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The Crystal Structure of Sodium Tricyanomethanide, $NaC(CN)_3$

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The structure of sodium tricyanomethanide, $NaC(CN)_3$, has been determined by X-ray diffraction techniques using three-dimensional Weissenberg data. The crystals are triclinic, space group $P\bar{1}$. The unit cell dimensions are: $a = 3.705$ Å, $b = 9.198$ Å, $c = 7.741$ Å, $\alpha = 105.76°$, $\beta = 100.16°$, $\gamma = 88.31°$, and $Z = 2$. All positional and thermal parameters have been refined to a final R index of 5.1% for 787 reflections of observable intensity. The symmetry of the $C(CN)_3^-$ ion is close to D_{3h}. There is a slight deviation from planarity, and a significant difference between the longest (1.164 Å) and shortest (1.145 Å) C–N bond distance. The average C–C distance is 1.408 Å. The Na^+ ion is coordinated to 6 nitrogen atoms, arranged in the form of a distorted octahedron.

Since the first X-ray structure information on the tricyanomethanide anion was reported,[1] a number of crystal structure investigations of this ion have been published or are in progress. Desiderato and Sass [2] found a non-planar anion in ammonium tricyanomethanide which, according to the authors, indicates a concentration of negative charge on the central carbon atom. Bugg and Sass [3] drew the same conclusion from the X-ray crystallographic investigation of pyridinium dicyanomethylide. Bugg [4] found, however, that the anion in potassium *p*-nitrophenyldicyanomethanide is planar. A three-dimensional investigation of potassium tricyanomethanide by Britton and Witt [5] showed that the anion deviated only slightly from planarity, while Bondi, Bonamico, Torelli and Vaciago [6] concluded that the tricyanomethanide ion is planar in the copper(II) compound.

The crystal structure investigation of the sodium tricyanomethanide was undertaken to obtain more precise atomic parameters for the anion. The announced refinement of the potassium salt [1] has been discontinued.

CRYSTAL AND X-RAY DATA*

$NaC(CN)_3$ was synthesized according to a description for the potassium salt by Trofimenko.[7] The light yellow needle-formed crystals always appeared as twins. Different solvents and growing conditions were tried, but we were unsuccessful in finding single crystals. The crystal used in this investigation (*ca.* 0.06 × 0.06 × 0.3 mm^3) had a very small twin satelite.

The crystals are triclinic and the space group is $P\bar{1}$. The unit cell dimensions were determined by least squares calculations ** based on measurements of 22 lines registered on a Guinier camera (5 % KCl for calibration, $a = 6.2929$ Å).[8]

The following values were obtained:

$a = 3.705$ Å (0.001); $b = 9.198$ Å (0.003); $c = 7.741$ Å (0.002); $\alpha = 105.76°$ (0.04); $\beta = 100.16°$ (0.05); $\gamma = 88.31°$ (0.06).
Formal e.s.d. in brackets.

a is the needle axis and (001) is the twin plane. This cell is transformed to the Dirichlets reduced cell by interchanging the b and c axis and making the angles acute. There are two formula units in the cell and the calculated density is 1.50 $g \cdot cm^{-3}$.

Integrated equi-inclination Weissenberg photographs of $h = 0$ through 3 were collected (Cu$K\alpha$ radiation). Two sets of films were taken, each with a 200° oscillation and with an overlapping region of 40°. The intensities were recorded on a microphotometer except for the weakest reflections which were estimated visually. For each layer the two sets were brought on a common scale by using the reflections of the overlapping regions. The mean values of the intensities were determined. Of the 999 possible reflections on these films 787 were observed. No correction for absorption was applied. Precession photographs of the $hk0$ and $h0l$ zones were taken with filtered Mo-radiation, and the intensities were registered on a recording photometer.

From the $h0l$ data and the Weissenberg diagrams it could be observed that the intensities from the small twin satelite were of negligible magnitude and no corrections were found necessary. Only the Weissenberg data were used in the three-dimensional refinement, except for 6 low angle reflections which were taken from the precession data. The data were recorded at room temperature.

STRUCTURE DETERMINATION

The structure was solved in two projections. The $0kl$ projection, which gives a well resolved electron density map, was refined to $R = 10$ % using isotropic temperature factors. The $h0l$ projection was analogously refined to $R = 8$ %. The different layers of the Weissenberg data were brought on a common scale by comparison with calculated structure factors based on the two-dimensionally refined structure. The initial R index was 15 %. Least squares

* With the exception of programs listed in footnote all programming has been carried out at this institute.

** Program written by R.A. Sparks.

refinement brought the R index down to 10 %. Anisotropic thermal parameters were then introduced and the refinement proceeded until an R index of 6.2 % was reached. At this stage the intensities were corrected for secondary extinction assuming a cylindrical crystal.[9] After two additional cycles of the least squares refinement the parameters converged with an $R = 5.1$ % for the observed reflections. The 212 non-observed reflections were then included as 1/3 of the minimum observable intensities.[10] Two more cycles resulting in an overall R index of 6.3 % did not give any significant shifts in the parameters, but improved the standard deviations by approximately 10 %. Observed and calculated structure factors are listed in Table 1. Non-observed reflections, marked U, are included with the values used in the least squares refinement.

A total difference Fourier synthesis* was calculated; the electron density varied between -0.29 — $+0.25$ e·Å^{-3} the standard deviation being 0.067 e·Å^{-3}.[11] No information about the distribution of the negative charge on the anion was obtained.

This least squares refinement program ** minimizes the sum $R_1 = \sum w^2 (K F_o - G|F_c|)^2$ by a full-matrix routine. Of the scale factors K and G, the latter is the adjustable parameter. A weighting analysis is included. The atomic formfactor for Na^+ was taken from *International Tables*,[12] and those for carbon and nitrogen from Hanson *et al.*[13]

The R index is defined by $R = \sum||F_o| - |F_c||/\sum F_o$. The weighting scheme $w = 1$ for $F_o \lesssim 7$, $w = 7\ F_o^{-1}$ for $F_o > 7$ gave nearly constant averages for $w^2\Delta^2$ in groups of increasing F_o's. (For non-observed reflections $w = 0.33$.)

The calculation of interionic distances revealed no contacts shorter than expected between non-bonded atoms.*** The ionic forces between the cation and the nitrogen atoms determine the crystal structure. A schematic drawing of the $0kl$-projection is shown in Fig. 1.

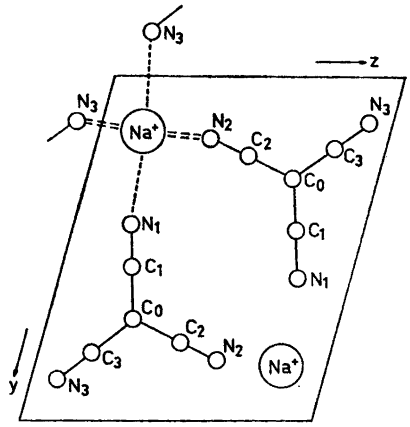
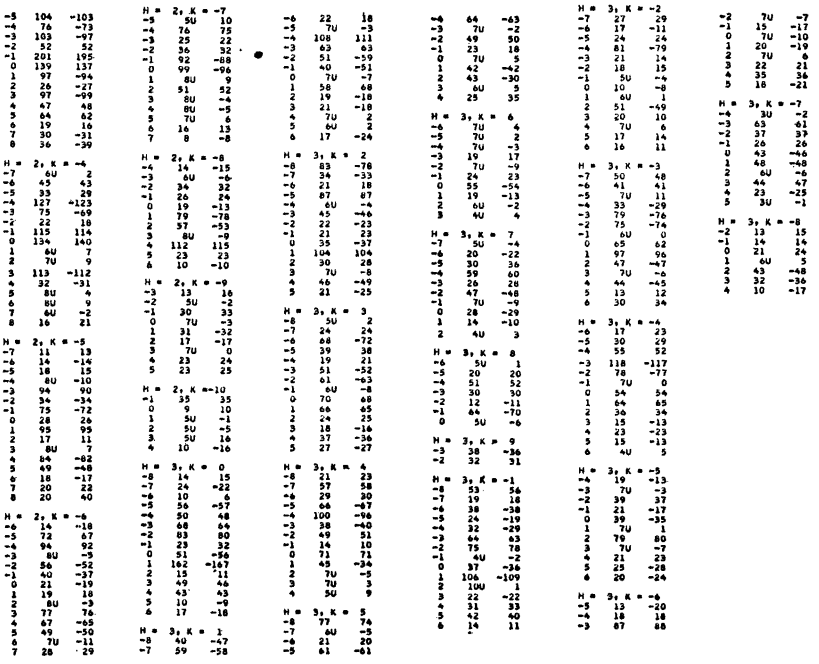


Fig. 1. A drawing of the structure of $NaC(CN)_3$ viewed along the a axis showing the numbering of atoms and the coordination (dashed lines).

* Program written by P.K. Gantzel and H. Hope, adapted for UNIVAC 1107 by H. Hope.
** Program written by P.K. Gantzel, R. A. Sparks, and K. N. Trueblood (IUCr World list No. 384), modified and adapted for UNIVAC 1107 by C. Rømming.
*** Program written at University of California, Los Angeles.

Table 1. Observed and calculated structure factors. The data are separated into groups having common values of h and k. The three columns in each group list values of l, 10 F_o and 10 F_c, in that order. Non-observed reflections, marked U, are included with the values used in the least squares refinement.

Table 1. Continued.



The anisotropic thermal parameters and the principal axes of the thermal vibration are given in Tables 2a and 2b, respectively. The atomic vibration tensors referred to crystallographic axes were obtained according to Scheringer [14] and the vibration tensors of the anion atoms were applied in a rigid body analysis of the translational and librational motion with refinement of the centre of libration.[15,16] The **S** tensor of Shoemaker and Trueblood [17] was not applied. The results are given in Tables 3a and 3b.* The **T** and ω tensors were calculated in orthogonal coordinates along the molecular inertial axes. This coordinate system has one axis (N) normal to the least squares plane through the anion, and another (L) along the $C_0 \cdots N_3$ direction with its origin at the centre of gravity. The translational motion is approximately isotropic. The librational motion is highly anisotropic, and the minute negative eigenvalue of ω (Table 3b) indicates that the treatment of the anion as a rigid body may be argued and that the internal vibrations also ought to be considered. The centre of libration is found 0.81 Å from the central carbon atom, close to the least squares plane. A line between C_0 and the centre of libration forms angles of 102° and 19° with $C_0 \cdots N_2$ and $C_0 \cdots N_3$.

The uncorrected values of the individual atomic positional parameters from the least squares refinement, the values corrected for libration, and the standard deviations estimated from the inverse of the matrix of the normal

* Program written by F. L. Hirshfeld, revised by F. Gram (IBM 1620 II).

Table 2a. The thermal parameters from the least squares refinement and their e.s.d.'s (all values $\times 10^5$). The temperature factor is given by exp− $(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)$.

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Na^+	5873	693	1190	556	1840	457
	106	13	20	50	63	24
C_0	5429	798	1205	171	1730	643
	247	33	47	128	155	61
C_1	6416	839	1443	193	1790	799
	272	37	52	141	173	69
C_2	4244	941	1195	324	1271	766
	230	34	48	127	149	63
C_3	4180	683	1071	387	1412	130
	222	30	44	114	140	56
N_1	10928	891	2525	894	3470	934
	330	37	70	160	232	79
N_2	7264	1378	1234	742	2243	859
	253	39	46	149	158	68
N_3	7606	842	1334	383	2467	723
	243	31	46	127	155	60

equations, are given in Table 4. The bond distances and angles are listed in Table 5. The uncorrected values and their formal e.s.d.'s from the least squares refinement are listed for comparison.

There is a slight deviation from trigonal symmetry of the anion. This may be demonstrated by the $N_1—C_0—N_2$ angle which is 118.2° (0.1°). The principal moments of inertia along the L and M axes (Table 3b) are significantly different, thus verifying the deviation. There are no significant differences between the C—C bond distances. The mean value is 1.408 Å. There is a significant difference, however, between the shortest and longest carbon-nitrogen bond length. Whether this difference is real and a consequence of the coordination, or is due to wrong corrections, has not been decided.

The anion is not quite planar. The central carbon atom and the three cyano carbon atoms are situated 0.039, 0.023, 0.020, and 0.019 Å above a plane through the three nitrogen atoms, respectively. The deviation of the different atomic positions from a least squares plane through the anion is shown in Table 6. The normal of the plane makes a 21.3° angle with the a axis, and the anion-anion distance is thus 3.45 Å. The shortest separations between atoms in adjacent anions are: carbon-carbon 3.47 Å, carbon-nitrogen 3.44 Å, and nitrogen-nitrogen 3.43 Å. The shortest sodium-carbon contact is 3.11 Å.

Table 2b. The principal axes of the atomic vibrations given by the components of a unit vector in fractional coordinates e_x, e_y, e_z; the corresponding root mean square amplitudes of vibration $(\bar{u}^2)^{\frac{1}{2}}$ (Å) and B(Å^2).

Atom	$(\bar{u}^2)^{\frac{1}{2}}$	B	e_x	e_y	e_z
	0.206	3.34	0.249	0.026	0.076
Na^+	0.173	2.38	0.058	0.059	−0.083
	0.159	2.00	−0.099	0.093	0.076
	0.200	3.14	0.234	0.023	0.090
C_0	0.178	2.50	−0.077	0.107	0.045
	0.162	2.08	0.121	0.029	0.092
	0.214	3.62	0.236	0.025	0.088
C_1	0.191	2.88	−0.130	0.073	0.089
	0.172	2.35	0.051	0.083	−0.054
	0.197	3.06	0.079	0.102	0.081
C_2	0.176	2.44	0.156	−0.048	0.088
	0.161	2.04	−0.211	0.000	0.065
	0.190	2.84	0.078	−0.054	0.097
C_3	0.174	2.39	0.225	0.066	0.047
	0.141	1.58	−0.137	0.074	0.084
	0.284	6.39	0.228	0.020	0.095
N_1	0.242	4.62	−0.149	0.003	0.097
	0.184	2.67	−0.032	0.111	0.018
	0.240	4.55	0.146	0.097	0.050
N_2	0.218	3.76	0.219	−0.057	0.039
	0.167	2.21	−0.077	0.013	0.121
	0.233	4.28	0.256	0.017	0.070
N_3	0.183	2.65	−0.063	0.105	0.064
	0.169	2.25	0.074	0.038	−0.098

The six nitrogen atoms coordinated with one sodium ion are all belonging to different anions and form a slightly distorted octahedron with edges ranging from 3.43 Å to 3.71 Å and angles (theoretically 60°) from 56.3° to 64.4°. There is a one, two, and three coordination of sodium about the nitrogens, N_1, N_2, and N_3, respectively. N_1 and N_2 are situated 2.45 Å and 2.44 Å from the plane through the four remaining nitrogen atoms in the octahedron. The position of the sodium ion is 0.08 Å out of this plane in the direction of N_1. This shows that N_1 which has one sodium ion as nearest neighbour, is more strongly bonded. The N···Na^+ distances and the corresponding C_0···N···Na^+ angles are listed in Table 7.

Table 3a. Components of atomic vibration tensors U_C ($\times 10^4$ Å^2) referred to crystallographic axes; as calculated from the thermal parameters of the least squares refinement (exp), the corresponding estimated standard deviations (esd), and as calculated from the rigid body parameters (RB).

Atom		U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Na	exp	408	297	361	47	133	82
	esd	7	6	6	4	5	4
C_0	exp	377	342	365	14	125	115
	esd	17	14	14	11	11	11
	RB	311	324	353	14	94	97
C_1	exp	446	359	438	16	130	144
	esd	19	16	16	12	13	12
	RB	493	325	499	41	159	122
C_2	exp	295	403	362	27	92	138
	esd	16	15	15	11	11	11
	RB	390	424	354	33	123	123
C_3	exp	290	292	325	33	102	23
	esd	15	13	13	10	10	10
	RB	288	329	337	6	88	90
N_1	exp	759	381	766	77	252	168
	esd	23	16	21	14	17	14
	RB	745	330	718	80	249	145
N_2	exp	505	590	374	64	162	154
	esd	18	17	14	13	11	12
	RB	484	591	360	59	155	135
N_3	exp	528	360	404	33	179	130
	esd	17	13	14	11	11	11
	RB	500	405	420	32	175	161

DISCUSSION

The three compounds $NaC(CN)_3$, $KC(CN)_3$, and $NH_4C(CN)_3$ are not isostructural. The coordination about the cation in the three compounds is shown in principle in Fig. 2. The 1-coordinated nitrogen in the sodium compound is 2-coordinated in the other compounds, thus both the potassium and the ammonium compounds have a coordination number of seven. Desiderato and Sass describe the coordination of NH_4^+ in $NH_4C(CN)_3$ as an 8-coordination. One of these $NH_4^+ \cdots N$ distances is, however, 3.52 Å compared with an average of 3.05 Å of the seven remaining coordination distances and 3.32 Å, which is the shortest approach of equally charged nitrogens.

Table 3b. The values of the principal moments of inertia in the anion (a.m.u. Å²), the unit vectors along the principal axes and the centre of gravity (fractional coordinates) defining the molecular coordinate system; the rigid body tensors and the centre of libration referred to this coordinate system.

Principal axes of inertia

Axis	Moment of inertia	x	y	z
L	169.5	0.0217	0.0647	−0.0828
M	174.8	0.0509	−0.0915	−0.0964
N	344.3	0.2686	0.0143	0.0493
Centre of gravity:		0.7202	0.7079	0.2978

Rigid body tensors

$$\mathbf{T} = \begin{pmatrix} 318 & -5 & -31 \\ & 305 & -26 \\ & & 274 \end{pmatrix} \times 10^{-4}\ \text{Å}^2 \qquad \omega = \begin{pmatrix} 12 & 155 & 15 \\ & 834 & 81 \\ & & 333 \end{pmatrix} \times 10^{-5}\ \text{rad}^2$$

	Eigenvalues	r.m.s. values	L	M	N
	0.034 Å²	0.18 Å	0.800	0.297	−0.522
T	0.031	0.18	0.456	−0.866	0.206
	0.025	0.16	0.390	0.403	0.828
	28.7 (°)²	5.4°	0.178	0.972	0.151
ω	10.5	3.2°	0.026	0.149	−0.988
	−0.5 ≈ 0		0.984	−0.180	−0.002
Centre of libration:			0.768	−0.255	−0.075

For the three structures the coordination distance decreases with increasing carbon-nitrogen-cation angle. This is also true for caesium tetracyanoquinodimethanide,[18] a somewhat similar compound, in which 8 nitrogen atoms form a cube shaped polyhedron about the Cs^+ ion. The coordination distances

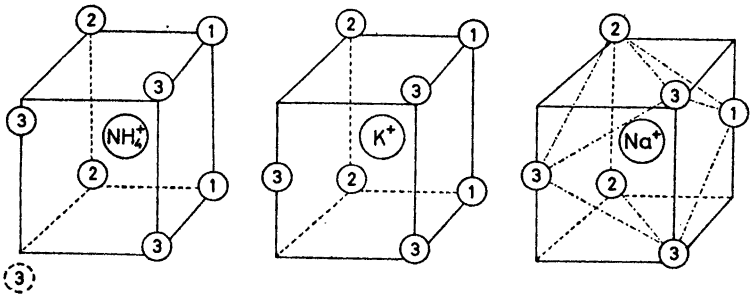


Fig. 2. The coordination of nitrogen atoms about the cation in the ammonium, potassium, and sodium tricyanomethanide.

Table 4. Uncorrected positional parameters from the least squares refinement and the parameters corrected for libration (in fractional coordinates). E.s.d.'s and corrections ($\times 10^4$) are given below the uncorrected and corrected parameters, respectively.

Atom	Uncorrected			Corrected		
	x	y	z	x	y	z
Na^+	0.8207	0.1596	0.1566			
	2	1	1			
C_0	0.7140	0.7077	0.2952	0.7140	0.7073	0.2955
	7	2	3	0	−4	3
C_1	0.7647	0.5516	0.2375	0.7646	0.5506	0.2379
	8	3	3	−1	−10	4
C_2	0.6416	0.7728	0.4722	0.6416	0.7724	0.4729
	7	3	3	0	−4	7
C_3	0.7491	0.7994	0.1810	0.7492	0.7996	0.1807
	7	2	3	1	2	−3
N_1	0.8075	0.4244	0.1939	0.8074	0.4230	0.1945
	9	3	4	−1	−14	6
N_2	0.5842	0.8241	0.6173	0.5842	0.8236	0.6184
	7	3	3	0	−5	11
N_3	0.7787	0.8754	0.0870	0.7789	0.8762	0.0863
	7	2	3	2	8	−7

Table 5. Bond distances and angles in the anion corrected for libration motion compared with the uncorrected values. E.s.d.'s are from the least squares refinement procedure.

Dist.	Corr. (Å)	Uncorr. (Å)	E.s.d. (Å)
C_0-C_1	1.405	1.401	0.003
C_0-C_2	1.411	1.407	0.003
C_0-C_3	1.409	1.401	0.002
C_1-N_1	1.145	1.141	0.003
C_2-N_2	1.151	1.149	0.003
C_3-N_3	1.164	1.157	0.002
Angle	**Corr. (°)**	**Uncorr. (°)**	**E.s.d. (°)**
$C_1-C_0-C_2$	119.2	119.4	0.2
$C_2-C_0-C_3$	120.1	120.1	0.2
$C_3-C_0-C_1$	120.7	120.5	0.2
$C_0-C_1-N_1$	178.7	178.7	0.3
$C_0-C_2-N_2$	179.0	179.0	0.2
$C_0-C_3-N_3$	179.8	179.8	0.2

Table 6. Deviations from the least squares plane through the anion: (0.2686 **a** + 0.0143 **b** + 0.0493 **c**) **r** − 3.252 = 0.

Atom	Deviation (Å)
C_0	−0.024
C_1	−0.008
C_2	−0.006
C_3	−0.005
N_1	0.015
N_2	0.014
N_3	0.014

Table 7. Coordination distances and the corresponding central carbon-nitrogen-cation angles.

Atom	N···Na$^+$(Å)	C_0···N···Na$^+$(°)
N_1	2.373	166.4
N_2	2.471	148.6
N_2	2.542	108.8
N_3	2.526	130.1
N_3	2.548	129.2
N_3	2.613	107.5

minus the respective cation radii of Na^+ (0.95 Å), K^+ (1.33 Å), Cs^+ (1.69 Å), and NH_4^+ (1.48 Å) are plotted as a function of the C—N···X^+ angle (Fig. 3). The plot shows a decrease of the reduced interionic distances with increasing angle, and a smooth curve enveloping the lower part of the assembly of the scattered points may indicate the variation with angle of the ionic radius of a partially charged nitrogen due to the lone pair electrons. The effect of the coordination number is ignored.

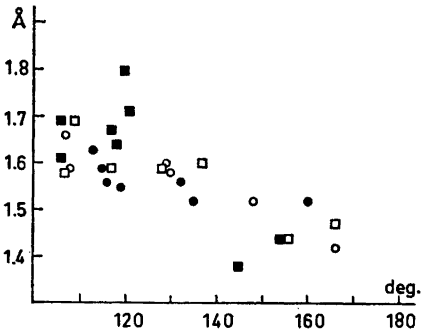


Fig. 3. The coordination distances minus the respective cation radii of Na^+ (○), K^+ (●), Cs^+ (■), and NH_4^+ (□); plotted as function of the C—N···X^+ angle.

The different degrees of deviation from planarity of the anion in the sodium, potassium, and ammonium compounds rather seem to be caused by the environment than by a significant amount of negative charge on the central carbon atom. In the potassium compound the largest deviation from a least squares plane through the anion is 0.015 Å. It is more reasonable to assume the explanation that the appreciable deviation from planarity found in the ammonium compound is due to the surroundings, than to explain the planarity found in the sodium and potassium compounds as a deviation from a stable pyramidal configuration. The normal of the least squares plane through the anion in $KC(CN)_3$ makes an angle of 25.6° with the shortest axis (3.89 Å) corresponding to an interanionic distance of 3.51 Å. The consequence is thus a less dense packing than in the sodium structure in which this distance is 3.45 Å, as the two compounds have approximately the same density. The corresponding angle for the anion in $NH_4C(CN)_3$ is 35.7° giving an interanionic distance of 3.14 Å. The parallel displacement of one anion with respect to the other makes this short interanionic distance possible. The environment of the anion in the ammonium compound is highly asymmetric. The shortest approach of atoms in adjacent anions is to be found between a cyano group in one anion and atoms in the equivalent anion one period along the *b* axis. C′(2) is 3.39 Å, 3.45 Å, and 3.46 Å away from C(0), C(1), and C(3), respectively, and N′(2) is 3.39 Å and 3.42 Å away from C(0) and C(2). If the anion in the ammonium compound had been situated in the least squares plane these distances would have been even shorter.

Molecular orbital calculations by Lofthus[19] on π-electron densities in the $C(CN)_3^-$ ion and pyridinium dicyanomethylide show that the charge densities on the central carbon atoms are approximately the same. The results are in close agreement with Enemark and Holm's[20] calculations on the tricyanomethanide anion. They have included σ-charge densities and find contrary to what Desiderato and Sass have concluded, a negligible net charge on the central carbon atom. The non-planar arrangement of the bonds to the central carbon atom in pyridinium dicyanomethylide is probably caused either by packing effects or by too close *ortho*-hydrogen cyanocarbon contacts. In potassium *p*-nitrophenyldicyanomethide[4] no significant deviation from planarity of the anion is observed. Other structures of this type which have been determined are N-methylphenazonium and caesium tetracyanoquinodimethanide,[18,21] where the distortions from planarity of the dicyanomethylene groups are by the authors ascribed primarily to the very rigid and close packing, and tetraphenylarsonium 1,1,4,5,5,-pentacyano-3-fluoro-2-azapentadienide,[22] where the anion is said to be definitely non-planar.

It seems that a deviation from planarity of the central carbon arrangement in this type of carbanions may well be explained in terms of a packing effect. On the four structure determinations of the $C(CN)_3^-$ ion there is an appreciable deviation from planarity in the ammonium compound only, while the deviation is negligible in the sodium, potassium, and copper(II) tricyanomethanide. The conclusion from spectroscopic investigations[23,24] that there is a planar trigonal equilibrium configuration of the ion, is therefore strongly supported.

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