PRNC=94 PUERTO RICO NUCLEAR CENTER NEUTRON DIFFRACTION PROGRAM PROGRESS SUMMARY REPORT NO. 5 M.L. KAY, S.F. KAPLAN, R. KLEINBERG, H.L. RITTER, J. ALMODOVAR, D.T. CROMER APRIL, 1967 OPERATED BY UNIVERSITY OF PUERTO RICO UNDER CONTRACT NO. AT (40-11-1833) FOR U.S. ATOMIC ENERGY COMMISSION ---Page Break--- Neutron Diffraction Program Progress Summary Report No. 5 M.L. Kay, S.F. Kaplan, R. Kleinberg, H.L. Ritter, J. Almodovar, D.T. Cromer April, 1967 ---Page Break--- Table of Contents Introduction === 1 Manganese Formate Dihydrate ~ Copper Formate Dihydrate 4 Liquid structures tables ~ References Figures Publication List ---Page Break--- The neutron diffraction group at the Puerto Rico Nuclear Center has worked on essentially two types of problems. The first is concerned with the chemical binding of atoms in crystals and molecules, and the second with the nature of ferromagnetism. Both problems are related to the spatial arrangement of atoms in molecules. If either x-rays or neutrons are scattered from crystals, patterns can sometimes be analyzed which show the arrangement of atoms in the crystal. The amplitude of x-rays diffracted from atoms is proportional to the atomic number of scattering atoms. Thus, if there are light and heavy atoms in the same compound, the contribution of the light atom is very weak and its position can be determined only with great difficulty. If neutrons are used, however, they are scattered by the mass of the atoms, and at a minimum, diffraction of neutrons by light elements compares favorably with that from heavier elements. There is also a neutron-electron spin interaction in compounds which possess atoms with unpaired electrons. Since the magnetic properties of substances are related to the way the electron spins are arranged within the crystal, neutron diffraction provides an accurate method for determining such spin arrangements (magnetic structures). ---Page Break--- Manganese Formate Dihydrate 1. Introduction In two papers by Okada (1965) and Okada, Kay, Cromer, and Almodovar (1965)

'The crystal structure and some of the electrical properties of copper formate tetrahydrate were described. The more common and more stable form of the plus two formates of ionic size similar to copper or manganese is a monoclinic dihydrate phase. Of known similar structure are the acenesium and manganese (Osaki, Takai and Watanabe) (1954), hereafter referred to as (oli), and copper (4 Puxowska-Strayzevsia (1965), hereafter referred to as 38), and nickel (Krogunnn and Mattes (1963). The present study defines the hydrogen positions in at least one of these compounds (manganese) which should be typical of the group. As might be expected from the greater stability of the dihydrates, the manganous formate shows a stable hydrogen bonding scheme with no disorder. The crystal structure of Mn formate dihydrate has been determined by iW (1964) and by Mascarenhas (1954). The cell parameters (a=6.06, b=7.29, c=9.60, β=97.7%) determined by the former authors were used in the present study. The space group is F2s/e. The structure consists of Mn atoms at cell corners related by symmetry to Mn atoms at face centers. A second set of M2 positions is found in band c face centers. In other words set one in the plane at x=0 and set two at x=1/2. The manganese atoms near x=0 are linked together by formates in the manner of copper formate. To complete the octahedron around Mn atoms, the second formate group links and is approximately perpendicular to (100). The structure is an atom in the stereogram in Fig. 1. ---Page Break--- The octahedron around Mn is completed by water oxygens. The environments around the two types of manganese are thus radically different from each other; one is octahedrally coordinated to six formate oxygens and the other to two formate oxygens and four water oxygens. This structure probably accounts for the two magnetic transitions (Abe, Vorigait, Matsmra, Torii and Yeemgnta (1961)) found in Mn formate at 5.7K and 1.7%.

According to Abe et al. (1964), cations on the first site only probably order first to Anions ionically at 5.7 and then totally at 1.7% TE. Bagerizentel, three zones of neutron diffraction data were taken on

Im(HC02)2*2ie0 crystals grown from water solution to find the hydrogen positions. A set of approximate parameters were found using (Ftype ~ Fyn,c,o) Fourier projections and refined by means of least squares (Roof, Craner, and Larson (1965)) to an R of 0.12. The final parameters are given in Table T and compared with the results of Gi. Except for the y coordinate of C2, all coordinates agree within 2σ. This discrepancy seems to affect only the O5-C2-OH angle as seen in Table Tia.

MIT Conclusions

The hydrogen bonding scheme indicated by OHW is correct. It might be noted that there are water-oxygen to oxygen approaches that could be conceivably hydrogen bonded of the order of 3.18. The hydrogen positions do not, however, indicate such linkages and difference maps show no significant extra hydrogen density not ascribable to anisotropic vibrations. It will be noted that the formate hydrogens have enormous temperature factors which are equivalent to root mean square amplitudes of vibration ---Page Break--- about 0.38. The stereo projection presented in Fig. 1 shows the formate hydrogens' surroundings are such as to permit a good deal of flapping; i.e., the distances to other atoms are felt greater than 2.58. We may then conclude that the hydrates' unique hydrogen bonding scheme and three-dimensional formate bridging array may be correlated with the greater stability of these compounds as compared to copper formate tetrahydrate.

Copper Formate Dihydrate

Introduction

During the early phases of the manganese formate work, Bukawsia-Strayzevake published a refinement of copper formate dihydrate from three zones of x-ray data. The refinement consisted of Fourier projections with the Co abstracted. No attempt was noted in the paper to either do a complete difference map or to even compute a Fe map to estimate series.

termination errors. The low R of «1 was probably due to the effect of the heavy copper scattering. Noted was the greater distortion of the octahedron of oxygens about copper in comparison to nickel (Kiroman and Matt (1965)) by elongation of two of the six Cu-O bonds (see table Tia). BS correlated the elongation of Cu-O with the elongation of O-O in the formate group. The extent of the elongation noted amounted to -2 and .058 differences in the carbon-oxygen approaches to the two formate groups (table Tia). Such differences imply a fair degree of difference of bond character between the two O-O bonds in a formate ion. The effect is somewhat larger than might be expected from the differences between the oxygen linkages to different types of copper orbitals. ---Page Break--- oS TL, results of Cu Formate Refinement For these reasons we refined the x-ray data by means of least squares using the program of Roof, Cromer and Larson (195) using a weighting scheme of We/(Fo + .cero?)}/2 with correction for anomalous dispersion for the copper, the results are given in Table IIfe together with the results of BS. The coordinates are translated to the origin used by GH and the atoms relabeled for easy comparison with the Hn formate work. The O-O distances in the copper formate hydrate now agree quite well with the O-O distances in other formates, including copper formate «Mi,0, within the standard deviations of the senate, BS reported a O2-O3 hydrogen bond of 2.99% of the O2 related to the O at 2.998 from Oye by the sixew axis transformation 18 chosen, O2-O3 distance of 2.76R found. This is the hydrogen bonded approach found in manganese formate. Anhydrous Copper Formate Introduction In three Dimensional Least squares refinements of the anhydrous copper formate x-ray data of Barclay, and ymnaré (1961) was min using the Roof, Cromer, and Larson (1965) program. Anisotropic temperature factors and an empirical extinction correction were included. The results were in very good agreement with the Barclay and Kennard Fourier results.

'There was, however, just enough change to bring all C-O distances within 2 standard deviations of the average, confining our results on Copper formate dihydrate. The new distances and angles are given in Table IIb and the positions and thermal parameters in IIIb. ---Page Break--- TZ, Conclusions The general molecular geometry of the various transition metal formates isomorphous with Mn has been determined by the various authors quoted. The study reported here defined the hydrogen bonding scheme. There are clearly some distortions, as expected in the octahedral surroundings of the metal ions. Fine details (i.e., of less than Ca. 0.05R) are not established in any of these compounds except possibly the i. It is clear that three-dimensional x-ray counter work and neutron data on a deuterated compound are needed. ums 1. Introduction In our 1966 Annual Report (FRICOh) and in Cromer, Kay, and Larson (1966) the structure of the alum (C&AL(SO,,),+12H,0) was described. Theg, and 7 alise structures as exemplified by My, at Wo,A1(90,,)pr22i,0 respectively, have now been completed. The x-ray work was done by Croaer and Larson at Los Alamos Scientific Laboratory. Neutron diffraction data was taken at PeRMCs to determine the hydrogen positions. The alums are a large class of double salts having the general formula AM BP9(ROy)*12iL0 where A can be Wi, Ci, Mis, Na, K, Hb, Cs; B can be AL, Ga, ce, Pe, V; R can be 8, Sa, or Be. These compounds are cubic with space group Pay (240 Table IV). The gross structures were originally determined by Lipson (1935) and refined in the present work. The type of alum formed depends on the size of the monovalent cation. If the cation is small, the 7 alum forms. The only known representative of this class is NeAL(Sq,)or2H0. The 4 alum forms if the cation is large and ---Page Break--- not the gi abun, which is by far the most common type, occurs if the cation is of intermediate size. In all the alums there are two crystallographically different water molecules, each associated exclusively.

with either the monovalent or trivalent cation, the trivalent cation is always surrounded by six water molecules in a nearly regular octahedron, but the orientation of the octahedron with respect to the cell axes is different in each of the three types. The monovalent cation in the 7 alums also has six waters in a nearly regular octahedron. The large cation in Falun can accommodate 12 oxygen neighbors. To attain this large coordination number, the water octahedron is compressed along the threefold axis and stretched out normal to this axis until it is nearly planar. The two ends of the resulting triconal antiprism are separated by only about 0.068. The sulfate groups at each end of this antiprism are then moved along the threefold axis toward the central cation until six sulfate oxygens are about the same distance from the cation as are the water molecules. A slightly distorted close-packed array of oxygens thus surrounds the cation. In the 7 structure, six water molecules approach the small sodium cation much more closely than in the other structure. This notion cannot take place unless the hydrogen bonding system changes. The most striking result is that the sulfate groups become oppositely oriented along the threefold axis. From Table IV, it may be noted that 640 on the threefold (XXX) axis (belonging to the sulfate group) is pointed towards the AT position at {a/2,2/2,1/2} in the 7 alums, but towards the AL at the cell origin in oh and Pato. IT Y-Kum: WakL(50,)9°22H,0 The atom positions found (Table IV) do not differ by more than about 0.058 from those reported by Lipson (1956). The hydrogen parameters are ---Page Break --- 4. tabulated in Table V. The interatomic distances and bond angles are given in Table VI. The standard deviations were calculated using the entire variance-covariance matrix and include the lattice parameter error. However, the values involving hydrogen atom positional parameters from the neutron data refinement assume no error in heavy atom positions. The water molecules about the

aluminum atom forms a perfect octahedron although this is not required by the crystal symmetry.

Whereas the principal orthogonal axes of this octahedron coincide almost exactly with the cell axes in equal, and within a few degrees in aluminum, the octahedron at the origin in 7 alum is rotated by 39.4° about the threefold axis of the b and diagonal of the unit cell. The Al-O distance to the surface in both Cs alum and Na alum. The octahedron of water about the sodium atom is somewhat distorted by being stretched out along the threefold axis of the cell. The angles in the sulfate group depart from those of a regular tetrahedron by a small but apparently significant amount. The differences, for equivalent angles, are in the same direction as those in Cs alum although the departure from tetrahedral symmetry of the sulfate group in Cs alum were not significant. The rigid body motion, discussed below, might well account for the apparent deviation from tetrahedral symmetry. The anisotropic thermal parameters for both neutron and x-rays work are given in Table VI and bs The anisotropic thermal parameters of the sulfate group do not seem to be consistent with the rigid body analysis given by Cruickshank (1956). We might expect a torsional oscillation of the group about the threefold axis. However, the major axis of the O(2) thermal ellipsoid, instead of being normal to the threefold axis is at an angle of 65.5°, we believe that this is a case in which the translational and the rotational motions are coupled. Thus the Cruickshank analysis does not apply. The O(2)-O(2) hydrogen bond ---Page Break--- is fairly short, 2.62Å, and the O(2)-I(1) distance is 1.64Å. The principal axis of the O(2) thermal ellipsoid makes an angle of 04.5° with the S-O bond and an angle of 79.9° with the O---I(4) bond. Thus a motion of 0.367Å (the maximum amplitude along this axis) changes the O---I(1) distance from O.0Å to 1.628Å, a small amount. However, if this 0.368 motion were to be directed along a line normal to both the S-O bond and the

threefold axis, the O--st(h) atatance would be reduced to 2.538. This sulfate group lies on the threefold axis so that a simple rotation about this axis will reduce three distances and we reason that the whole group therefore is forced to translate along the threefold axis whenever it rotates about this axis. Figure 2 shows a stereo view of a portion of the structure. A sulfate group is at the center of the figure and the direction of view is normal to the threefold axis and in a plane containing the S-O(1) and S-O(2) bonds. The ellipsoids have been derived from the neutron diffraction data and are scaled so that their axes are two times the thermal amplitude. The aluminum atom and its water neighbors are at the top center and the sodium atom with its neighbors is at the left. The notion of O(2), 20 as to avoid the hydrogen neighbor can be clearly seen. This figure was produced by the 80-020 microfilm plotter using a code recently developed by Larson (1966). The fact that the thermal motion of O(2) was found to be essentially the same from both the x-ray and neutron diffraction measurements is strong evidence that the apparent motion is not an artifact resulting from systematic error in the data. Corrections to the S-O bond lengths were computed according to the in-phase or "riding motion" assumption of Busing and Levy (1964). The S-O bond lengths appear to be about 0.015% shorter than those found in Cs alum (CKL) ---Page Break--- 10. Due to the uncertainty in the thermal motion correction, the difference is probably not significant. Further analysis is planned. The O-H distances, except for O(2)-H(4), show the usual feature that bonds determined from x-ray data are shorter than those determined by neutron diffraction. The O-H bonds in this structure are all nearly the same and do not show any particular correlation of long O-H bonds and short O-O hydrogen bonds. As in alum, water (1), which is associated with the monovalent cation, forms hydrogen bonds that link O(2) of one sulfate group with O(2).

of another. 'Again, as in alum, water (2) forms hydrogen bonds with O,(2) and water (1). In the present cage, there is a strong indication that shorter hydrogen bonds tend to be more nearly linear. TIT qh, -Abuns ND,AL(SO4)p*22020 Deuterated ammonium alum was selected as an alum to study by neutron diffraction for several reasons could be located accurately. In addition, the

nature of the disorder of the ammonium ion, which must exist if the space group is to regain Pa, could also vary average centric 've determined. 'The ammonium ion can attain the new symmetry by rotating freely or by randomly assuming either of two representations. It was also of interest to verify the sulfate group disorder found by Larson 'and Cromer (1966) by means of x-ray diffraction and to determine whether any water molecule disorder is coupled with the sulfate disorder. Refinement of the structure A full matrix least squares refinement including anteo-temperatures factors was carried out. This refinement led to R = 9.2%/qy AB in the case of the x-ray refinement, the thermal parameters for the sulfate oxygens were ---Page Break--- quite large and quite anisotropic. Other thermal parameters behaved normally. 'The ellipsoids are somewhat smaller than those obtained from the analogous x-ray refinement of ordered Mii, alum but the directions of the axes are in good agreement for the two cases. 'A difference Fourier clearly revealed small peaks from disordered sulfate 'oxygen atoms. A model with a fraction, k, of reversed sulfate oxygen atoms was then refined as in the x-ray refinement of K alum (Larson, and Cromer, (1966)). However, no attempt was made to displace /2 ND, groups along the threefold 'axis as was done for the potassium atom in K alum. This additional disorder would have introduced too many highly correlated parameters. Anisotropic 'thermal parameters for the reversed oxygen, Og(2)', did not remain real. A refined refinement was made with the reversed oxygen atoms isotropic and all other atoms.

anisotropic. 'The final R index was 8.4°/., The final parameters are given in Table VIII, The disorder parameter is 0.116 1'0,c22 in good agreement with the value indicated by the x-ray data for My alum. In Tables V, and following, the symbols O(2)' and O(2)' are used for the reversed sulfate oxygen atoms and D' is used for the half deuterium atoms in the disordered ND group. 'The various interatomic distances and bond angles and their standard deviations are given in Table IX. The anisotropic thermal parameters were transformed to obtain the thermal ellipsoid parameters, which are given in Table X. 'The O-D bond lengths have been corrected assuming that D rides on O (Busing and Levy, 1964). No such corrections were made to N-O and N-D ends because of uncertainties introduced by the disorder. Agreement of these distances with those from the x-ray study of the alums (Larson and Cromer, 1966) is reasonable except for three striking exceptions. 'The O(2)-O(2) and O(2) hydrogen bond distances are both about 0.05Å longer than in Wi, alum. 'The Al-O(2) distance found is 1.065 Å ± 0.008Å. If these differences are real, they must arise from an isotope effect of 0.005Å whereas the analogous distance found by x-rays in Mi, alum is a different effect. To settle the question, an x-ray study was made of deuterated ammonium alum and a refinement was made exactly as had been made for Hi, alum. There were no significant differences between these (Cromer and Cromer, 1966). The x-ray studies of Mi, and WD, alums, As another check to determine if we had systematic errors in either our x-ray work or neutron work, the neutron data for Ne alum (Cromer, Key, and Larson, 1966) were refined while letting all parameters vary. (In the original work heavy stone had been held in fixed positions for the neutron refinement). Unfortunately, only limited three-dimensional data were available and there were only 144 observations to determine the parameters. Nevertheless, the refinement converged satisfactorily and the parameters did.

not change very much. The resulting A1-0,(2) distance was 1.679 ± 0.014, in good agreement with the x-ray value. In this case, no systematic difference existed in the two techniques. We believe that the most likely reason for the difference between the present neutron results and the x-ray results is that the model used for refinement does not properly account for the disorder. Thus, the systematic error exists in the least-squares model and not in either of the experimental methods. No provision for positional disorder of the nitrogen atom in the ammonium ion was made in either

refinement. In the refinement of K alum, the potassium disorder was coupled with sulfate group disorder. Neglect of this disorder would have a much greater effect on the neutron because the deuterium contributes a large amount to the scattering whereas the hydrogen atoms are practically negligible in x-ray results. The x-ray results are probably correct and the neutron results are in error. Scattering. We therefore reason that the x-ray interference fouriers revealed no significant features and there is no evidence for water disorder coupled with sulfate group disorder. Further, there is no marked anisotropy in the thermal motion of the deuterium atoms which might suggest positional disorder. As noted by Croner and Larson (1966), the sulfate oxygen atom can form hydrogen bonds with the same hydrogen atoms whether the oxygen atom is in position 0,(2) or 0,(2)'. The two distances in the ND4 group differ by 0.0868, but this difference is probably not real. The longer distance has a large standard deviation and further, there is the possibility for some positional disorder of the ND4 group coupled with the sulfate group disorder. Although the thermal ellipsoid of the nitrogen atom suggests this possibility, the ellipsoids of the deuterium do not. The ND4 group perhaps has to move very little to accommodate the reversed 0,(1) atom because either an O-D bond can form (the O---H distance is 2.648) or the Van der Waals radius of the nitrogen.

aton is smaller along a symmetry axis of the I group than in other attractions. The O-D bonds of water (1) are significantly shorter than those of water (2). The difference is related to the strength of the hydrogen bonds, the shorter O-D bonds being associated with longer hydrogen bonds. The shorter hydrogen bonds also tend to be closer to linearity. A stereo drawing of a portion of the structure is shown in Fig. 3. The ammonium ion at 1/2, 1/2, 1/2, (4n has two orientations) is shown with its six water neighbors in the lower right front. The octahedron about the aluminum at 2/2, 0, 1/2 is shown at the lower left front. The sulfate group (in the normal orientation only) is in the middle. All water molecules hydrogen bonded to the sulfate group are shown. ---Page Break--- Except as noted below, all calculations were performed with an TE¥-709% using codes written by Larson, Roof, and Cromer (1963, 196%, 1965). Magnetic Structures I Introduction Recent nuclear magnetic resonance measurements by Pence, Miédents, Mi Saffar, and Kleinberg (196+) have confirmed that CoC,' 6Hg0 and MCL, 6,0 are antiferromagnetic of point group 2/m at liquid helium temperatures, and that each salt has one of three possible magnetic structures. In order to uniquely determine the correct structures, a neutron diffraction study was required. The results of such a study are described in the present report. Crystals of NiClg'61,0 and CoClg' 61,0 are monoclinic, and according to Mizuno (2960, 1962) belong to the space C2/m. There are two molecules in the unit cell. For the nickel salt a-20.25A, b-7.05A, c-6.57A, and β-122910!. For the Co salt a-10.34, b-7.405, c-6.67, and β-12°20". The metal ions are situated on inversion centers at the cell corners, and at the centers of the a:b faces. They are octahedrally coordinated to four oxygen and two chlorine atoms as shown in Fig. 4. These oxygens form a slightly distorted square, with the cations at the center, while chlorines are located on the two normals to the oxygen plane. The remaining two

Water molecules of the formula unit are located in the mirror plane and are relatively free, but do take part in the hydrogen bonding scheme. The local magnetic fields at the proton positions have been measured by applied and zero field methods at 1.1% by Spence, Middents, El Saffer, and Kleinberg (1964). By applying magnetic symmetry theory to these data, it was found that the possible magnetic space group symmetries which describe the magnetic ordering are P2,/%, C2/e, and T2/c. Clearly, O2/e and T2/e are equivalent, but generate different structures when applied to an axial vector. ---Page Break--- -5- In a classic monoclinic cell. Conditions for magnetic Bragg scattering from the (102) zone, for the three different structures that are generated by applying each of these symmetries to the usual monoclinic cell, are that by B be even-even, even-odd, and

odd-odd respectively. The crystals of Hici,/65,0 grown from aqueous solution at about 30°C had the habit described by Groth (1906). An approximately cylindrical crystal was suspended from a titanium-silicon crystal mount that was attached to the tail of an ocean dewar. At 4.2% the crystal was aligned, and nuclear peace was measured on the (10%) zone to give a set of approximate unit cell dimensions. A subsequent search for magnetic reflections showed that the space group is Tqo/e, with the magnetic structure as given in Fig. 5. This structure consists of antiferromagnetic (O01) planes with an antiferromagnetic coupling between planes, to give a magnetic cell that is twice the size of the chemical cell, along the c axis. The angle between the spin direction and the magnetic axis was determined as that angle which produced a minimum value for the function BEIF. It was found to be 22.5° from the a! axis towards the a axis, with a standard deviation of about 1.0°. The observed magnetic form factor values of HIE" are shown in Fig. 6. The solid line is the spherical part of the magnetic form factor, calculated by Watson and Freeman (1961), and is seen to give a good

fit with the date. mis result differs from having a spin direction along the a' axis as has been proposed from susceptibility measurements by Reseda, Kobayshita, and Date (1959) and Flippen and Friedberg (1960). On the other hand, there is good agreement with the results of the antiferromagnetic resonance and magnetic tongue measurements of Date (1965), who has found the spin easy axis to be 25°45 from the HE Coty: Sti0 Neutron diffraction data have been taken at 4.2% and 25% on the ext (oR) tone. Only reflections of the type odd-odd were observed; therefore, the space group is I,0/e and the structure, except for spin direction, is as shown in Figs 5. The magnetic structure consists of antiferromagnetic (OOL) planes, with an antiferromagnetic coupling between the planes, to give a magnetic cell that is double the chemical cell, in the c direction. As the nuclear intensity data have not yet been refined, the set of experimental structure factors was scaled by dividing each number of the set by the sum of the set. The observed structure factors were then compared to sets of calculated structure factors, which were scaled in the same way. The angle between the spin direction and the c axis is defined to be the angle at which $J\_ip = Fel$ to the minimum. This angle was found to be about 25°. There may be quite a large standard deviation on this angle, as it was quite difficult to determine the background scattering at 4.2%. The form factor curve for this angle is shown in Fig. 7. We note that the path engineering for (202) was quite large because of the crystal shape, thus giving the low observed value. The reflections (105) and (305), which have such large error flags, clearly have intensities that are only about 3% of the largest observed intensity. IV Conclusion There are four possible interplanar superexchange paths in the cobalt chloride hexahydrate. They are shown in Fig. 1b. The paths 1-5 are essentially the same as in cobalt chloride dihydrate, where they serve to

strongly coupled ferromagnetic chain. In that case, they have the smaller exchange energy. Short range order is attributed to the strong Co-Cl-Co intrachain coupling. The magnetic structure does not stabilize until the energies of the paths 1-5 predominate over the thermal energy. This occurs at about 17.5% in the case of the cobalt chloride hexahydrate; there is no strong contact coupling along the axis. Instead, there is the very weak Co-O-Co coupling along path 5 in Fig. Thus, in the hexahydrate, we expect antiferromagnetic sheets to be formed in the (001) plane, at about 17%, as a consequence of the paths. As the temperature is decreased, there is increasing short range order until about 2.25 K, where the weak interplanar forces bring three-dimensional stability to the structures. This conclusion is illustrated in the specific heat data of cobalt chloride hexahydrate, as measured by Robinson and Friedberg (1960). They found in these that approximately 52% of the entropy of the transition was obtained above the Néel temperature, and it is seen that the limiting entropy in 2 is reached in the vicinity of the hydrate's melting temperature. Except for the difference

in spin direction, the magnetic ordering in Micip: 6110 is the same as for the CoClp'6#0. The spin direction for the former is at 1972.59 from the a! axis, and for the latter salt is approximately along the c axis. This changing of the cation in these two isostructural salts does not change the magnetic ordering. It is of interest now to study the antiferromagnetism in HBr 6H0, cconry-61,0 and WiCig" 2Hg0, and it is planned to do so as soon as the crystals are available. The experimental data for the magnetic studies on Co and Micip+6H2O were taken from the U.S. Naval Research Reactor and analyzed and interpreted at PRIC. ---Page Break--- Ferroelectrics. In an effort to provide the solid state physicist at PARNC (and other laboratories) with the electronic geometrical knowledge with which to interpret their electrical measurements,

the crystal structure determinations of several ferroelectric compounds of interest have been undertaken. I had, Data has been taken on NeliQ, about 15°C below the transition temperature. While a rigorous analysis awaits further data correction, preliminary results do not seem to indicate any strongly preferred vibrational mode that would account for the phase transition. Such motions may show up after an extinction correction is made or at higher temperatures. Data will be taken at higher temperatures. The room temperature structure of Halis(se05) has almost been completed. Preliminary results show that the heavy atom positions determined by Unteriitner (1966) are correct and those by Chou and Chotang (1957) contain errors. Hydrogen positions will be available shortly. Preliminary measurements show HHS('S605') space group P2 with a = 6.32, b = 16.11, c = 6.24, Z = 8 molecules/unit cell. Liquid structures It was hoped to determine the structure (coordination) of molten S0C2y. If different isotopes of Sn with substantially different scattering lengths may be used, then the neutron diffraction patterns may be combined to yield separate parts of the radial distribution function. ---Page Break--- Age measurements have been made of the scattering lengths of all available isotopes of Sn. These are: d = 50.02, dT = 64.02, dE = 50.06, dE = 604.00, dO = 64.07, dE = 155.05, dI = 459.02. The above isotopes account for 96% of the isotopic content of natural tin. If only isotopic incoherence is included, the total scattering cross section is 4.62 x 10^-4 cm and the coherent cross section 4.61 x 10^-5 cm. Measurements given in Bacon (1962, Neutron Diffraction) indicate 4.9 and 4.6 for the above cross sections, respectively. While the former figure may be due to experimental error, there is also a possibility of nuclear spin incoherence. If this is the case, the effect should be fairly substantial because odd nuclei account for only about 15% of the total natural isotopic abundance. Because of

the closeness of the scattering lengths of the Sn Isotopes to each other it was decided to pursue the solid work with Cu Oh using Cu, Ow, CuPS, Ci, 1°, C1°" which have scattering lengths of «79, -67, Ld, +%, 1.18, .26700" en, respectively. ---Page Break--- ECEESE RE RE RERPREG -20- table-I Structural Parameters for Vin Formate « 'are given first, followed Parameters from neutron data Sckay parameters (Oi) in parentheses. Gy, and Hi, are water oxygens and hydrogens, respectively x ° 6 + 0564, 0015 (038) -3265t. 0012(.325) 09854, 0012(..096) +0868, 0012(..084) sWshat, 0012(.439) +2035%, 0016(.213) «2700, 0012 (.267) 4az0t, 0015(.420) x ° 3 «P23ut. 0019(-220) 61344, 0017(.622) +2067t, 0020(-102) +2682, 0021(..266) Text. 0021(.723) -6565t, 0019(..656) +8262, 0020(.485) 2 ° ° -27agt. 0015(-274) hszet, 0012(.435) -2035%.0013(.204) hoot, 0012(.398) -ke0bt, 0012(.420) 926 0028(.49%) +0656, 0015(..066) +2958. 0015 (.296) +227, 006 +3958. 008 +051, 05 0091. 008 -2614. 005 ake, 002 Ja by standard deviations followed by B a.283(2.2) 2.42.5(2.2) 2.78.3(2-2) 2.2%.2(2.8) 2.1t.2(1.8) 2.2t.2(2.7) 2,54.2(2.3) (1.5) Pat 2.24.3(1.8) 3.08, 3(2.4) ete 6.38.9 3.945 3.98.6 3.285 ---Page Break--- a 'ape 118 Bond Distances and Angles in Manganese and Copper Formate Dihydrates with Standard Deviations. X-Ray results for i (Oifl) and previous X-ray refinement of the copper compound are given in parentheses. Corrections to bond distances involving hydrogens due to the H "ricing" on the Oxygen are given in

brackets. MANGANESE COPPER I Metal-Oxygen Octahedra m0 eaietoe (2.15) 2.30. xb (2.26) m-ce 2.135%017 (225) 2.982, 026 (2.02) mao Bento — (222) 201s. ony (2.01) o1-sa-ce ites (7) o.ab7 (90) o1-a-o8 hts (80.2) eu 5t6 wo conna-ob eats (03) 6.82.7) 12-05 aaigte.3s (2.22) 2.364.019 (2.35) 1-4 2.21St.or3 (2,24) 2.0Mbt, 017 (2.02) 12-02 2.165t.05 (2.36) a.97ht. 5 91) OHO 6.64.5 (1.0) 81.6.6 () Osa 2 t9.rh6 (2.8) B96 wn tee 69.085 (09.8) en5t (69) IX FORMATE GROUPS co 1,264.02 (22) 1,261.05 (2.29) c1-ce Leite (2.25) 2.30808 (39) ca-ca-ce qak.5t.2

(226) 2 (2) tpl 1.054, 052.20) OL-ch-Hpl 126. 545.8 cencheFpl 126,.983.8 ---Page Break--- te. Table Tia Contd. 2-03 azstce (1425) 1.26405 (uz) C2=04 1.26.02 (u21) 128, 05 (2) 2tig2 05 (2.06) 05=C2=04 123.0814 (123) azote (aah) 052g mister ObacBatig2 1ag.5t2.2 TXT HYDROGEN BONDS AND WATER MOLECULES ota Loot ob [2402 a2 934.08 nay HOH aoste ano aphce (213) 2.7oh02 (2.85) Hal-ob Lptos a,a-i-08 asots aoe 2.19.08 (2.81) 2.tat.oe (2.62) y2-0e 2.008.053 oa-ii2-02 arrts Opts 1954.05 (961 othe 098.05 (-09) wpa 07 582.3 ozo (2.61) aris (2.79) OL 1.864.05 O2-HyS-O1, viet 02-05 2.75t.02 (27) 2.67802 (2.76) ORE 1.86%.05 OyarHy05 arete ---Page Break--- 3. 'Table IIe Bond Distances and Angles with Standard Deviations in Anhydrous Copper Formate 'Old Keray refinenent results are in parentheses TONS DISTANCES STANDARD DEVIATIONS cu - 1 Lesh (2.985) +009 cu - 2.939 (2.982) +008 ca LgvL (1.986) +007 cu = L952 (2.98) +009 ou - 2.415 (2.391) +008 cu - cu 3.432 (3.035) +005; a-o 2.2k3 (2.255) Os ao 2.269 (1.295) +03, 2 - 02 1.250 (1.270) +035, ca = ob 267 (2.208) ok a1 - cu - ce B34 (U9) 35 = ca - 98.9 (95) BD ceca oh 9 (69) oT > = cu ob 81.8 (66) 235 a-a-o 320.9 ze Lo = co- h 323.6 ze ua ---Page Break--- the 'Beble IIT Rarefined Parameters and Standard deviations from Yeoray Date of original parameters in parentheses Atos x y 2 B ou, ° ° ° hia owe 3 6 ° uta a -022,005(.055) zat. ood(.2n7) ——«2964.003(.302) EES. ce w322t,005(.303) «6064, 004(.600) «Wast.o0R(.422) 2a a +0p't.o0e(.098) teat. oce(.096) «zat. ooe(.223) 205 oe 20U5%,000(.007) 264. 003(.267) sant ove(sbai) 93 3 s430%.002(.425) « 7058.005(.705) «HOR#, 000.405) 2.683 oF +20KE,002(.204) «35%,003(.356) saKE.O0n(.495) Leeks oa .2.5t,002(.209) hgh 002fshub) 0754. 002(.075) 2.383 oe wast ov{4ia} Bow 00a205) ssa5 one 3.36) 2h ---Page Break--- 'Table 111 refined Parameters and Standard Deviation from X-Ray data of Barclay and Kennard Original Parameters are in Parentheses x y z oo +2150 (.1252). 0002 +0587 (-0506), 0008 +0062 (0065). 0005 a +0292, (-02u%)..000 13254 (.3226). O25

4.2614 (.2625). 00 cy +2172 (.2710).000 -1540 (.1595)- 0015 +2549 (-2536).0082 a +0096 (095). 0005 +2433 (.2427).0009 +2605 (.2622). 0008 ce +2225 (2285). 0006 10215 (.0262).00L0 -195. (.1950)-003 ° +0070 (0061), 0005 +0(U4 (.07U2)..0009 ago (.2902). 0012 oe HUB (,2493). 0005, e121 (-12207).0009 -+ 1869 (~+1459). 0025, 'Thermal Parameters po fa po fa Bs fe ca oon 00h. 207 0002 =, 0005 +0006 ot +0028 00K +0066 =.0007 +0021 ~-00s 2 0005 +008 +0103, 001g. =, 0008 +0085 a 001g +0043 039 00a =, 0015 -.0029 cy +0021 008g +0105 0002 =, 00K 0024 3 +0004 +005 +0083, 10051 =. 0024 -.0002 ob + 00n4 +0087 +0196 +0002 +002 +0005 ---Page Break--- Table IV cent Date and Heavy Atom Parameters for the Alums Studied by Neutron Diffraction (in order of +1 cation size) Sample 0AN(S0y)o-12H,0 ——ND4AL(SO,)g- 21240 CAAL(SO, pr 120 Space Op. ® was 32.352 GLE) — (65, 05, -3) —_— (4,752) —_ (©, 0, 0) — 3(4,%,%) 2652 +5090 327 C2) (sie) BAS 2408 2596 og(2)X 295% opae +2009 y 2105 260% whan 2 saubt ans 36 aytayx +0167 +0456 --1595 x + oho; 305 +9507 2 518 2510 28h ofa)x "Ba sone 3s Y +9596 ~- 0069 -.0080 Zz +9573 156 +0000 ---Page Break--- 2 rey Hydrogen parameters in Hak1(50)p.12%i20 from Least squares toa QQ) Ht) (3) (4) refinement of neutron diffraction data, 0.5958(21) 0.4709(22) 0.5504(25) 0.5926(21) Zz 0.5272(20) 0.3225(21) 0.2031(15) 0.3822(15) 0.3665(21) 0.3808(20) 0.4999(2) 0.2204(25) ---Page Break--- 28. 'Table VI interatomic distances and angles in 1eA1(50, }g- 121.0 standard deviation, in parentheses, apply to the rightmost digit

Distances corrected for thermal motion were in parentheses Distances and angles from neutron data are in underlined. 12-65,(2) 1.881(2) 8 Yan604(2) 2.455(2) 'The sulfate group 8-09(1) 46a (4) (aks) '8-30,(2) ash (2) (48) 4(2)-30(2) 2.589(4) 04(2)-20,(2) 2.366(4) eter molecules (2-02) 0.184) 2.915(24) Gov) o(2)-H(2) 0.05(4) 0.991(24) G54) 4f2)-H03) 0.924) 0.916(27) (009) ay(2)-t4) a.01(s) 0.991(20) 1.006 {2)-A1-04(2) O42) -Ad-04(2) y(2)-He-0,(2.) 4 (2)-ta=0y(2) 05(2)=8-04(2) 0¢(2)-8-09(2)

9(2)=04(2)-04(2) 04(2)=04(2)-04(2) Og(2)-09(2)-04(2) H(2)-04(2)-H(2) u(3)-0,(2)-1H(4) 90, 0(2)° 90, 0(2) 85.0(2) 95-02) aa0.a(2) 108.9(2) 60.0 60.3(2) 59.42) 20302) ---Page Break--- Hydrogen bonds a(2)-30 (2) 0,(2)--3 H(2) Og(2) -0,(2) (2) -2(2) og(2) -04(0) (2) -H(4) (2) -04(2) (2) -H(3) 29. 'Table VI Conta. 2. TH7(4) g(2)-#(2) -04(2) 2.703(25) 2,822(3) (2) -H(2) -94(2) 2.901125) 2.605(5) og(2) -1(4) -04(2) 1.655(20) 2.64903) (2) 83) -04(2) 2.613037) 16005) 3563) aik(2) 376(2) ---Page Break--- Atos Ha, 09(2) 0,2) (2) a,(2) "30 Teble THe 'Toermal elipeotds in Tak2(50y Jp 2250 from X-ray data Standard deviations, in parentheses, apply to rightsont digit Direction angles relative to crystel axes ne dap Tate & « a x 0.270(5)R 2, 26(23)R? shee he? shi? 0,160(3) 2.55(8) 0,160(3) 2.55(8) oat} Re 5he7 he She 0, 1404 3} 1.54(5) 0,140(3) 1.54(6) cable) 265) oe a 3} ei ais ' le 113(7) 2. 36(393 Sh ST shy 2a BB) 0: or 2ehh} 10.62(21) 2a} 115(1) h(a orients) 200} ua 88) tt +h, 76(26) 43) 133(2) 98(5) 2.63(12) 53(6) ato ag 3 0.27a(4) 2. 34(12) au(u) 12e(10) 'ho(6 | T 6) 61 SH ERR I a} Sif} 0, 206(5 | 3.35(10) 95(4) 568 Bath) ---Page Break--- ae able VITO 'Thermal ellipsoids in MeAl(S0,)p-12120 from neutron diffraction date Standard deviations, in paréntheret, apply to the rightmost digit Direction angles relative to crystel axes 2S Aton pplitede BL L a 2 Ma 0. 05(24)R 0.5025)R* shi? she hie o.20(6) 3.3(20) 0.2016) 3:3(20) a 0.4;(20) 2.3(20) she hi she 0,0:20) 2.5033 @.05(40) 0n5(a5, 8 22M} 1.529) sh het sh ol20(h) 3.502) 0.204) 5.5002) 1 0.2065; 5-2(36 Shed MT SAT oat) oo} a3 onan(5: w50a) o,(2 0.36(2 n.6(35 21a 234) 76(5) fe) 355} 2.3 3 a} 30(20) 204(24) . 0-10(8 0.816) silt) a3) 20(20) o.20(2 3.010 aué7) (aT) 86(59) iil aaa} 3h} Sie} "Bae owe} Ona L6(6 913) 0047) 2209: 2 0.123 1.216) 364) aaah) 306 (24) owe) oH Sun 36033 ae) (55) orb: 2M) we 105036) xe) #Q) 0.324) 8.3(20) 23(20) 9n(26) 61(20) ovzats) 3.0035, 901238) 2(5310) 8o{539) 212500) Bots: 1i(20) athe) 23656) #2 0.35(4) 9.4(22) mwas) 0035 1) oz tisha 0.1465)

1.6(32) aah) a1) tea) 6) 0.20(3) 3.3(33) elee) —ate0} 201139) 508) an9) S345) pha 3109 0.2163 5.834) 65a 6o(a5 ie2(a2) 0. 22(3; 3.9(20) 29(299) 5705) a(S) i) ova 3 3.5(02) 37488) 551 3} 89(87) o.22(h 119) 65(29) 39(a7 46(a7) ---Page Break--- (oLyre- (ga)ozt — (6a)egt- ae)nee —Cn)96S (BE)LOH (966 (LYS —(EnJens (YAS (ED LOE (g6)e08- 3 (€s)ocL —(Ano6n——(esoeL (lero — (@a)6i2- (96)CET- —(6S)9L (659669 (an)ets (esr (syn get (East (96602 (ETEK (cunt (6LJowe(ULYEHT (Seen (BEEK Cone (onort (Fours (seple (oor)90¢ —(en)see = (9LYELB— (Gn) ' (eed ba ty (eorex % (es)t99 my ye eotst sy (endone (6669 (962)k2 (6te)Nee —(noa)OGeT (Late —_—(28T)96 ety ay (oscjese- Ts (eer eset ty sy (Gor)obt wy Ts (cages my ey (ootdoct- % (ondont gry ex rx ote ore (new exe (siK6r6"0 (6) 00 (9)erge"0 (5166270 (ge)enée"0 (yxét9ro (e2yatsro an ° (S)p2n0°o (ge)sticro (e)6192"0 mL oxL xt (er)segsro alt ° z a19Tp eomUsya 91 04 Arde 'cosayyuared uF 'suoTINTASD BIVENS o%aat-?("os)tv"an 105 sxeyoeured sarenbs 3009] ILIA a1eea (ez)gtt"o = (s)gsetro (n)lz02"0 (g)tx00"o- (9)<gtt'o, (s)oLto°0 (a)igror0 (ec pntero (o)eere-0 (ogyasiero Oa (g)rore"o (6r)S084"0 (rayeuryro ant ° x sto to 8 sa (ta X w ood ---Page Break--- e 'Tabte Tx Intevetcnic distances and bond angles tn 10,40(59,); standard deviations, in perertheses, appay to the rightaoet eight Distances corrected Fer thermal ceviow are in parentheses a6 0,(2) 1.065(5)8 0,(2)-41-0,(2) 90.6(2)° ,(2)-A1-0,(2) 03.2(2) 6 Oh) 3-057) (2-8-0, (2) Toe2(1) 0, (2)-1-0,0) 204.00) ¥.04(2)! 2.64(13) §D, croup m-2D(1)* 1,204(45) D(1)*=H=D(2) 102.8 (4), w~60(2)" 1,016(16) 'D(2)*=t-D(2)* 15.99) 80, group 8+05(2) 2-49(2) 04(2)=8+05(2) 109.4(6) 8-0,(1)' 21439(23) 0,(2)=8-0,(2) 1109.6(3) 8-0,(2) 16443(9) 0g(2)"=8+0,(2)* oe(2) 8+0,(2)" sba(4)

0,(2)'-8-04(2) a6(2) Water molecules Q(2)-0(1) 0.934(9)8 (2)+0,(1)-D(2) 106.19)" (0.965) 0,,(2)-D(2) 0.954(9)8 (0.97%) 0,(2)-D(5) 0.993(7) (3)=0,(2)=D(4) 12106.2(7) (2.008) 04(2)-0(4) 0.908(7) (2.005) ---Page Break--- og(2)93 0,02) 9,(2)-°D() 05(2)=04(2) 0,(2)-0(2) 09(2)-0,(2) o(2)-0(4)

0,(2)"=04(2) 0,(2)'-D(2) 0(2)"-04(2) 0,(2)'-D(4) 9,(2)-9,(2) 0,(2)-008) he 'Table 1X continued 2.T1e(a2) 9g(2)-23)-0,02) 2.06321) 2.7119) 0,(2)-D(2)-0,(2) 2.652(20) 2.6178) 04(2)-D(4)-0,(2) 1.643(9) 2,05(4) og(2)*-D(2)-0,(2) 1.99(4) 2.714) 0,(2)"=D(4)-0(2) cat) 2.652(9) 9,(2)-D(3)-0,(2) 1.662(9) 163.6(9) 168.219) 269.17) 3512) 62(2) 1ihe(6) ---Page Break--- "35+ rabie x Thermal ellipsoids in ND,A2(S0,),°220,0 Standard deviations, in parentheses, apply to the rightmost digit rea. 0. Direction angles relative to cell axes Aton haplitle y a ® 7 ¥ wex(2)AM2T)AP hee oh mT 8) 2.703) : - - 80) 2.703) : : - Da)! 285) 6.0023) ST ht oht 223) 8.3027) - - - +32(5) 8.327) * - - D(2)*se7te) 5.79) 2825) rar) 222) wee) -3.6(8)— 20719) 37(20)——90(a7) s(2) 7.812) 6920) x(a) 2222) a 0x(28)—0.(6) SHAT 7 ut 22) 1.24) - - : 22) 1.204) - : : s 0.183) 2.58) ST MT SMT one) 1.004) - - : o.1(2) 1.04) : - - 002) nse) .6) OT ot MT 0.25() 5.05) : - - 0.25(1) 5.065) : - - o,(2) oar) 2.203) 336) 303(6) 287) 22) 5.706) 6(4) "6(3) 624) 0.09(2) o.(2), (7) 1224) 316) (2) ovena)—5.6(3) 308) 726) 218) 0.2K) 1.62) —_98(28) zo(12)T2(20) 0.16(2)2.(3) 4820) sete) 34) oy@) ora) eee) 337) 9x27) 029) os) 1.8(2) 8927) 30(24)——200(23) 0.10(2) 0.8(2) mn x(a) 26(8) ---Page Break--- om pa) (2) (3) D(a) Jeph tate 0.1802) 0,22(2) 0.26(2) 0.2202) 0.2702) 0.2703) 0.15(2) 0,20(2) 0.22) 0.16() 0.20) 0.18(2) Table x continued 2.43) 4.063) 5.5(8) 3-9(4) 2.63) 5.108) 1.12) 3.1(5) 346) 2.0(2) 3.66) 2.6(2) 3360 Direction angles relative to cell axes @ B holo) e9(9) (6) 4x9) #300) 232(8) ar(s) 207(8) 75) o(an) 16(8) 59(26) 63(8) 4e(a0) aer(9) asco) 20(6) 207(6) 7318) alto) sia) 'itao) 29(20) 65(a2) 7 62(6) 67(a1) (6) sx(9) (6) r(7) 0e(7) 325(li2) 36(40) 329(27) yo1(20) na(a7) ---Page Break--- 20. 37- RECRENCES Abe, He, Morigali, He, Matewura, 14, Toril, Ke, Yamagata, K., J+ Physs Soc. Sapan 715 (1964) Barclay, G.Ae, Kennard, C.H.L., J. Chem Sor 3209 (1962) Puckews! steayzewsiia, Ye, Acta Cryste 19, 357 (1965) Busing, WR, Lew, Ae, Acta Cryst. 27, 1b2 (1965) Chou, Kud+, and

Fanc, YoChe, KO Hovek Suns Pao 299 (195%) Cromer, Kay and Larson, Acta 0: + An press (1960) Cromer, Dots, Kay, Mole, and Larsen, A.C. Acta Cryst, 22 33 (1966) Crusevshank:, Dnt, Acta Cryst. 2, 154 (1956) Date, Me, Private Communication (1966) Fuippen, R.B., and Friedberg, S.A., J. Appl. Phys. Suppl. 32, 333 (1960) Groth, Py Chem Keyste, T, 27 (1960) Meinberg R., J. Appl. Phys. To be published March (1967) 9. (1966) Kroguann, K., and Mattes, B., Zeit. fur Krist., 120, 292 (1965) Meinberg R., Bull. Am. Phys. Soc. II, Larson, A.C., and Cromer D.T., Acta Cryst. in press (1965) Larson, A.C., unpublished (1965) Larson, A.C., Roof, R.B. Jr., Cromer, D.T., Los Alamos Scientific Laboratory Reports 297, 305, 3255, 3195, 5259, 5309, 5310 (1985, 1964, 1965) Lipson, H., Proc. Roy. Soc. A152, 5h (1956) Mizuno, J., J. Phys. Soc. Japan 26, 1547 (1962) Mizuno J., J. Phys. Soc. Japan 15, 2422 (1960) Mascarentas, Y., Anais da Acad. Brasileira de Ciencias, 35, 260 (1964) Clade, K., Phys. Rev. Letters 15, 252 (1955) Oeada, K., Kay, Mels, Cromer, D.T., Alnoovar, I., J. Chem. Phys. 61, she (1965) Osaki, K., Heliad Ye, Watanabe, T., J. Phys. Soc. Japan 16, 957 (1965) Osaki, K., Maral, Y., Watanabe, T., J. Phys. Soc. Japan 19, 727 (1964) ---Page Break--- B. Robinson, H., Friedberg, S., Phys. Rev. 121, oe (1960) Roof, R.B., Cromer, D.T., Larson, A.C., 3259 Los Alamos Scientific Lab. (1965) Spence, R.D., Miadents, F.S., Z. Saffar, Z., and Kleiner, R., J. Appl. Phys. (1964) Supp. 35, 05% (1965) Unterlitner, F., Chaya, Y., Pepinsiy, R. (To be published) Watson, R.E., Freeman, S.A., Acta Cryst. 24, 27 (1961) ---Page Break--- 2396 Figures Stereoscopic Figures (1,2,3) should be viewed with one's eyes decoupled. The right side of the figure should be viewed by the right eye and the left side by the left eye. A piece of paper extending from the nose to the figure with an edge between the halves of the figure and one eye on each side of the paper, or a commercial viewer, is of aid. ---Page Break--- Figs 1

Stereogram of Manganous Phosphate Dihydrate, View is

slightly off (100) direction. ---Page Break--- ne Fig. 2 Stereogram of Sodium Alum Ellipsoids are scaled to twice the RMS amplitude of vibration. ---Page Break--- are scaled to twice the RMS amplitude of vibration Fig. 3 Stereogram of Deuterated Ammonium Alums, Ellipsoids ---Page Break--- Figs b Sketch of Color Xicig*61ig0 show: metal ton and possible exchange peti. cndings of the ---Page Break--- VV V derangement of magnetic moments in ¥iClp-0Rg0 for the magnetic space group 1,2/e. The angle between the Magnetic moment and the a axis is approximately 10°, Fig. 5 ---Page Break--- 5. Form factor for ¥i2* in NiClg*6H20, from data taken at 1.5°K, Solid line is the theoretical curve for the spherical part. All indices are Based on the magnetic unit cell. Fig. 6 ---Page Break--- Magnetic form factor of CoClg: 6H20 (BL, c)*25° ---Page Break--- oe Publication List 1. Crystal structure by Neutron Diffraction and the Antiferroelectric Phase Transition in Copper Formate Tetrahydrate, K. Okada, MI. Kay, De. Cromer, T. Almodovar, J. Chem Phys. Wh, 1643 (1966). 2 Magnetic Structure of the Zeta Phase of Pdgiine Alloy, JA. Gonzalo and ME. Kay, J. Phys Soc. Japan 21, 1606 (1965). 3. Refinement of the Structure of D-Tartaric Acid by X-Ray and Neutron Diffraction, Y. Okaya, MR. Semple, and MI. Hay, Acta Cryst 21, 257 (1966). 4. Refinement of the Alum structure by X-ray and Neutron Diffraction Study of CsA1(904)e+22%0, « Hidun, DAE, Cromer, MI. Kay, A.C. Larson 21, 383 (1966). 5. Refinement of the Alum structures by X-Ray and Neutron Diffraction of NaA(804)s°12He0, / Alum, D.T. Cromer, Me, Kay, and A.C, Larson, Acta Cryst, in preparation. 6. Refinement of the Alum structures, IV Neutron Diffraction study of Deuterated Ammonium Alum NDsA1(S0s)-12D20 and Alum, Df. Cromer, and MI. Kay, Acta Cryst, in preparation. The Magnetic Structure of MiCle*6H10, R. Kleinberg, J. Appl. Phys, To be published March, 1967. Papers presented at meetings 2. Crystal structure by Neutron Diffraction and Antiferroelectric properties Of Copper Formate Tetrahydrate, K.

Okada, M.I. Kay, D.T. Cromer, I. Almodovar, 'American Crystallographic Assn. Meeting, Austin, Texas, Feb., 1966. 2. Refinement of Alum structures, D.T. Cromer, M.I. Kay, A.C. Larson 'American Crystallographic Assn. Meeting, Austin, Texas, Feb., 1965. 3. Magnetic Structure of Zeta Pd, J.A. Gonzalo and M.I. Kay, International Union of Crystallography, International Congress, Moscow, USSR, May, 1966. 4. Magnetic Structure of CoCl2-6H2O, R. Kleinberg, presented at American Physical Soc. Meeting, Mexico City, Aug., 1966. 5. Magnetic Structure of NiCl2-6H2O, R. Kleinberg, 12th Annual Conference of Magnetism and Magnetic Materials, Washington, D.C., Nov., 1966. 6. 'The Hydrogen Positions in Formate HCOO-, M.I. Kay, I. Almodovar, and S.F. Kaplan, American Crystallographic Assn. Meeting, Atlanta, Georgia, San., 1967. ---Page Break---