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TECHNICAL REPORT ARLCD-TR-77066

INVESTIGATION OF PROPELLANT AND EXPLOSIVE
SOLID SOLUTION SYSTEMS II X-RAY STUDIES

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Report ARLCD-TR-77066	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) INVESTIGATION OF PROPELLANT AND EXPLOSIVE SOLID SOLUTION SYSTEMS II X-RAY STUDIES	5. TYPE OF REPORT & PERIOD COVERED	
	6. PERFORMING ORG. REPORT NUMBER	
7. AUTHOR(s) Scott I. Morrow Joseph J. Campisi Arthur J. Bracuti	8. CONTRACT OR GRANT NUMBER(s) AMCMS Code 501A.11.84400 DA Proj No. 1T061101A91A	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Feltman Research Laboratory Picatinny Arsenal Dover, NJ 07801	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
11. CONTROLLING OFFICE NAME AND ADDRESS ARRADCOM ATTN: DRDAR-TSS Dover, NJ 07801	12. REPORT DATE March 1978	
	13. NUMBER OF PAGES 36	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) ARRADCOM ATTN: DRDAR-LCE Dover, NJ 07801	15. SECURITY CLASS. (of this report) Unclassified	
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release, distribution 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) Ammonium Perchlorate (AP) Addition Compound 3KN•AN Potassium Perchlorate (KP) Oxidizer solid solutions Ammonium Nitrate (AN) Isomorphism Potassium Nitrate (KN)		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Debye Scherrer and diffractometer powder techniques were used to character- ize isomorphous oxidizer solid solutions. Those involved were: AP-KP, AN-AP, AN-3KN•AN, KN-3KN•AN, and AN-KP. In most cases we showed that the x-ray powder patterns were different from physical mixtures with the same relative amounts of constituents. Observations were made on the shift of major diffraction lines with introduction of an isomorphous oxidizer component into another oxidizer		

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20. ABSTRACT (Continued)

host crystal. The x-ray powder patterns helped to illustrate the unique nature of the solid solutions involved. Some theoretical speculation was made on the possibility of the existence of superstructure in such materials, particularly the AP-KP solid solutions.

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INTRODUCTION

The Feltman Research Laboratory had been conducting a long-term study of isomorphous oxidizer solid solutions or mixed crystals (refs. 1 and 3 through 6). These crystals have potential applications in solid propellant rocket motors and gun propellant systems. The synthesis of these oxidizer solid solutions is described in reference 7.

One of the more interesting aspects about the characterization of the experimental solid solutions is their structures. In ammonium perchlorate-potassium perchlorate (AP-KP) materials, both crystals are closely related isomorphously; at normal temperatures they are orthorhombic, but at higher temperatures they become cubic. According to Simanova and Shul'ts (ref. 8), when this salt pair is cocrystallized from water at 25.0°C, a continuous series of mixed crystals is formed over the whole composition range. However, it appears that little effort has been made to study these and other similar oxidizer solid solutions by direct methods.

It is particularly desirable to carry out x-ray diffraction studies of mixed crystals to determine their structure wholly or in part. AP-KP solid solutions (where no discontinuities in structure were to be expected over the whole compositional range at normal temperatures) required a precision lattice constant study. A precision technique was needed because of the relatively small difference in unit cell dimensions of AP and KP (ref. 9).

It is not advantageous for most purposes to use single crystal x-ray techniques for microcrystalline products like ours because there is some uncertainty in using any single crystal to represent the aggregate. The preferred method is to pulverize the product and analyze it by the Debye Scherrer or diffractometer x-ray methods. (This is a less useful approach for determining structure than single crystal methods.) It is possible to make certain limited speculations about the changes in structure of one component upon the introduction of another into its crystal lattice based upon x-ray powder pattern data. Also, the use of powder methods is advisable in cases like ours where a complete structure determination is not intended.

There has been some interest in connection with this work in speculating whether structural ordering exists in some of the products. This subject has been discussed for a gold-copper alloy solid solution, AuCu_3 , by Guinier (ref. 2). If the alloy is quenched from a temperature above 425°C, its x-ray diffraction pattern displays face-centered cubic symmetry as do the diffraction patterns of gold or copper. On the other hand, if the

alloy is cooled gradually instead of being rapidly quenched, the x-ray diffraction pattern is more complex. This is a direct result of the ordering of the structure. The additional new diffraction lines are due to x-ray reflections from new planes of atoms formed by the ordering process. The term "superlattice" is used to describe this formation of a secondary structural arrangement by the process of ordering.

From the standpoint of structure, all of the solid solutions used in this study represented considerably less ideal cases for x-ray studies than the gold-copper one of Guinier. For this and other reasons, a rigorous study of the subject of order vs disorder was not attempted here. However, the problem was considered and is discussed in this report.

RESULTS AND DISCUSSION

Order vs Disorder

Generally speaking, the accepted view of solid solutions is that there are two types, substitutional and interstitial. This study deals with substitutional solutions. From the standpoint of structure, one can consider that, in the formation of substitutional solid solutions, each crystallographically equivalent site in the unit cell is filled with a statistical atom. This indicates that sites are randomly filled with host and substituted atoms. The type of lattice involved can have an important bearing upon the order in a solid solution. With primitive lattices a substitutional solid solution will be disordered, but both disordered and ordered (superlattice) substitutional types can occur with non-primitive lattices.

For example, when an ionic compound AB crystallizes in the cubic system, two possible ionic configurations are the CsCl structure and the NaCl one. The CsCl (fig. 1) has a primitive lattice with the cation, A^+ , occupying the (000) site and the anion, B^- , the $(\frac{1}{2} \frac{1}{2} \frac{1}{2})$ site. If a small amount of the cation component C^+ is substituted for the A^+ cation, there will be random distribution of C^+ cations on (000) sites throughout the entire crystal. On the basis of the representative unit cell (fig. 2) however, each (000) site can be considered filled by a statistical AC^+ cation. If there is no limitation upon substitution, then a continuous disordered $A_x C_{1-x} B$ solid solution series is formed in which $x = 0$ to 1. The resulting simple cubic x-ray diffraction patterns of the solid solution series has unit cell parameters which lie between those of the pure compounds AB and CB.

On the other hand, the NaCl structure (fig. 3) is based on a non-primitive, face-centered cubic unit cell. In this case the cation A^+ occupies two sets of equivalent positions. One cation occupies the corner position (000), and three cations occupy the face-centered positions, $(1/2 \ 1/2 \ 0)$, $(0 \ 1/2 \ 1/2)$, and $(1/2 \ 0 \ 1/2)$. The anions occupy the $(1/2 \ 1/2 \ 1/2)$, $(1/2 \ 0 \ 0)$, $(0 \ 1/2 \ 0)$, and $(0 \ 0 \ 1/2)$ positions. In this situation, cationic substitution could occur either randomly over all the lattice sites, or selectively over one set of equivalent sites. If the substitution is completely random over all sites, a continuous disordered solid solution series results. The x-ray diffraction patterns in this case show systematic absences of reflections indicating face-centered cubic symmetry.

If substitution is selective to the face-centered positions, an ordered $A_x C_{4-x} B_4$ ($X = 1$ to 4) solid solution series occurs, terminating in the formation of the compound, AC_3B_4 , as is shown in figure 4. If selective substitution occurs on corner positions, as illustrated in figure 5, an ordered $A_{4-x} C_x B_4$ ($X = 1$ to 0) solid solution series results, forming the compound A_3CB_4 . Ordered substitution, or superlattice formation, usually changes the symmetry of the crystal; in this case, from face-centered cubic to simple cubic.

In theory, the presence of ordered substitution is easy to detect by means of x-ray diffraction. The x-ray patterns will exhibit simple cubic symmetry because of the superlattice reflections. In the example of the NaCl, there will be systematic reflection absences in the x-ray pattern of AB. In both cases the length of the edge of the unit cell is identical. The intensity of this set of lines varies directly with substitution, and reaches a maximum at complete substitution. Furthermore, the intensity of the set of superlattice lines will depend upon the difference in atomic numbers of cation A and cation B. If both cations have similar atomic numbers the superlattice lines will be faint. If they differ markedly the superlattice lines will be stronger.

The forms of ammonium perchlorate (AP) and potassium perchlorate (KP) which exist at normal room temperature are simple orthorhombic (Pnma) that have one set of equivalent sites available for substitution, and no superstructure is expected. The high temperature form of each compound, however, crystallized in the face-centered cubic NaCl structure. In this case there is a definite possibility for the occurrence of superlattices in the solid solutions. Since high temperature x-ray studies have not been conducted it was not possible to find evidence for this. In the more complex cases of the other types of solid solutions no speculations were made about whether ordered arrangements and superlattices might be present.

Ammonium Perchlorate (AP)-Potassium Perchlorate (KP)

The preparation of these AP-KP solid solutions as well as other experimental ones by isothermal cocrystallization has been described in reference 7. Our x-ray studies were limited to three different compositions of AP and KP, namely those with AP-KP weight ratios of 3:1, 1:1, and 1:3.

Comparisons of interplanar d spacings and relative intensities of beams of x-rays diffracted from them (of AP, KP, physical mixtures, and corresponding solid solutions) are shown in tables 1, 2, and 3. The data is represented graphically in figure 6. The two strongest lines of AP, at 4.58 and 3.25 Å appear in both physical mixtures and solid solutions of corresponding compositions, namely those with weight ratios of 3:1-AP:KP and 1:1-AP:KP. In the case of the KP-rich mixture 1:3-AP:KP, the only d spacing of AP present in the physical mixtures is the principal one at 4.60. It has a slightly altered value in the corresponding 1:3 mixed crystal, 4.53 vs 4.58 for pure AP, for instance. Precise measurement of relatively subtle changes in interplanar spacings caused by substituting the KP isomorph into the crystal lattice of AP is essentially beyond the scope of this investigation. It would be difficult to distinguish between the 3:1-AP:KP physical mixture vs the solid solution on the basis of their x-ray patterns alone. The 1:1 and 1:3 physical mixtures vs their corresponding solid solutions can be differentiated easily on the basis of their x-ray diffraction patterns. Thus, in the latter two cases we were able to demonstrate readily, by simple x-ray techniques, the dissimilarity of the solid solutions in question.

Ammonium Nitrate (AN)-Ammonium Perchlorate (AP) Solid Solutions

Synthesis of this series of solid solutions by isothermal cocrystallization is discussed in reference 7. Four different solid solution compositions having weight ratios of AN-AP of 8:1, 1:1, 1:3, and 1:5 were investigated by x-ray. The x-ray diffraction patterns of this series is shown in figure 7. It is evident that although the d spacing in the AN:AP solid solutions with an 8:1 ratio of ingredients are somewhat different from pure AN, there is a strong resemblance to the diffraction pattern of AN. The pattern of the 1:1 material is unlike either AN or AP. The solid solutions which are AP-rich, with AN:AP weight ratios of 1:3 and 1:5, have patterns similar, but not identical, to that of AP.

Figure 8 shows the x-ray diffraction patterns of physical mixtures of AN and AP corresponding to the solid solutions in figure 7. Both x-ray diffraction patterns are shown in figure 9 so their differences can be seen. None of the patterns of the solid solutions are identical to those of the corresponding physical mixtures. The patterns of two AP-rich solid solutions

of nearly the same composition are compared in figure 10 to a physical mixture of similar composition. Despite their similar chemical composition, the x-ray patterns of the two solid solutions are significantly different. Also they are not the same as those of the corresponding physical mixtures. Figure 11 illustrates the manner in which the d spacings with the greatest diffraction intensities of pure AN and AP vary with introduction of increasing amounts of the other component. The intensities of the five major diffraction lines of AN, 4.95, 3.96, 3.09, 2.72, and 2.26 either disappear or fall to low values in the 1:1 solid solution. The same is true of the seven strongest diffraction lines of AP. A unique line, 2.59, appears in the AN:AP 1:5 solid solution.

In figure 12, the changes in the relative intensities of the three strongest diffraction lines of AN as AP is introduced isomorphously into its crystal lattice are compared to those of physical mixtures of similar compositions. Obviously, the three strongest lines in the solid solutions are different from those in the corresponding physical mixtures. Figure 13 shows the behavior of the three strongest diffraction lines of AP in the series of solid solutions vs physical mixtures. These last two figures support the evidence for the difference of the structure of the AN-AP solid solutions from either pure AN or pure AP.

Ammonium Nitrate (AN)-Potassium Nitrate (PN)

Reference 7 describes the preparation of solid solutions and the addition compound, $3\text{KN}\cdot\text{AN}^*$, formed by isothermal cocrystallization from the ternary system AN-KN- H_2O at 25.0°C. Two series of solid solutions occur here: AN with the compound $3\text{KN}\cdot\text{AN}$, and KN with $3\text{KN}\cdot\text{AN}$. We were particularly interested in isolating and obtaining the x-ray pattern of the addition compound, $3\text{KN}\cdot\text{AN}$. This is shown in figure 14, along with the patterns of the respective pure components and a physical mixture with the same relative amounts of components as in the addition compound. As might be expected, the pattern of this addition compound $3\text{KN}\cdot\text{AN}$, more nearly resembles that of the principal component, KN than that of AN. The pattern differs from those of the corresponding physical mixture and the pure components.

Figure 15 shows not only the same patterns as in figure 14, but also those of three different solid solutions which are also unlike their corresponding physical mixtures. The pattern of the solid solution containing 88.1% KN resembles and shows the influence of the $3\text{KN}\cdot\text{AN}$ addition compound upon its structure. Significantly, the x-ray pattern of a solid solution containing only a small amount of AN (5.5% by weight) differs considerably from

* $3\text{KN}\cdot\text{AN}$ is the addition compound composed of three molecules of KN and one of AN.

the x-ray pattern of pure KN. At the other end of the composition range of the solid solutions it can be seen that introduction of 31.4% by weight of KN into AN also causes substantial alteration of the diffraction pattern when compared to that of pure AN.

Ammonium Nitrate (AN)-Potassium Perchlorate (KP)

KP was dissolved in molten AN to make a solid solution with an AN-KP weight ratio of 3:1 (ref. 7). The diffraction pattern of this material, as well as that of a physical mixture with the same relative weights of components, is given in table 4 and figure 16. Figure 16 shows the strongest diffracting lines of AN and KP are absent in the solid solution. It is quite evident upon further comparison that the solid solutions patterns are quite unlike the corresponding physical mixture. The x-ray patterns of the solid solutions are different from their respective physical mixtures and, by inference, so is the arrangement of the respective atoms and ions in their structural lattice networks.

CONCLUSIONS

Though rigorous structural determinations are desirable wherever possible for characterizing new crystalline material, the use of simpler x-ray technique was effective in these studies. The x-ray powder data was helpful in characterizing the experimental oxidizer solid solutions and one addition compound as being unique materials. The major result of this investigation clearly shows that these solid solutions are entirely different compositions of matter than even the most finely ground physical mixtures containing the same relative amounts of components. Thus, theoretical expectations are confirmed.

Table 1

X-ray diffraction data of AP, KP, and their physical mixtures

AP		3:1 - AP:KP		1:1 - AP:KP		1:3 - AP:KP		KP	
d	I/I_0 *	d	I/I_0	d	I/I_0	d	I/I_0	d	I/I_0
		5.82	30						
4.58	100	4.60	100	4.58	40	5.68	26		
						4.53	38	4.47	30
3.92	43	3.93	52						
3.72	33	3.74	36			3.67	68	3.63	29
		3.63	85	3.62	66				
		3.50	30	3.48	100	3.53	100	3.49	100
3.25	51	3.26	64	3.25	28	3.40	29	3.36	31
		3.15	34	3.14	45	3.18	91	3.15	78
						2.92	48		
2.97	42	2.99	54					2.89	67
		2.91	34					2.83	28
2.60	29	2.60	31	2.88	28				
						2.18	20	2.17	24
								2.16	22
						2.14	21	2.12	25

*I is the height of the diffractometer peak for the d spacing in question.
 I_r is the highest peak of all observed for the sample.

Table 2

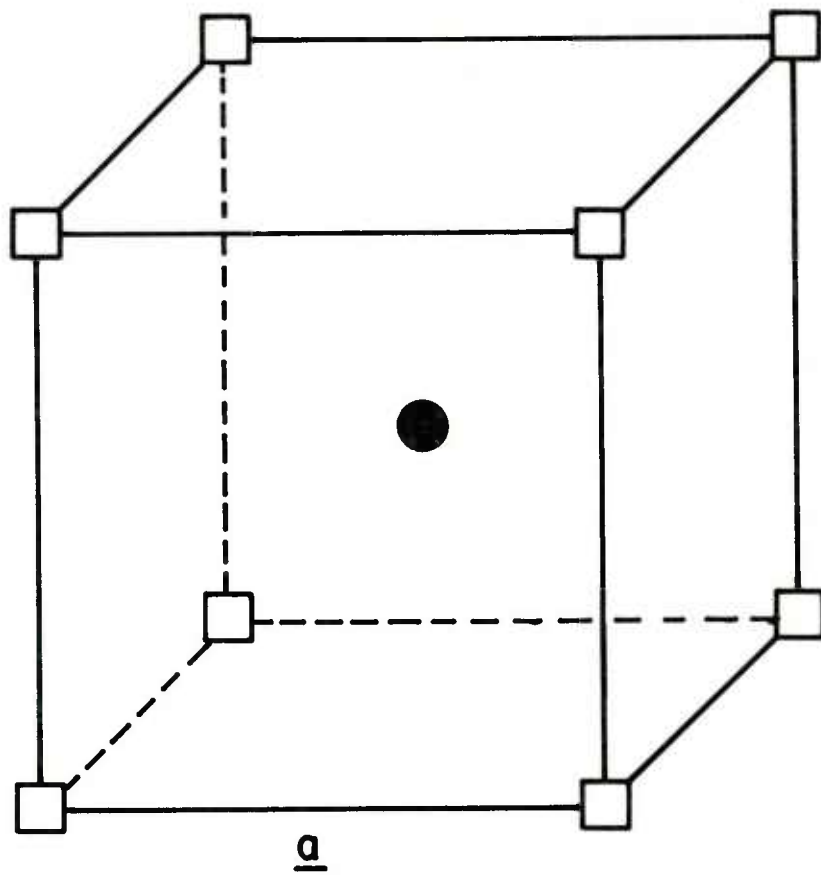
X-ray diffraction data of AP, KP, and their solid solutions

AP d	I/I_0	3:1 - AP:KP d	I/I_0	1:1 - AP:KP d	I/I_0	1:3 - AP:KP d	I/I_0	KP d	I/I_0
		5.82	36	5.79	26				
4.58	100	4.61	99			5.68	26		
		4.57	100			4.53	38	4.47	30
3.92	43	3.93	60	3.88	38				
3.72	33	3.74	38	3.70	44	3.67	68	3.63	29
		3.62	100	3.59	93				
3.25	51	3.25	70			3.53	100	3.49	100
				3.23	80	3.40	29	3.36	31
						3.18	91	3.15	78
2.97	42	2.98	56	2.95	66	2.92	48		
				2.89	36	2.86	30	2.89	67
								2.83	28
2.60	29	2.60	30	2.58	33				
				2.17	24	2.18	20	2.17	24
								2.16	22
						2.14	21	2.12	25

Table 4

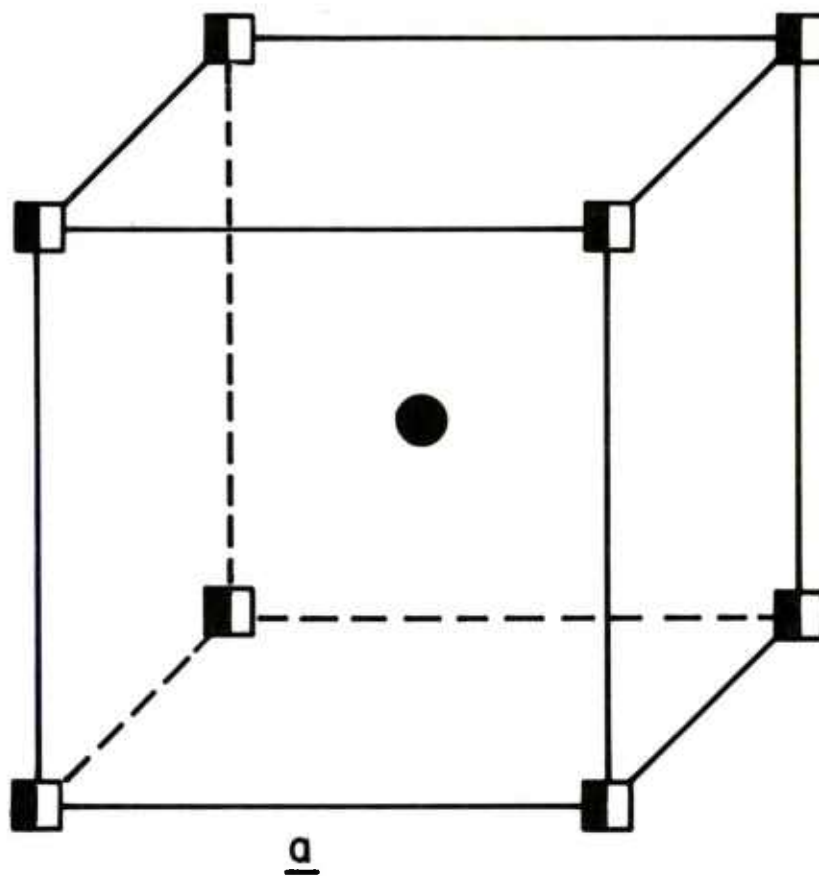
X-ray data of 3 AN: KP solid solution and
3 AN, KP physical mixture

<u>Solid solution</u>		<u>Physical mixture</u>	
d	Rel int	d	Rel int
4.53	40	4.92	86
3.90	78	3.97	44
3.56	36		
3.39	51	3.49	30
3.24	65		
3.21	100	3.15	21
		3.09	100
2.94	27	2.88	72
2.83	30	2.74	76
2.62	69		
2.55	61	2.40	25
2.32	28	2.26	28
2.26	28		



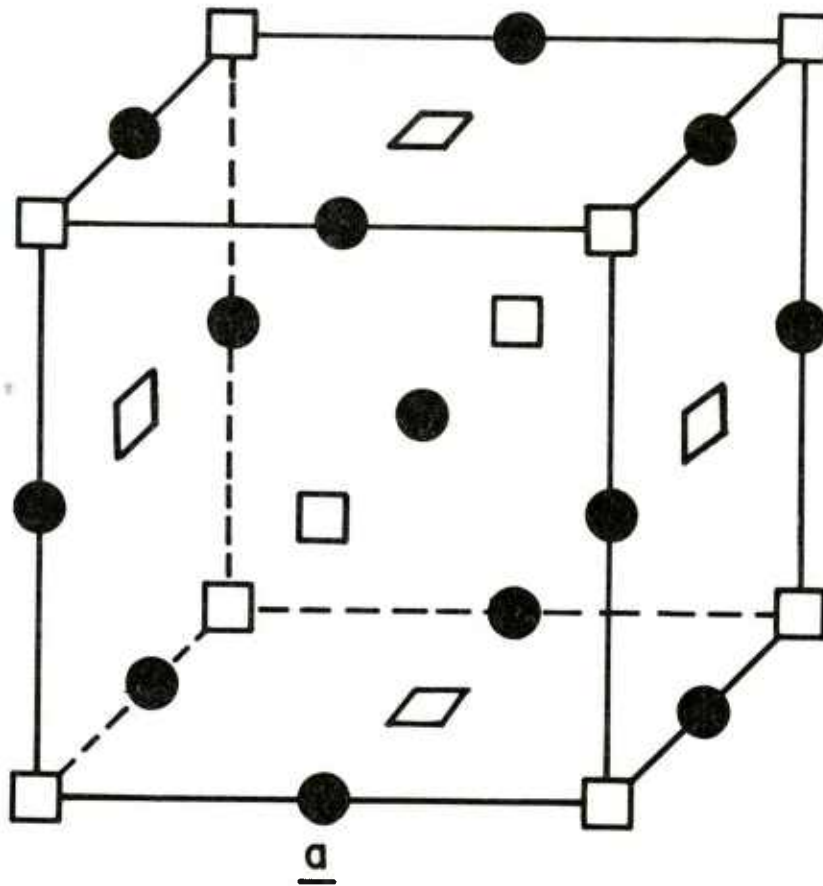
- CATION A^+ ON (000) SITE
 ANION B^- ON $(\frac{1}{2} \frac{1}{2} \frac{1}{2})$ SITE

Figure 1. Representative unit cell of Compound AB (simple cubic CsCl structure type)



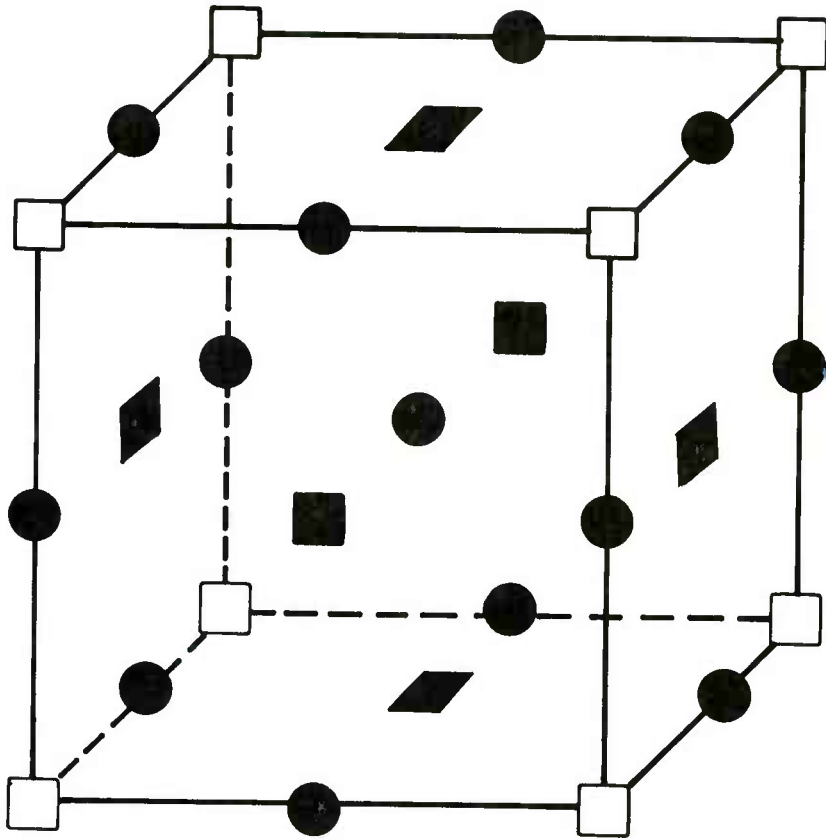
- STATISTICAL $[AC]^+$ CATION ON (000) SITE
 ANION B^- ON $(\frac{1}{2} \frac{1}{2} \frac{1}{2})$ SITE

Figure 2. Representative unit cell of disordered $A_{1-x}C_xB$ solid solution (simple cubic CsCl structure type)



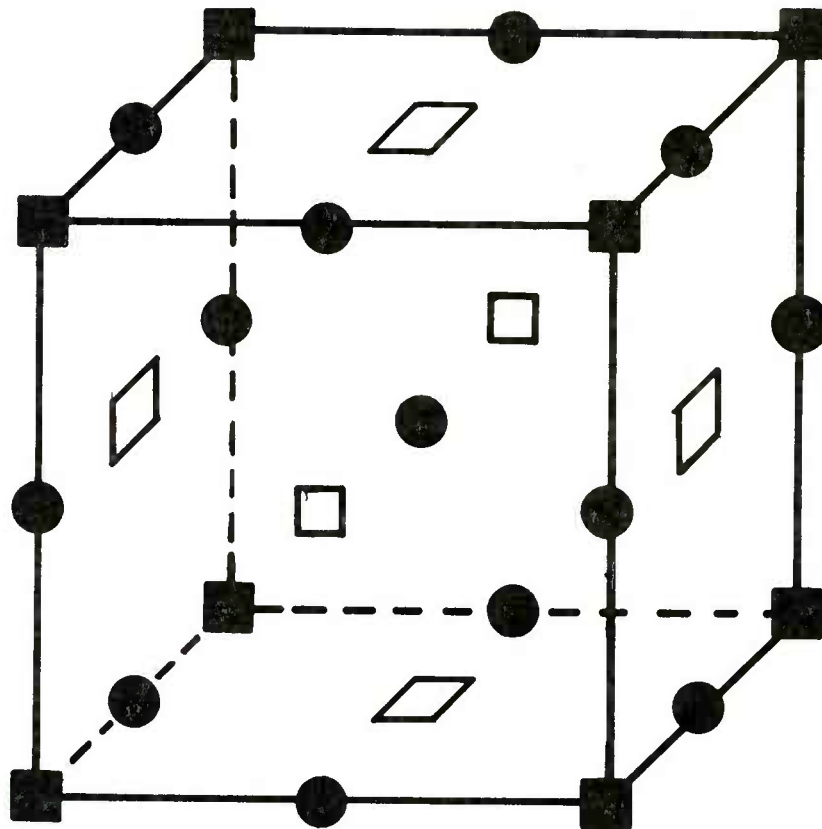
- CATIONS A^+ ON (000) SITE AND $(\frac{1}{2}\frac{1}{2}0)$, $(0\frac{1}{2}\frac{1}{2})$, $(\frac{1}{2}0\frac{1}{2})$ SITES
- ANIONS B^- ON $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$ SITE AND $(\frac{1}{2}00)$, $(00\frac{1}{2})$, $(0\frac{1}{2}0)$ SITES

Figure 3. Representative unit cell of Compound AB
(face-centered cubic NaCl structure type)



- CATION A^+ ON (000) SITE
- CATION C^+ ON $(\frac{1}{2}\frac{1}{2}0)$, $(0\frac{1}{2}\frac{1}{2})$, $(\frac{1}{2}0\frac{1}{2})$ SITES
- ANION B^- ON $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$ SITE AND $(\frac{1}{2}00)$, $(00\frac{1}{2})$, $(0\frac{1}{2}0)$ SITES

Figure 4. Representative unit cell of ordered solid solution AC_3B_4 (simple cubic superstructure)



- CATION A^+ ON $(\frac{1}{2} \frac{1}{2} 0)$, $(0 \frac{1}{2} \frac{1}{2})$, $(\frac{1}{2} 0 \frac{1}{2})$ SITES
- CATION C^+ ON (000) SITE
- ANION B^- ON $(\frac{1}{2} \frac{1}{2} \frac{1}{2})$ SITE AND $(\frac{1}{2} 00)$, $(00 \frac{1}{2})$, $(0 \frac{1}{2} 0)$ SITES

Figure 5. Representative unit cell of ordered solid solution A_3CB_4 (simple cubic superstructure)

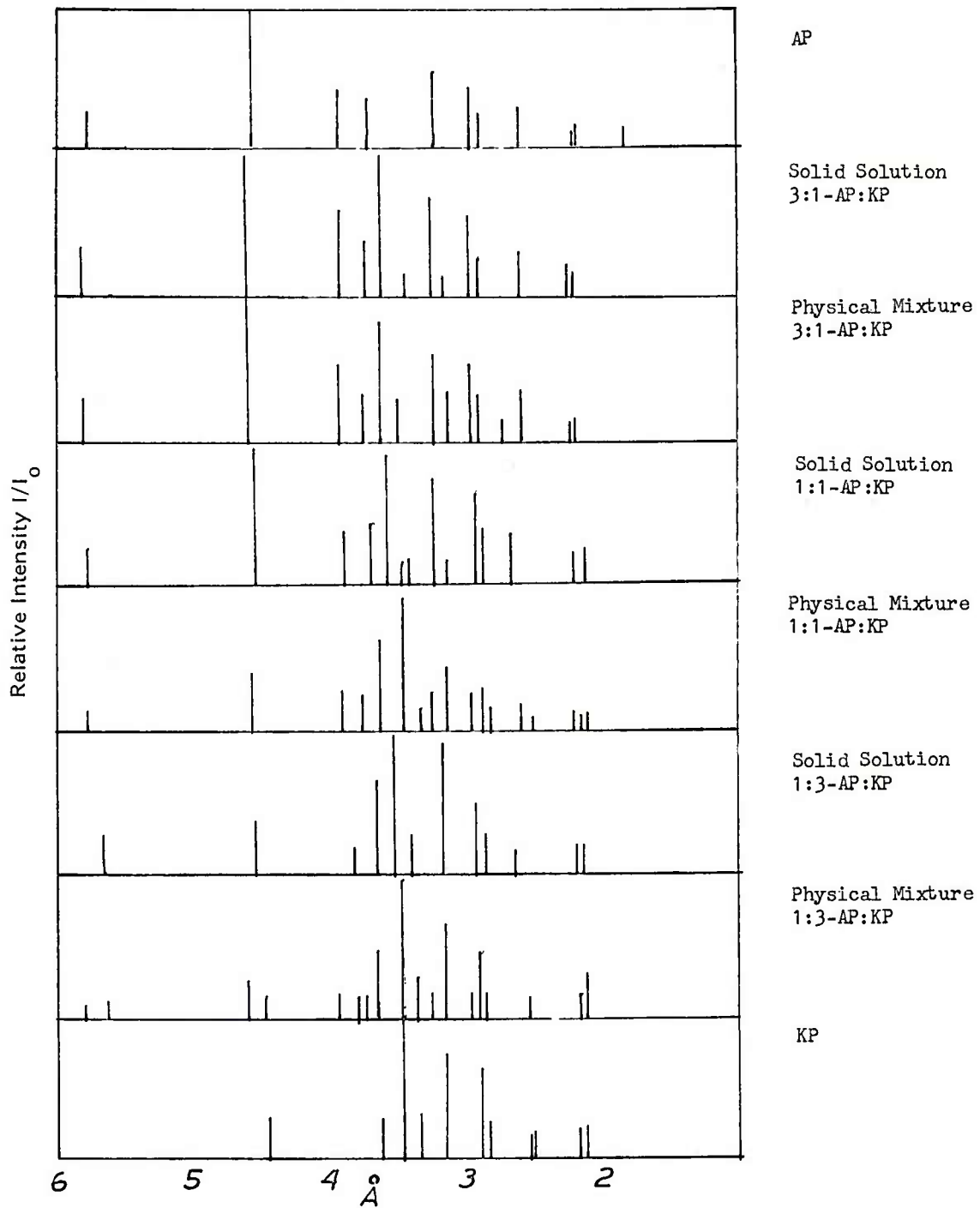


Figure 6. Comparison of x-ray powder patterns of AP, KP, and their physical mixtures and solid solutions

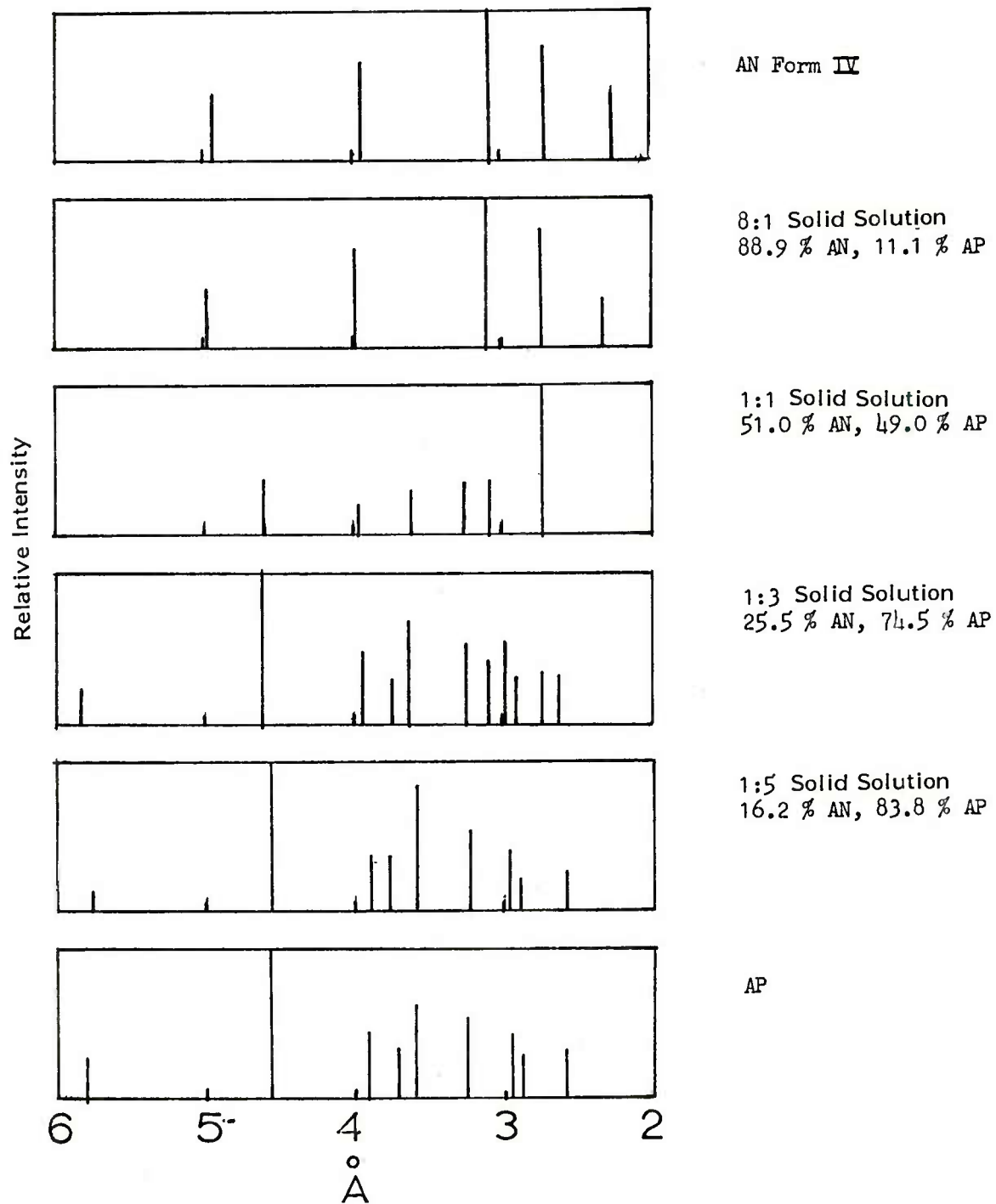


Figure 7. X-ray diffraction data for AN, AP, and a series of their solid solutions

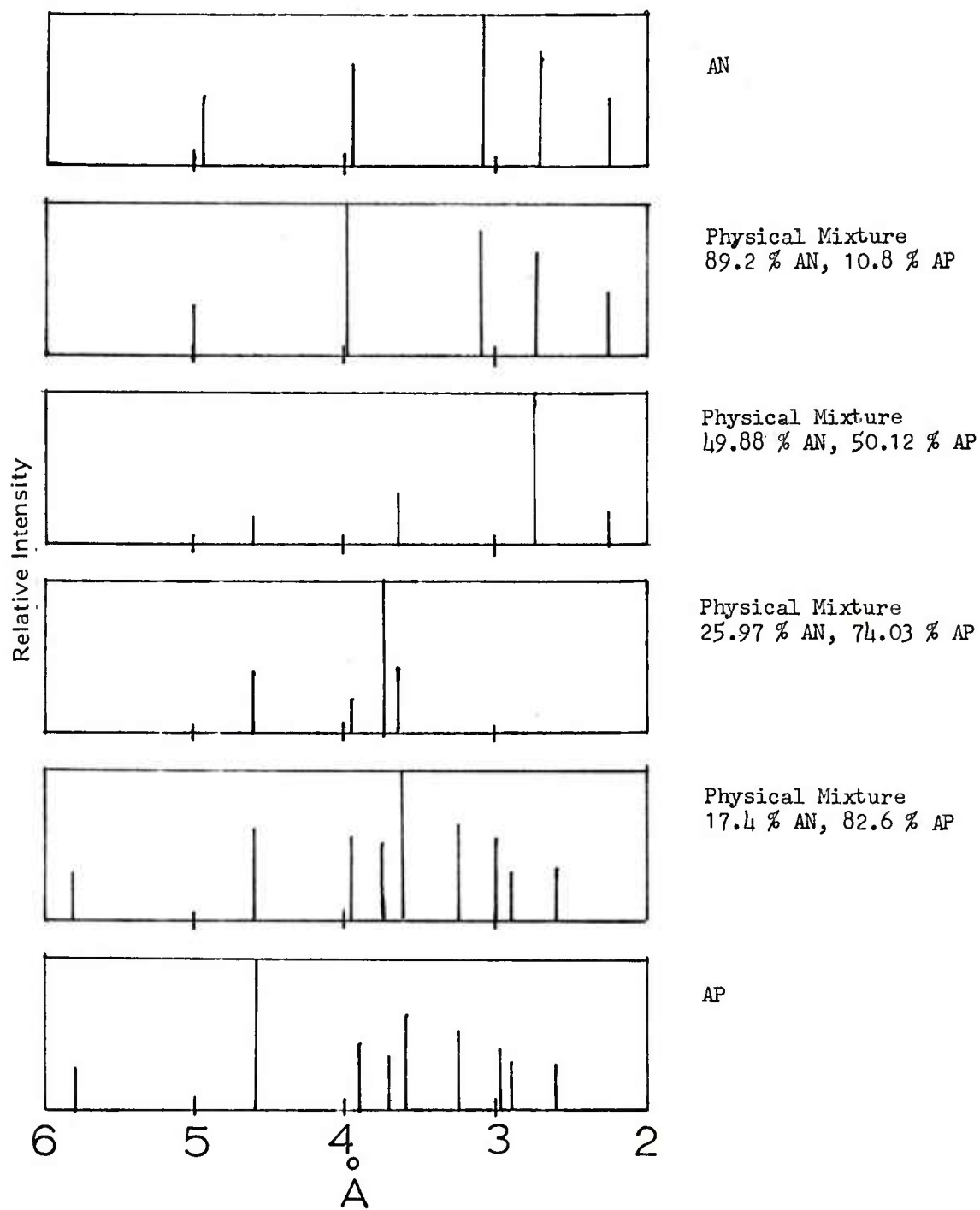


Figure 8. X-ray diffraction data for AP, AN, and a series of their physical mixtures

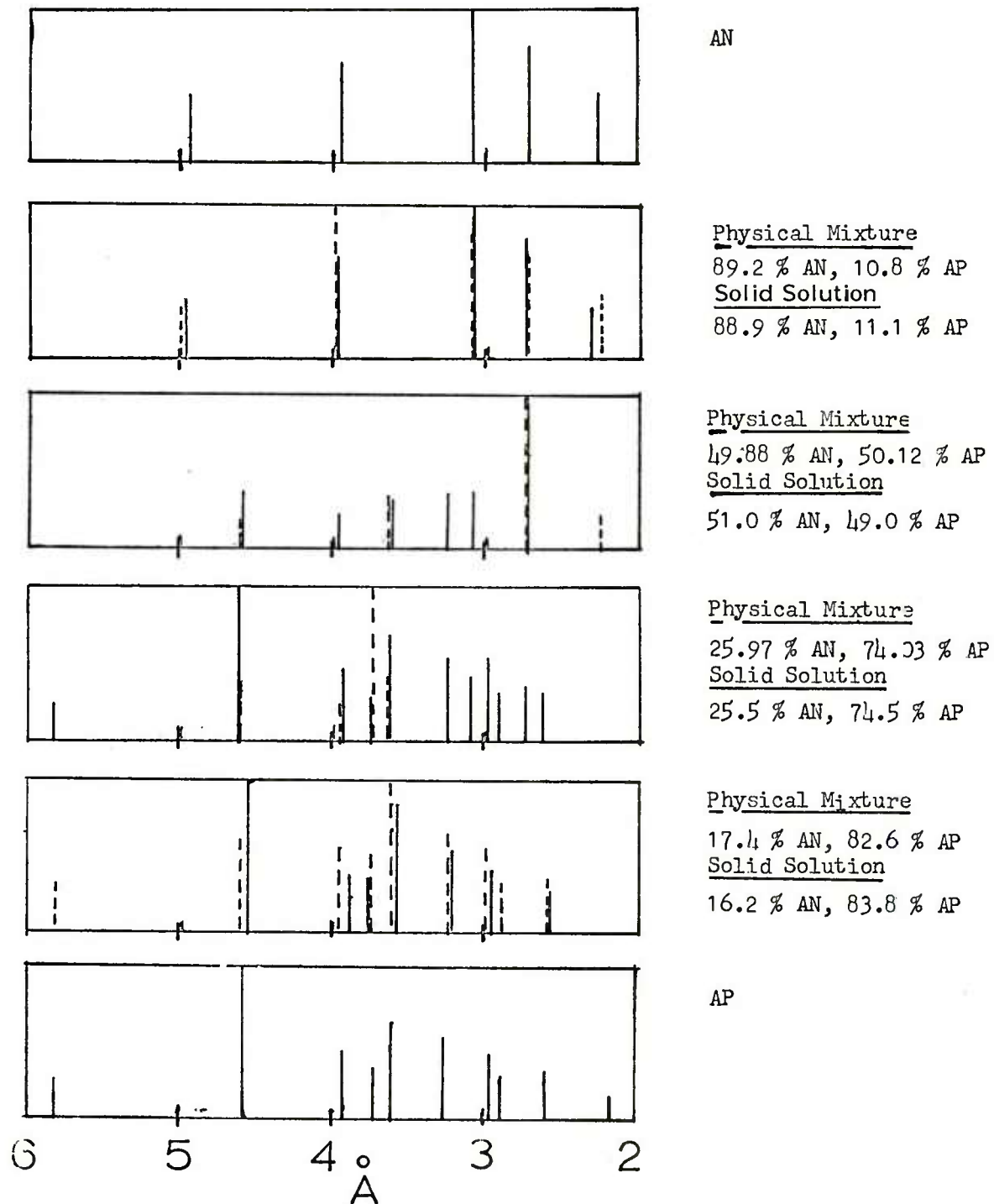


Figure 9. Comparison of x-ray patterns of corresponding physical mixtures and solid solutions of AN and AP

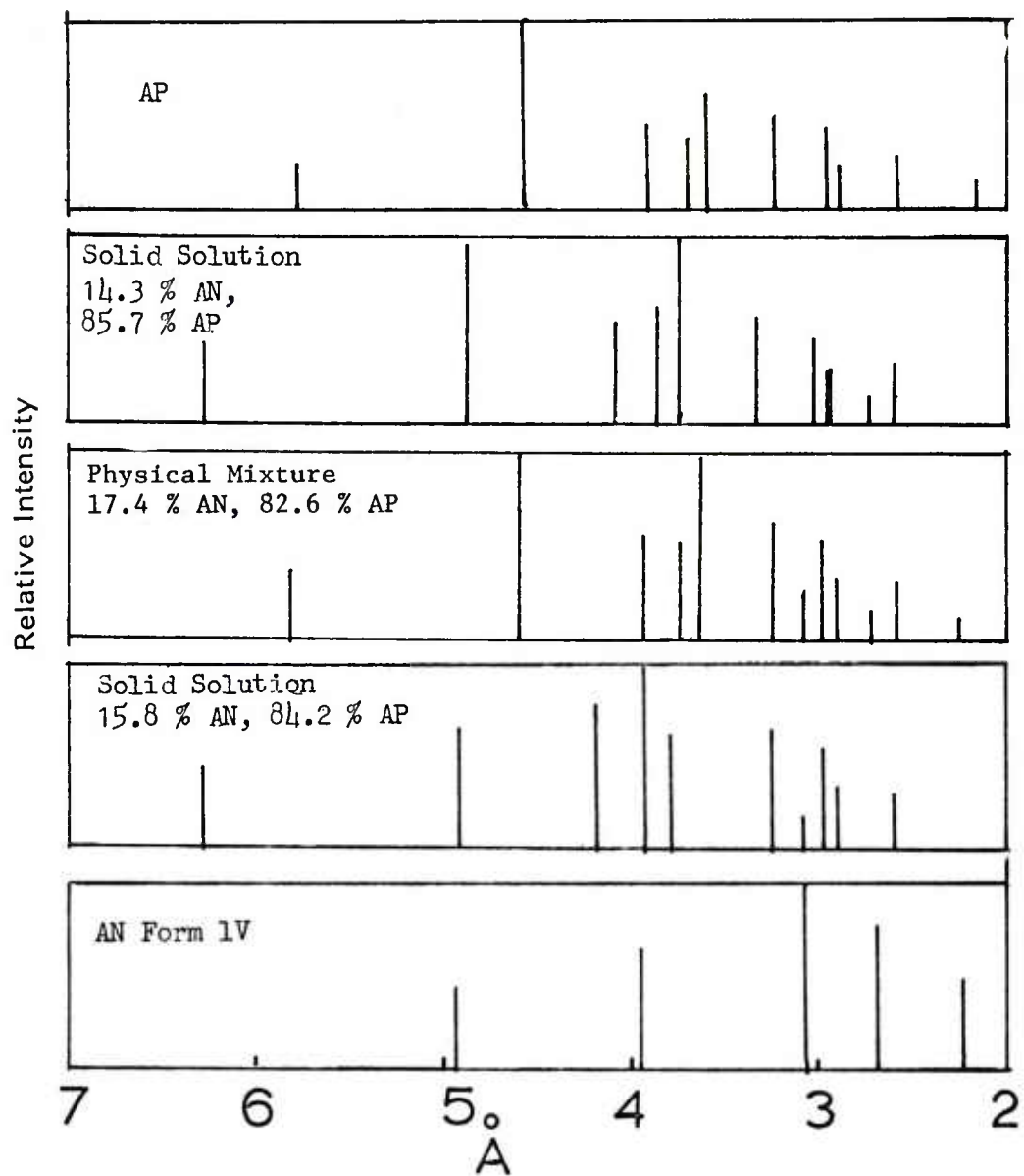
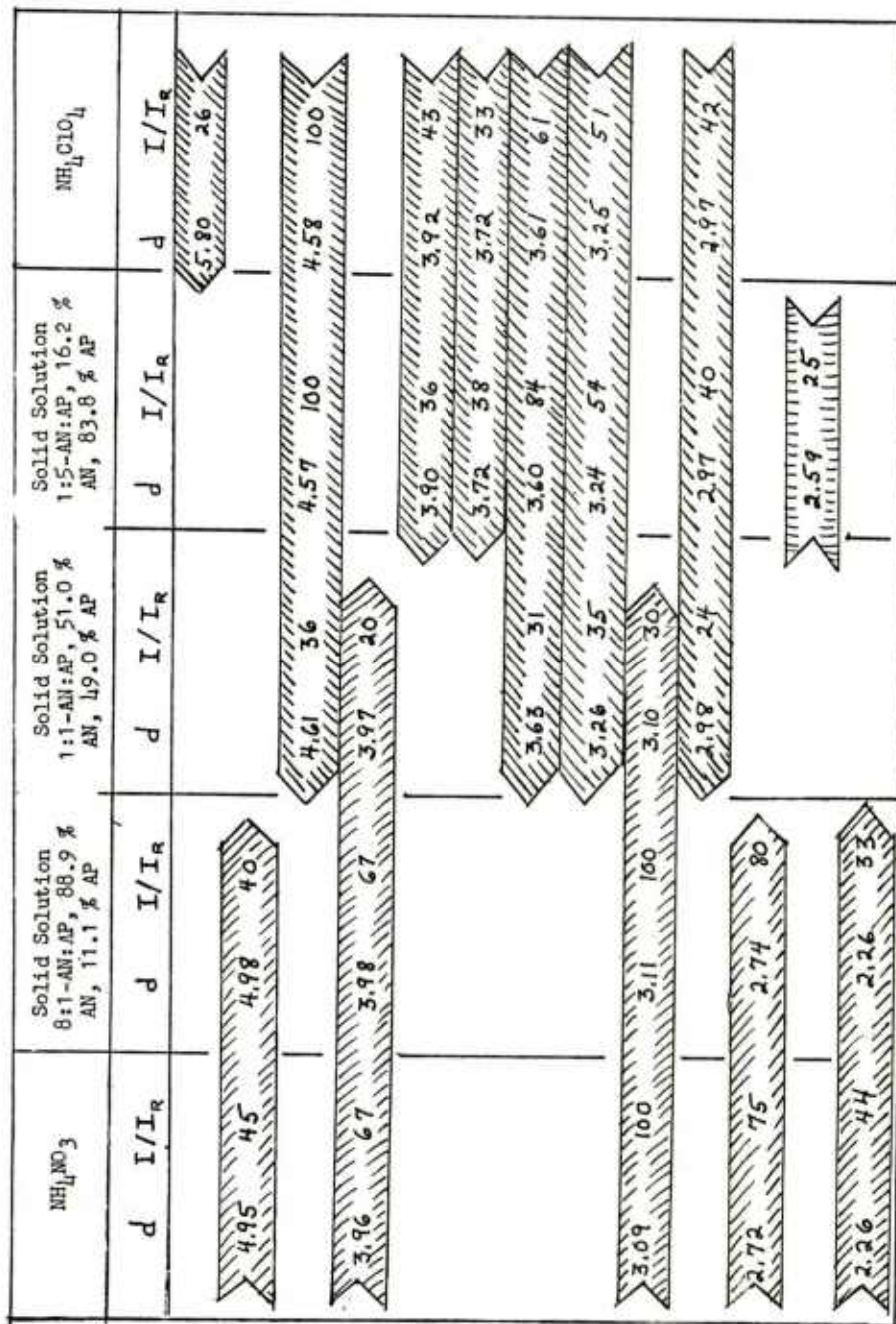


Figure 10. X-ray diffraction patterns of AN, AP, and AP-rich physical mixtures and solid solutions



Note: I/I_R is the ratio of the intensity of a given X-ray reflection for a d spacing related to the strongest reflection obtained for any d spacing in the sample.

Figure 11. Change in d spacings and intensities of corresponding x-ray reflections in AN, AP, and their solid solutions

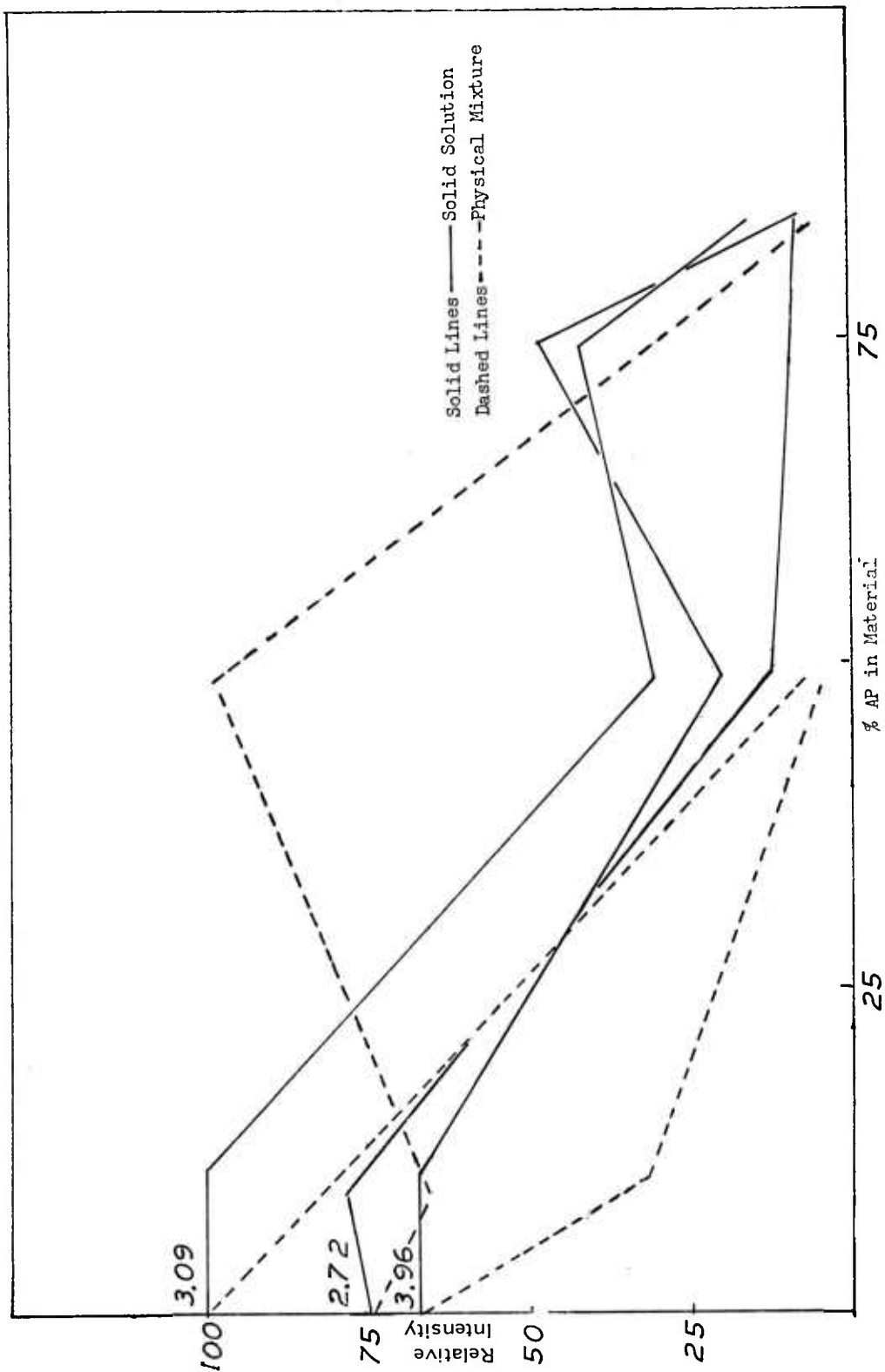


Figure 12. Comparison of relative intensities of the three strongest x-ray diffraction lines of AN with those for the same d spacing and a series of AN-AP solid solutions and corresponding physical mixtures

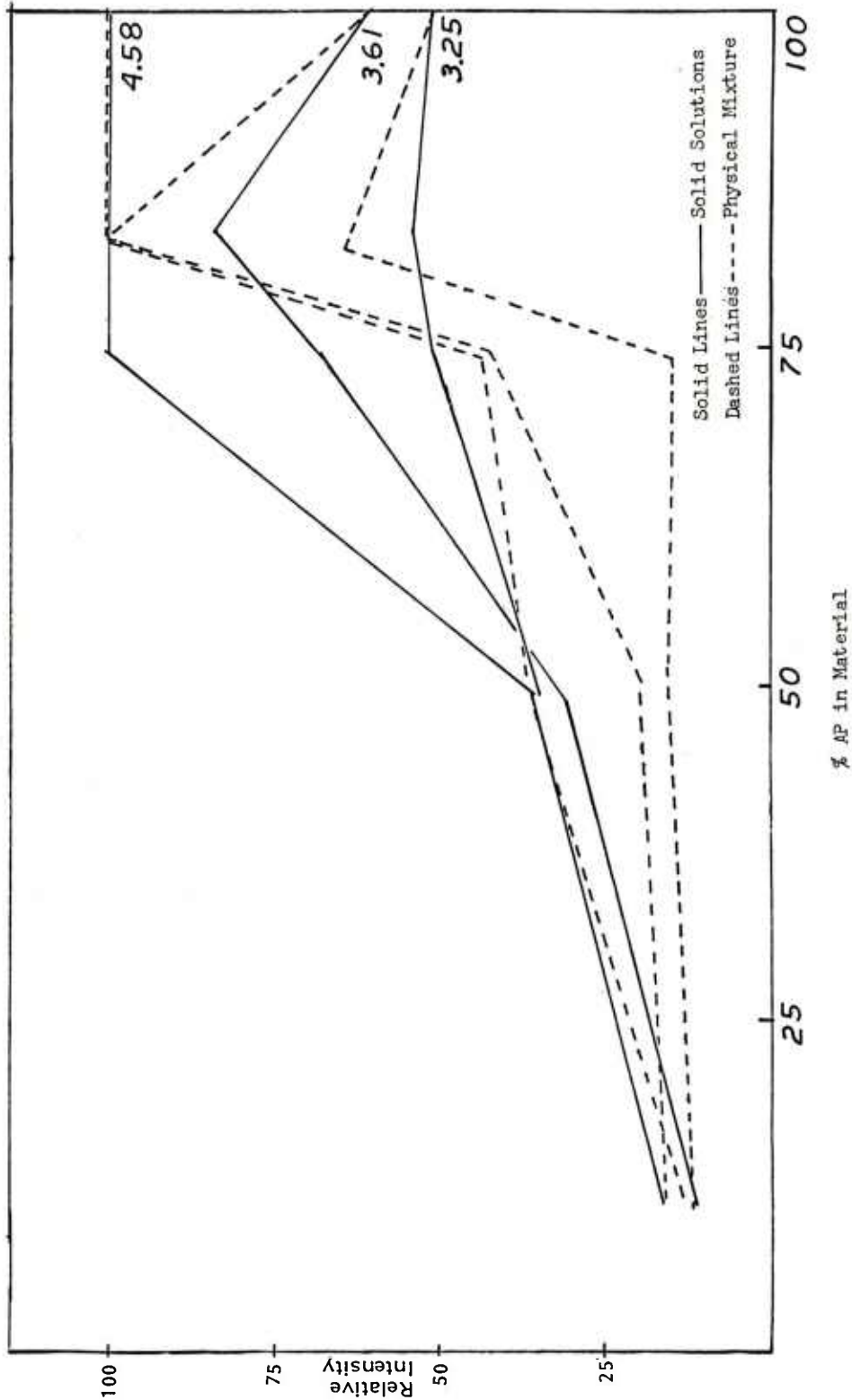


Figure 13. Comparison of relative intensities of the three strongest x-ray diffraction lines of AP with those for the same d spacing in a series of AN-AP solid solutions and corresponding physical mixtures

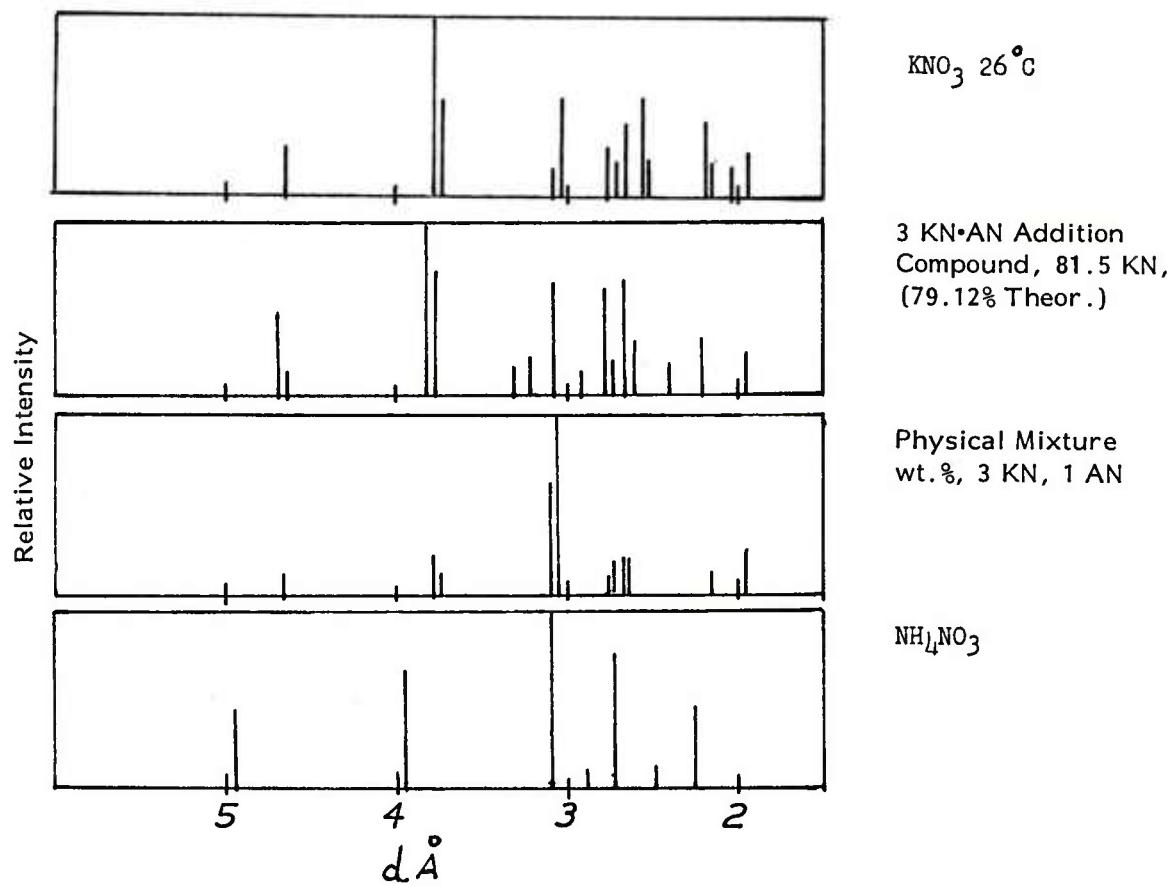


Figure 14. X-ray pattern of 3KN•AN addition compound vs components and corresponding physical mixture

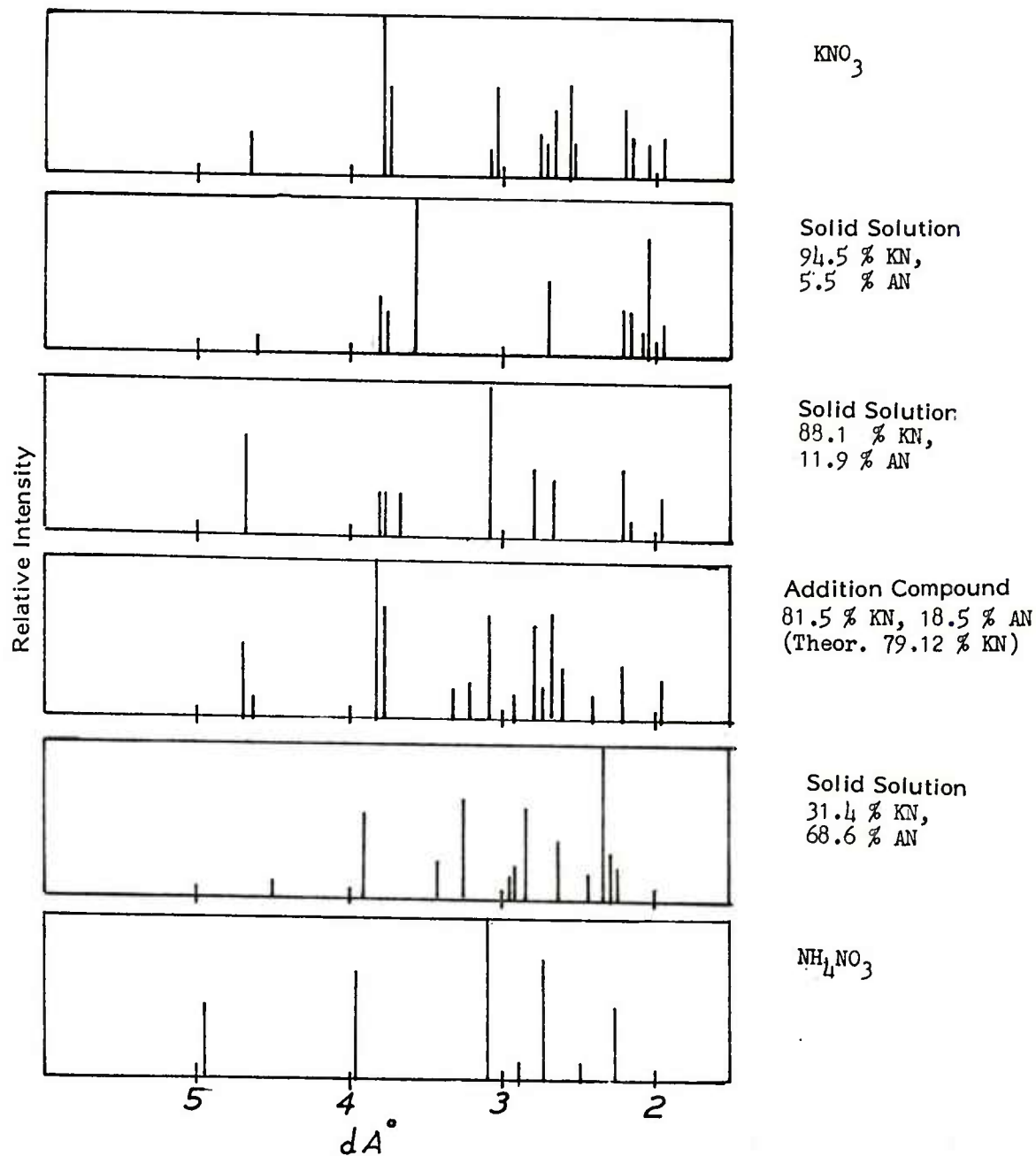


Figure 15. X-ray diffraction patterns of AN, KN, and addition compound $3KN \cdot AN$ and their solid solutions

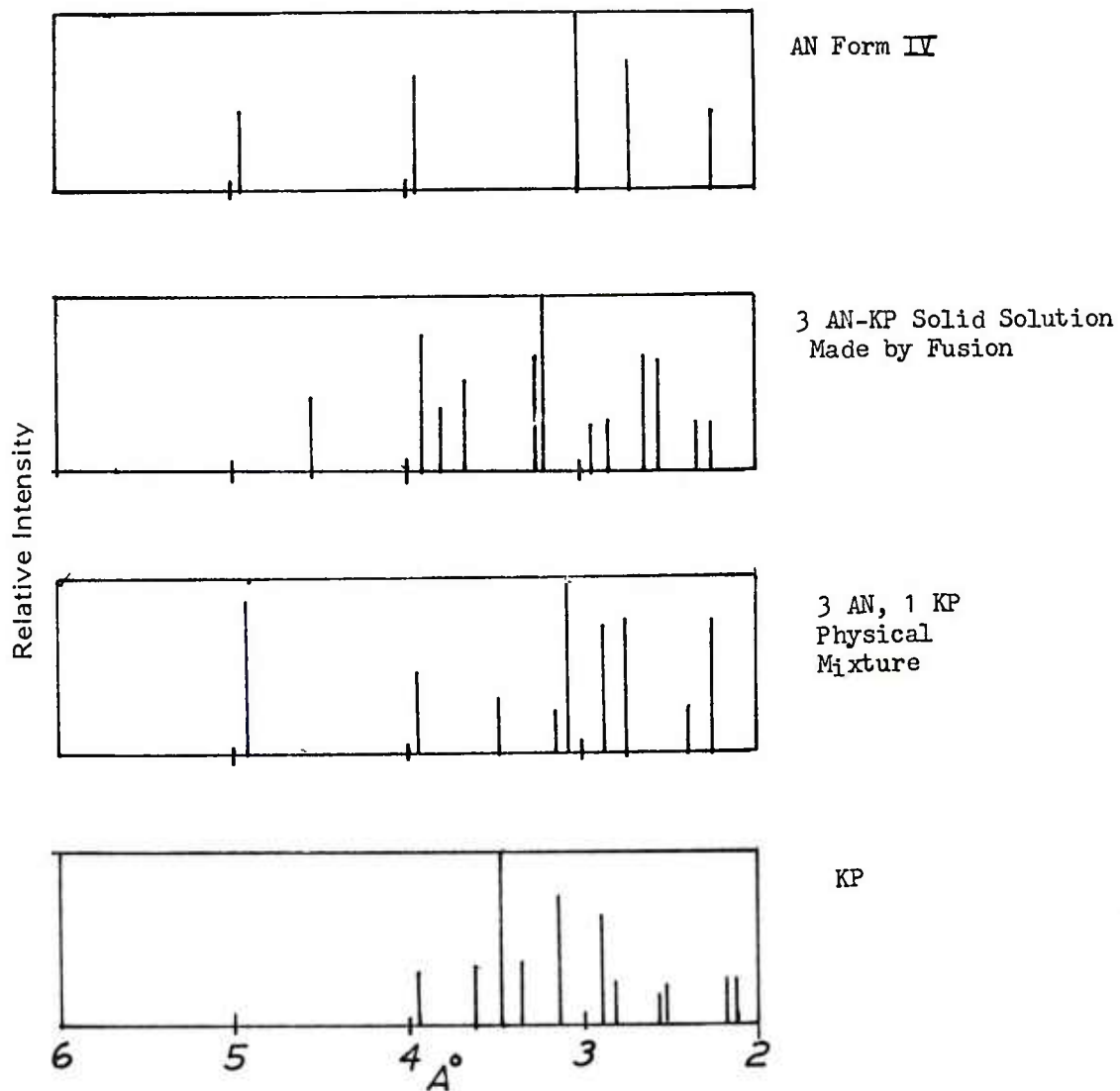


Figure 16. X-ray diffraction patterns of AN, KP, and solid solutions and their physical mixtures

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