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The Crystal Structure of Potassium *p*-Nitrophenyldicyanomethide

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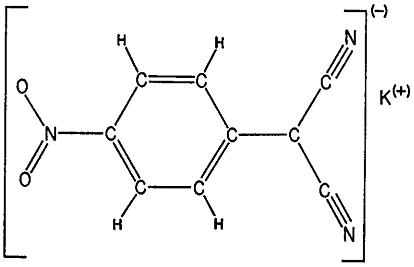
The crystal structure of potassium paranitrophenyldicyanomethide has been solved from three-dimensional X-ray diffraction data. The crystals are orthorhombic, space group $P2_12_12_1$ (D_2^4), with $a=21\cdot749 \pm 0\cdot001$, $b=11\cdot416 \pm 0\cdot001$, $c=3\cdot771 \pm 0\cdot001$ Å. With four molecules per unit cell the calculated density is 1·598 g.cm^{-3}; the observed density is 1·61 g.cm^{-3}. The final conventional R value is 5·8%. The carbanion is considerably distorted from C_{2v} symmetry within the phenyl ring; in addition the cyano groups and the nitro groups are significantly displaced from the ring plane.

Introduction

We have determined the crystal structure of potassium *p*-nitrophenyldicyanomethide (I) $KC_9H_4N_3O_2$, as part of a program of research on the structures of carbanions.



(I) Potassium *p*-nitrophenyldicyanomethide.

Experimental

A sample of $KC_9H_4N_3O_2$ was kindly given to us by Dr H.D.Hartzler of E.I. du Pont de Nemours and Company. The sample consisted of dark red crystals which were too small to use for diffraction study. Study needles were grown from solution by slow evaporation of acetonitrile from a mixture of acetonitrile and chlorobenzene. The needles are easily cleaved along planes perpendicular to the needle axis. Several crystals were cleaved and the segments were mounted along directions both parallel and perpendicular to the needle axis.

Oscillation, Weissenberg and precession photographs showed the Laue symmet ryto be *mmm* (D_{2h}). The needle axis was chosen as the c axis. The space group is $P2_12_12_1$ as derived from the systematic absences:

$h00$ absent if $h=2n+1$
$0k0$ absent if $k=2n+1$
$00l$ absent if $l=2n+1$

Accurate values for the unit-cell parameters were obtained from two Straumanis-type Weissenberg photographs taken about the b and c axes. The positions of 51 $hk0$ and 43 $h0l$ reflections were measured with a traveling microscope and a calibrated steel scale, and the Bragg angles were included in a least-squares determination of the unit-cell constants and the eccentricity and absorption factor. The final cell parameters and their estimated standard deviations are (Cu $K\alpha$ = 1·54051 Å):

$$a=21\cdot749 \pm 0\cdot001 \text{ Å}$$
$$b=11\cdot416 \pm 0\cdot001$$
$$c=\ \ 3\cdot771 \pm 0\cdot001$$

The absorption and eccentricity corrections were small. The density calculated on the basis of four $KC_9H_4N_3O_2$ molecules per unit cell is 1·598 g.cm^{-3}; the density measured by flotation is 1·61 g.cm^{-3}.

Two sets of intensity data were collected. The first set was collected visually, using a needle shaped crystal approximately 0·04 mm in diameter and 2 mm in length mounted along the c axis. Multiple film equi-inclination Weissenberg photographs of the $l=0$, 1, 2 and 3 layers were taken with filtered Cu $K\alpha$ radiation; the intensities were estimated visually by comparison with a standard intensity strip and were corrected for Lorentz–polarization effects. The different layers were correlated by comparison with a composite photograph which contained carefully timed exposures of a 40° segment of each layer. These visually estimated intensities were subsequently used in the determination and initial refinement of the trial structure.

The second set of data was collected on a Datex-automated General Electric XRD-5 diffractometer

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using a crystal 0·09 mm long and 0·05 × 0·05 mm in cross section. All data were collected with nickel-filtered copper radiation and a xenon-filled proportional counter. The intensities were measured using a θ-2θ scan at a scanning speed of 1° per minute. Background was counted for 100 seconds at each end of the scan. The scan range was adjusted to account for α_1–α_2 splitting. The 550 reflections was monitored every fifteen reflections. Intensity measurements were made for each of the 1219 reflections in the region between $\theta = 3{\cdot}5°$ and $\theta = 78{\cdot}2°$; this covered approximately 92% of the copper sphere. Intensities which were less than the standard deviation of the background were measured at least six times; multiple intensity measurements were also made for several strong and medium intensity reflections. The individual reflections were assigned intensity values, I, according to the equation

$$I = \left[S - \left(\frac{B_1+B_2}{2t}\right)\left(\frac{r}{m} - k\right)\right] g(\mathrm{Lp})^{-1} \qquad (1)$$

where S is the total scan count; B_1 and B_2 are the background counts; t is the time spent counting the background – in this case, $t = 100$ seconds; r is the 2θ scan range; m is the 2θ scanning speed – in this case, $m = 1°$ per minute; k is a time lag constant which was found experimentally to be 0·05 second; Lp is the Lorentz–polarization correction factor; g is a scale factor.

Standard deviations in intensity measurements

The integrated intensity values derived from the diffractometer measurements were assigned standard deviations, $\sigma(I)$, according to the equation

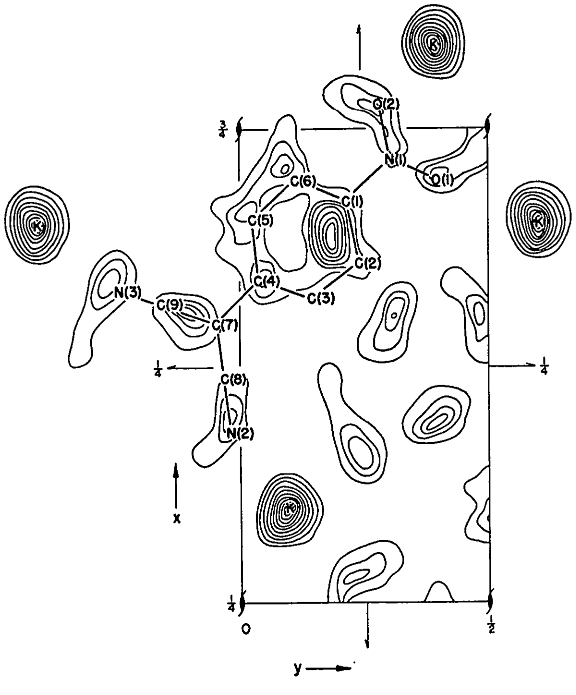

Fig. 1. Superposition projection on to (001).

$$\sigma^2(I) = \frac{g^2}{\mathrm{Lp}^2}\left\{\frac{1}{n}\left[S + (B_1+B_2)\left(\frac{r}{2mt} - \frac{k}{2t}\right)^2\right] + (dS)^2\right\}. \qquad (2)$$

In equation (2), d is an arbitrary constant, n is equal to the number of independent measurements of the reflection intensity, and the other terms are as previously defined. The factor $(dS)^2$ is introduced to account for absorption, extinction and instrumental errors which might be expected to be proportional to the reflection intensity (Peterson & Levy, 1957); standard deviations were calculated assuming d values of 0·00, 0·01, 0·02 and 0·03. For reflections measured more than once, the S in expression (2) was replaced by the average scan count and B_1 and B_2 were replaced by the respective average background counts.

The trial structure

The photographic intensity data were used for the determination and initial refinement of the trial structure. The calculations were carried out on the Rice computer. All reflections were given equal weights during the initial least-squares refinements of the trial structure; the quantity minimized was $\Sigma\,|F_o - 1/kF_c|^2$.

Trial x and y potassium ion coordinates were determined from an unsharpened [001] Patterson map. Trial x and y coordinates for the remaining heavy atoms were then readily obtained from a fourfold sum function superposition of unsharpened Patterson maps translated to the trial potassium positions. The Patterson maps were translated by the method described by Lipson & Taylor (1958). This method involves the convolution of the Patterson function with a peak function which is zero everywhere except at the potassium positions. The peak function can be effectively represented by the electron density distribution of point scatterers positioned at the trial potassium coordinates. The transforms, $S(h,k,l)$ of the superposition are then given by the relationship:

$$S(h,k,l) = F(h,k,l)^2 \,.\, G(h,k,l)\,;$$

$F(h,k,l)$ is the observed structure factor amplitude and

$$G(h,k,l) = \Sigma_j \exp[2\pi i(hxj + kyj + lzj)]\,,$$

where the summation includes the trial potassium coordinates. Fig. 1 shows the [001] superposition projection.

The x and y coordinates and isotropic temperature factors for the heavy atoms were refined by full-matrix least-squares to an R index of 13%. Trial z coordinates were then assigned to the potassium ions by considering the Harker peaks in a sharpened [010] Patterson map. The z coordinates of the other heavy atoms were determined from packing considerations; the anion was positioned in a manner which would allow the cyano nitrogen atoms and the nitro oxygen atoms to assume reasonable contacts with the potassium ions.

The heavy atom positional parameters and isotropic temperature factors were refined by full-matrix least squares. The scale factors for the individual layers were empirically adjusted during the refinement. In this manner the trial structure was refined until the R index was 14%.

The three-dimensional refinement with the diffractometer data

Using the diffractometer data, the three-dimensional refinement of the structure was carried out mainly by full-matrix least squares. All calculations were performed on an IBM 7094 computer using the CRYRM Crystallographic Computing System (Duchamp, 1965). The quantity minimized was $\Sigma w\left(F_o^2-\frac{1}{k}F_c^2\right)^2$. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1962).

During the refinement, most reflections were assigned weights equal to $1/\sigma^2(I)$ where $\sigma^2(I)$ was calculated according to equation (2) (see *Experimental*). Reflections which were measured in 2θ regions where the background count was unusually high were assigned zero weights. Intensity measurements were hindered by background radiation in two manners: (1) reflections occurring at very low values of 2θ were measured in regions where white radiation and air scattering created a high and inconsistent background; and (2) a number of $hk4$ reflections were measured at crystal settings which gave rise to large amounts of diffuse scattering from the brass pin to which the crystal was mounted. In the later stage of the refinement, the 031 reflection was assigned zero weight because it appeared to be suffering from secondary extinction. A total of seventy-two reflections were assigned zero weights.

Several independent least-squares refinements were performed with different weighting schemes. The weighting schemes were varied by successively assigning values of 0·00, 0·01, 0·02 and 0·03 to the parameter d in equation (2).

The first refinement was carried out using standard deviations calculated with $d=0{\cdot}0$. Heavy atom positional parameters and anisotropic temperature factors were refined to an R index of about 7%. The hydrogen atom coordinates were then determined from a three-dimensional difference Fourier map, and additional least-squares refinements of all coordinates, heavy atom anisotropic temperature factors and hydrogen atom isotropic temperature factors, reduced the conventional R index ($\Sigma|F_o-F_c|/\Sigma F_o$) to 5·8%; the final R index based on $F^2(\Sigma|F_o^2-F_c^2|/\Sigma F_o^2)$ was 5%. In the final least-squares cycle no parameter shift exceeded one-tenth the value of its estimated standard deviation. The 'goodness of fit', $[\Sigma w(F_o^2-F_c^2)^2/(m-s)]^{\frac{1}{2}}$ is 1·9; this deviation from unity suggests that the standard deviations are not adequately represented by equation (2) calculated with $d=0{\cdot}0$. Table 1 lists the average residuals $|(\overline{w(F_o^2-F_c^2)})|$ for the reflections within several structure factor ranges. There appears to be a definite trend of increasing residuals corresponding to increasing structure factor amplitudes. The introduction of d values greater than 0·0 decreases the weights of the larger structure factors relative to those of the smaller structure factors. Since this effect tends to equalize the residuals, we felt that it would be worthwhile to refine the structure with weighting functions determined from d values greater than 0·0.

Table 1. *Reflection statistics*

The average residuals are from the least-squares refinement using weights equal to $1/\sigma(I)$; $\sigma(I)$ was calculated by equation (2) (see text) assuming that $d=0{\cdot}00$.

F_o range	Number of non-zero weight reflections in the range	Average residual for the range = $\overline{\lvert w(F_o{}^2-F_c{}^2)\rvert}$
0–5	281	1·08
5–10	372	1·28
10–15	234	1·47
15–20	105	1·93
20–25	57	1·71
25–30	34	1·88
30–35	28	2·18
35–40	16	2·31
40–55	17	2·06
55–80	13	3·10

Full-matrix least-squares refinements were then carried out using sets of standard deviations calculated with the d value in equation (2) equal to 0·01, 0·02 and 0·03. The heavy atom coordinates and anisotropic temperature factors and the hydrogen coordinates were refined; the hydrogen atom temperature factors were held stationary. In the final refinements, no parameter shift exceeded one-tenth of its estimated standard deviation. Table 2 describes the results of these additional refinements using the different weighting schemes. The conventional R index decreased as the d value increased. The 'goodness of fit' decreased to a value of 0·9 corresponding to the d value of 0·03. Table 2 also lists the maximum and root-mean-square parameter shifts resulting from changes in the weighting schemes; these shifts represent the changes produced in the structure by changing from $d=0{\cdot}0$ to $d=0{\cdot}01$, 0·02 and 0·03. In all cases, the maximum heavy atom shift was in the z coordinate of the atom N(2). Although the shifts displayed by atom N(2) are significantly larger than the estimated standard deviation of its coordinates, the root-mean-square shifts do not appear to be significant.

We feel that, in spite of its failure to represent completely the errors in the structure factors, a weighting function calculated with $d=0{\cdot}00$ is reasonably acceptable. Considering the small size of the crystal used for the data collection, errors due to absorption are probably negligible. Therefore, we have chosen to report the results for the refinement based on standard deviations derived from equation (II) with the arbitrary $(dS)^2$ term omitted. The final heavy-atom parameters and their estimated standard deviations are listed in Table 3. The hydrogen atom parameters and their estimated

Table 2. *Results of least-squares refinements using different weighting schemes*

The weights were equal to $1/\sigma(I)$, with $\sigma(I)$ calculated by equation (2) (see text) assuming different values of the constant d.

			Heavy atoms (excluding K⁺)			Hydrogen atoms		
d	$R=\frac{\Sigma\lvert F_o-F_c\rvert}{\Sigma F_o}$	Goodness of fit	R.M.S. shift	Maximum shift	(Shift parameter)	R.M.S. shift	Maximum shift	(Shift parameter)
0·00	5·78	1·91						
0·01	5·52	1·55	0·003 Å	0·009 Å	$(Z_{N(2)})$	0·04 Å	0·06 Å	$(Z_{H(4)})$
0·02	5·44	1·20	0·004	0·012	$(Z_{N(2)})$	0·05	0·10	$(Z_{H(1)})$
0·03	5·42	0·94	0·004	0·012	$(Z_{N(2)})$	0·06	0·11	$(Z_{H(2)})$

Table 3. *The final heavy atom parameters and their estimated standard deviations*

The values have been multiplied by 10^4.

The temperature factors are in the form $T=\exp(-\beta_{11}h^2-\beta_{22}k^2-\beta_{33}l^2-\beta_{12}hk-\beta_{13}hl-\beta_{23}kl)$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	7083 (1)	3962 (2)	−2488 (8)	22 (1)	53 (2)	1084 (33)	−11 (3)	−1 (9)	68 (20)
O(2)	7796 (1)	2871 (2)	−0216 (10)	12 (1)	98 (3)	1042 (34)	−20 (2)	−22 (10)	64 (26)
N(1)	7238 (1)	3039 (3)	−0987 (11)	16 (1)	61 (3)	652 (38)	−8 (3)	15 (10)	−39 (21)
N(2)	4362 (1)	−0168 (3)	0497 (10)	14 (1)	60 (3)	784 (38)	0 (2)	−25 (11)	47 (23)
N(3)	5843 (1)	−2390 (2)	4830 (13)	18 (1)	54 (3)	76 (37)	8 (3)	26 (12)	53 (25)
C(1)	6803 (2)	2177 (3)	−0117 (12)	11 (1)	50 (3)	369 (35)	−11 (2)	21 (10)	42 (24)
C(2)	6183 (2)	2370 (3)	−0983 (12)	14 (1)	44 (3)	501 (44)	7 (3)	6 (10)	66 (20)
C(3)	5765 (6)	1508 (3)	−0197 (13)	11 (1)	50 (3)	534 (38)	9 (3)	19 (11)	30 (24)
C(4)	5935 (2)	0441 (3)	1340 (10)	11 (1)	42 (3)	493 (39)	5 (3)	−1 (9)	−4 (18)
C(5)	6569 (2)	0281 (3)	2132 (12)	15 (1)	49 (3)	543 (39)	8 (3)	−26 (10)	55 (20)
C(6)	6990 (2)	1143 (3)	1458 (10)	10 (1)	59 (3)	573 (41)	1 (3)	−23 (9)	43 (22)
C(7)	5500 (2)	−0459 (3)	2083 (10)	12 (1)	44 (3)	453 (36)	1 (3)	−10 (10)	−3 (19)
C(8)	4868 (2)	−0314 (3)	1235 (11)	17 (1)	37 (3)	443 (41)	−3 (3)	0 (10)	−27(18)
C(9)	5677 (2)	1538 (3)	3612 (10)	13 (1)	43 (3)	432 (40)	−6 (3)	26 (9)	−28 (19)
K	3563 (0)	0995 (1)	5412 (2)	16 (0)	59 (1)	505 (7)	9 (1)	9 (3)	0 (5)

Table 4. *The final hydrogen parameters and their estimated standard deviations*

The positional parameters have been multiplied by 10^3.

	x	y	z	B
H(1)	602 (1)	312 (2)	−195 (8)	−0·3 (0·7) Å²
H(2)	535 (1)	169 (2)	−057 (10)	0·5 (0·7)
H(3)	670 (2)	042 (3)	316 (10)	2·6 (1·1)
H(4)	744 (1)	111 (3)	231 (9)	1·6 (0·8)

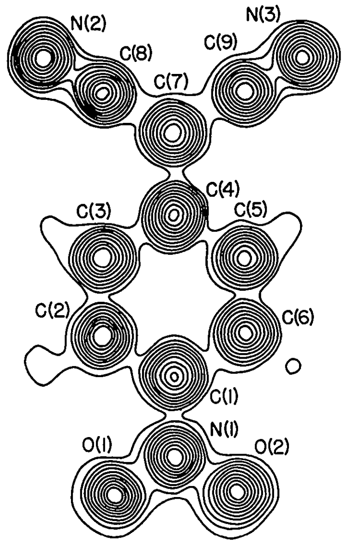

Fig. 2. The composite of the electron density map in the least-squares plane of the benzene ring. Contours are at intervals of 1 e.Å^{-3}, beginning with the 1 e.Å^{-3} contour.

standard deviations are listed in Table 4. Observed structure factors and their estimated standard deviations (calculated from equation (2) assuming $d=0{\cdot}0$) are given in Table 5 along with the calculated structure factors and phase angles.

At the conclusion of the refinement, an electron density map and a difference map, each in the plane of the anion, were calculated. These are shown in Figs. 2 and 3.

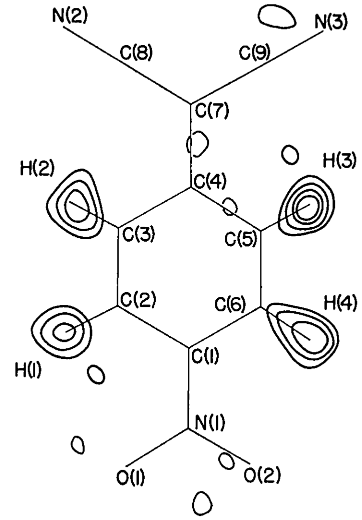

Fig. 3. The composite of the final difference map in the least-squares plane of the benzene ring. Contours are at intervals of 0·1 e.Å^{-3}, beginning with the 0·2 e.Å^{-3} contour.

Table 5. *Observed and calculated structure factors and phase angles*

Reading from left to right, the columns contain values of h, $10F_o$, $10\sigma(F_o)$, $10F_c$ and 10 times the phase angle (°). Values of $10F_o$ reported as --- represent intensity values observed negative and where arbitrarily set equal to zero.

H 0 0				
2	302	***	289	180
4	489	***	472	180
6	283	1	279	180
8	361	1	357	180
10	113	3	102	180
12	67	5	79	180
14	667	2	672	0
16	149	4	147	180
18	57	4	61	180
20	38	14	36	0
22	51	10	56	0
24	113	4	118	180
26	20	6	1	180
H 1 0				
27	106	2	106	270
26	70	4	76	180
25	42	9	92	270
24	78	5	74	0
23	130	4	125	90
22	63	8	59	0
21	10	22	30	90
20	84	6	74	180
19	26	9	26	90
18	119	5	112	180
17	111	5	115	90
16	160	4	165	0
15	280	3	277	90
14	122	4	122	0
13	343	2	339	270
12	451	2	442	180
11	93	4	84	90
10	---	***	9	180
9	406	1	404	90
8	254	2	261	0
7	27	4	19	270
6	303	1	305	180
5	266	***	238	270
4	384	***	390	0
3	313	***	320	270
2	33	***	49	180
1	208	***	193	90
H 2 0				
0	83	***	57	0
1	301	***	302	270
2	586	***	611	0
3	795	***	822	90
4	110	3	113	180
5	241	2	245	90
6	471	1	469	0
7	627	1	630	270
8	38	8	35	180
9	124	3	129	90
10	386	2	381	180
11	102	4	109	90
12	76	5	67	0
13	164	3	168	270
14	91	5	84	0
15	67	3	71	270
16	36	6	22	0
17	263	3	261	90
18	65	9	68	180
19	106	6	113	90
20	292	3	301	0
21	182	4	182	270
22	54	9	59	180
23	43	4	38	90
24	44	9	57	180
25	39	9	26	90
26	68	4	66	180
27	67	3	72	270
H 3 0				
26	131	2	129	180
25	52	6	52	270
24	34	11	45	0
23	103	5	105	270
22	101	5	108	0
21	77	7	65	90
20	132	4	133	180
19	124	5	135	90
18	56	10	62	0
17	347	3	344	90
16	94	6	92	180
15	159	4	165	270
14	168	3	163	180
13	127	4	118	90
12	292	2	297	180
11	115	4	123	270
10	173	3	179	0
9	348	2	344	270
8	788	1	787	0
7	204	2	206	90
6	703	1	708	180
5	358	1	357	90
4	143	2	145	180
3	546	1	549	90
2	232	2	236	0
1	334	1	336	270
H 4 0				
0	96	3	83	0
1	---	***	3	270
2	363	1	362	0
3	402	1	402	90
4	114	3	126	0
5	362	2	359	90
6	243	2	250	180
7	305	2	311	270
8	31	5	48	180
9	7	23	19	90
10	163	3	164	0
11	89	5	83	90
12	191	3	190	0
13	238	3	235	270
14	283	3	281	180
15	36	14	32	90
16	---	***	6	180
17	217	3	218	90
18	261	3	271	0
19	35	14	1	90
20	128	***	141	180
21	119	5	118	270
22	25	7	31	0
23	55	7	71	90
24	49	7	54	0
25	29	4	24	90
26	16	6	19	180
H 5 0				
25	---	***	11	90
24	7	13	3	180
23	35	11	45	270
22	72	6	63	180
21	---	***	2	90
20	87	6	84	180
19	253	3	252	90
18	104	5	118	180
17	69	8	75	270
16	50	11	48	0
15	102	6	97	270
14	33	7	23	180
13	73	7	60	90
12	108	5	102	180
11	194	3	199	90
10	44	9	10	180
9	245	2	249	270
8	157	3	160	180
7	106	4	97	270
6	252	2	242	0
5	750	1	750	90
4	35	9	5	0
3	366	2	363	270
2	118	3	114	180
1	301	2	302	270
H 6 0				
0	332	2	328	180
1	244	2	242	90
2	322	2	314	0
3	187	2	175	90
4	328	2	331	0
5	43	3	52	270
6	247	2	248	180
7	136	3	141	90
8	211	3	216	180
9	110	4	111	270
10	16	12	11	180
11	182	3	178	270
12	162	4	165	0
13	90	6	83	90
14	64	9	53	180
15	190	4	193	90
16	81	7	90	0
17	92	10	90	270
18	146	4	139	0
19	48	10	45	270
20	124	4	126	180
21	109	4	111	90
22	77	5	82	180
23	54	6	57	270
24	13	8	9	180
25	24	***	2	90
H 7 0				
24	78	3	77	180
23	43	6	28	90
22	86	4	81	180
21	---	***	7	270
20	99	5	104	0
19	27	7	6	90
18	51	4	52	0
17	---	***	15	90
16	210	3	208	180
15	231	3	231	270
14	301	3	307	180
13	170	4	171	90
12	228	3	213	0
11	53	10	66	90
10	---	***	14	0
9	57	9	60	270
8	201	3	203	180
7	49	4	44	90
6	325	2	322	0
5	36	5	22	270
4	89	5	90	180
3	150	3	156	90
2	370	2	365	180
1	187	3	195	270
H 8 0				
0	195	3	193	0
1	272	3	268	90
2	12	17	13	0
3	124	4	115	270
4	172	3	172	180
5	81	6	74	90
6	---	***	23	0
7	228	3	232	90
8	61	9	29	180
9	118	5	113	270
10	29	8	11	0
11	222	3	223	270
12	35	6	26	180
13	23	10	17	90
14	130	4	113	0
15	171	4	172	90
16	86	6	91	0
17	61	8	72	90
18	109	4	103	180
19	---	***	8	270
20	37	4	1	180
21	57	2	65	90
22	95	3	87	180
H 9 0				
21	---	***	8	90
20	39	7	24	180
19	53	6	54	270
18	---	***	13	0
17	34	12	28	270
16	106	4	101	180
15	158	4	169	90
14	69	3	77	180
13	219	3	218	270
12	66	8	73	0
11	120	5	128	270
10	44	12	43	180
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9	27	7	25	240
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13	65	8	57	126
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8	177	4	167	145
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9	92	6	88	322
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12	27	9	12	187
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23	72	3	70	319
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9	141	4	146	347
10	148	4	158	41
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12	45	11	34	326
13	173	4	181	151
14	172	4	148	228
15	184	4	192	125
16	168	4	164	327
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25	117	2	129	349
H 4 1				
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9	137	3	140	56
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11	126	4	121	354
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17	243	3	238	347
16	196	4	166	47
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14	152	4	148	83
13	136	4	131	188
12	185	3	180	302
11	240	2	242	179
10	330	2	323	176
9	113	3	114	7
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7	120	3	124	342
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11	337	2	324	191
12	161	3	155	23
13	170	3	134	44
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16	46	5	39	219
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13	163	4	157	0
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7	192	1	198	208
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6	199	3	199	176
7	140	4	141	268
8	207	3	196	167
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7	97	5	96	112
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2	39	12	46	27
1	85	6	97	179
0	59	8	51	180
H 7 3				
0	91	6	84	90
1	127	4	139	232
2	32	7	33	330
3	82	6	86	125
4	12	19	14	250
5	168	4	182	42
6	111	2	114	95
7	34	14	36	305
8	77	6	72	78
9	93	5	71	309
10	67	7	62	273
11	55	7	55	66
12	95	4	92	237
13	109	4	108	126
14	73	2	76	35
15	99	3	103	244
16	96	4	46	34
H 6 3				
18	53	4	52	65
17	10	11	17	315
16	57	6	43	9
15	127	3	138	202
14	60	6	62	189
13	20	9	29	238
12	85	5	84	52
11	71	7	66	84
10	78	6	72	310
9	113	5	114	161
8	128	4	132	269
7	152	4	147	305
6	110	5	112	54
5	116	5	116	44
4	121	5	131	83
3	51	11	59	137
2	78	7	61	321
1	100	6	103	255
0	106	5	93	180
H 5 3				
0	63	9	61	90
1	77	7	88	190
2	163	4	164	6
3	201	3	194	19
4	67	8	78	101
5	139	4	130	116
6	119	5	123	167
7	29	8	50	121
8	161	4	163	344
9	53	10	45	318
10	4	63	10	354
11	95	6	96	335
12	98	5	78	208
13	38	5	33	171
14	23	8	13	196
15	63	6	68	165
16	95	4	92	36
17	53	6	47	26
18	17	7	20	124
19	---	***	24	345
H 4 3				
20	22	9	13	56
19	40	6	44	101
18	40	3	37	108
17	74	3	63	107
16	91	5	89	233
15	44	4	44	271
14	114	4	119	338
13	182	3	184	213
12	185	3	195	93
11	52	10	62	154
10	43	12	57	160
9	125	5	126	18
8	92	6	92	351
7	83	7	86	274
6	139	4	135	43
5	61	9	50	106
4	115	5	114	274
3	38	6	23	332
2	235	3	239	243
1	73	8	71	285
0	109	5	100	0
H 3 3				
0	267	3	271	270
1	261	3	266	66
2	147	4	150	54
3	85	7	87	65
4	159	4	162	86
5	72	8	69	278
6	85	7	83	300
7	102	6	96	188
8	91	6	78	14
9	84	7	81	193
10	45	5	42	61
11	68	8	71	38
12	114	5	110	198
13	133	4	126	300
14	86	6	88	274
15	67	7	73	121
16	57	8	62	96
17	35	11	38	19
18	88	4	91	81
19	30	10	45	257
20	54	4	47	304
H 2 3				
21	48	4	63	131
20	27	10	15	22
19	59	6	65	157
18	73	5	76	189
17	65	6	70	286
16	60	7	70	81
15	44	10	54	2
14	144	4	136	23
13	65	8	70	147
12	43	12	28	297
11	115	5	115	211
10	167	4	180	125
9	174	4	175	317
8	42	13	63	331
7	78	7	61	85
6	109	6	116	328
5	131	5	132	183
4	155	4	151	196
3	107	6	100	339
2	28	9	36	260
1	73	8	88	334
0	188	4	196	0
H 1 3				
0	17	15	22	270
1	129	5	130	353
2	56	10	54	167
3	143	4	128	176
4	119	5	119	50
5	120	5	103	283
6	178	4	173	341
7	190	4	193	19
8	105	6	87	269
9	57	10	67	85
10	95	6	93	114
11	59	9	54	191
12	96	6	86	55
13	44	12	42	305
14	62	8	71	236
15	52	9	38	89
16	51	9	63	184
17	117	4	114	169
18	60	6	49	34
19	120	3	125	292
20	118	3	126	318
21	111	2	115	21
H 0 3				
21	55	4	64	90
20	44	3	44	270
19	15	10	10	270
18	98	4	96	90
17	32	13	10	270
16	116	4	118	90
15	12	10	23	270
14	22	10	15	90
13	38	14	38	90
12	86	6	77	270
11	202	3	204	270
10	28	9	30	270
9	70	8	66	270
8	219	3	218	90
7	236	1	237	90
6	149	4	131	270
5	67	9	63	270
4	63	9	56	270
3	216	3	210	270
2	71	8	72	90
1	50	12	69	90
H 0 4				
0	60	9	57	0
1	201	3	192	180
2	58	9	60	180
3	87	6	93	180
4	76	7	74	180
5	86	6	79	0
6	74	6	65	0
7	43	10	27	180
8	38	13	40	180
9	74	6	80	180
10	98	***	113	180
11	51	***	61	0
12	---	***	16	0
13	50	***	84	0
14	42	***	31	0
15	60	***	56	180
H 1 4				
14	56	***	83	81
13	---	***	63	304
12	54	***	76	209
11	48	***	51	190
10	110	***	123	331
9	40	10	29	236
8	69	6	67	106
7	86	5	78	332
6	67	7	57	146
5	49	9	48	252
4	67	7	69	249
3	52	9	36	141
2	54	9	40	331
1	59	4	61	79
0	71	7	50	90
H 2 4				
0	38	12	17	180
1	62	8	73	255
2	135	2	135	98
3	78	6	67	148
4	65	7	67	237
5	24	18	22	54
6	77	6	77	267
7	63	7	67	227
8	57	7	70	68
9	42	***	53	126
10	91	***	96	175
11	111	***	72	33
12	74	***	96	223
13	54	***	105	288
14	---	***	31	0
H 3 4				
13	---	***	47	68
12	---	***	25	45
11	99	***	103	185
10	---	***	54	106
9	53	***	64	254
8	81	***	89	339
7	110	4	116	342
6	28	15	28	242
5	20	20	25	156
4	44	10	48	155
3	82	6	84	188
2	59	8	55	21
1	61	7	69	349
0	46	10	46	270
H 4 4				
0	17	24	31	180
1	20	21	35	42
2	54	8	46	118
3	35	11	10	202
4	76	6	84	296
5	75	5	84	178
6	65	***	72	257
7	131	***	146	269
8	103	***	108	163
9	---	***	55	354
10	10	***	36	76
11	---	***	28	96
12	31	***	38	282
H 5 4				
11	---	***	15	175
10	72	***	83	95
9	---	***	35	215
8	62	***	79	230
7	26	***	78	226
6	78	***	86	274
5	50	***	62	76
4	148	***	168	44
3	41	***	37	22
2	46	9	73	11
1	101	4	103	281
0	77	5	85	270
H 6 4				
0	32	***	67	180
1	25	***	66	31
2	---	***	55	277
3	---	***	41	26
4	32	***	25	280
5	148	***	159	242
6	---	***	32	203
7	---	***	15	193
8	---	***	22	282
9	---	***	68	334
H 7 4				
7	49	***	77	147
6	30	***	63	343
5	---	***	26	58
4	---	***	27	32
3	68	***	80	346
2	60	***	73	149
1	---	***	49	173
0	---	***	14	270

Table 6. *The shorter van der Waal contacts*

K–O(1)(*i*)	2·815 Å	N(3)–C(9)(*d*)	3·470 Å
K–O(2)(*g*)	2·875	N(3)–O(2)(*a*)	3·009
K–O(2)(*h*)	2·783	C(2)–N(2)(*c*)	3·288
K–N(2)(*d*)	2·907	C(4)–C(3)(*d*)	3·436
K–N(2)	2·865	C(6)–C(1)(*d*)	3·414
K–N(3)(*f*)	2·879	C(9)–C(7)(*d*)	3·445
K–N(3)(*b*)	2·995	H(1)–N(2)(*c*)	2·38
O(1)–N(2)(*c*)	3·480	H(2)–N(3)(*b*)	2·84
N(1)–O(1)(*d*)	3·391	H(3)–O(2)(*a*)	2·98
N(2)–N(3)(*b*)	3·203	H(3)–H(4)(*a*)	2·50

(*a*) $-x+\frac{3}{2},\ -y,\ z+\frac{1}{2}$
(*b*) $-x+1,\ y+\frac{1}{2},\ -z+\frac{1}{2}$
(*c*) $-x+1,\ y+\frac{1}{2},\ -z-\frac{1}{2}$
(*d*) $x,\ y,\ z+1$
(*e*) $x,\ y,\ z-1$
(*f*) $-x+1,\ y+\frac{1}{2},\ -z+\frac{3}{2}$
(*g*) $x-\frac{1}{2},\ -y+\frac{1}{2},\ -z$
(*h*) $x-\frac{1}{2},\ -y+\frac{1}{2},\ -z+1$
(*i*) $-x+1,\ y-\frac{1}{2},\ -z+\frac{1}{2}$

The crystal packing

Fig. 4 shows the (001) projection of the crystal structure. The crystal packing permits close contacts between the potassium ions and the nitro oxygen atoms and cyano nitrogen atoms. The shortest van der Waals contacts are listed in Table 6. The 2·38 Å distance between H(1) and N(2) from a symmetry related molecule is unusually short, but all other contacts are compatible with the accepted van der Waals radii. Molecules related by the c translation exhibit an interplanar spacing of 3·42; the dihedral angle between the molecular plane and the (001) plane is approximately 25°.

Structure of the carbanion

The bond distances and angles for the anion are shown in Fig. 5. The unsymmetrical appearance of the anion

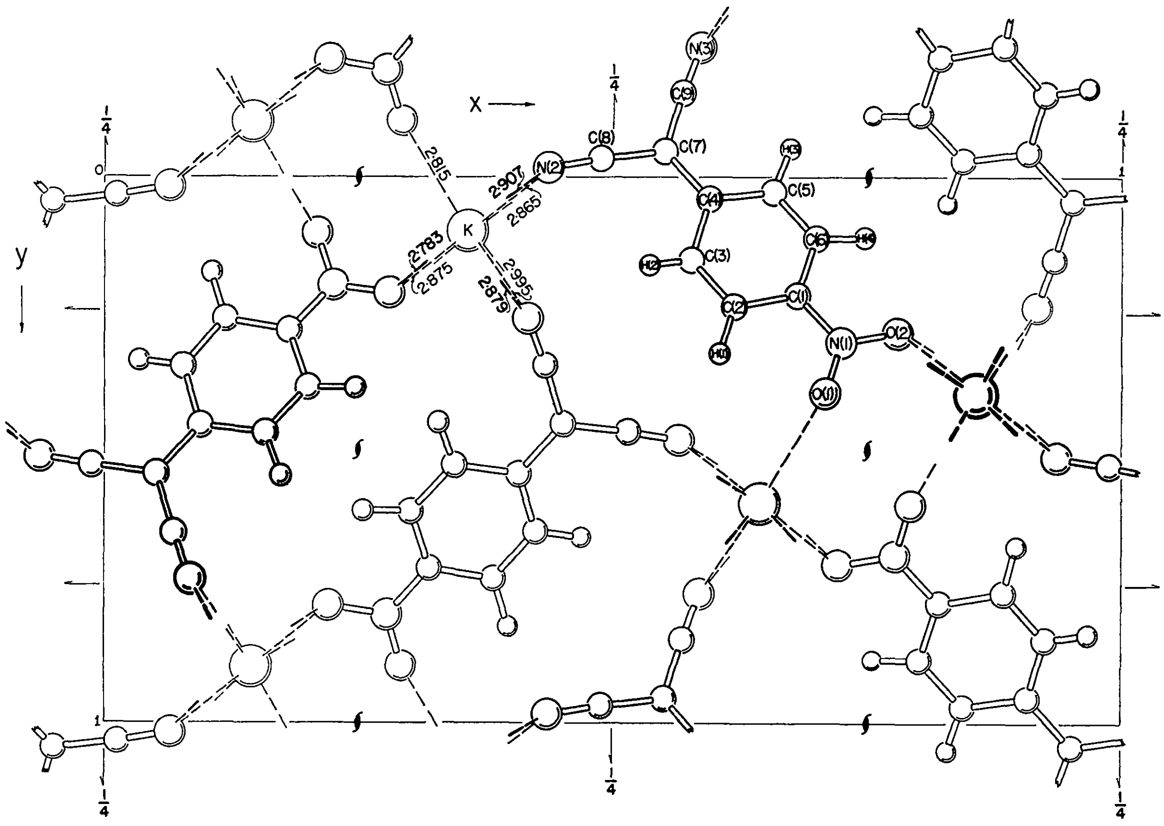

Fig. 4. The crystal structure projected on (001).

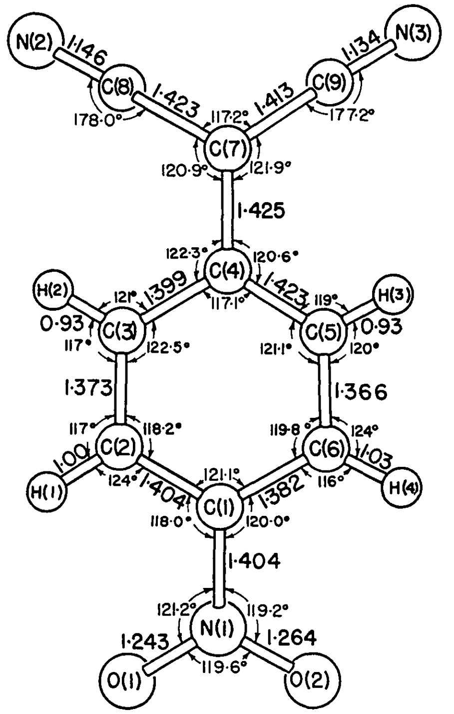

Fig. 5. Bond lengths and angles in the anion.

Table 7. *Deviations from the least-squares planes through the anion*

	Plane *A* Deviation	Plane *B* Deviation
O(1)	−0·106 Å*	−0·129 Å*
O(2)	−0·054*	−0·076*
N(1)	−0·058*	−0·076*
N(2)	−0·040*	−0·013
N(3)	−0·043*	−0·014
C(1)	−0·004	−0·012
C(2)	−0·009	−0·014
C(3)	0·011	0·016
C(4)	0·003	0·012
C(5)	−0·007	−0·002
C(6)	0·010	0·006
C(7)	−0·004	0·015
C(8)	−0·020*	0·004
C(9)	−0·023*	0·002
H(1)	0·05*	0·04*
H(2)	0·10*	0·11*
H(3)	−0·01*	0·00*
H(4)	0·14*	0·13*

* Atoms excluded from the calculation of the least-squares plane. The equations of the least squares planes, with the coefficients of *X*, *Y*, and *Z* direction cosines with respect to the crystallographic axes, are:

Plane *A*: $-0{\cdot}1516X+0{\cdot}3928Y+0{\cdot}9070Z=-1{\cdot}3036$ Å
Plane *B*: $-0{\cdot}1434X+0{\cdot}4332Y+0{\cdot}8897Z=-1{\cdot}0851$

Table 8. *The magnitudes and the direction cosines of the principal axes of the thermal ellipsoids*

The direction cosines are with respect to the crystallographic axes.

Atom	Axis i	$B_i(\text{Å}^2)$	q_i^a	q_i^b	q_i^c
O(1)	1	6.27	-0.054	0.171	0.984
	2	4.40	0.954	-0.283	0.102
	3	2.48	-0.295	-0.944	0.148
O(2)	1	6.36	-0.195	0.527	0.827
	2	5.00	0.208	-0.802	0.560
	3	1.91	0.958	0.282	0.047
N(1)	1	4.05	0.404	-0.483	0.777
	2	3.20	-0.572	0.530	0.626
	3	2.78	0.714	0.697	0.062
N(2)	1	4.65	-0.194	0.251	0.948
	2	3.03	-0.206	-0.956	0.211
	3	2.51	0.959	-0.154	0.238
N(3)	1	5.21	0.268	0.218	0.939
	2	3.41	-0.907	-0.272	0.322
	3	2.65	0.325	-0.937	0.125
C(1)	1	2.98	-0.496	0.854	0.159
	2	2.43	0.573	0.185	0.798
	3	1.42	-0.652	-0.487	0.581
C(2)	1	3.31	0.377	0.546	0.748
	2	2.59	-0.865	-0.080	0.495
	3	1.86	0.330	-0.834	0.442
C(3)	1	3.35	0.380	0.494	0.782
	2	2.58	-0.307	-0.730	0.610
	3	1.84	-0.873	0.472	0.125
C(4)	1	2.81	-0.040	-0.069	0.997
	2	2.35	0.572	0.816	0.080
	3	1.85	-0.819	0.573	0.007
C(5)	1	3.47	-0.495	0.219	0.841
	2	3.15	0.702	0.617	0.238
	3	1.94	0.512	-0.708	0.486
C(6)	1	3.60	-0.177	0.555	0.813
	2	2.84	-0.239	-0.825	0.512
	3	1.81	0.955	-0.104	0.278
C(7)	1	2.64	-0.361	-0.126	0.924
	2	2.32	0.191	0.960	0.206
	3	2.13	0.913	-0.250	0.322
C(8)	1	3.17	0.994	-0.105	0.034
	2	2.60	-0.065	-0.311	0.948
	3	1.84	0.089	0.945	0.315
C(9)	1	2.98	0.668	-0.295	0.683
	2	2.25	-0.531	-0.832	0.160
	3	1.98	-0.521	0.470	0.712
K	1	3.53	0.718	0.677	0.165
	2	2.89	0.126	-0.358	0.925
	3	2.56	-0.685	0.643	0.342

is unexpected and somewhat embarrassing; however, considering the estimated standard deviations in bond lengths and angles, the apparent anomalies seem to be significant. The distortion of the anion persisted through the least-squares refinements using the visual data and through refinements using the diffractometer data with different weighting schemes.

An especially unusual feature of the structure is the trend of relatively long lengths for the bonds parallel to the N(2)–C(8) bond. These bonds are also nearly parallel to the *a* axis, suggesting that the unit-cell dimensions might be in error. However, since the unit-cell dimensions were measured on a precision Weissenberg camera and were later verified with the diffractometer – in both cases using the same crystal which was subsequently employed for the collection of intensity data – it seems unlikely that the unit-cell constants are significantly incorrect. Thermal motion in the **a** direction is not especially pronounced and does not provide an obvious explanation for the unusual bond lengths.

Table 7 lists deviations from least-squares planes through the anion. The atoms of the phenyl ring and atom C(7) lie in the same plane. The bonding to atom C(7) is pyramidal with the cyano groups displaced significantly from the plane through the phenyl ring. Similar deviations from planarity have been reported for the crystal structures of other carbanions (Bugg & Sass, 1965; Desiderato & Sass, 1965). The atoms of the nitro group are also significantly displaced from the phenyl plane.

Thermal vibrations

The ellipsoids of thermal vibration are described in Table 8. The thermal motions are in general agreement with those that would be expected on the basis of the anion geometry and the crystal packing. The largest components of vibrations are approximately perpendicular to the anion plane. The most severe thermal motion is displayed by the cyano nitrogen atoms and the nitro oxygen atoms; the thermal vibrations of the other atoms are relatively small.

No attempt was made to correct the bond lengths for the effects of thermal vibration.

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