In 2-MPNA the *N*-H proton is directed toward the center of a face of the $SbBr_6^-$ octahedron. Of the three possible H · · · Br contacts, the shortest, 2.9 (1) Å, also involves an equatorial bromine atom; this N-H · · · Br angle is 154 (4)°. A slightly shorter distance, 2.8 (1) Å, is also observed in 2-MPNA and involves a ring *C*-H with a terminal bromine atom of the tribromide ion; the C-H · · · Br angle is 134 (4)°. These H · · · Br contacts are marginally shorter than the sum of the appropriate van der Waals radii (3.15 Å[35] or 3.05 Å[36]), indicative of very weak interactions. By contrast, certain pyridinium complexes[7,42,43] exhibit relatively strong N^+-H · · · Br-(M) hydrogen bonds with H · · · Br separations ranging from 2.3 to 2.6 Å.

In conclusion, the following remarks may be made with regard to differences in bonded and nonbonded interactions in the two MPNA analogs. (1) The $SbBr_6^-$ ion in both structures exhibits tetragonal deformation from octahedral symmetry by a *lengthening* of two bonds. The difference between the lengths of the axial and equatorial bonds is smaller in 4-MPNA (Δ = 0.007 (3) Å) than in 2-MPNA (Δ = 0.018 (3) Å). (2) The average length of the bond in the $SbBr_6^-$ ion is 0.010 (3) Å longer in 4-MPNA than in 2-MPNA. (3) The bond in the tribromide ion is also longer in 4-MPNA, by 0.007 (4) Å. (4) Bromine · · · bromine separations along the · · · $SbBr_6^-$ · · · Br_3^- · · · chains are 0.029 (4) Å shorter in 4-MPNA. And (5) the axes of the anions along the · · · $SbBr_6^-$ · · · Br_3^- · · · chains are more nearly collinear in 4-MPNA than in 2-MPNA. Clearly, differences between the two structures are small. It would appear, in fact, that these differences are much too small to account for the change in absorption by the compound relative to others in the series. Subsequently, examination of the quinolinium analog should prove enlightening in view of the anticipated structural change necessary to accommodate the larger cation.

Registry No. $(RH)_2Sb^VBr_9$, RH = 2-methylpyridinium, 12798-43-5; $(RH)_2Sb^VBr_9$, RH = 3-methylpyridinium, 12798-44-6; $(RH)_2Sb^VBr_9$, RH = 2-bromopyridinium, 12798-41-3; $(RH)_2Sb^VBr_9$, RH = 2-chloropyridinium, 12798-42-4; $(RH)_2Sb^VBr_9$, RH = 2,4-dimethylpyridinium, 12798-47-9; $(RH)_2Sb^VBr_9$, RH = quinolinium, 12798-48-0; $(C_6H_7NH)_2Sb^VBr_9$, 12798-45-7.

Acknowledgments. We thank Mobil Research and Development Corp. for their financial support of this research. Helpful discussions with Professors R. A. Jacobson and L. S. Bartell and with A. W. Peters are gratefully acknowledged.

(41) This and the other H · · · Br distances discussed here are based on Sb-Br and Br-Br bond lengths corrected for librational and "riding" motions, respectively. The N-H and C-H bonds were assigned lengths of 1.05 and 1.10 Å, respectively. Standard deviations in the H · · · Br distances were estimated to be 5 times those for C · · · Br distances.

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Contribution from the Los Alamos Scientific Laboratory,
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Bonding Trends in M(III) Transition Metal Hexacyanides. Crystal Structures of $Cs_2LiM(CN)_6$ (M = Mn, Fe, Co)[1]

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The hexacyano complexes $Cs_2LiM(CN)_6$ (M = Mn, Fe) have been prepared and their crystal structures determined. The structure of $Cs_2LiCo(CN)_6$ has been redetermined. Least-squares refinement of three-dimensional X-ray data led to conventional *R* factors of 2.02, 1.58, and 1.74 and weighted *R* factors of 2.05, 1.69, and 1.60% (142, 149, and 149 observed reflections) for Mn, Fe, and Co, respectively. The $Cs_2LiM(CN)_6$ salts crystallize in the space group *Fm*3*m* (four molecules per unit cell) with unit cell dimensions of *a* = 10.667 (3), 10.571 (1), and 10.495 (1) Å for Mn, Fe, and Co, respectively. The M-C bond lengths decrease significantly proceeding from Mn through Co [r_{MC} = 1.976 (4), 1.926 (3), and 1.886 (3) Å]. The C-N bond lengths do not change significantly in the series. These results are discussed in terms of changes in the M-CN σ and π bonding.

Introduction

The structures of hexacyano complexes of Mn(III), Fe(III), and Co(III) have been studied extensively. The tripotassium salts $K_3M(CN)_6$ are known to be isomorphous.[3,4] Unfortunately, polytypism[4,5] and the low site symmetries for the $M(CN)_6^{3-}$ moieties have prevented the determination of significant trends in the M-C and C-N bond lengths in this series of complexes.[6] Early structural studies of the Prussian Blue analogs ($M_3[M'(CN)_6]_2 \cdot xH_2O$, where M = Fe(II), Co(II), etc. and M′ = Fe(III), Co(III), etc.) were limited to powder methods as single crystals could not be obtained.[7] Recently,[8] single crystal studies have been made of Prussian Blue

(1) This work was performed under the auspices of the U. S. Atomic Energy Commission.

(2) To whom correspondence should be addressed.

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(6) Using lattice constants determined from powder diffraction data Chadwick and Sharpe were able to demonstrate that the size of the $M(CN)_6^{3-}$ moiety in $K_3M(CN)_6$ is in the order Cr > Mn > Fe > Co: B. M. Chadwick and A. G. Sharpe, *J. Chem. Soc. A*, 1390 (1966).

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analogs of cobalticyanide. Various other salts[9] of complex cyanides of M(III) transition metals have been investigated, but no systematic study has been made of an isostructural series.

A comparison of the M–C and C–N bond lengths for $M(CN)_6{}^{3-}$ complexes should provide useful trends which can possibly be correlated with changes in the electronic structure of first-row transition metal cyanides. In proceeding from Mn through Co M–CN σ and π bonding are both expected to increase,[10] thereby increasing the M–C bond order and decreasing the M–C bond length. However, for the C–N bond the σ- and π-bonding changes oppose one another. Thus, it should be possible to estimate relative changes in M–CN σ and π bonding in the $M(CN)_6{}^{3-}$ (Mn through Co) series by correlating significant changes in the M–C and C–N bond lengths.

For accurate comparisons of M–C and C–N bond lengths the $M(CN)_6{}^{3-}$ salts studied must be isomorphous so as to avoid structural variations resulting from packing differences. In addition, the $M(CN)_6{}^{3-}$ moiety site symmetry should be octahedral (O_h) in order to avoid the distortions from O_h molecular symmetry characteristic of the tripotassium salts.[11] Recently,[12] Wolberg reported that $Cs_2LiCO(CN)_6$ crystallizes in the space group $Fm3m$ with the $Co(CN)_6{}^{3-}$ moiety occupying a site of O_h symmetry. The suitability of this salt for detailed vibrational analysis[13,14] led to the preparation of $Cs_2LiM(CN)_6$ salts where M = Cr, Mn, and Fe. Preliminary crystallographic data indicated that the new dicesium lithium salts were isostructural with $Cs_2LiCo(CN)_6$ for Mn and Fe. The Cr salt, however, crystallizes in the tetragonal cell $P4_22_12$. This prompted a detailed structural study of this series of hexacyano complexes (Mn through Co), which is the topic of the present report. In order to minimize systematic differences in data collection, the data for $Cs_2LiCo(CN)_6$ were collected again and the structure refined.

Experimental Section

The dicesium lithium salts $Cs_2LiM(CN)_6$ (M = Cr, Mn, Fe, Co) were prepared by combining aqueous solutions of the stoichiometric molar ratios of the individual salts $Li_3M(CN)_6$ and $Cs_3M(CN)_6$. The tricesium and trilithium salts were synthesized from the $K_3M(CN)_6$ salts by ion exchange on Li^+- and Cs^+-replaced hydrogen-form resin (Bio-Rad AG 50W-X16). The potassium salts $K_3M(CN)_6$, for Cr,[15] Mn,[16] and Co[17] were prepared using the usual literature methods while $K_3Fe(CN)_6$ was obtained commercially from Baker. Single crystals of the Cr, Fe, and Co salts were obtained by slow evaporation of aqueous solutions of the respective $Cs_2LiM(CN)_6$ complexes. Decomposition was a problem for the manganese salt since an excess of CN^- could not easily be maintained in the solutions.[16] However, suitable crystals of the Mn salt were obtained by rapid evaporation of an aqueous solution of $Cs_2LiMn(CN)_6$.

The crystals used in this study were parallelepipeds with the following dimensions: Mn, 0.06 × 0.11 × 0.12 mm; Fe, 0.10 × 0.09 × 0.08 mm; Co, 0.11 × 0.11 × 0.07 mm. Precession photographs (Mo Kα radiation) showed the crystals to be cubic, with systematic absences indicating the possible space groups $Fm3m$ ($O_h{}^5$), $F\overline{4}3m$ ($T_d{}^2$), or $F432$ (O^3). The structures were refined in the centrosymmetric space group $Fm3m$.[12]

After alignment of the crystals on an automatic Picker four-circle diffractometer, 12 reflections with 2θ in the range 50–56° were centered (Mo Kα radiation, takeoff angle 1.0°). These reflections were used in least-squares refinements of the cell parameters and the orientation matrices. The room-temperature unit cell constants and their standard deviations for Mn, Fe, and Co salts are $a = 10.667$ (3), $a = 10.571$ (1), and $a = 10.495$ (1), Å, respectively. Mosaicities of the crystals were checked by ω scans of several reflections using a takeoff angle of 1.0°; peak widths at half-height never exceeded 0.1°.

One octant of data was collected for each crystal using Mo Kα radiation and a 5° takeoff angle and a single-crystal graphite monochromator. Crystal data were collected to 70° in 2θ. A symmetric θ–2θ scan range of 2° was used and the count was taken in 0.05° steps, for 2 sec at each step. The background was counted for 20 sec at each end of the scan. Of the 176, 171, and 166 reflections measured, totals of 142, 149, and 149 reflections with intensity above 3σ ($I \geqslant 3\sigma(I)$) were obtained and used in the structure refinement for Mn, Fe, and Co respectively. I is the intensity after background correction and $\sigma(I) = [(T + B) + [0.015(T - B)]^2]^{1/2}$ where T is the total count and B is the estimated background.[18] The intensities of standard reflections, counted after every 25 reflections, showed no significant change during data collection for any of the three complexes.

All three data sets were corrected for Lorentz and polarization effects and absorption corrections were made by the Busing and Levy method[19a] using Larson, Cromer, and Roof's version of Burnham's program.[19b] For the Mn, Fe, and Co complexes the linear absorption coefficients (Mo Kα radiation) are 83.2, 84.5, and 85.9 cm^{-1} and the calculated transmission factors were in the ranges 0.365–0.551, 0.431–0.531, and 0.432–0.558, respectively.

Refinement of Structures. Wolberg's structure was used as a starting model for full-matrix least-squares refinements. The function minimized was $\Sigma w^2(|F_o| - |F_c{}^*|)^2$, where w^2 is the weight defined as $1/\sigma^2(F_o)$ and $F_c{}^* = KF_c/[1 + gLp[2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2]F_c{}^2]^{1/2}$ in which K is a scale constant, Lp is the Lorentz–polarization factor, g is the extinction coefficient,[20] and F_c is the usual calculated structure factor.

Neutral atom scattering factors[21] were used for all atoms and anomalous dispersion corrections were made for Cs and M atoms.[22] The structures were refined in the centric space group $Fm3m$ to weighted R factors, $R = \Sigma w||F_o| - |F_c||/\Sigma w|F_o|$, of 2.05, 1.69, and 1.60% for Mn, Fe, and Co respectively. Final R' factors, $R' = [\Sigma w^2(F_o - F_c{}^*)^2/\Sigma w^2F_o{}^2]^{1/2}$, were 2.11, 1.85, and 1.84%. Anisotropic temperature parameters were refined for C and N atoms. Extinction was important for Fe and Co (as judged by Hamilton's significance tests[23]) and extinction coefficients, g, were included in the refinements for these salts. A correction for extinction was not necessary for $Cs_2LiMn(CN)_6$. The errors in an observation of unit weight for the Mn, Fe, and Co salts are 1.67, 1.55, and 1.64. Analysis of this function as a function of $|F_o|$ and $\lambda^{-1} \sin\theta$ showed no significant trends indicating that while the absolute weights are too high, the relative weights are correct.

Final difference Fourier maps showed residuals centered around the metal atoms which ranged in value from −1.1 to +1.1 e/Å³. With the exception of the region around the Cs atom positions the highest residuals elsewhere were about 0.35 e/Å³. The final positional parameters, temperature factors, and extinction coefficients g are given in Table I.

Discussion

The pertinent interatomic distances are presented in Table II. The bond lengths given by Wolberg[12] and those presented in Table II for $Cs_2LiCo(CN)_6$ agree within the limits of error.

The M–C bond lengths show a monotonic decrease in pro-

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[9] $H_3Fe(CN)_6$: M. Pierrot, R. Haser, C. E. DeBroin, and R. Kern, *Bull. Soc. Fr. Mineral Cristallogr.*, 91, 535 (1968). $D_3Co(CN)_6$: H. U. Gudel, A. Ludi, P. Fisher, and W. Halg, *J. Chem. Phys.*, 53, 1917 (1970); $Ag_3Co(CN)_6$ and $H_3Co(CN)_6$: A. Ludi, H. U. Gudel, and V. Drorak, *Helv. Chim. Acta.*, 50, 2035 (1967).

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[20] (a) W. H. Zachariasen, *Acta Crystallogr.*, 23, 558 (1967); (b) A. C. Larson, *ibid.*, 23, 664 (1967).

[21] P. A. Doyle and P. S. Turner, *Acta Crystallogr., Sect. A*, 24, 390 (1968).

[22] D. T. Cromer, *Acta Crystallogr.*, 18, 17 (1965).

[23] W. C. Hamilton, *Acta Crystallogr.*, 18, 502 (1965).

Table I. Final Least-Squares Parameters for $Cs_2LiM(CN)_6$ [a]

	x [b]	β_{11} [c]	β_{22}	β_{33}	B(Li) [d]	B(Cs) [d]	B(M) [d]	g [e]
$Cs_2LiMn(CN)_6$					1.67 (19)	4.26 (2)	1.63 (3)	
C	0.1853 (3)	81.4 (22)	81.4	46.6 (30)				
N	0.2926 (4)	186.3 (43)	186.3	48.0 (30)				
$Cs_2LiFe(CN)_6$					2.21 (16)	3.70 (1)	1.36 (2)	9.28×10^{-6}
C	0.1822 (3)	62.6 (16)	62.6	41.3 (22)				
N	0.2908 (3)	131.2 (27)	131.3	41.4 (23)				
$Cs_2LiCo(CN)_6$					2.28 (23)	3.32 (2)	1.16 (2)	1.67×10^{-4}
C	0.1797 (3)	48.7 (13)	48.7	37.9 (21)				
N	0.2891 (3)	105.4 (22)	105.4	37.6 (20)				

[a] In this and subsequent tables, the standard deviation in the last significant figure is given in parentheses. [b] (0, 0, x) etc. in 24(e). [c] Anisotropic temperature factors are given by $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33}]$. [d] The Li, M, and Cs atoms occupy special positions (0, 0, 1/2), (0, 0, 0), and (1/4, 1/4, 1/4). [e] g is the secondary extinction coefficient; not included in refinement for $Cs_2LiMn(CN)_6$ (see text).

Table II. Interatomic Distances (Å) for $Cs_2LiM(CN)_6$

Atoms	Mn	Fe	Co
C-N	1.145 (6)	1.148 (5)	1.148 (5)
M-C	1.976 (4)	1.926 (3)	1.886 (3)
Li-N [a]	2.212 (5)	2.212 (3)	2.213 (3)
Cs-N [b]	3.798 (1)	3.762 (1)	3.733 (1)
Cs-C	3.834 (1)	3.805 (1)	3.783 (1)

[a] Li atom surrounded by octahedral array of N atoms. [b] Cs atom surrounded by array of 12 CN moieties. The next nearest neighbor to Cs is greater than 5 Å away.

Table III. M-C Bond Lengths (Å) Corrected for Thermal Motion [a]

Metal	S^0 [b]	S^R	S^I
Mn	1.976	1.989	2.010
Fe	1.926	1.936	1.953
Co	1.886	1.893	1.908
(Mn-C)-(Co-C)	0.090	0.096	0.102

[a] See ref 24. [b] S^0 = uncorrected M-C bond length; S^R = M-C corrected using riding model; S^I = M-C corrected assuming independent motion.

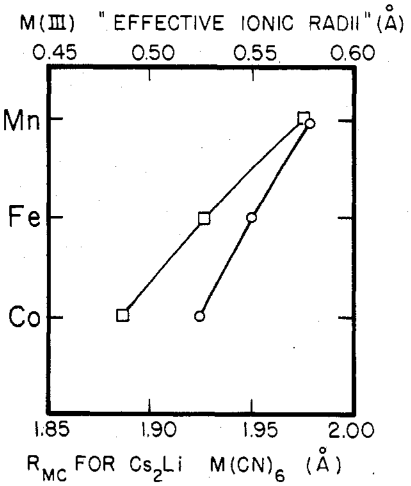

Figure 1. Comparison of M-C bond lengths in $Cs_2LiM(CN)_6$ (□) with Shannon and Prewitt's "effective ionic radii" (○) for M(III).[25] The ionic radii selected correspond to low-spin M(III) in octahedral environments.

ceeding from $Cs_2LiMn(CN)_6$ through $Cs_2LiCo(CN)_6$. The total decrease [0.090 Å] and the successive bond length decreases [Mn → Fe, 0.050 (5) Å; Fe → Co, 0.040 (4) Å] are all significant. When the M-C bond lengths are corrected for thermal motion,[24] Table III, the decrease through the series is even greater.

It is of interest to compare the M-C bond length changes in $Cs_2LiM(CN)_6$ (M = Mn → Co) with changes in the M(III) ionic radii. A comparison of M(III) ionic radii[25] and the M-C bond length in $Cs_2LiM(CN)_6$ is given in Figure 1. The M-C bond length in $Cs_2LiM(CN)_6$ is shown to decrease faster than the ionic radii. The overall decrease in ionic radii (*ca.* 0.055 Å) is about half that of the M-C bond in the series. Therefore, the decrease in the M-C bond length in $M(CN)_6{}^{3-}$ complexes in proceeding from Mn to Co cannot be attributed entirely to a decrease in the ionic radius of the transition metal.

In terms of a molecular orbital model the change in M-C bond length can be attributed to changes in M-CN σ and π bonding. The difference between transition metal-fluoride bonds, M-F, and transition metal-cyanide bonds, M-CN, results primarily from the availability of orbitals in the latter which are suitable for back-bonding. If one assumes that the σ-bonding changes for M-F and M-CN bonds in proceeding from Mn through Co are roughly the same, then differences between changes in the M-F and M-CN bond lengths can be attributed to M-CN π bonding. Thus, the comparison between M(III) ionic radii and M-CN bond lengths presented in Figure 1 provides an estimate of the relative change in σ and π bonding for the hexacyano complexes. The above considerations indicate that changes in the σ and π bonding in $M(CN)_6{}^{3-}$ affect the M-C bond by roughly the same amount.

The C-N bond lengths in $Cs_2LiM(CN)_6$ complexes do not change in proceeding from Mn through Co unless corrections are made for thermal motion in which case the change is not statistically significant. The absence of a change for the C-N bond lengths lends support to the suggestion that M-CN σ bonding increases at roughly the same rate as M-CN π bonding (see above). That is, the increase in C-N bond order resulting from increased M-CN σ bonding is compensated by the decrease in C-N bond order resulting from increased M-CN π bonding as concluded earlier from infrared studies.[10] The C-N bond length is a poor measure of small changes in C-N bond order and a better measure of the C-N bond order will be provided by accurate potential constant determinations.[26]

A striking feature of the structures presented here is the high thermal motion of the Cs atoms (the mean-square amplitudes of vibration are given in Table IV). This unusual thermal motion results from the Cs atoms occupying holes which are too large. In all four structures the Cs atom nearest neighbor distances (Cs-N) are significantly larger than the combined Cs and N atom atomic radii (3.24 Å).[27] However,

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Table IV. Root-Mean-Square Amplitudes of Vibration (Å) for for $Cs_2LiM(CN)_6$

	Mn	Fe	Co
Li	0.145 (16)	0.167 (12)	0.170 (17)
Cs	0.232 (2)	0.216 (1)	0.205 (1)
M	0.144 (3)	0.131 (2)	0.121 (2)
C(max)[a]	0.216 (6)	0.188 (5)	0.165 (4)
C(min)	0.164 (10)	0.153 (8)	0.145 (8)
N(max)	0.327 (8)	0.272 (6)	0.242 (5)
N(min)	0.166 (10)	0.153 (8)	0.145 (8)

[a] Maximum direction for C and N atoms is perpendicular to the C-N bond while minimum direction is coaxial with the C-N bond.

the Li-N bond lengths in $Cs_2LiM(CN)_6$ are comparable to the combined Li and N atomic radii (2.1 Å) and are constant through the series Mn → Co. The foregoing indicates that the $Cs_2LiM(CN)_6$ crystal structures are held together by strong Li-N bonds while the Cs atoms do little more than occupy space. This is in agreement with the high Li-N and low Cs-N force constants observed for $Cs_2LiCo(CN)_6$.[14]

The Cs atom thermal motion is shown to decrease monotonically in the series Mn → Co as do the mean amplitudes of vibration perpendicular to the C-N bond for both C and N atoms.

The decreases in Cs, C, and N atom thermal motion are to be expected since the structures are becoming more tightly packed (and the Cs atom hole size is reduced) as indicated by the monotonic decreases in the M-C and Cs-N bond lengths.

It should be possible to obtain meaningful potential constants for this series of compounds since lattice perturbations can be included[13] and since isotopic vibrational data can be obtained.[26] It will, no doubt, be useful to correlate the bond lengths obtained in this study with potential constants for the $Cs_2LiM(CN)_6$ complexes.[28]

Registry No. $Cs_2LiMn(CN)_6$, 37164-28-6; $Cs_2LiFe(CN)_6$, 37164-29-7; $Cs_2LiCo(CN)_6$, 23591-91-5.

Acknowledgment. The help of D. Cromer, Los Alamos Scientific Laboratory, is gratefully acknowledged.

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[28] A supplementary structure factor table will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth St., N.W., Washington, D. C. 20036. Remit check or money order for \$3.00 for photocopy or \$2.00 for microfiche, referring to code number INORG-73-283.

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Angeli's Salt. Crystal Structure of Sodium Trioxodinitrate(II) Monohydrate, $Na_2N_2O_3 \cdot H_2O$[1]

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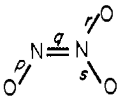
Received July 10, 1972

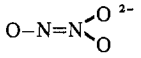
The structure of sodium trioxodinitrate(II) monohydrate, $Na_2N_2O_3 \cdot H_2O$, has been determined by X-ray diffraction methods. The salt crystallizes in the orthorhombic system, space group *Pbcm*, $a = 6.359$ (2), $b = 9.765$ (4), $c = 6.677$ (9) Å. The unit cell contains four formula units. The structure was solved by direct methods and refined by full-matrix least squares to an R index of 0.032. The anion is planar with the structure



Bond distances and angles are as follows: $p = 1.347$ (4), $q = 1.264$ (5), $r = 1.310$ (4), $s = 1.322$ (4) Å; $(pq) = 112.9$ (3), $(qr) = 118.4$ (3), $(qs) = 122.5$ (3)°. The water of hydration is hydrogen bonded to O(s) with O· · ·O = 2.784 (3) Å. The shortest Na–O contact distance is 2.35 Å.

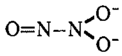
Introduction

Angeli's salt, $Na_2N_2O_3$, was first described by Angeli[2] in 1896. He proposed the structure



$$\mathrm{O{-}N{=}N}\begin{smallmatrix}\nearrow \mathrm{O} \\ \searrow \mathrm{O}\end{smallmatrix}{}^{2-}$$

for the anion. Over the years other atomic arrangements have been suggested, and considerable effort has been expended on this problem.[3-6] Based on the uv spectrum of solutions of the salt Addison, *et al.*,[3] have concluded that the anion contains an N=N double bond. Hendrickson, *et al.*, in an X-ray photoelectron spectroscopic study[6] determined the presence of two chemically nonequivalent N atoms, and presented the formula



$$\mathrm{O{=}N{-}N}\begin{smallmatrix}\nearrow \mathrm{O^-} \\ \searrow \mathrm{O^-}\end{smallmatrix}$$

as the most probable.

It seemed to us that an X-ray study would represent the best approach to this structural problem. We now report our results for the monohydrate of Angeli's salt, $Na_2N_2O_3 \cdot H_2O$.

Experimental Section

Angeli's salt was prepared according to the procedure of Addison,

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[1] Abstracted from the M.S. thesis of M. R. Sequeira, University of California, Davis, Calif., 1971.

[2] A. Angeli, *Gazz. Chim. Ital.*, **26**, 7 (1896).

[3] (a) C. C. Addison, G. A. Gamlen, and R. Thompson, *J. Chem. Soc.*, 338 (1952); (b) H. R. Hunt, Jr., J. R. Cox, and J. D. Ray, *Inorg. Chem.*, **1**, 938 (1962).

[4] P. E. Sturrock, J. D. Ray, J. McDowell, and H. R. Hunt, Jr., *Inorg. Chem.*, **2**, 649 (1963).

[5] R. D. Feltham, *Inorg. Chem.*, **3**, 900 (1964).

[6] D. Hendrickson, J. M. Hollander, and W. L. Jolly, *Inorg. Chem.*, **8**, 2642 (1969).