitrate lines. Figure 3 shows the identification of a sample of black powder.

In instances where the unknown is only a fragment or a small crystal, the Gandolfi camera is used. Three crystal fragments of different explosives approximately 0.2 mm in diameter were submitted for identification. X-ray data was obtained for each and the explosives were identified as TNT, RDX, and potassium chlorate. The data was entered on the Gandolfi tape (tape 4) for future reference. Then, a test case was run on data obtained from another TNT crystal; Figure 4 shows the identification.

DISCUSSION OF RESULTS

The X-ray data from calculated powder diffraction patterns, which has been included in our file of explosives, is accepted by the Joint Committee on Powder Diffraction Standards, who include the data in the Powder Diffraction File, where it is treated in the same manner as experimentally obtained data. The standard reference patterns, used in the Powder Diffraction File, consist of spectra calculated from refined crystal structure determinations (Ref 6, 7).

Data from the structural determinations done by workers in this laboratory (Ref 8-12) is used to provide calculated patterns of lead azide, ammonium nitrate, TNB, RDX, and β -HMX for the master file. This data is highly reliable since it requires pure substances of uniform particle size, and since corrections for absorption, Lorentz polarization, and temperature factors have been made. This data is ideal for comparison with laboratory and production samples. Changes caused by preferred orientation and particle size are readily observable.

The identification of crystals and crystal fragments with the Gandolfi camera is a relatively new field. TNT, RDX, and potassium chlorate were the first explosives studied. The data obtained from all three X-ray films was superior to that of powder samples. In both TNT and RDX, additional reflections were observed in the high 2θ region, where the greatest resolution and accuracy are attainable. Gandolfi data will greatly aid the identification of explosives after a bomb blast, when any small fragments of the original explosive are available.

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12. Choi, C. S. and Boutin, H. P., *Acta Cryst.* B26 part 9, 1235 (1970)



UNKNOWN		BLACK PDR				
D-VALUE	PEAK INT.	D-VALUE	H	K	L	PEAK INT.
4.650	45.00	4.640	-0	-0	-0	40.00
4.590	23.00	4.590	-0	-0	-0	23.00
3.820	15.00	3.830	-0	-0	-0	20.00
3.750	100.00	3.750	-0	-0	-0	100.00
3.420	7.00	3.430	-0	-0	-0	9.00
3.200	12.00	3.200	-0	-0	-0	12.00
3.060	10.00	3.070	-0	-0	-0	10.00
3.030	65.00	3.030	-0	-0	-0	60.00
2.770	40.00	2.760	-0	-0	-0	34.00
2.700	20.00	2.700	-0	-0	-0	20.00
2.660	50.00	2.660	-0	-0	-0	50.00
2.640	60.00	2.650	-0	-0	-0	63.00
2.630	30.00	2.630	-0	-0	-0	34.00
2.410	10.00	2.410	-0	-0	-0	10.00
2.380	5.00	2.380	-0	-0	-0	5.00
2.330	14.00	2.330	-0	-0	-0	14.00
2.290	10.00	2.290	-0	-0	-0	7.00
2.190	60.00	2.190	-0	-0	-0	60.00
2.160	34.00	2.160	-0	-0	-0	34.00
2.070	20.00	2.070	-0	-0	-0	15.00
		2.050	-0	-0	-0	27.00

Fig 3 The identification of an explosive mixture

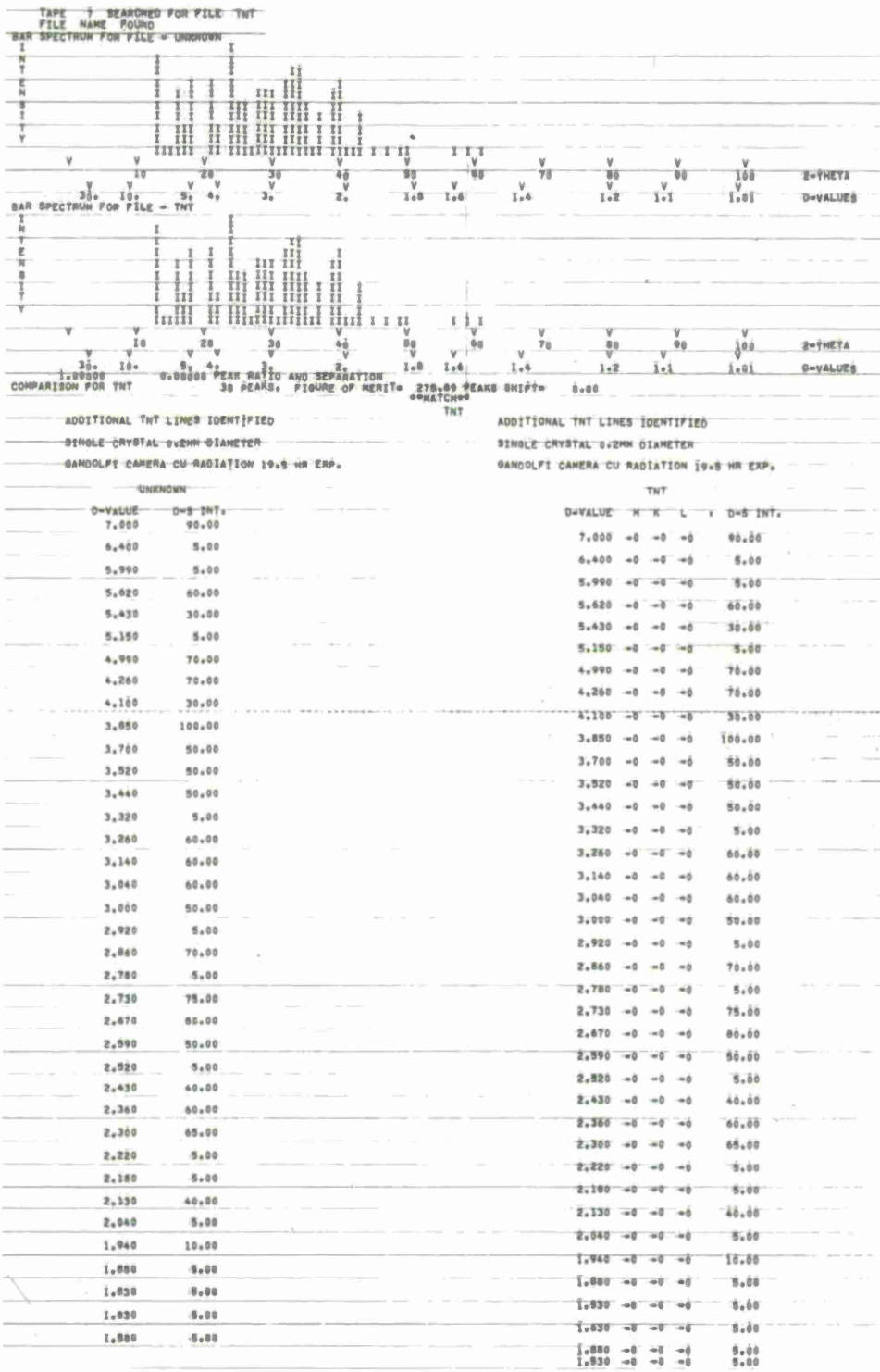
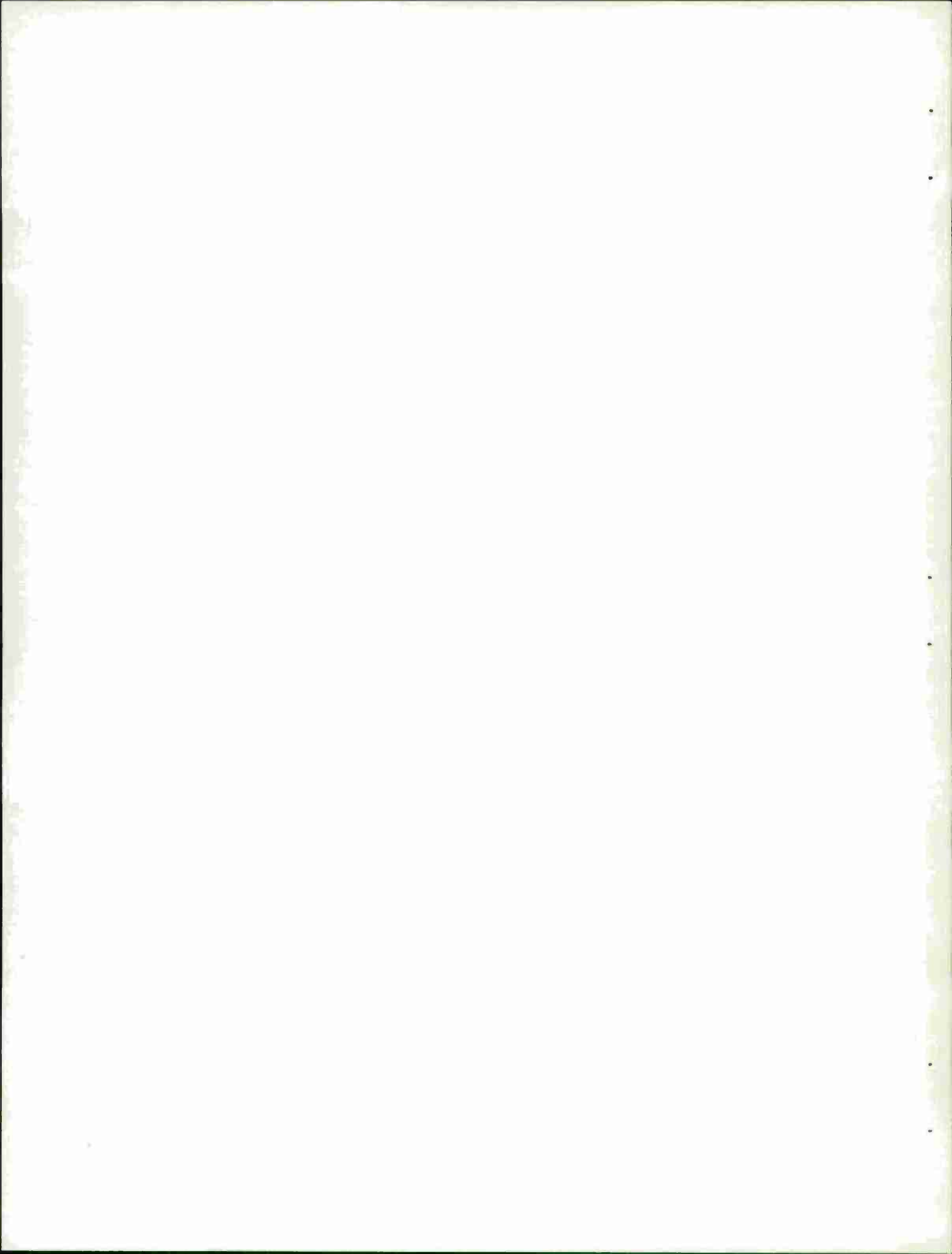
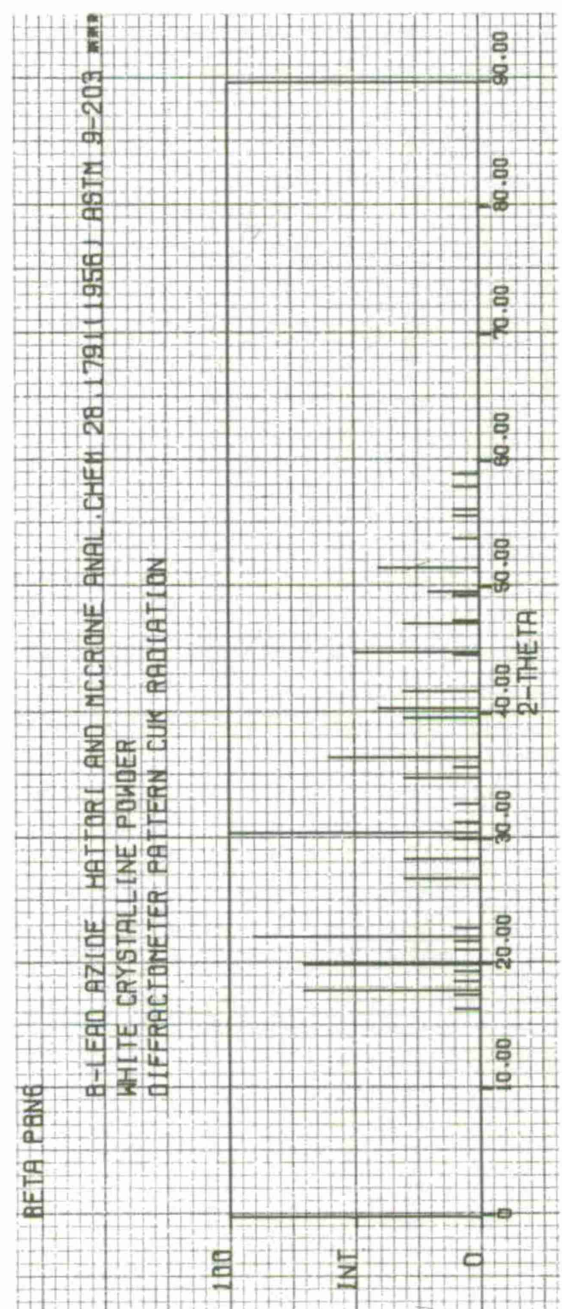
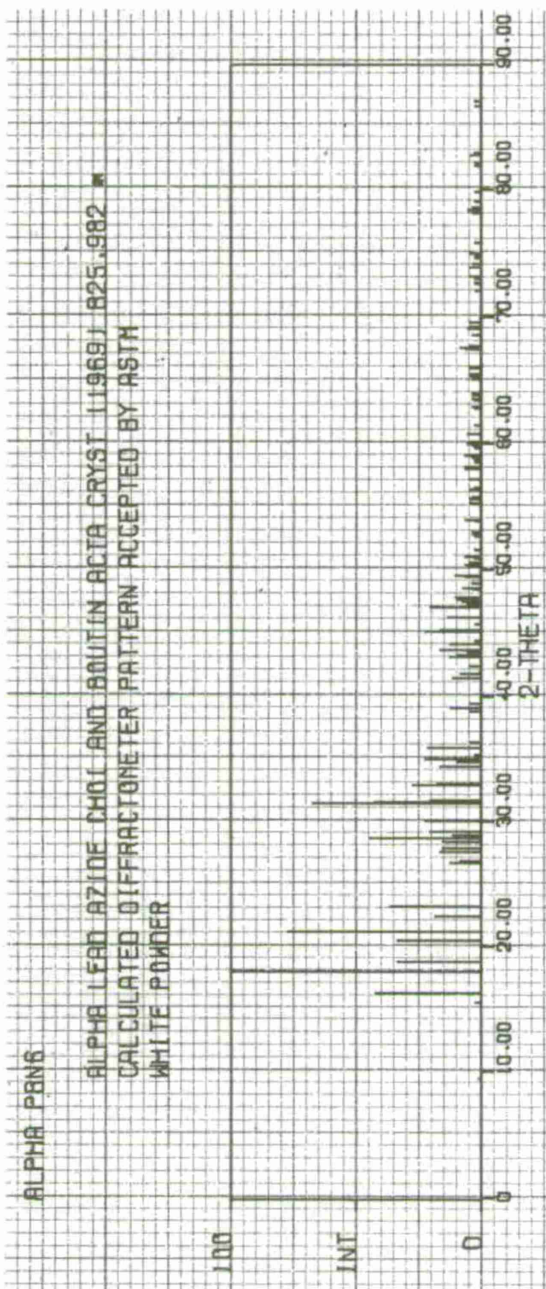


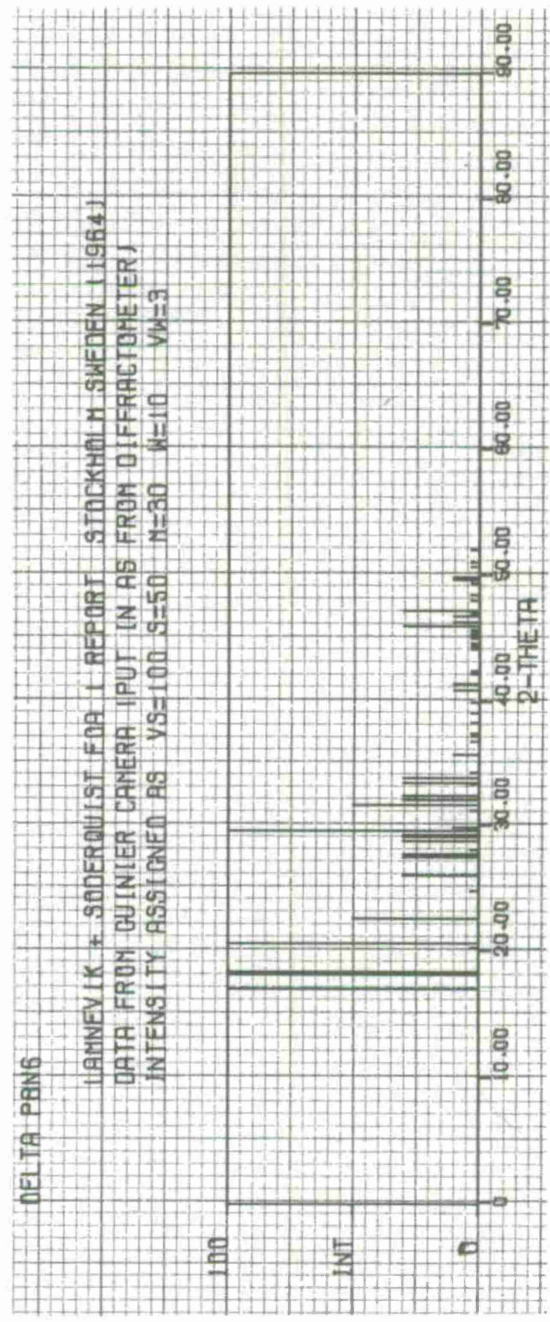
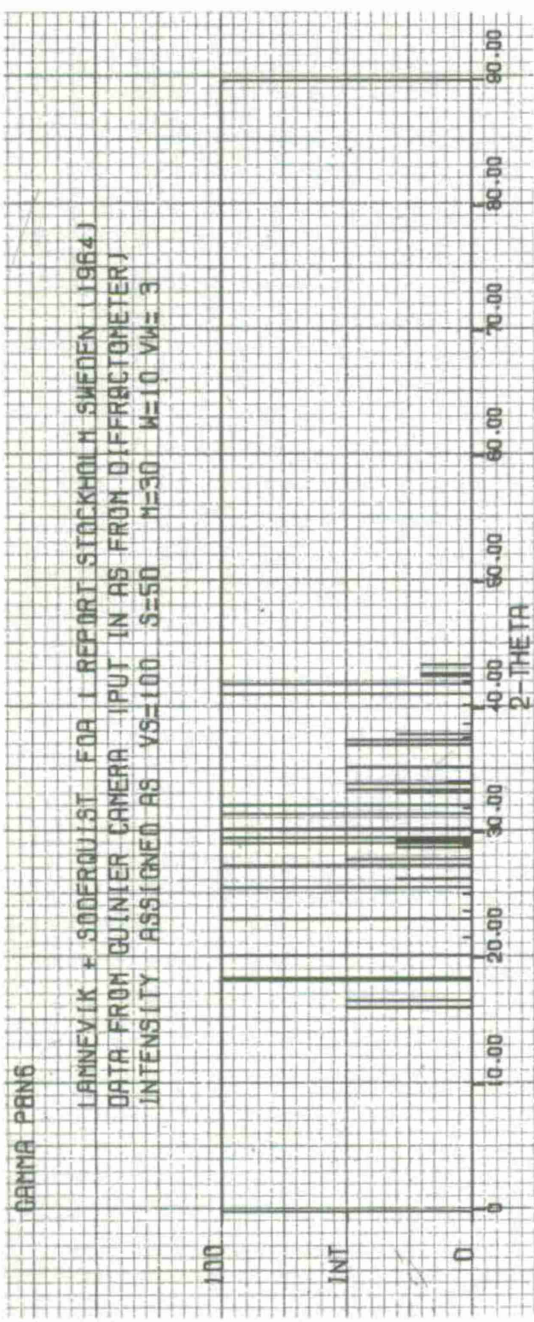
Fig 4 The identification of an unknown crystal with the Gandolfi camera



APPENDIX 1

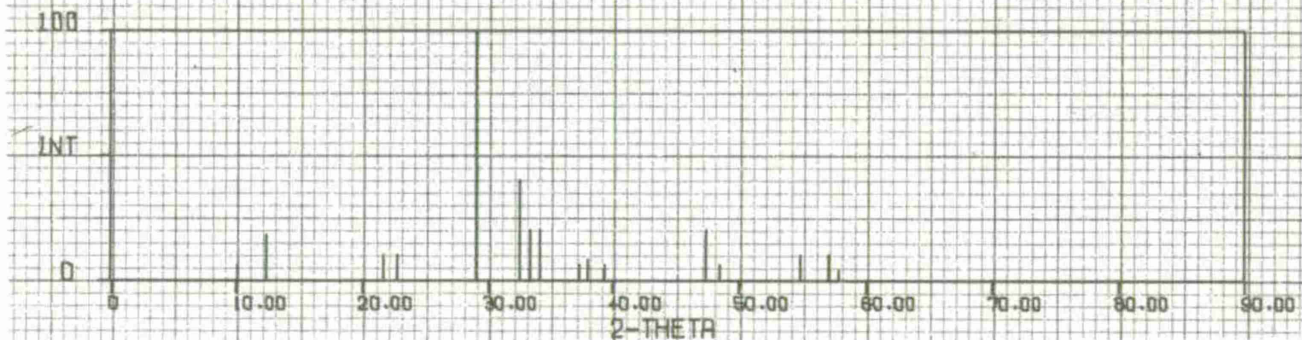
COMPUTER FILES OF X-RAY DIFFRACTIONS
OF ENERGETIC MATERIALS





BASIC PBN6

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WHITE POWDER UNIFORM PARTICLE SIZE
DIFFRACTOMETER PATTERN



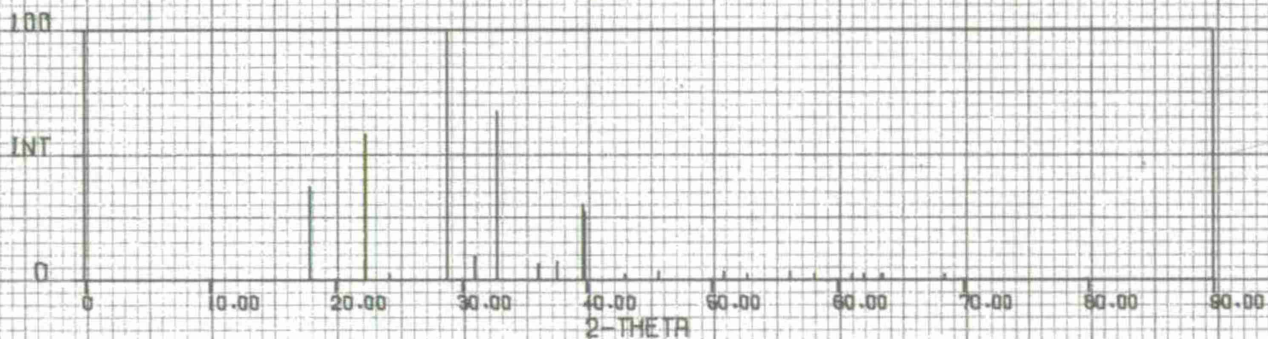
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GRAY COLOR
DATA FROM DIFFRACTOMETER COPPER RADIATION



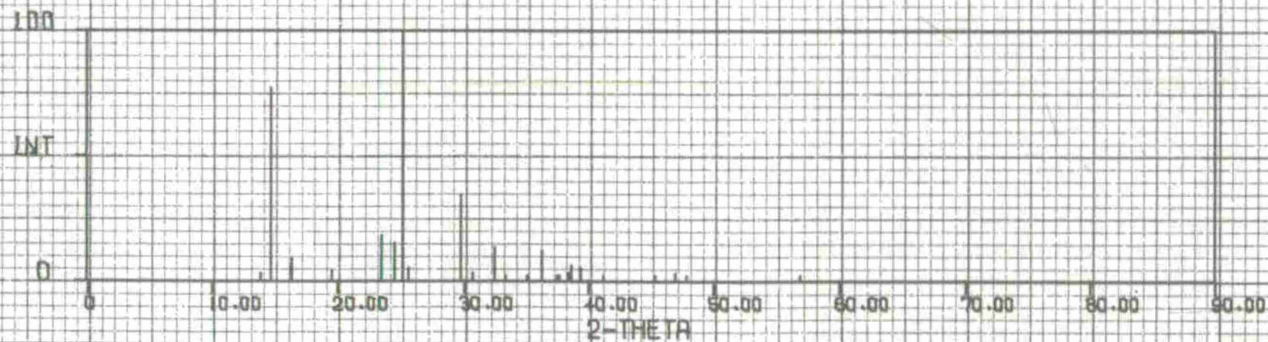
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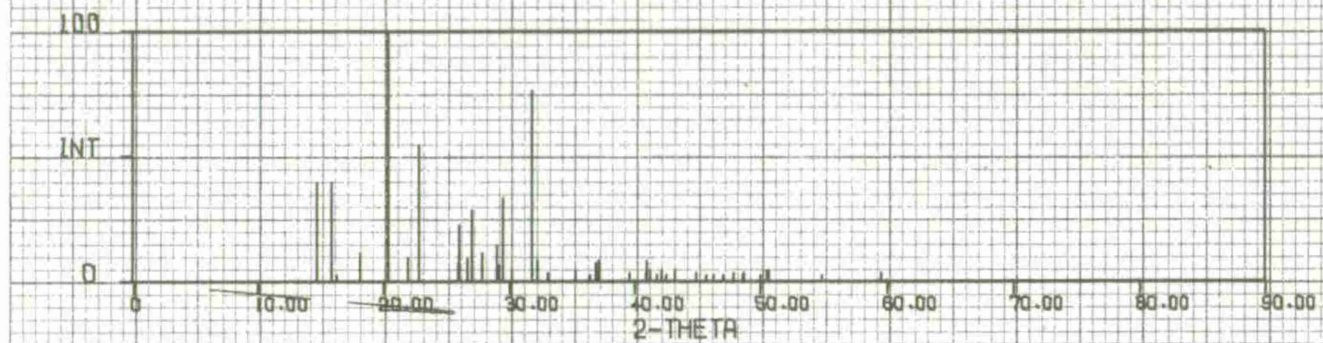
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WHITE FINE POWDER LANS-2652
GOOD DATA (LOS ALAMOS)
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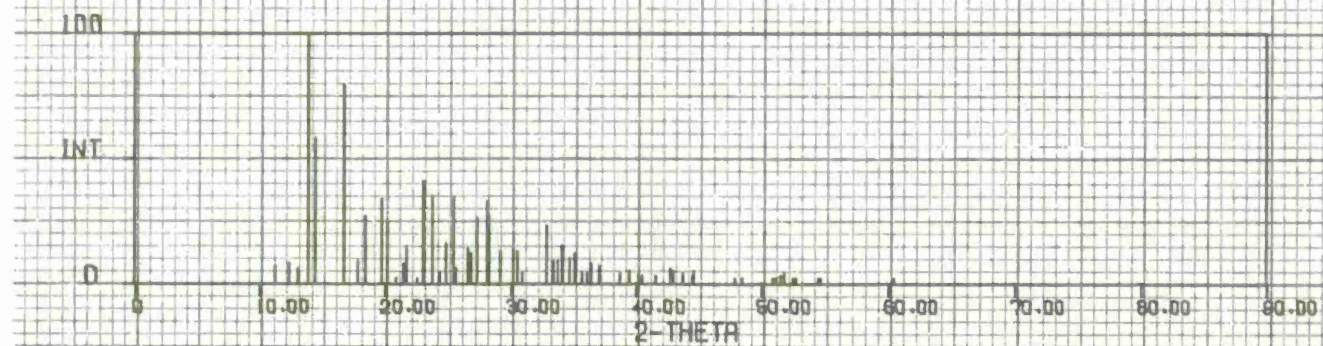
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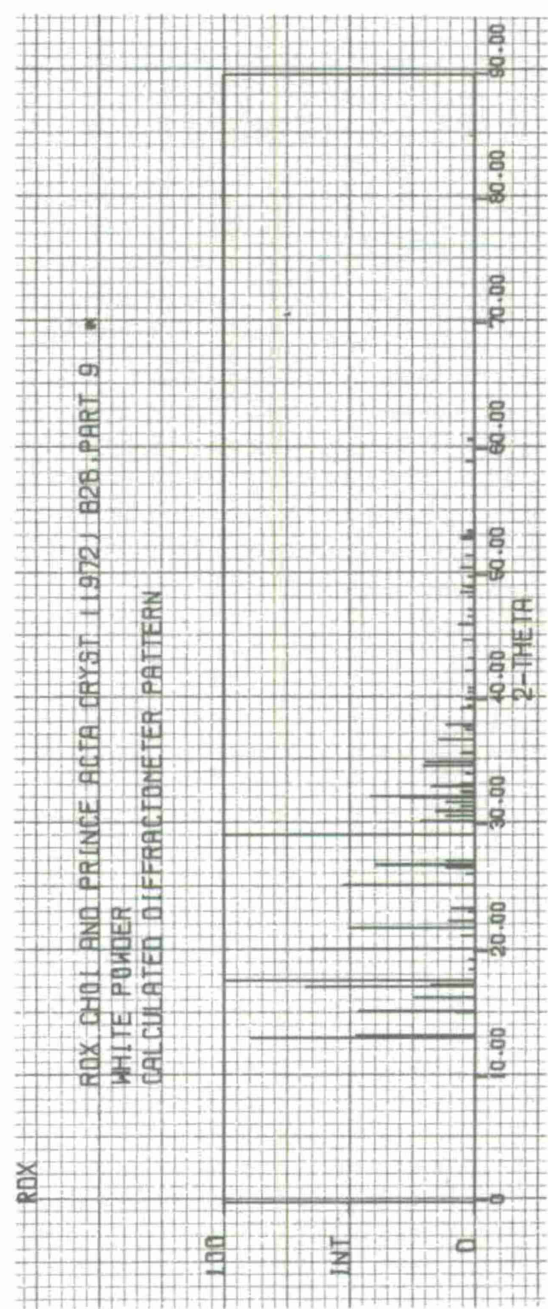
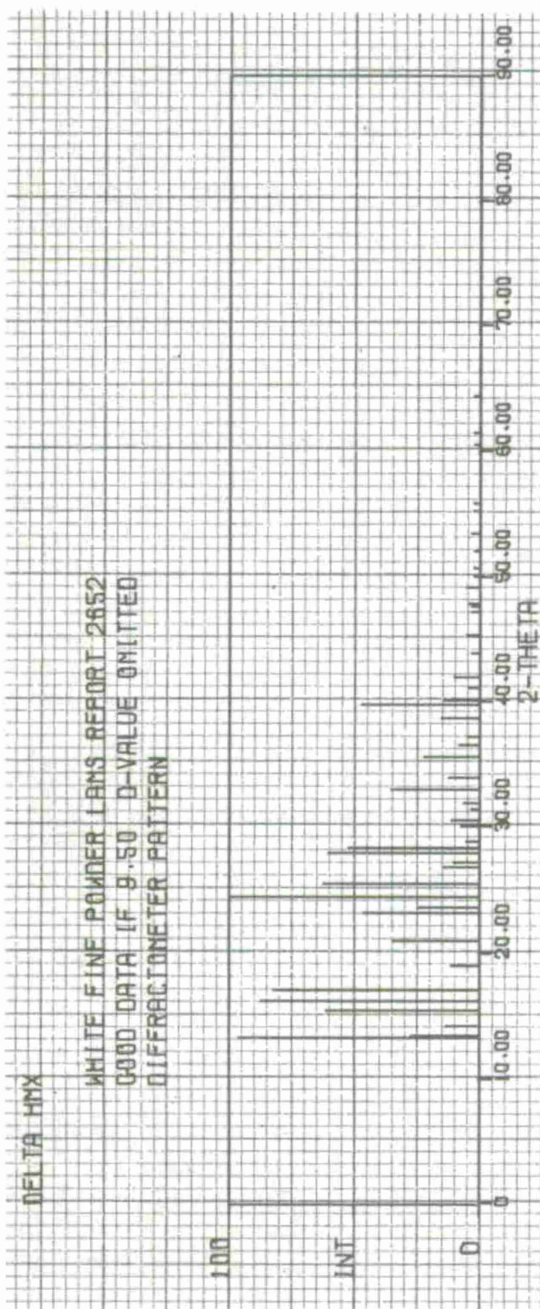
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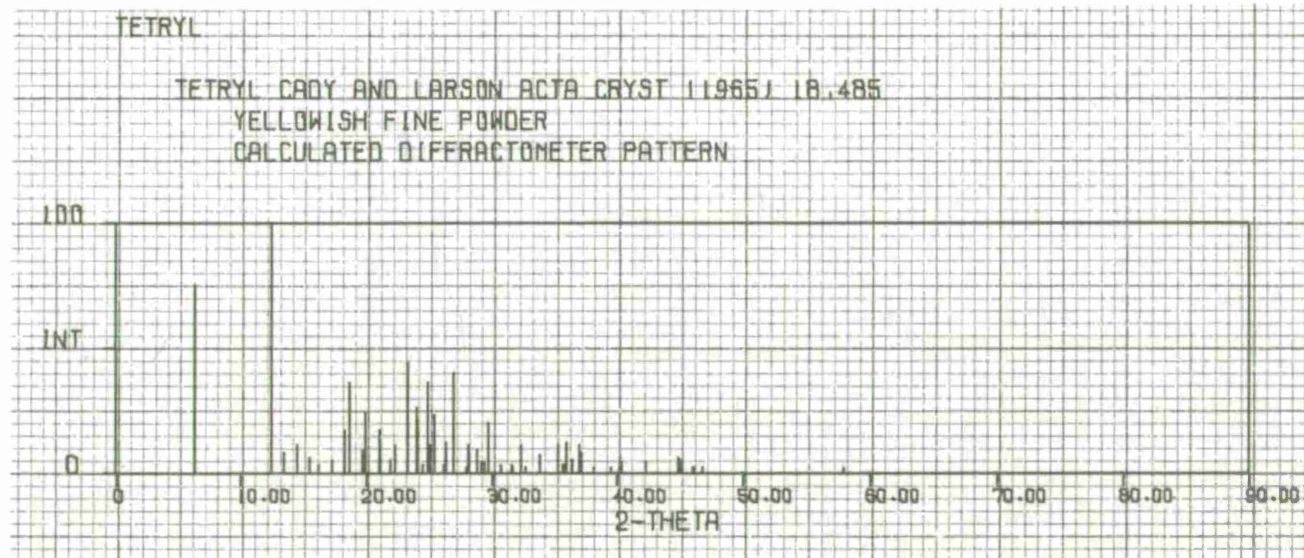
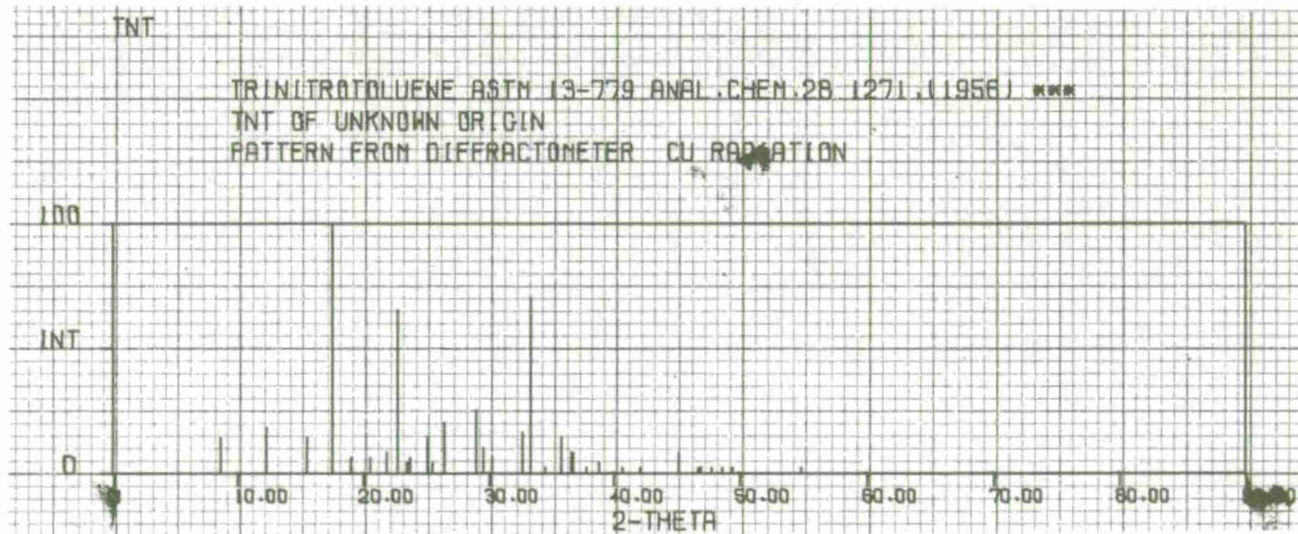


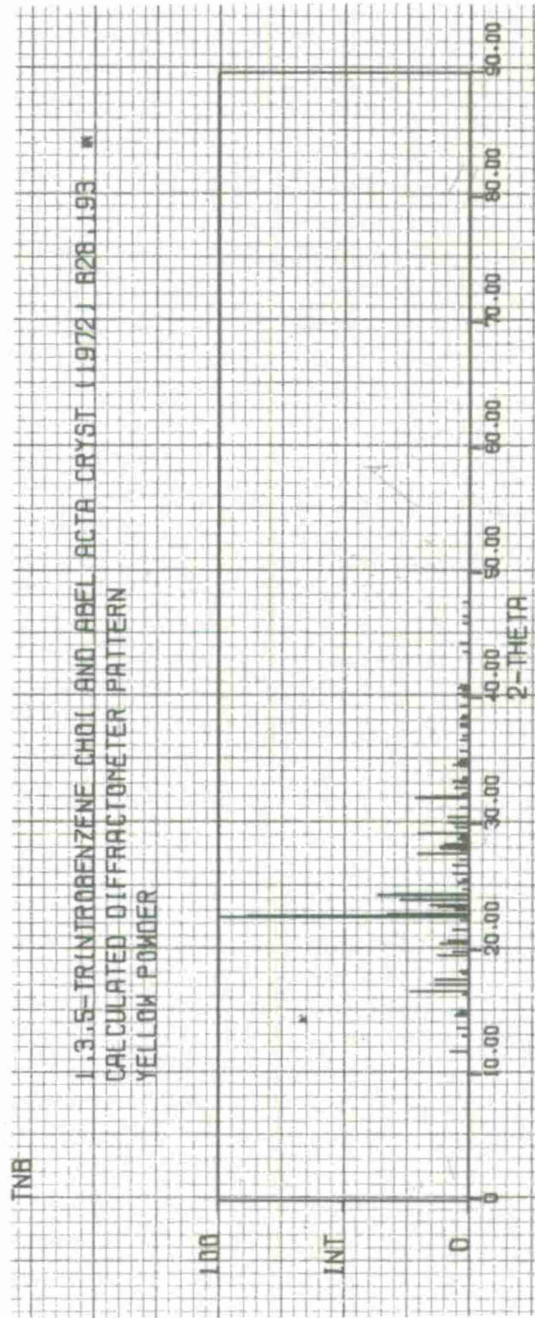
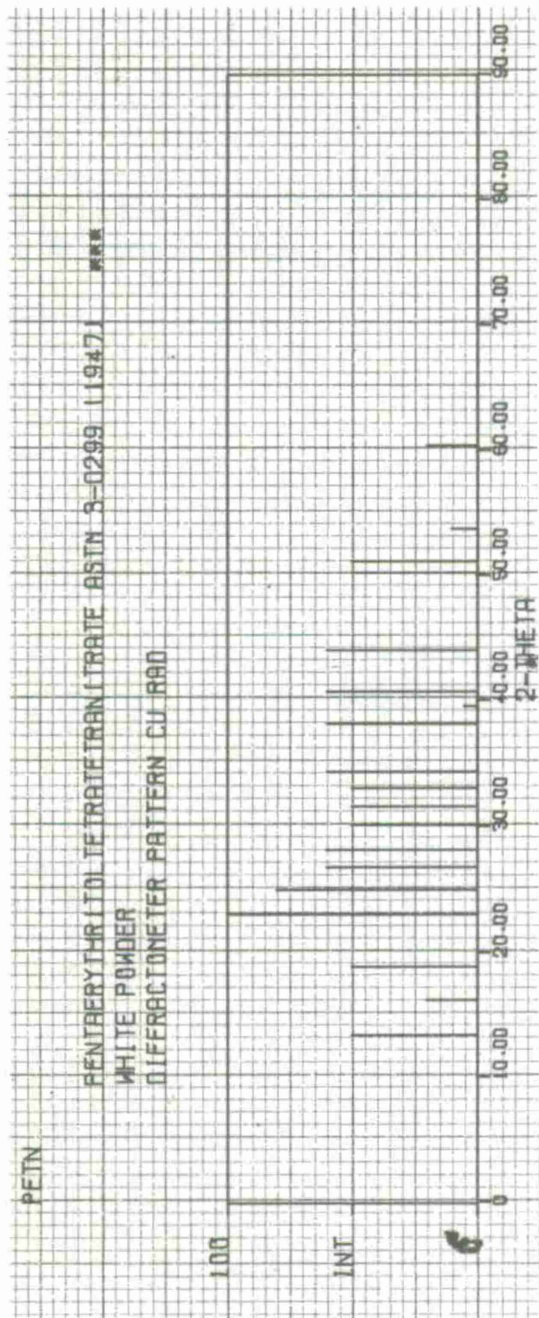
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WHITE FINE POWDER LAMS-2652
DATA CHECKED IN THIS LAB
DIFFRACTOMETER PATTERN (ROTATING SPECIMEN HOLDER)



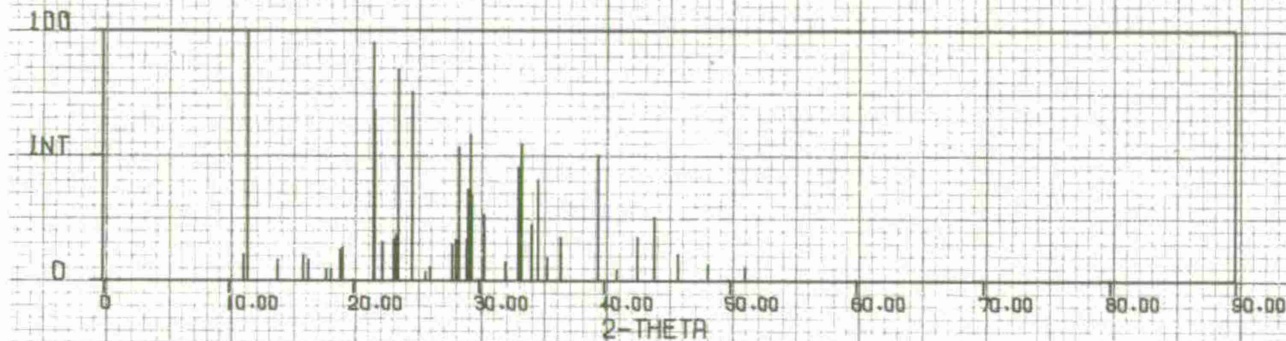






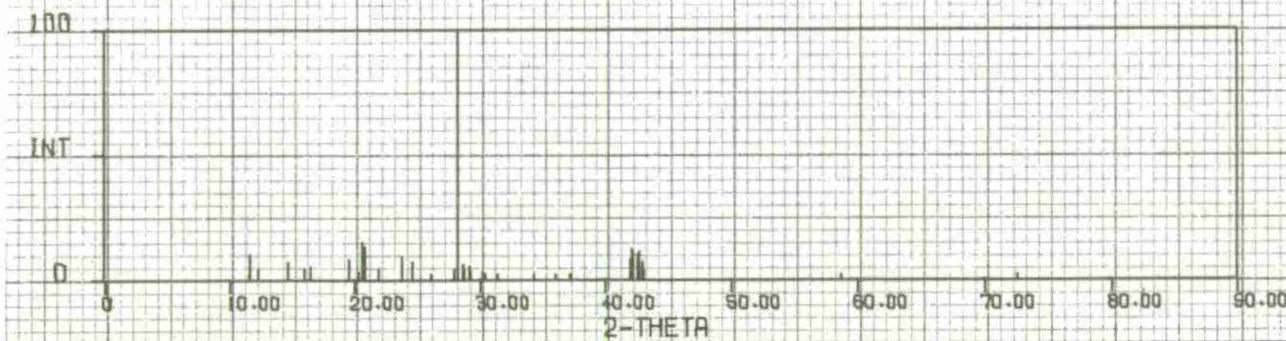
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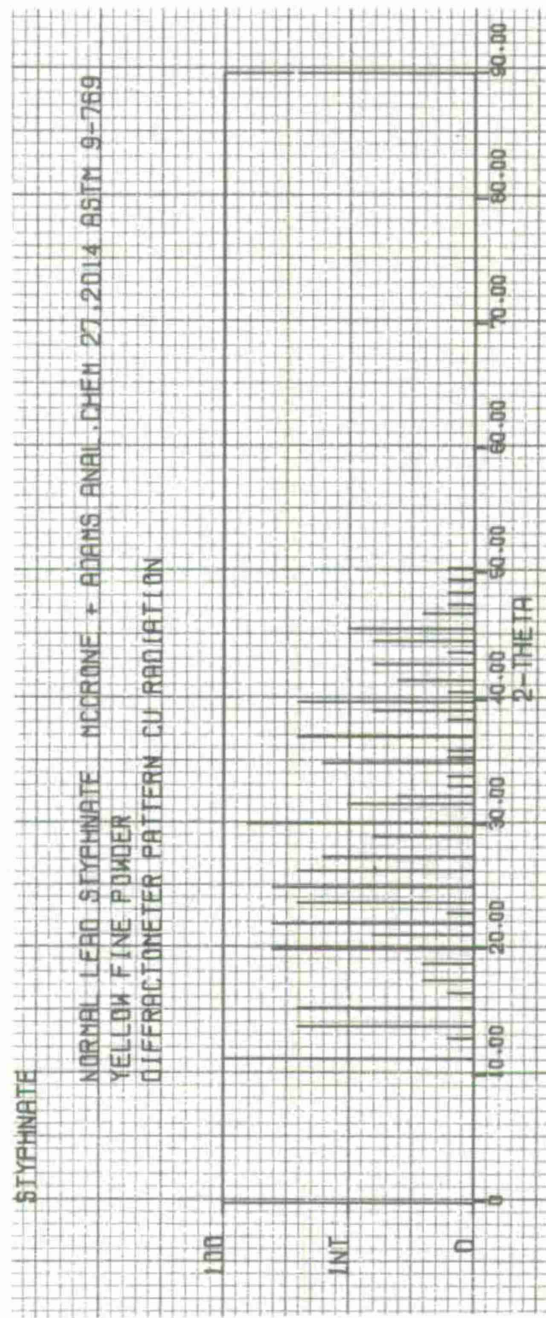
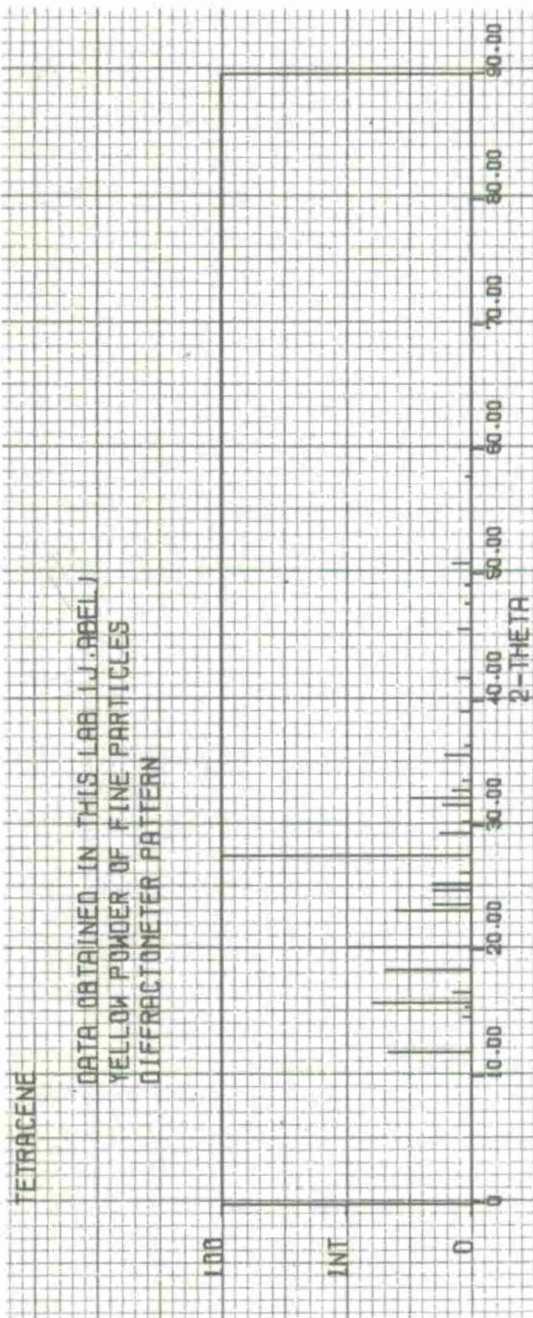
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YELLOW FINE POWDER
DIFFRACTOMETER PATTERN CUK RADIATION

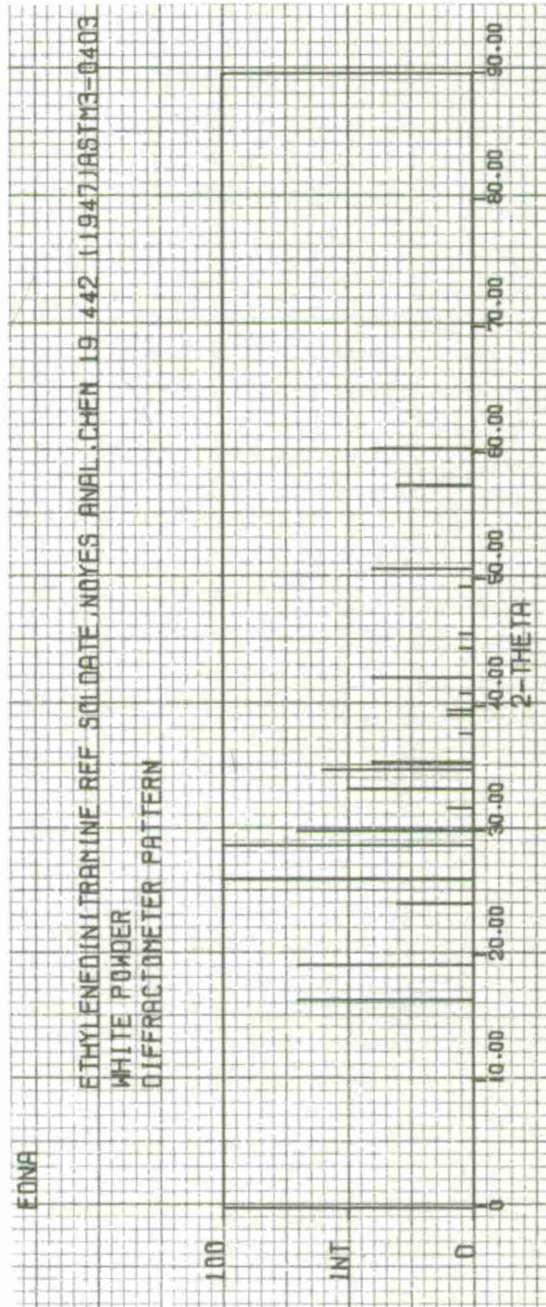
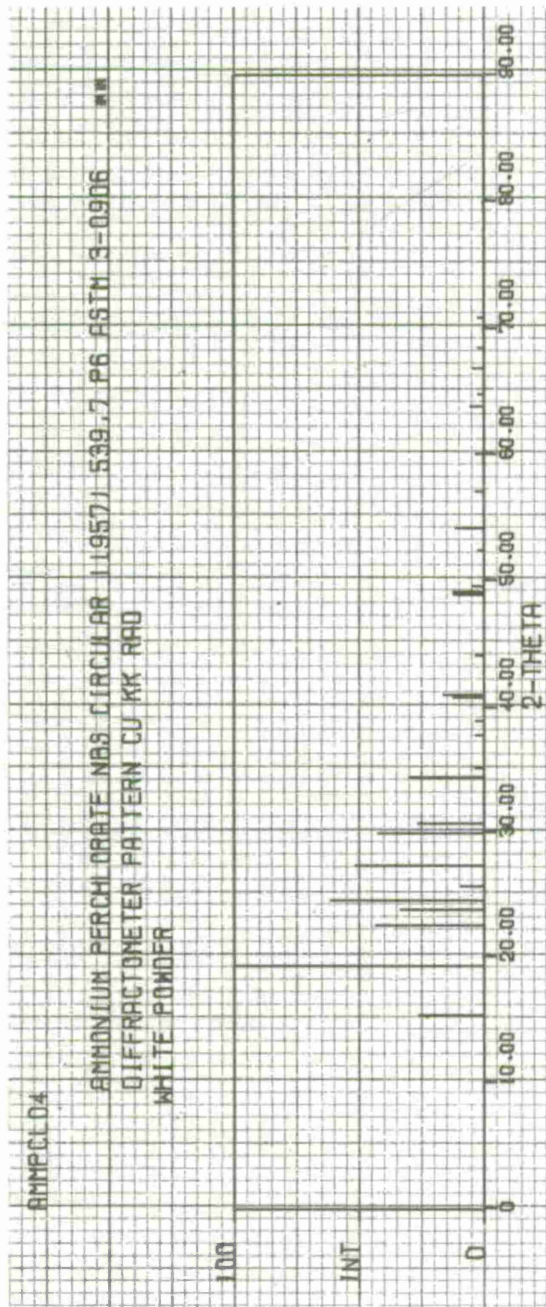


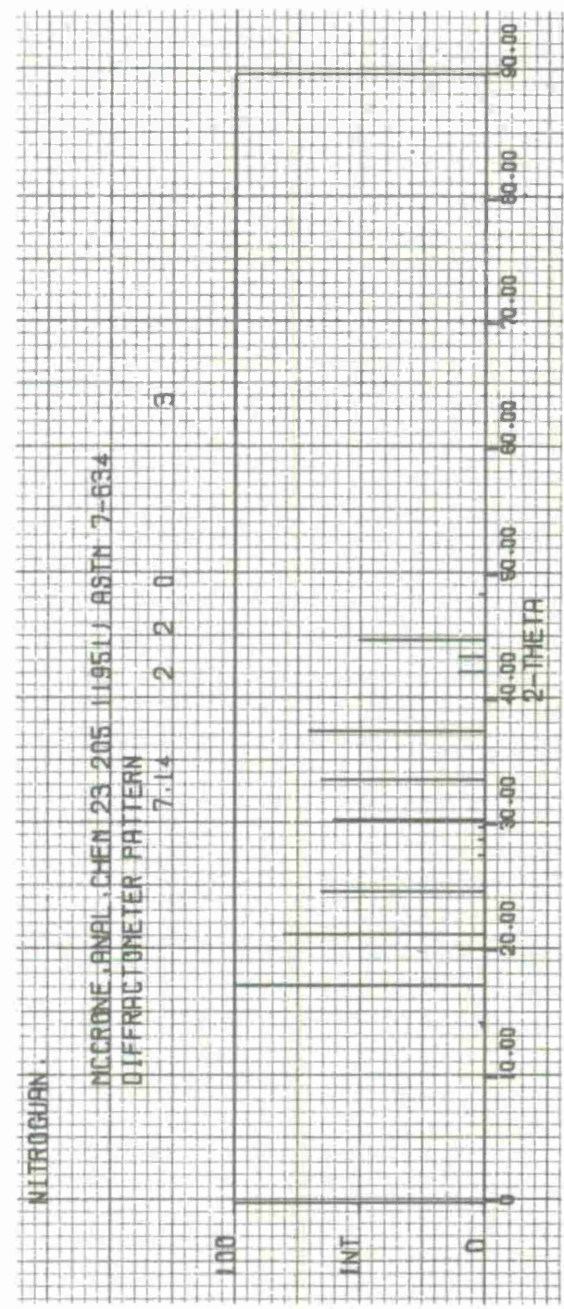
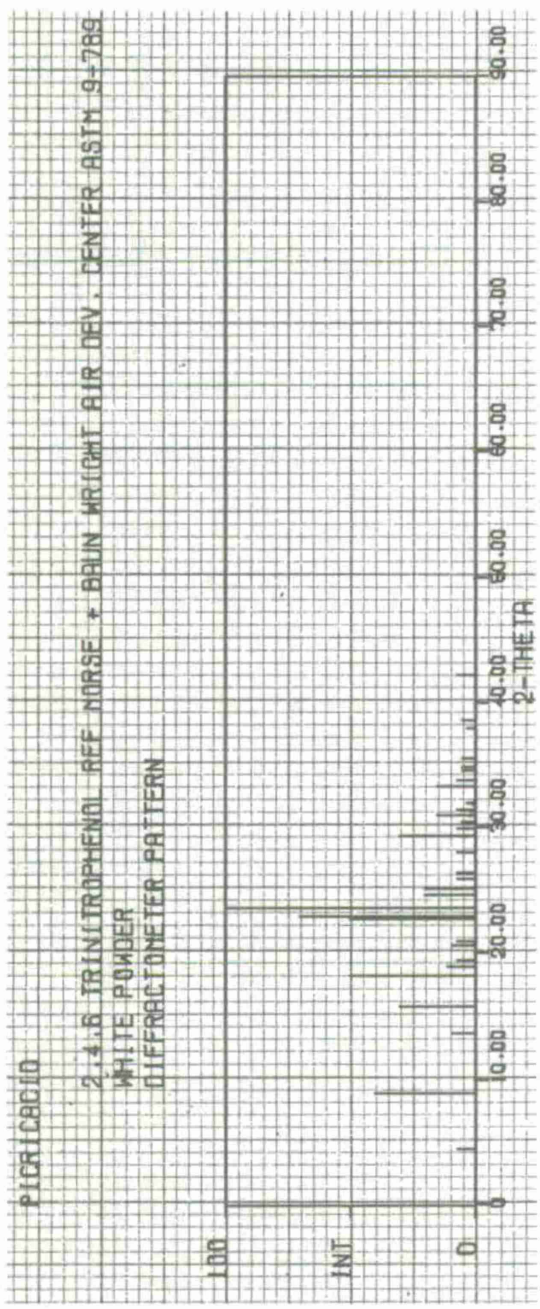
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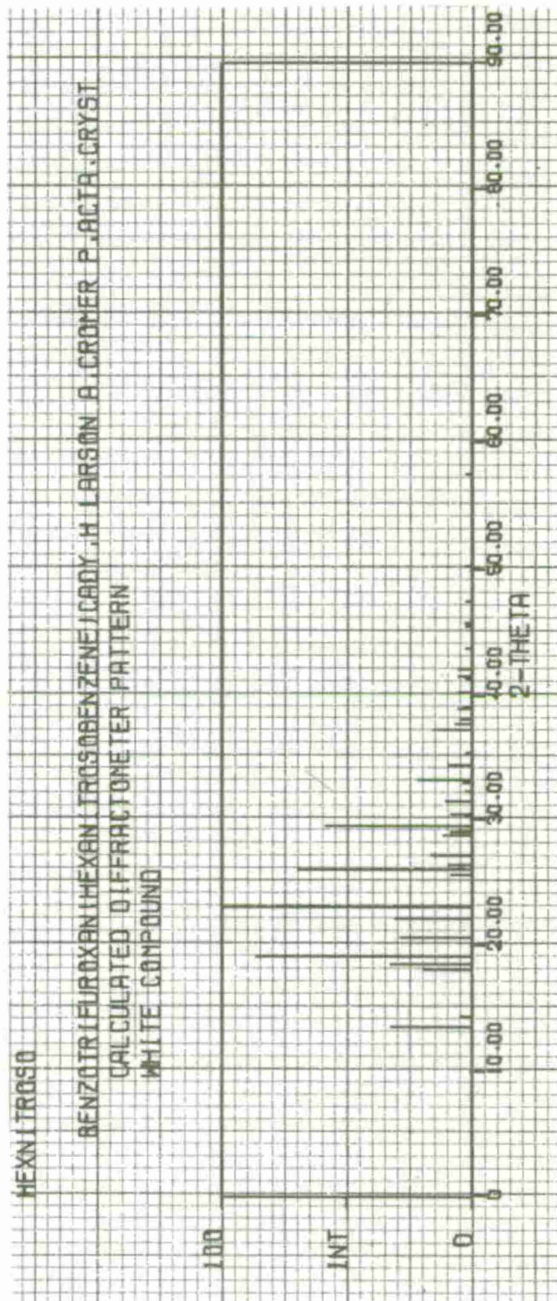
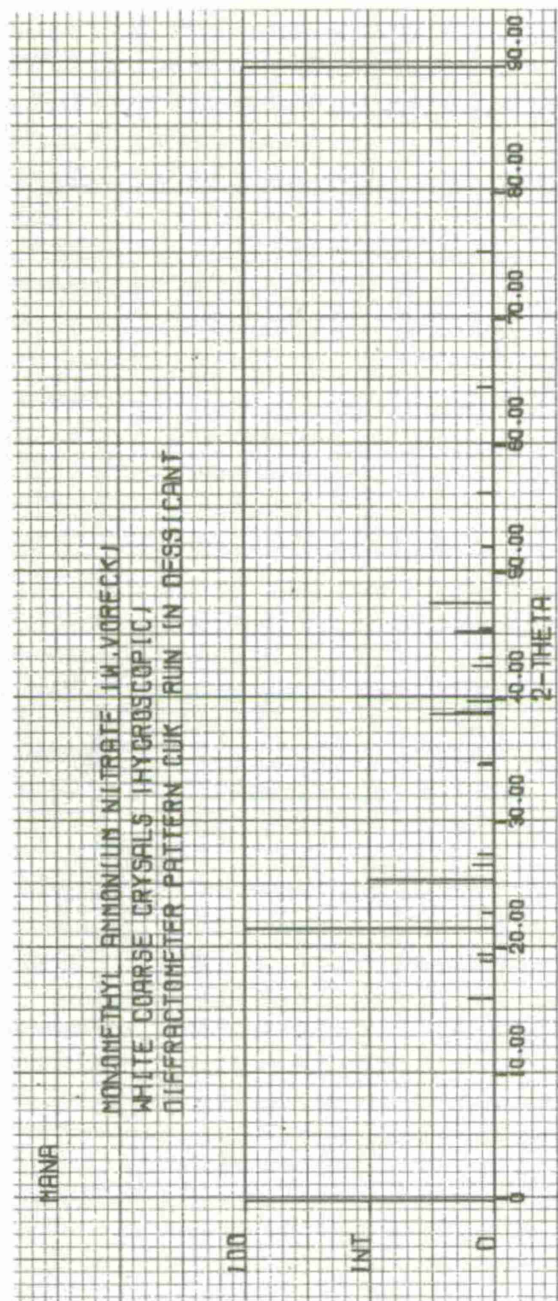
TATB CADY AND LARSON ACTA. CRYST. (1965) 18,485 *
YELLOW FINE POWDER
CALCULATED DIFFRACTOMETER PATTERN

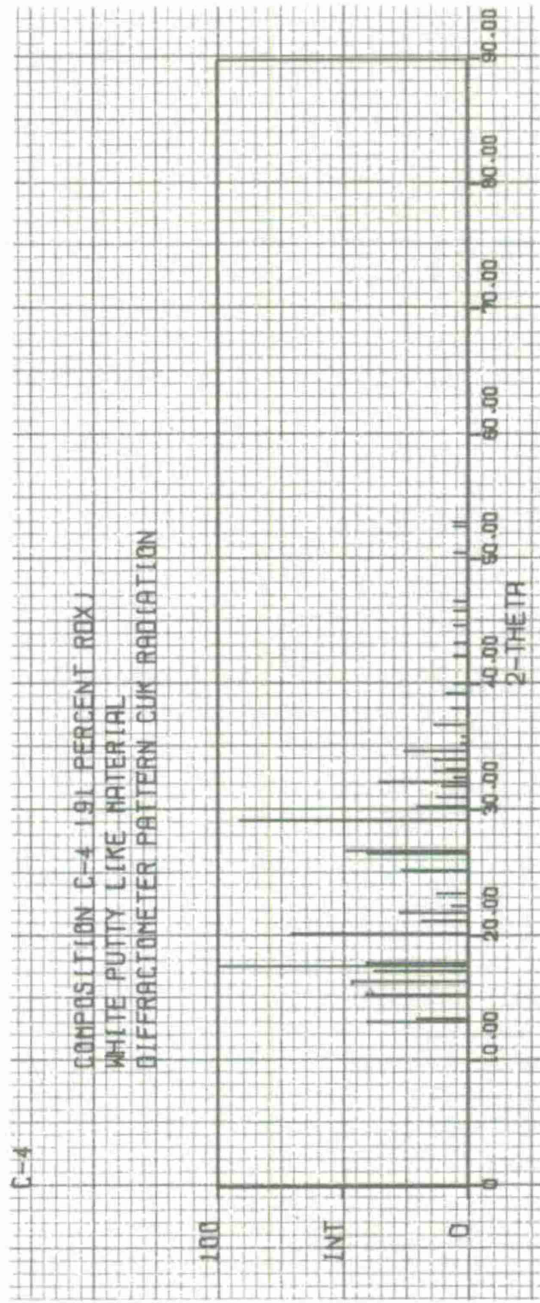
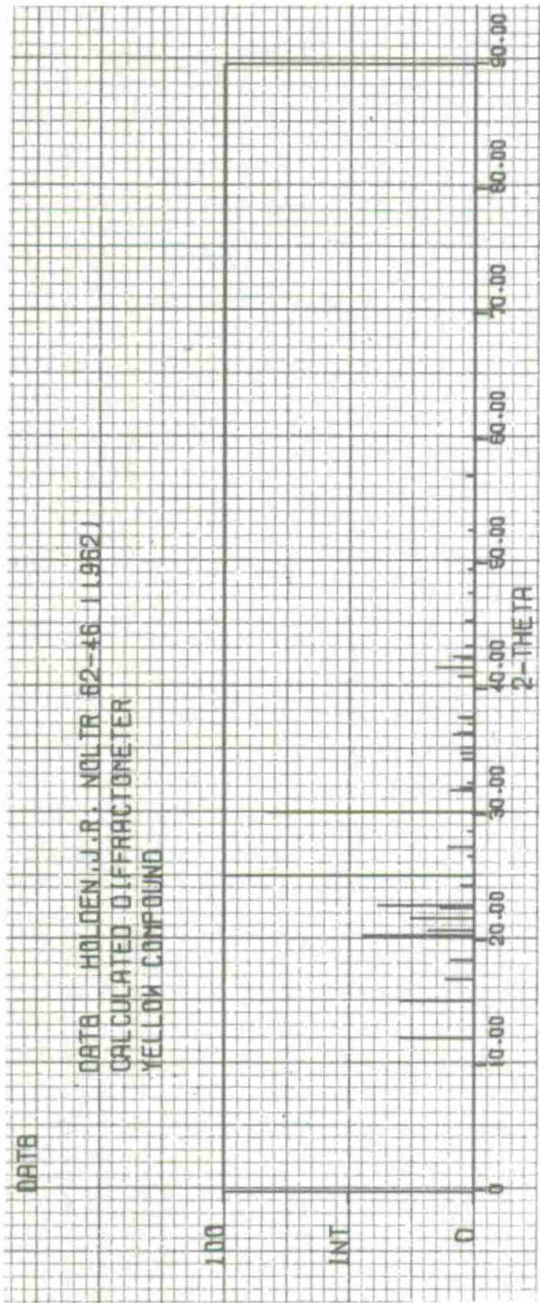


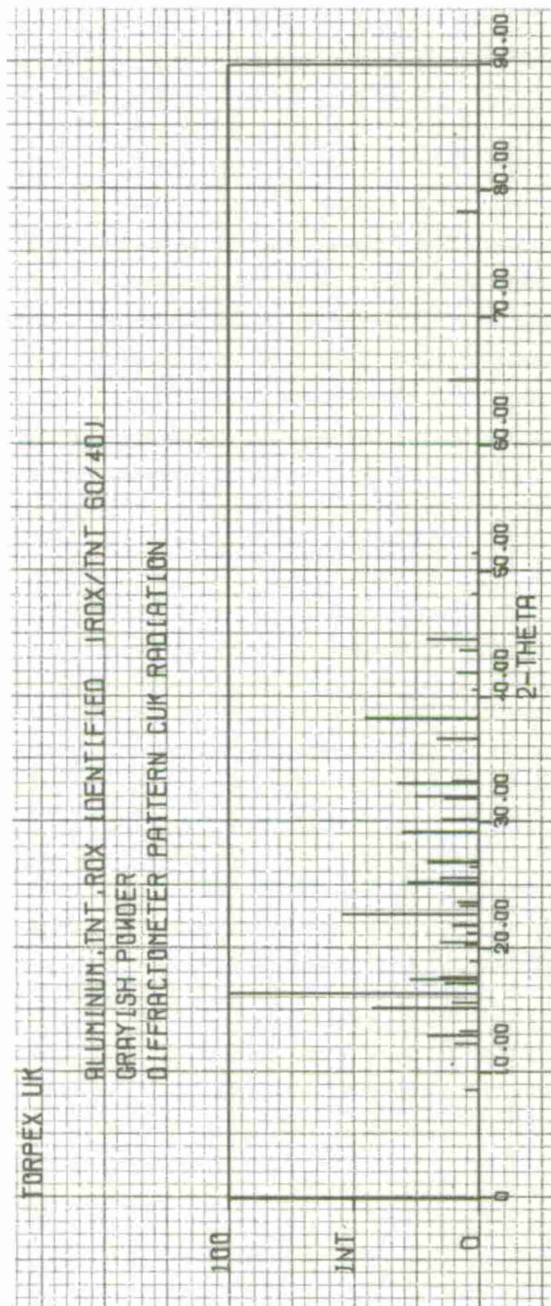
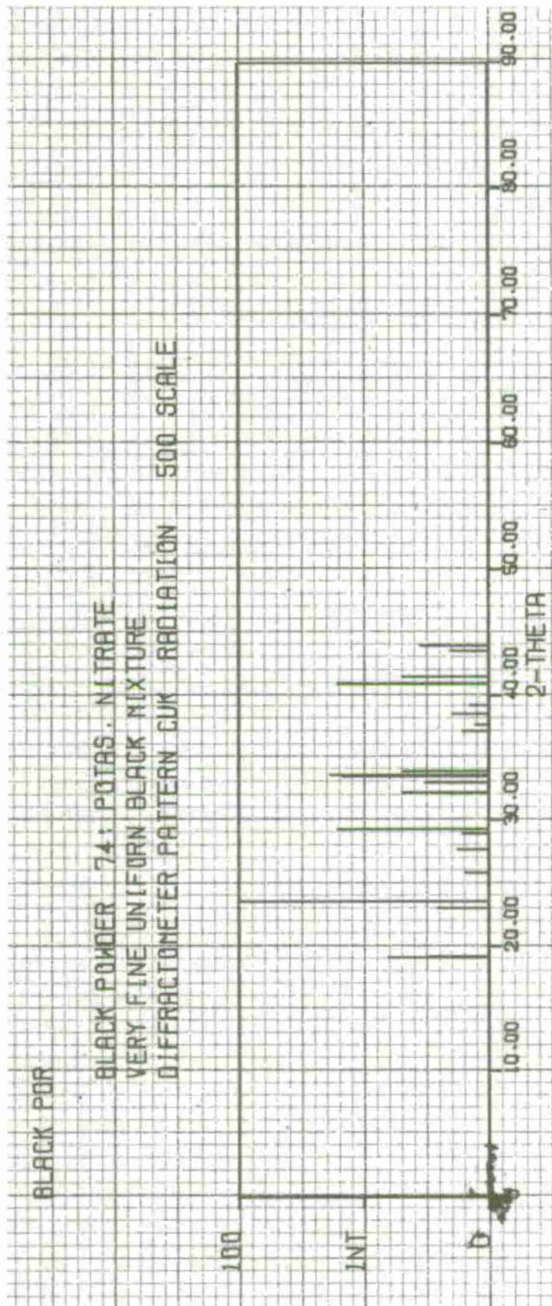


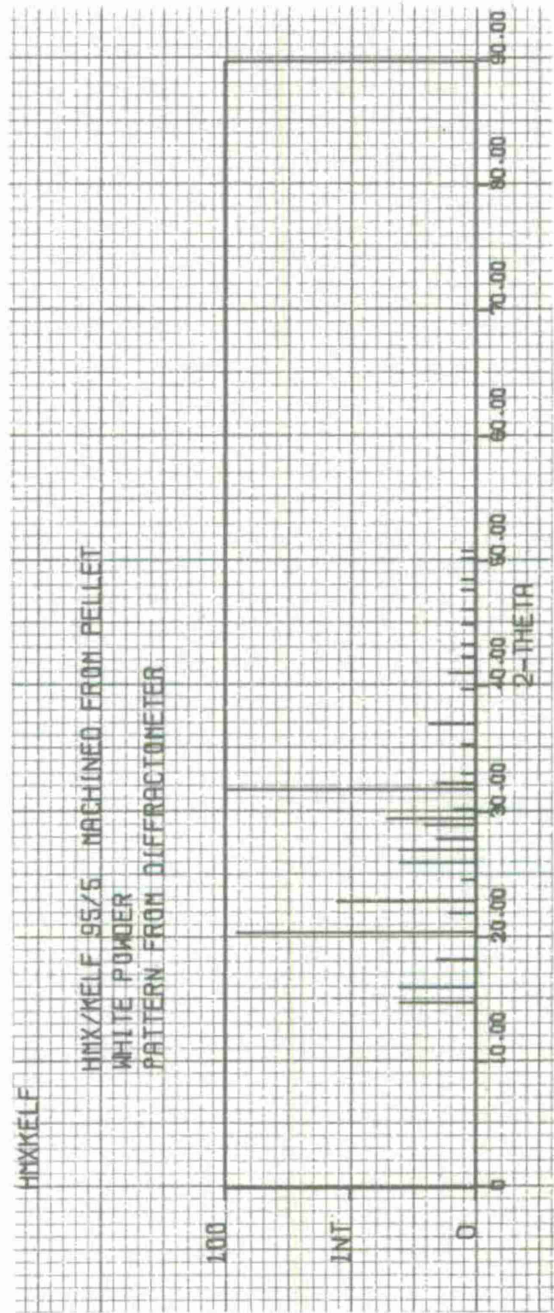
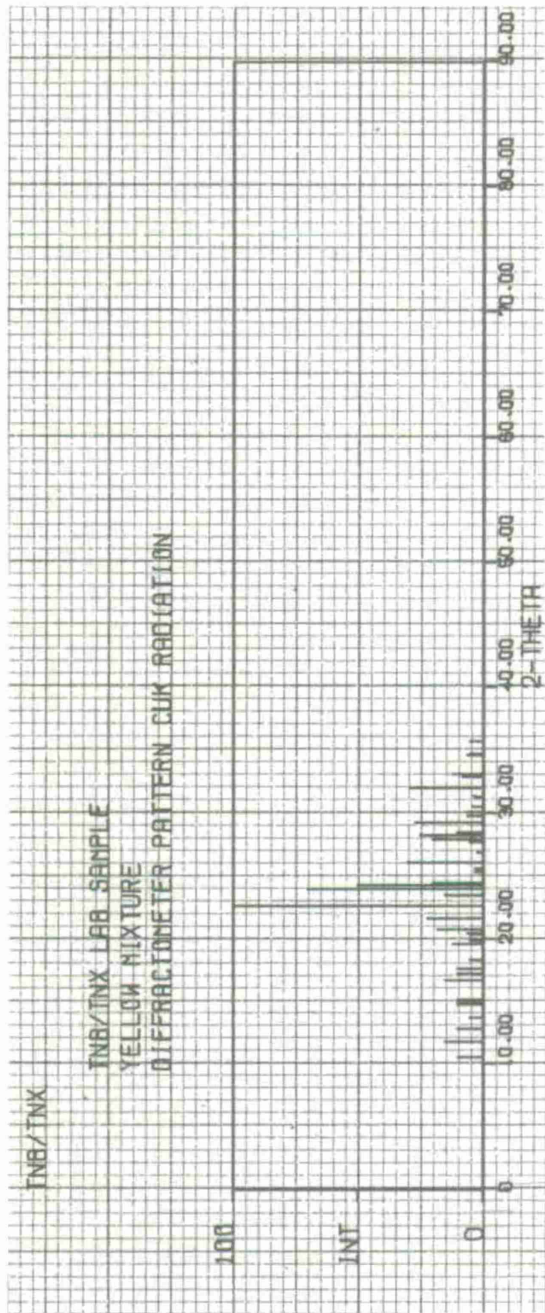


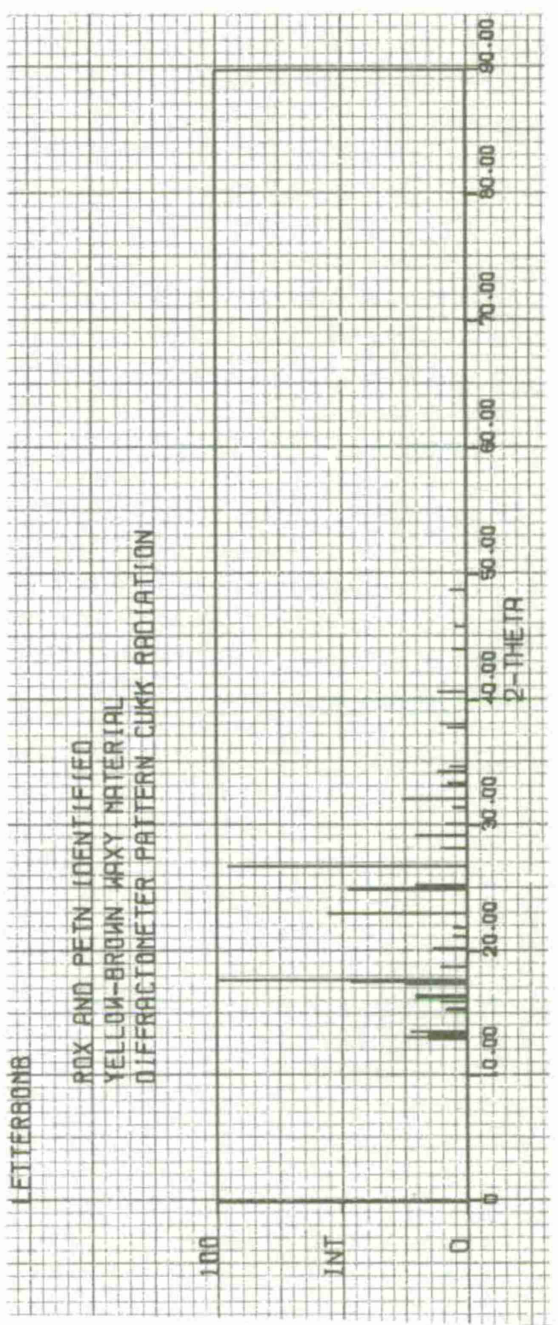
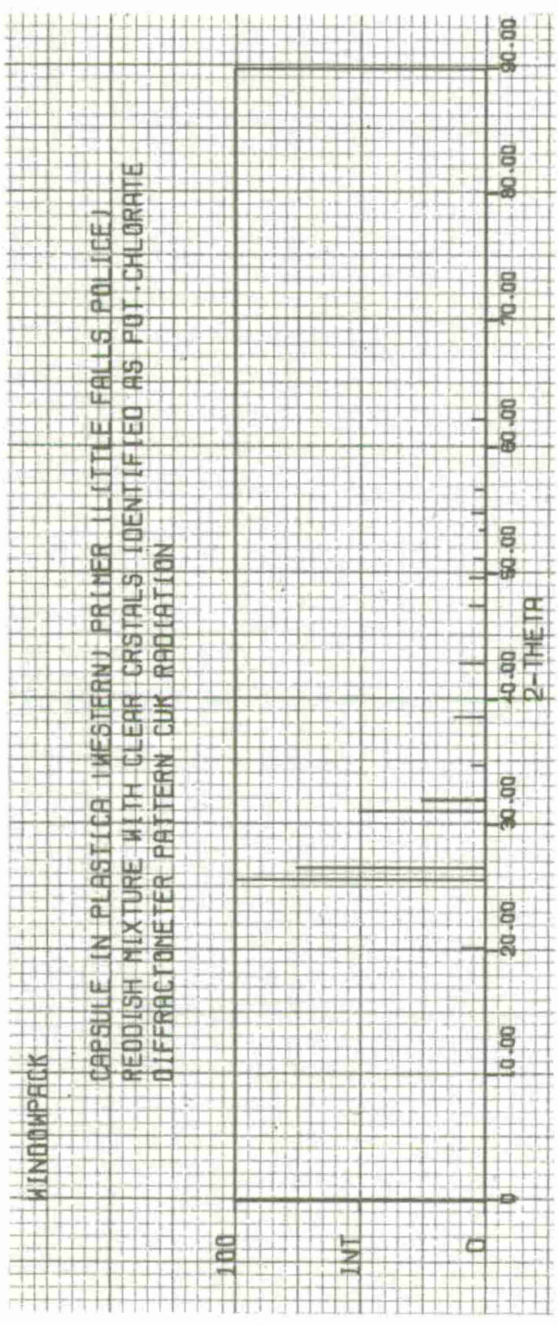


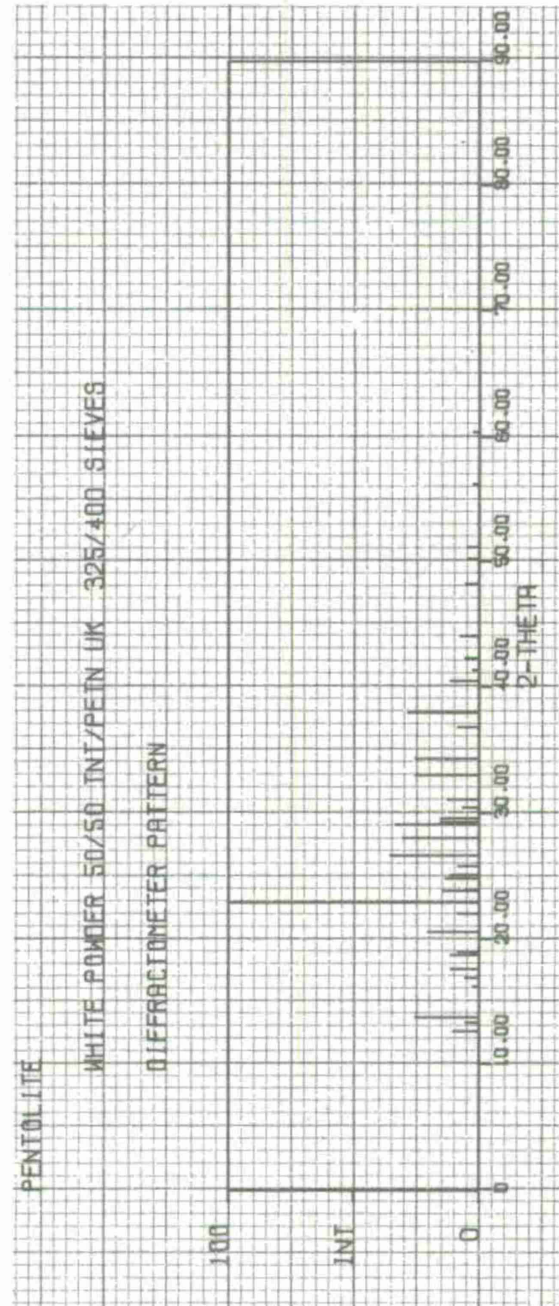
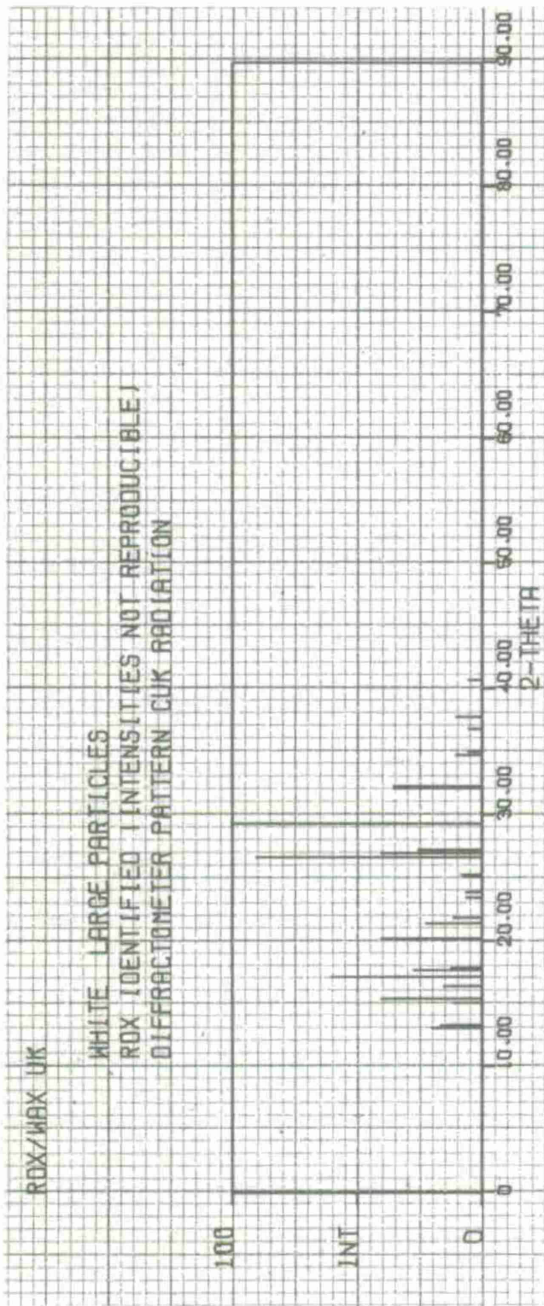


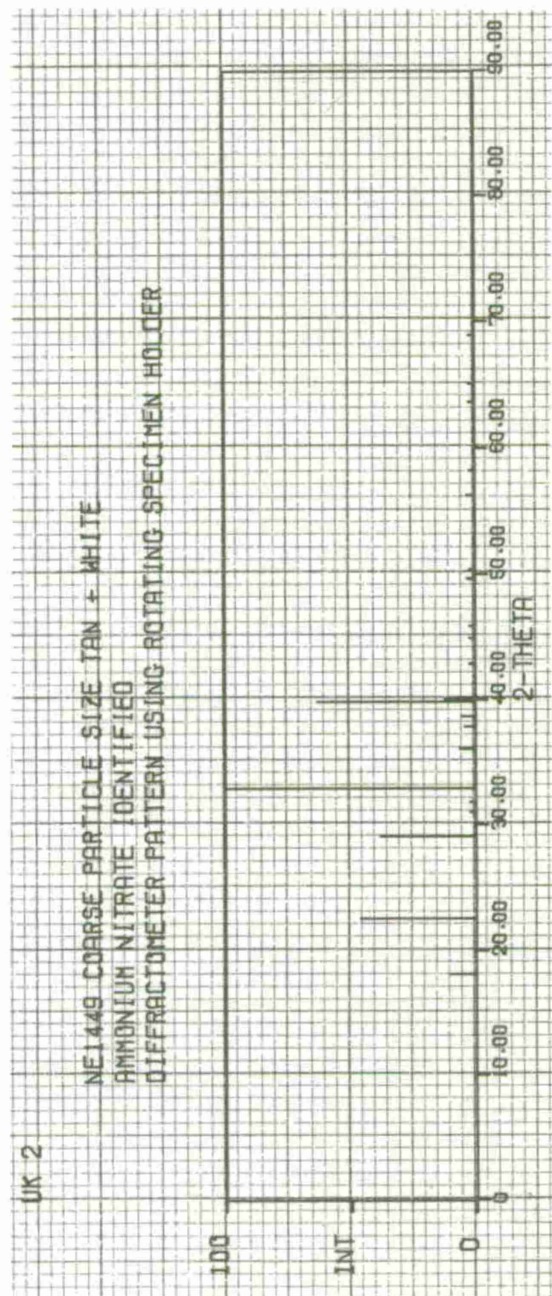
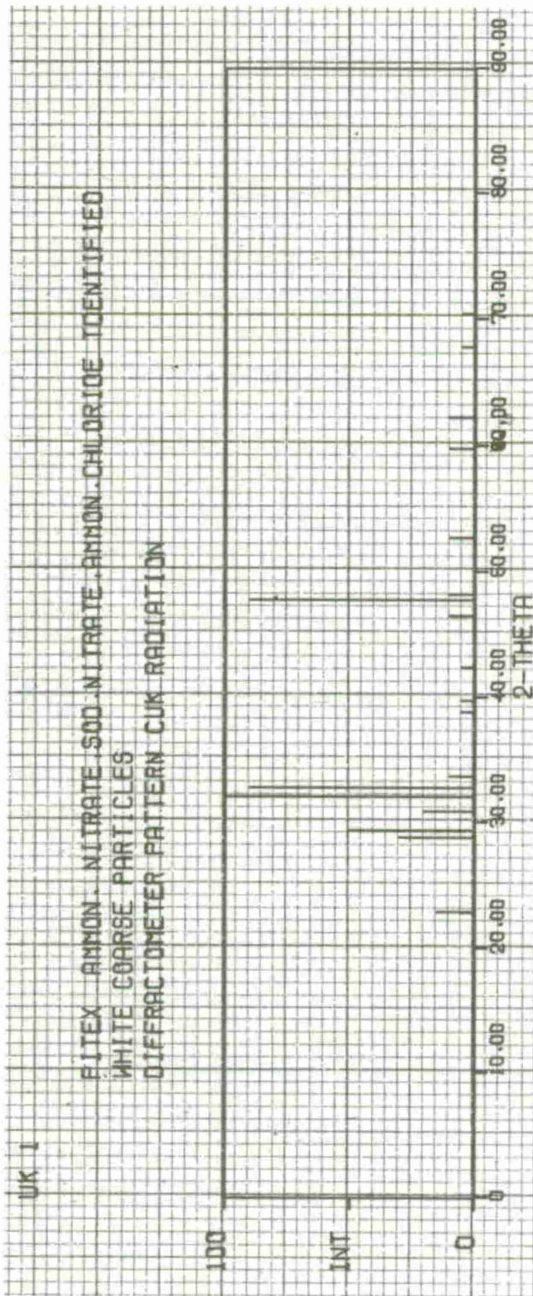


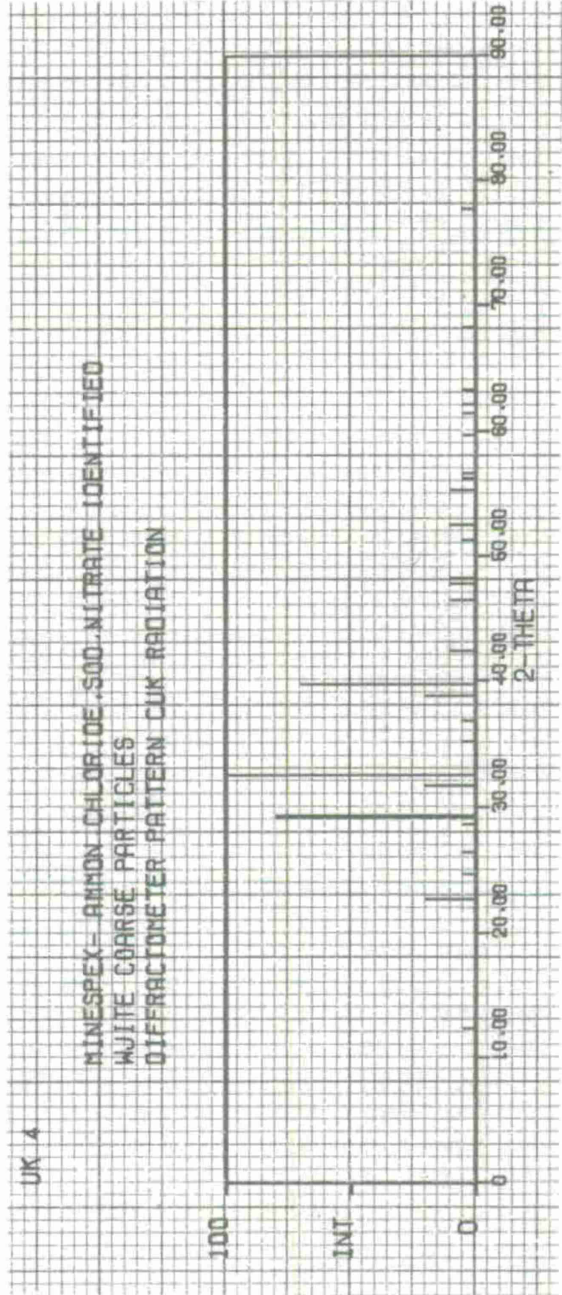
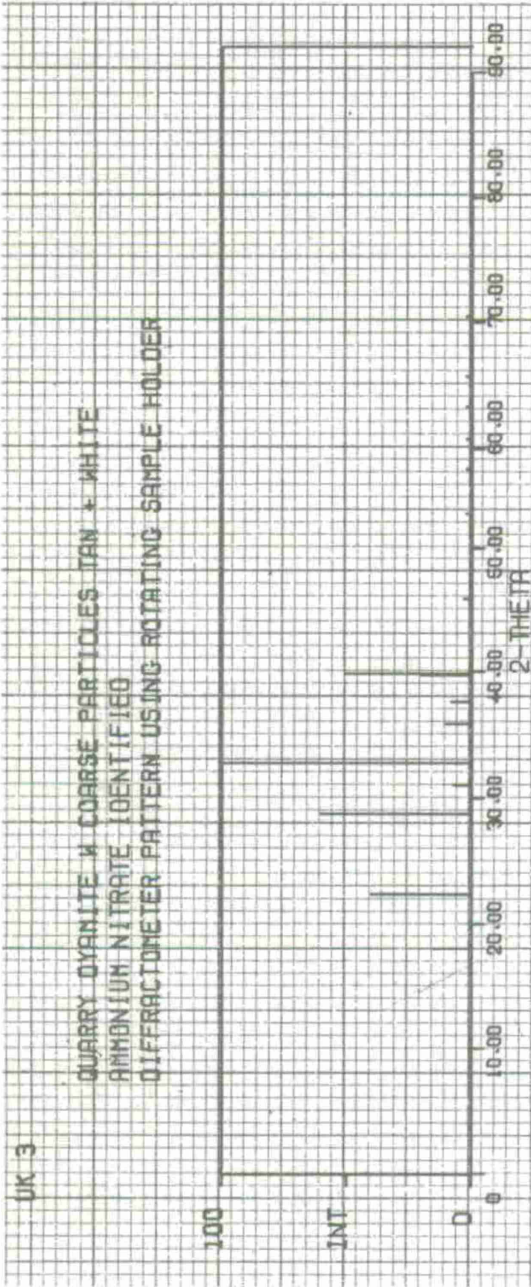


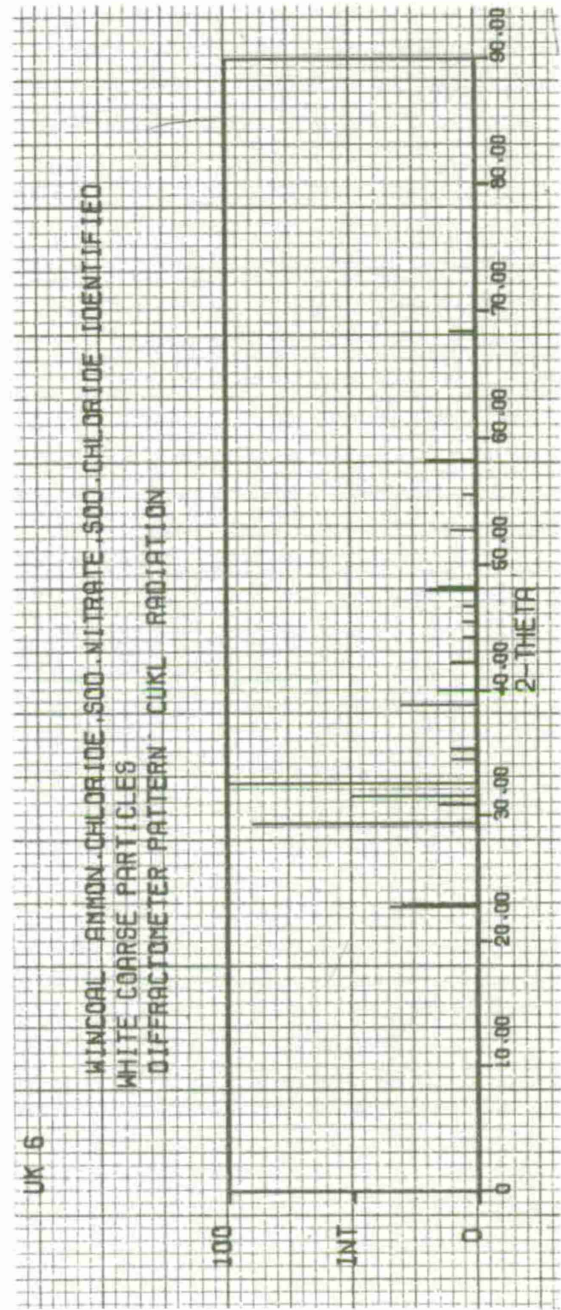
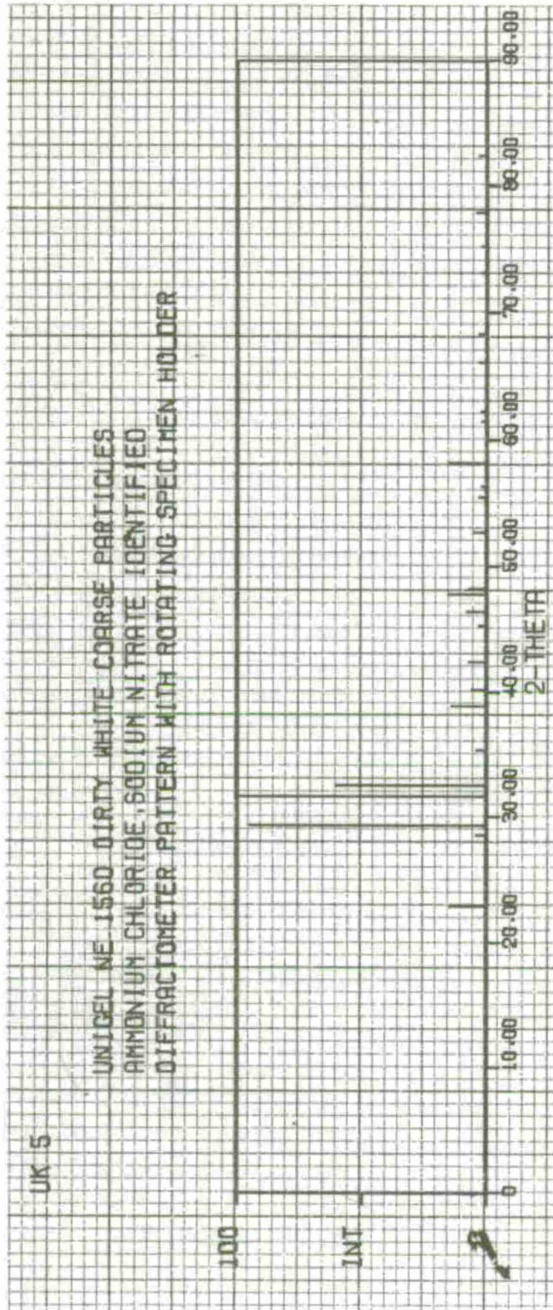


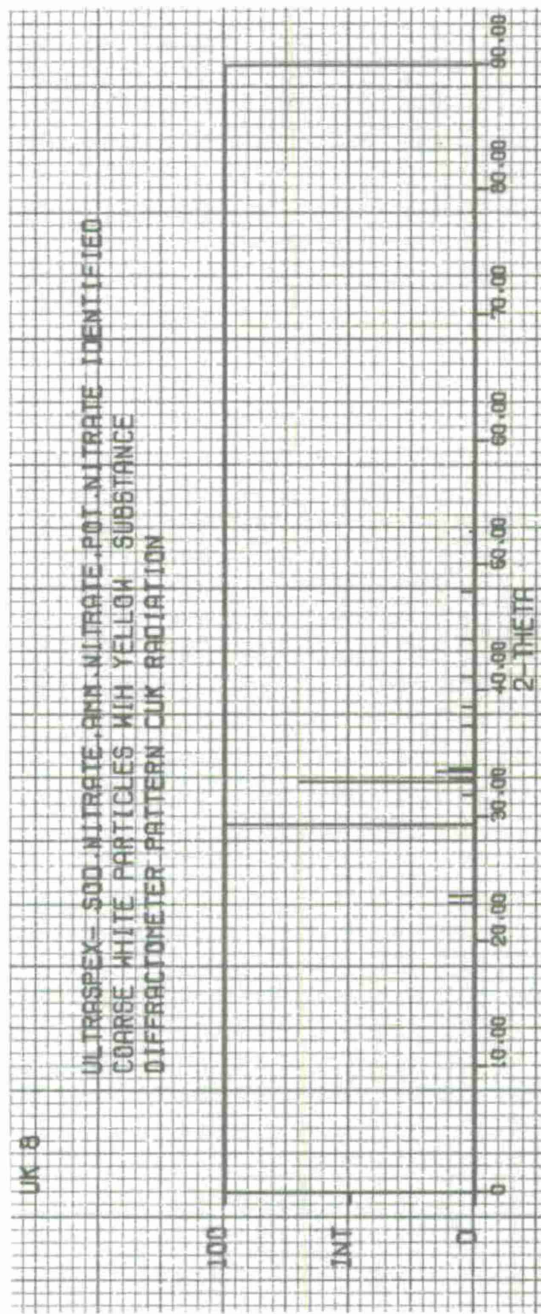
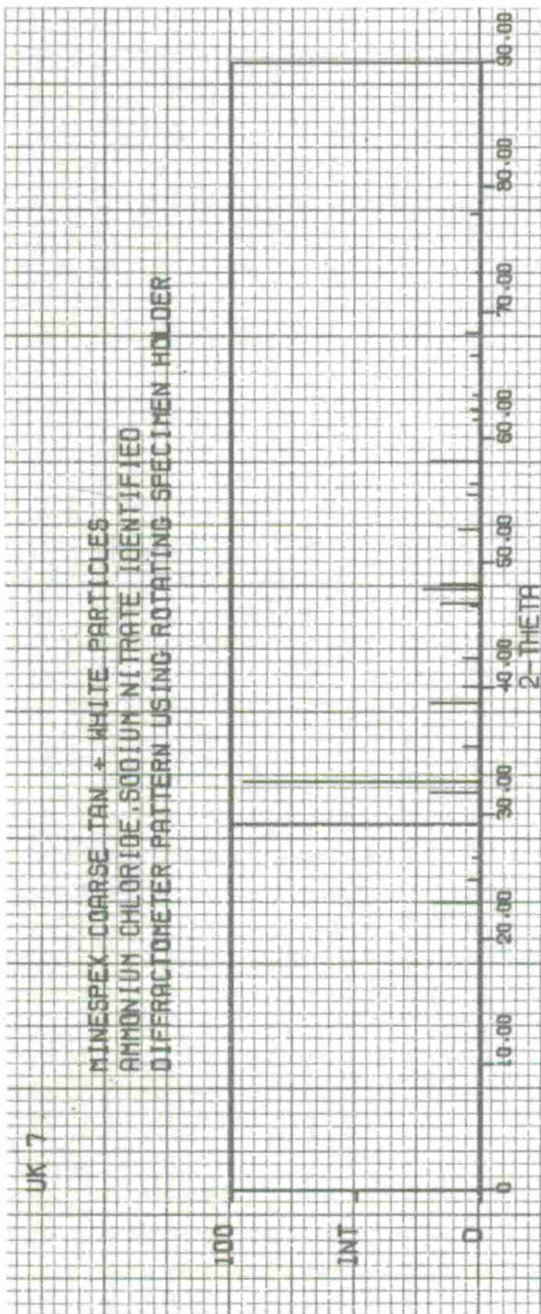


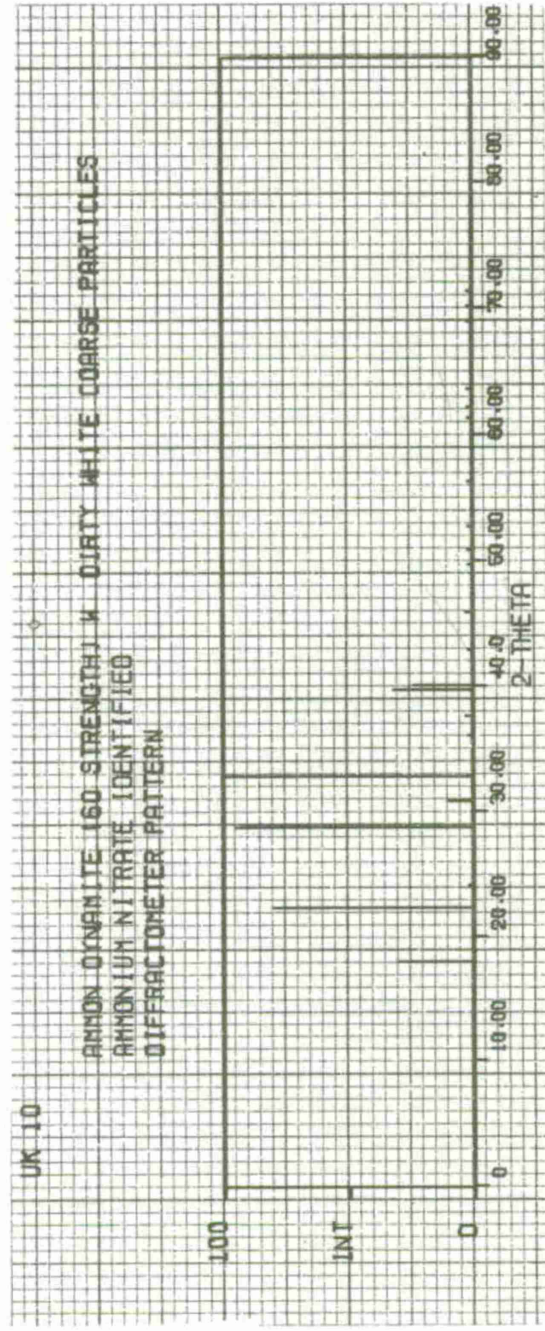
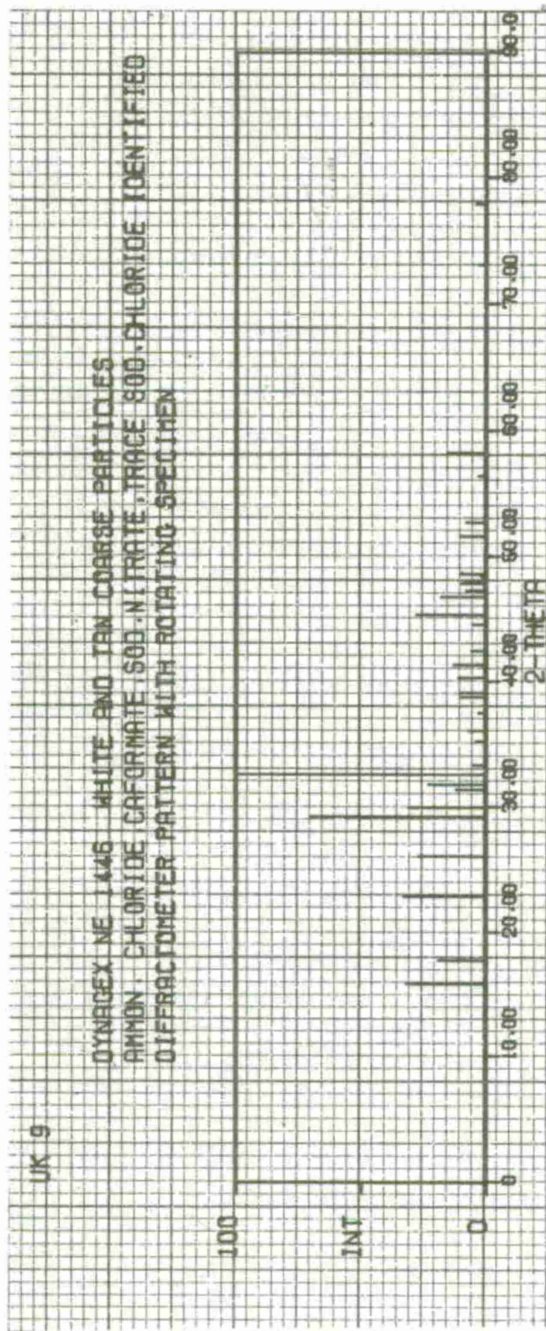


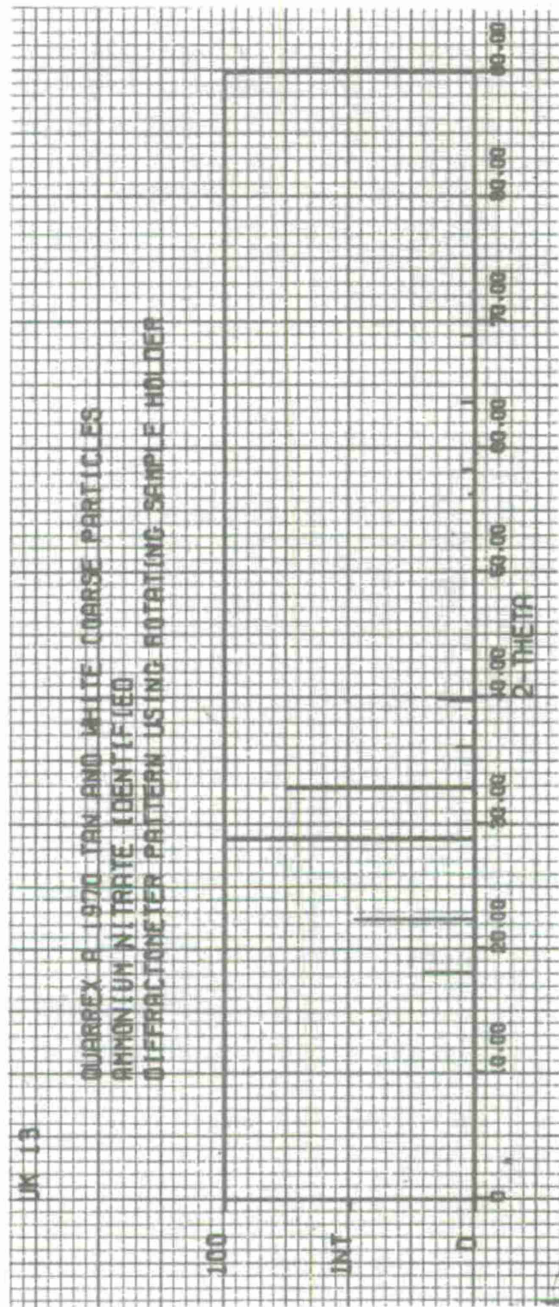
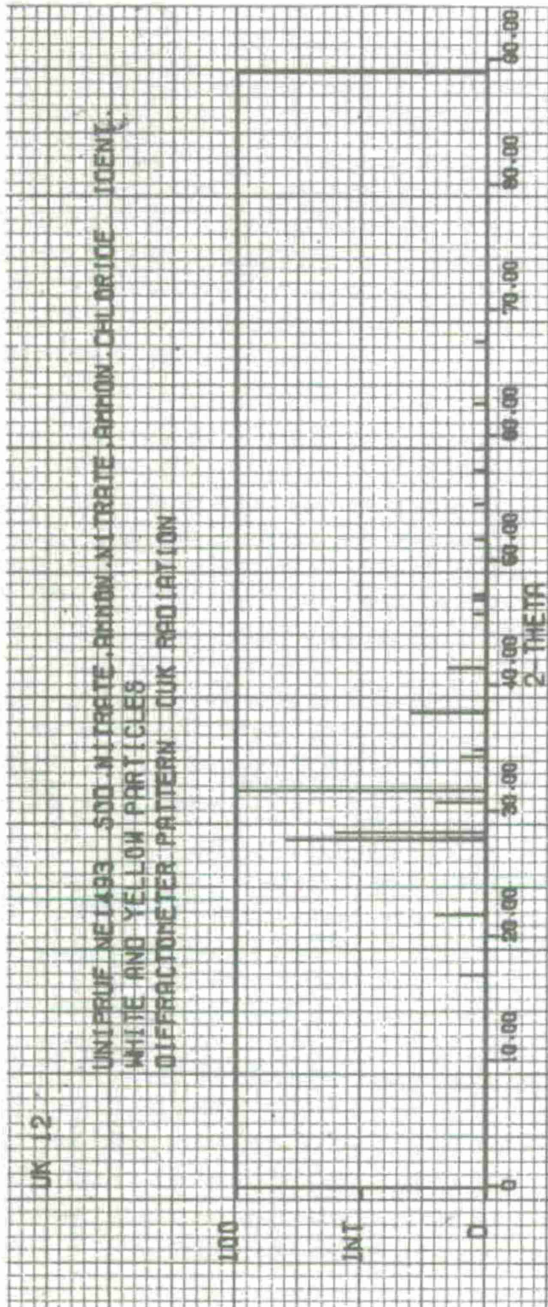


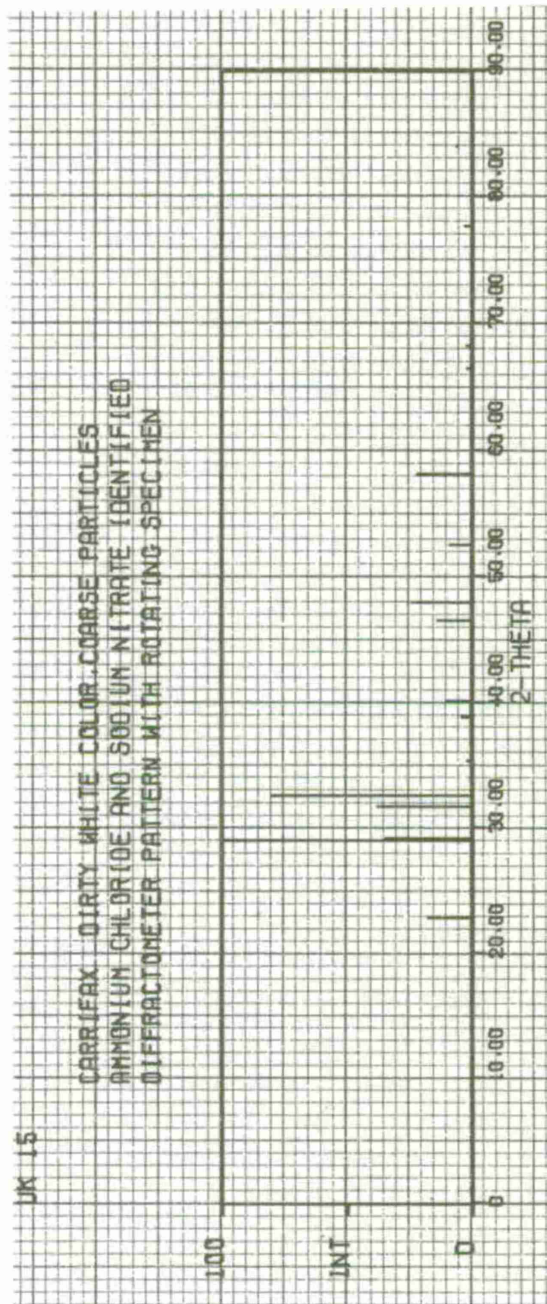
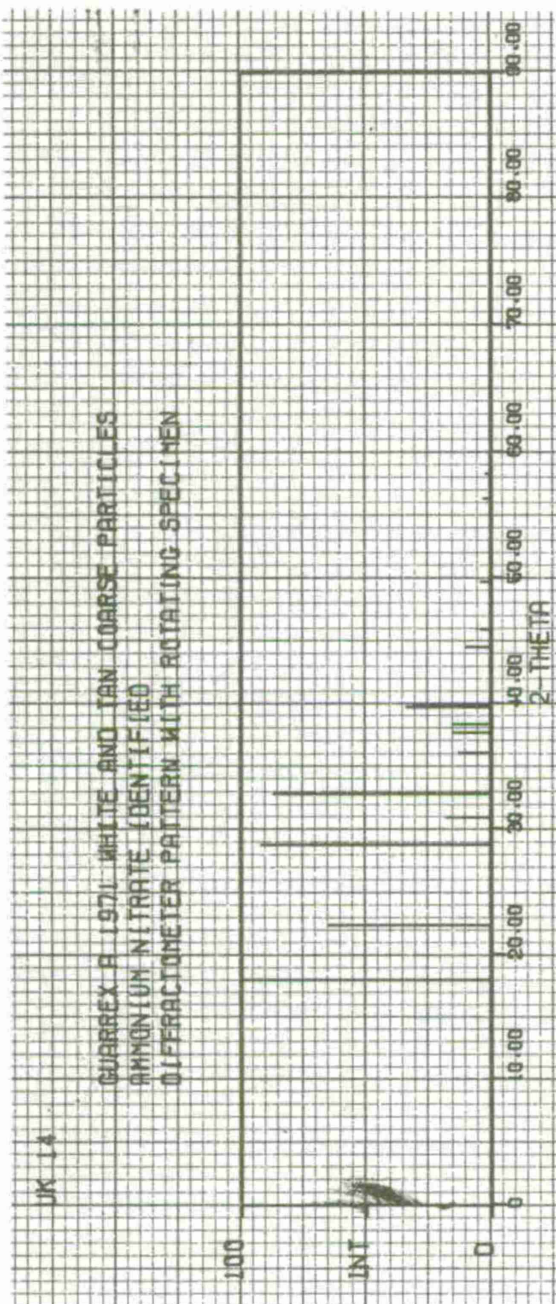


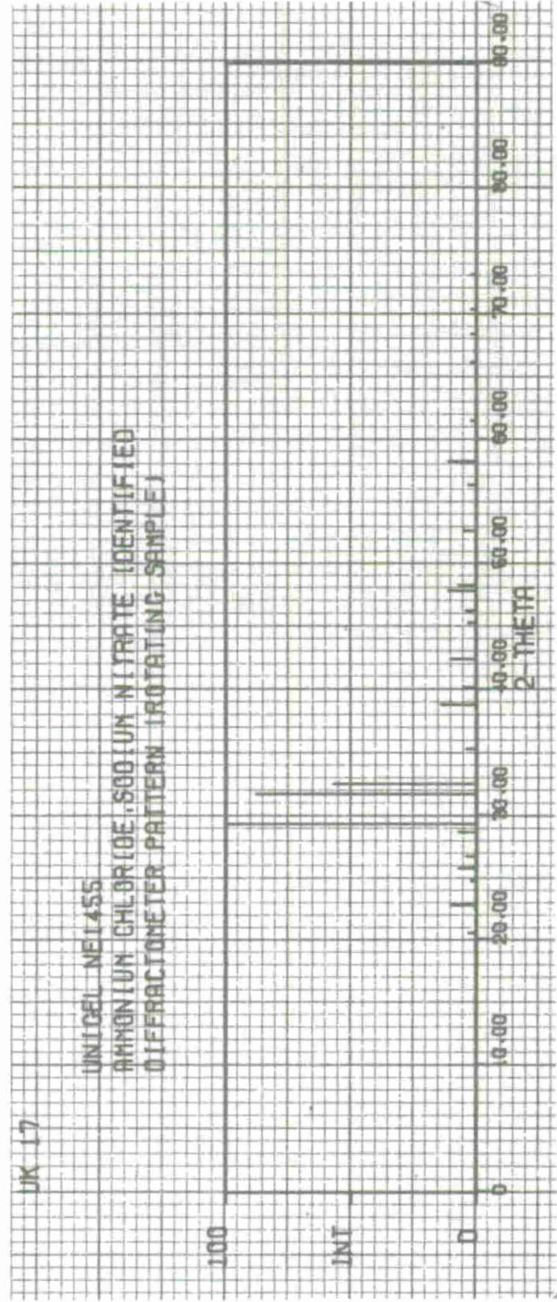
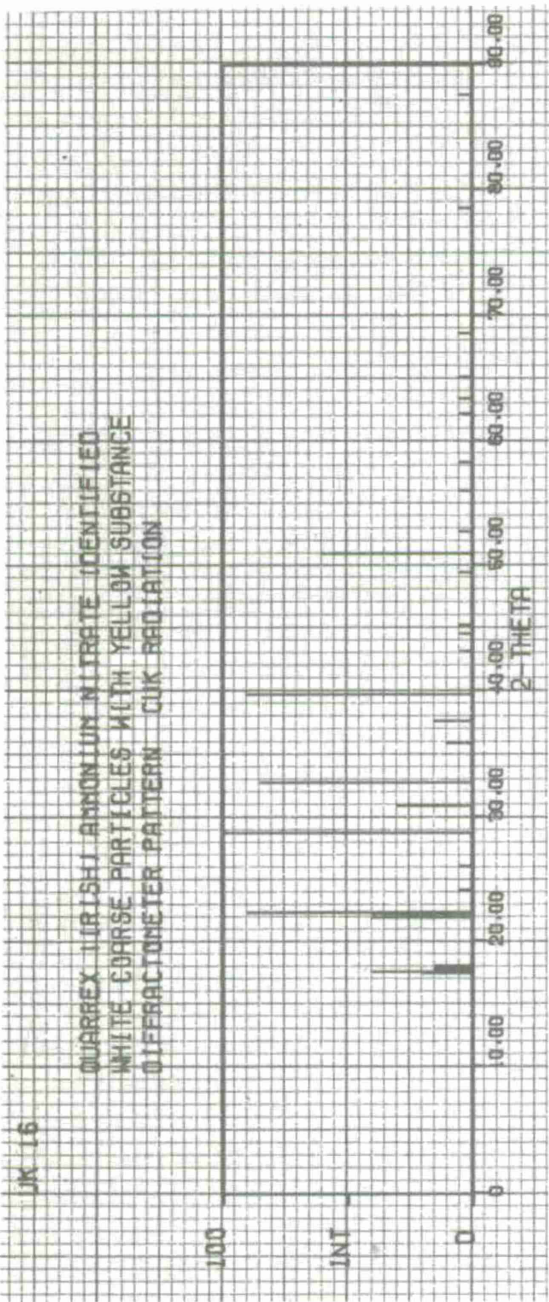


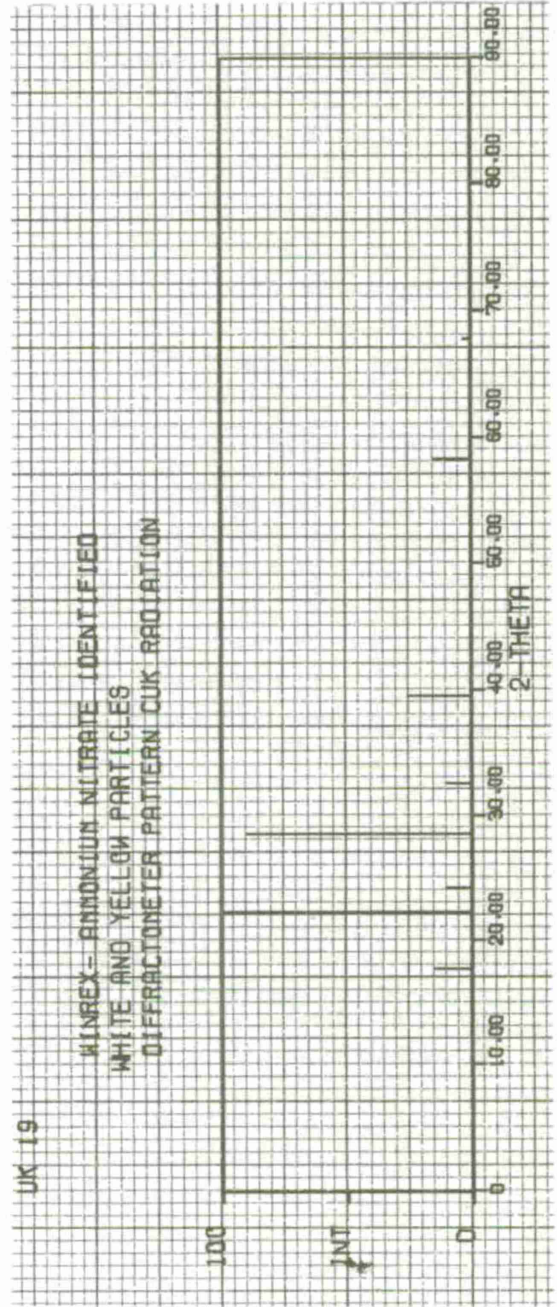
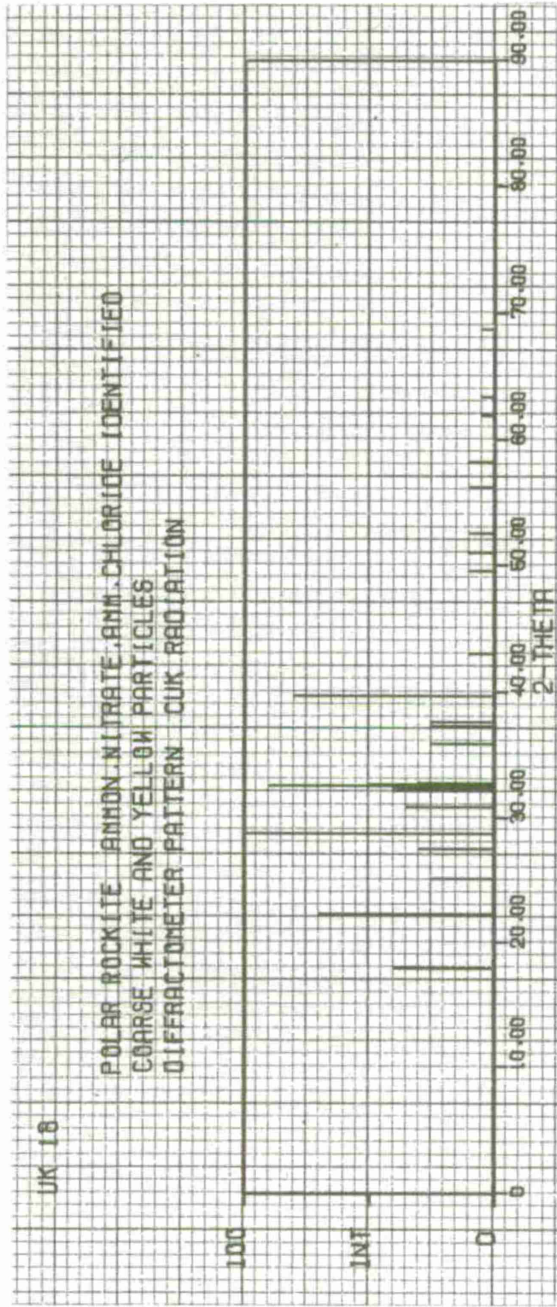


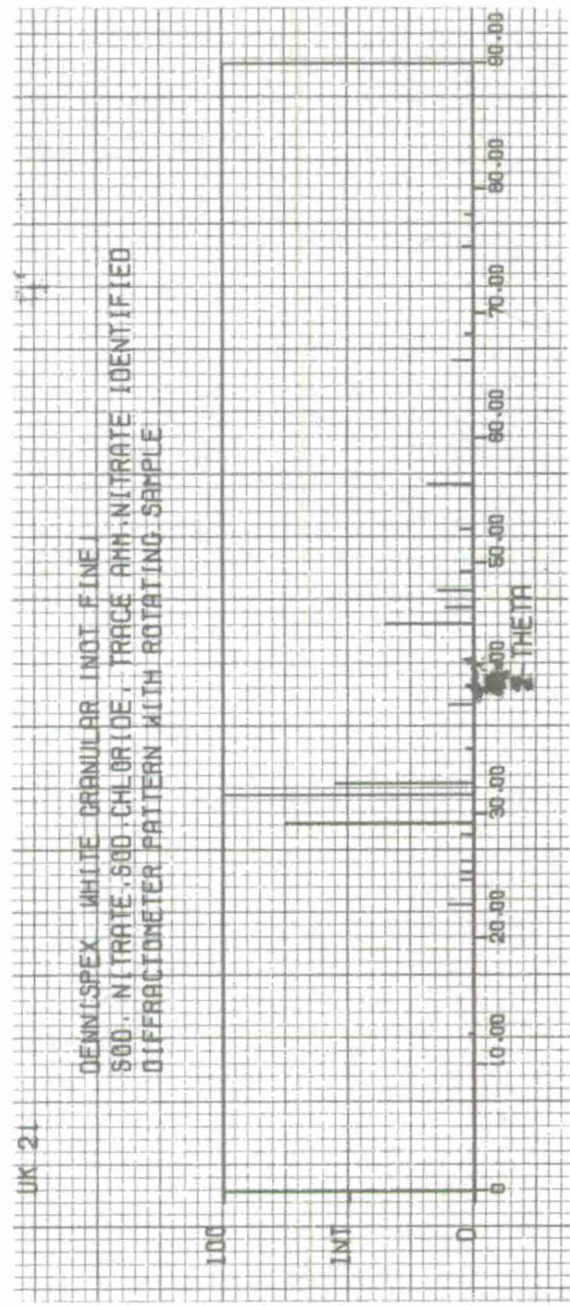
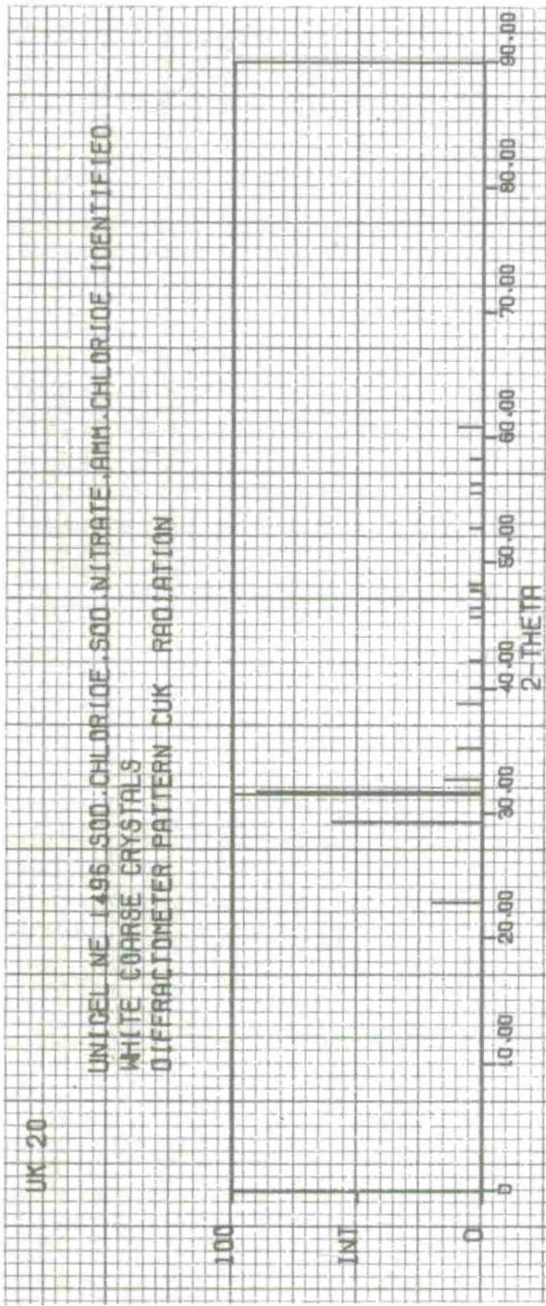


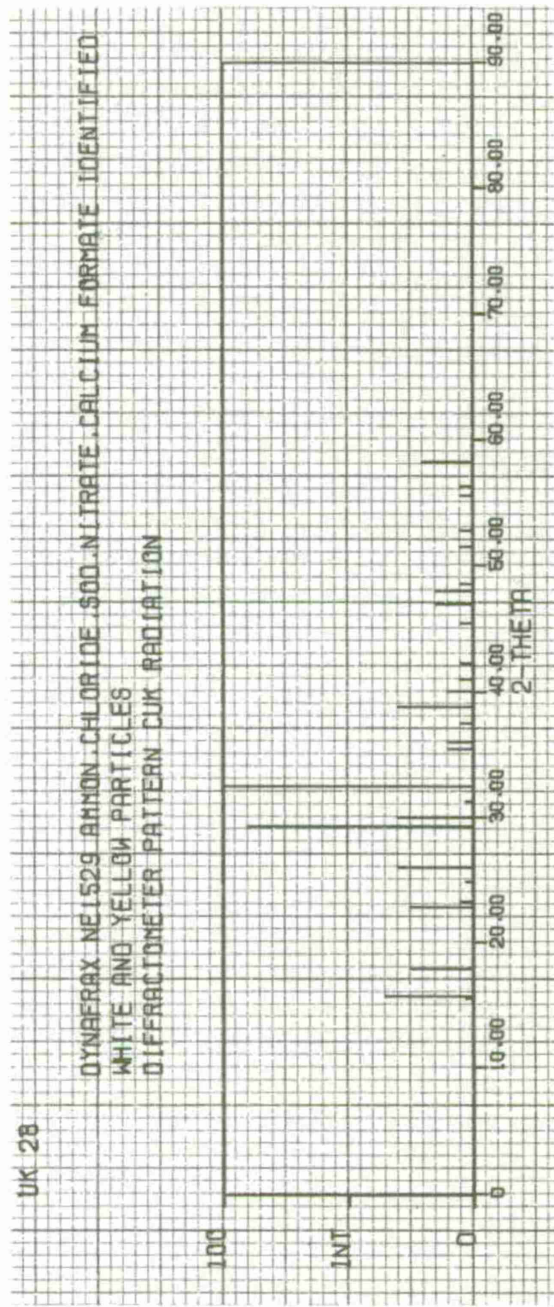
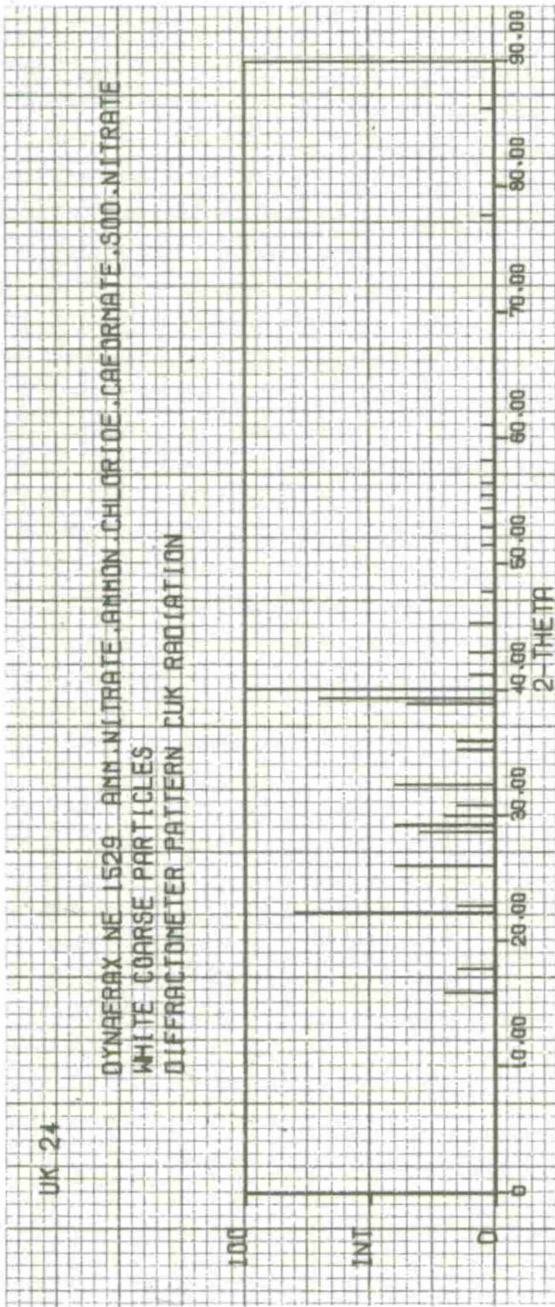


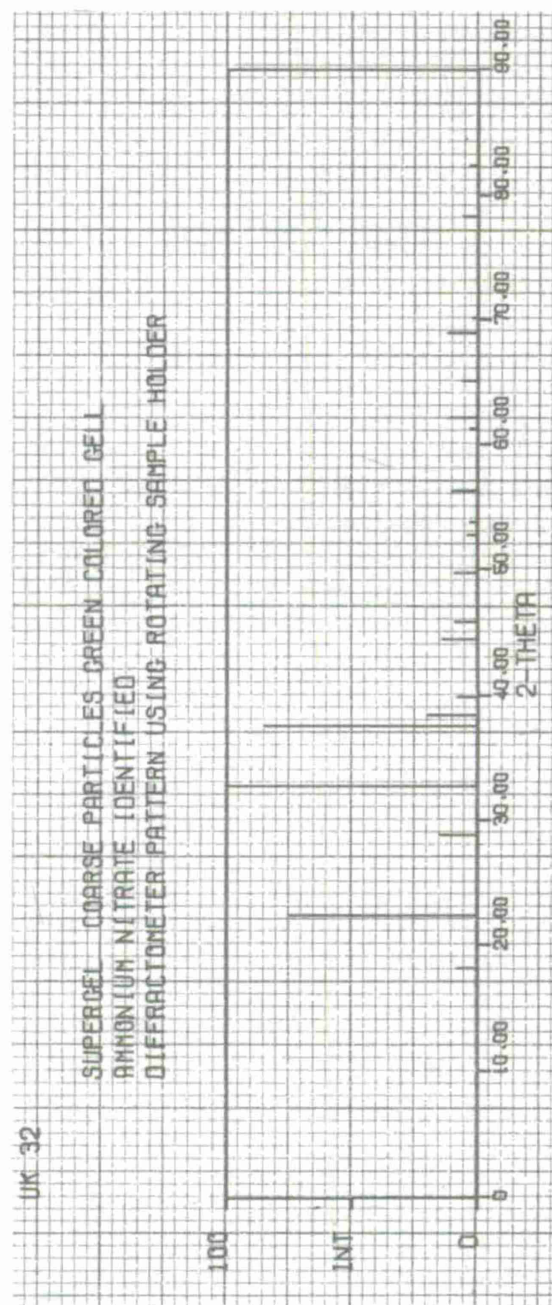
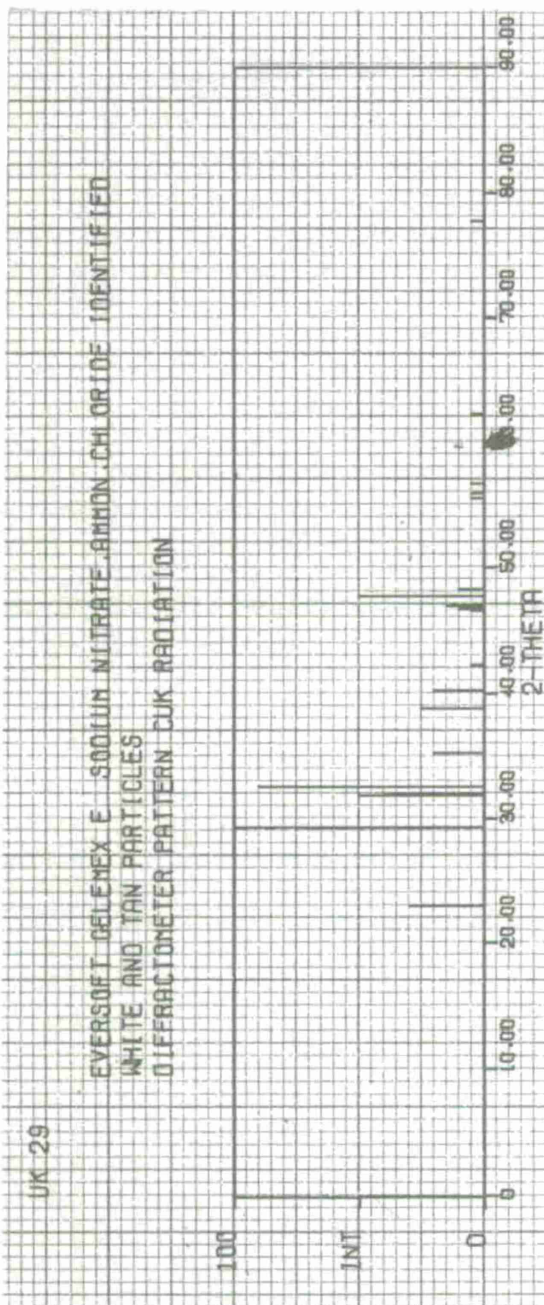






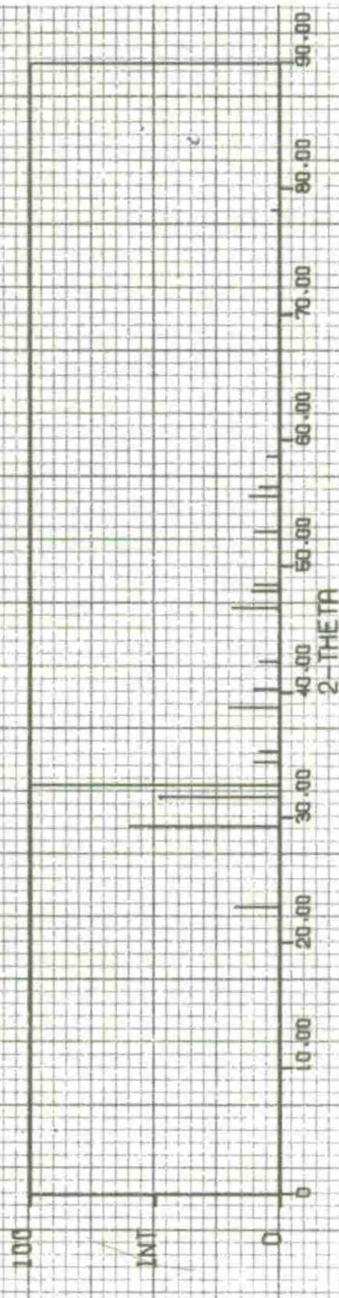






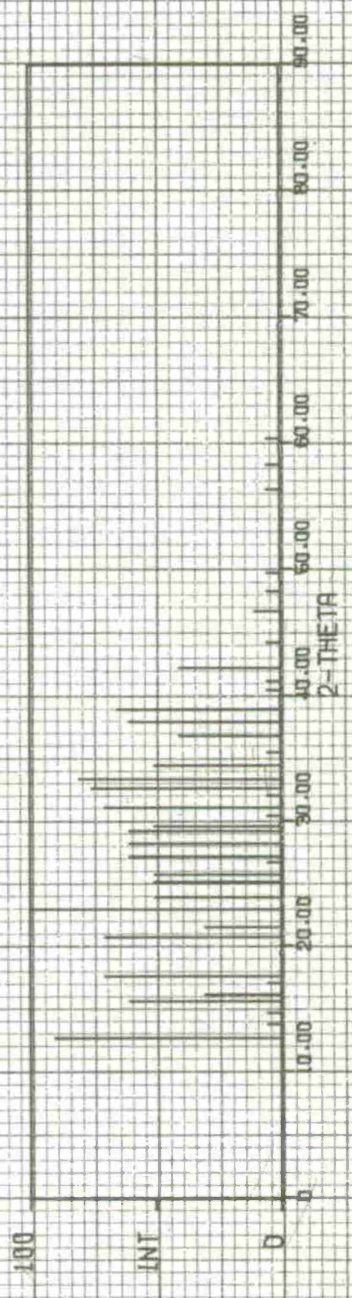
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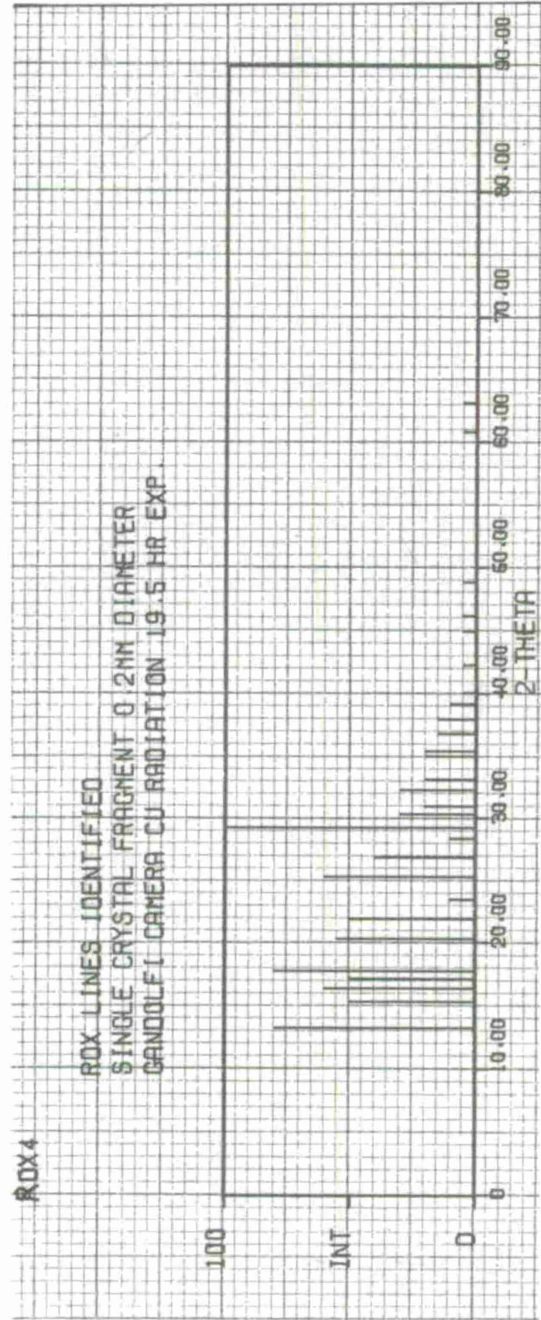
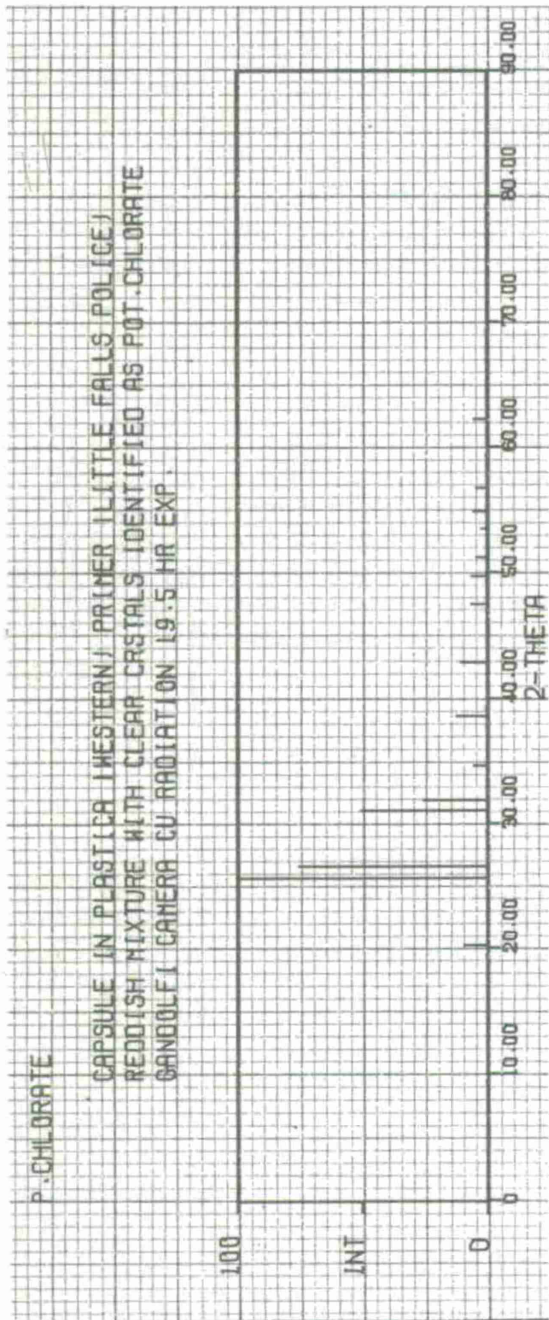
MINCOAL A COARSE WHITE PARTICLES NAT 3000 FOR X-RAY
SODIUM NITRATE IDENTIFIED, AMMON CHLORIDE POSSIBLE, 7 UNIDENTIFIED LINES
DIFFRACTOMETER PATTERN ROTATING SAMPLE

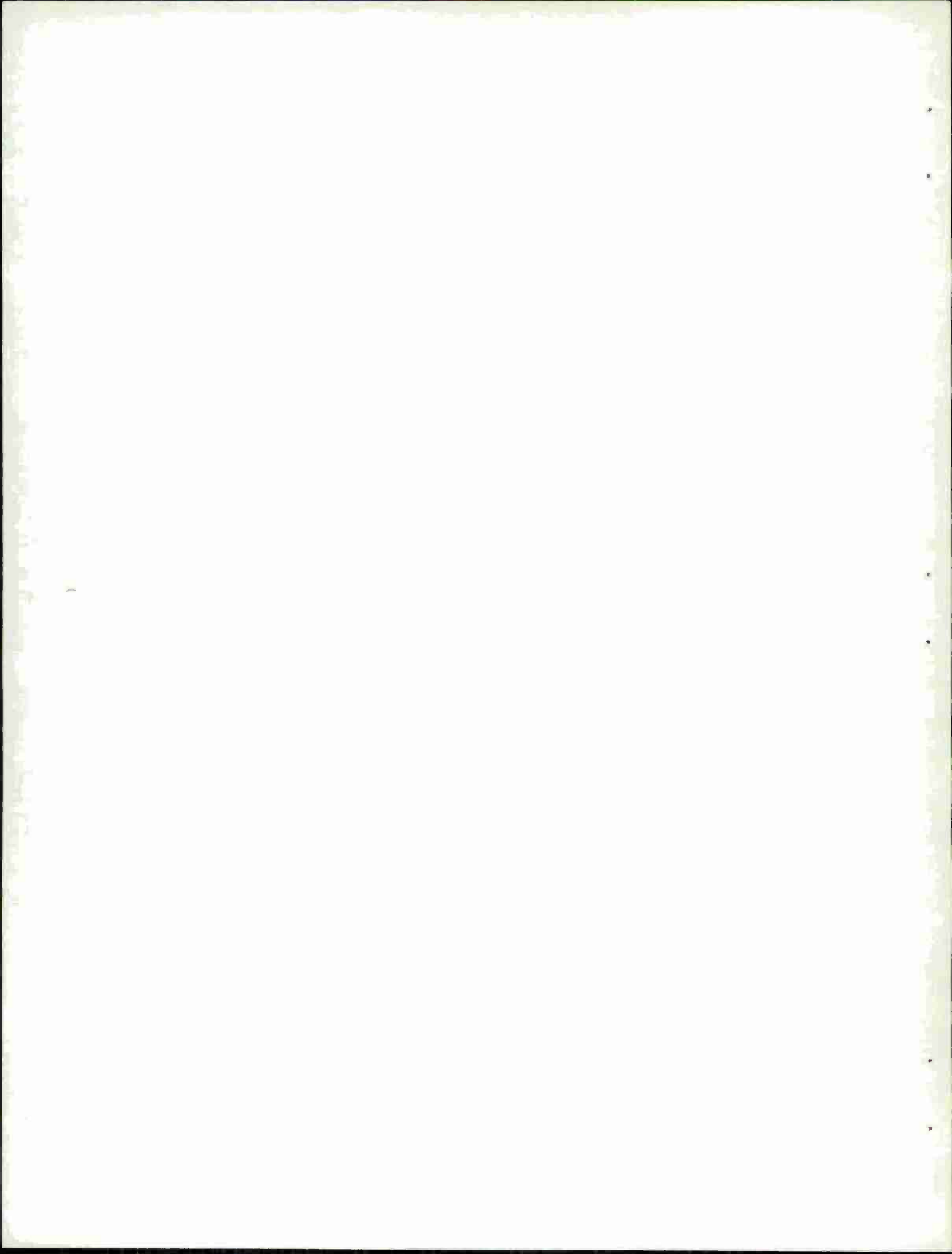


TNT4

ADDITIONAL TNT LINES IDENTIFIED
SINGLE CRYSTAL 0.2MM DIAMETER
GANDOLFI CAMERA CU RADIATION 19.5 HR EXP.



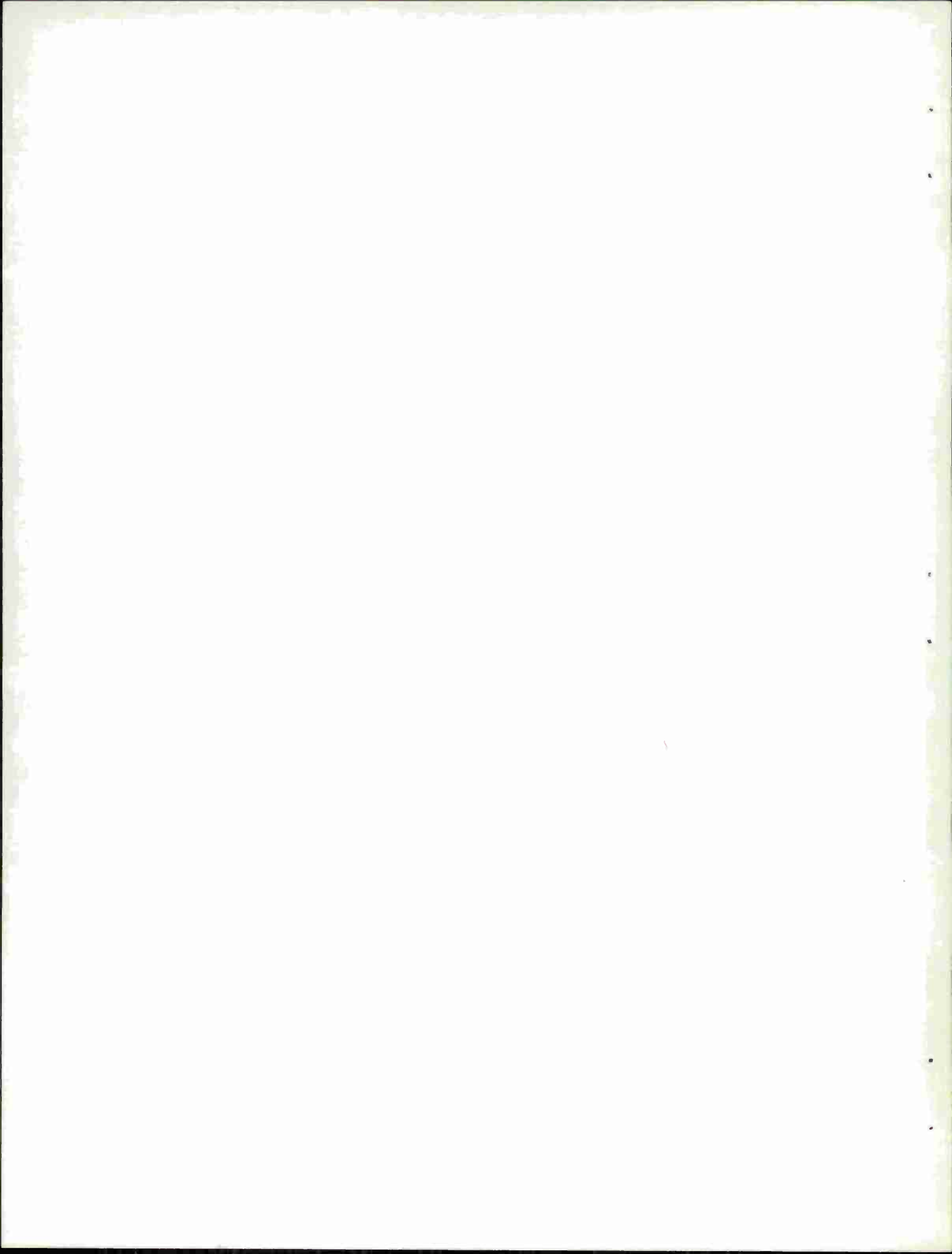




APPENDIX 2

OPTION CARD FORMAT

<u>Column</u>	<u>Digit</u>
1	1 Read Input Data from Cards
2	1 Print Input Cards
3	1 Produce Bar Graphs
4	1 2-Theta Corrected 2 2-Theta For Peak 3 d-spacing
5	1 Integrated Intensity 2 Peak Height Intensity 3 Debye-Scherrer or Gandolfi Intensity 4 Ignore Intensities
6	1 Compare Data with Catalog Files
7	1 Add Data to Catalog Files
8-9	Omit
10	1 Card must contain 1



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