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Crystal Structures of Condensation Products of Malononitrile

II. The Potassium Salt of 1,1,3-Tricyanopropanone

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The crystals are monoclinic, space group $P2_1/c$, with cell dimensions $a = 8.28_1$ Å, $b = 11.83_7$ Å, $c = 7.43_2$ Å, and $\beta = 101.2_7°$. 1575 reflections were recorded as observed on an automatic four-circle diffractometer. The structure was refined by full-matrix least squares technique ($R_w = 3.5$ %, $R = 3.2$ %). Apart from the cyano group and hydrogen atoms of the $CH_2(CN)$ group, the anion is nearly planar; the dihedral angle $O-C-CH_2-(CN)$ is 15°. No significant differences in the $sp-sp^2$ C–C and sp^2-sp^2 C–C bonds (1.412 Å) are observed. The K^+ ion is coordinated to two oxygen atoms and four nitrogen atoms, arranged in the form of a distorted trigonal prism.

This work was undertaken as part of a program of X-ray crystallographic studies on condensation products of malononitrile. Structure determinations of the potassium salt of 2-cyanomethyl-1,1,3,3-tetracyanopropene [1] (potassium 2-dicyanomethyl-1,1,3-tricyanopropenide) and the 2-amino-1,1,3-tricyanopropene [2] molecule ("dimeric malononitrile"), have also been carried out. These will be referred to as $KC_9N_5H_2$ and $C_6N_4H_4$, respectively.

The synthesis of 1,1,3-tricyanopropanone has been reported.[3] The potassium salt of 1,1,3-tricyanopropanone ($KC_6N_3OH_2$) is conveniently synthesized by a base catalysed condensation reaction of malononitrile and K_2CO_3. It can also be formed by hydrolysis of $C_6N_4H_4$.[4]

CRYSTAL DATA

$KC_6N_3OH_2$ was synthesized by E. G. Iversen by reaction between K_2CO_3 and malononitrile in a boiling ethanol-water solution.

The compound crystallizes from methanol as light yellow-coloured needles. The crystals are monoclinic, with space group $P2_1/c$. Unit cell dimensions determined on a manual four-circle diffractometer, (CuK radiation), with estimated standard deviations, are $a = 8.2814(17)$ Å, $b = 11.8373(25)$ Å, $c = 7.4322(17)$ Å, and $\beta = 101.27(1)°$. c is the needle axis. There are four formula units in the cell ($\varrho_{calc} = 1.598$ g cm^{-3}, $\varrho_{obs} = 1.57$ g cm^{-3}).

Table 1. Observed and calculated structure factors. (The five columns list values of h, k, l, 10 F_o, and 10 F_c.)

h	k	l	Fo	Fc
0	0	2	46	48
0	0	4	788	- 781
0	0	6	188	- 186
0	0	8	114	120
0	0	10	65	64
0	1	1	50	39
0	1	2	506	474
0	1	4	14	6
0	1	5	218	- 215
0	1	6	34	34
0	1	8	82	81
0	1	9	56	55
0	2	0	574	- 558
0	2	1	282	- 282
0	2	2	454	- 445
0	2	3	558	- 549
0	2	4	314	307
0	2	5	82	81
0	2	6	133	134
0	2	7	104	102
0	2	8	36	- 31
0	2	9	29	16
0	3	1	197	- 205
0	3	2	184	- 172
0	3	3	126	126
0	3	4	43	- 42
0	3	5	210	208
0	3	6	86	- 90
0	3	7	38	38
0	3	9	104	- 99
0	4	0	362	341
0	4	1	854	827
0	4	2	30	26
0	4	3	391	383
0	4	4	27	6
0	4	5	69	- 67
0	4	6	41	43
0	4	7	106	- 106
0	4	8	79	- 76
0	5	1	142	- 135
0	5	2	556	544
0	5	3	50	48
0	5	4	230	231
0	5	5	127	- 128
0	5	6	128	- 132
0	5	7	22	- 23
0	5	8	101	- 101
0	5	9	74	74
0	6	0	305	320
0	6	1	199	- 194
0	6	2	66	54
0	6	3	272	- 276
0	6	4	271	- 270
0	6	5	24	24
0	6	7	35	32
0	6	8	57	58
0	7	1	340	339
0	7	2	214	- 214
0	7	3	93	- 94
0	7	4	144	- 145
0	7	5	115	- 119
0	7	6	73	72
0	7	8	105	103
0	8	0	288	- 292
0	8	3	73	- 74
0	8	4	100	98
0	8	6	36	36
0	8	7	133	133
0	8	8	55	- 56
0	9	1	185	- 183
0	9	2	80	79
0	9	3	41	40
0	9	4	75	75
0	9	5	69	68
0	9	6	62	- 62
0	9	7	53	50
0	9	8	64	- 59
0	10	0	44	- 48
0	10	1	202	- 207
0	10	2	40	- 34
0	10	3	230	229
0	10	4	66	64
0	10	5	141	- 140
0	10	6	76	73
0	10	7	109	- 109
0	10	8	29	- 28
0	11	1	82	78
0	11	2	137	134
0	11	3	30	26
0	11	4	36	37
0	11	5	76	- 74
0	11	6	36	- 43
0	11	7	36	- 39
0	12	0	73	71
0	12	4	52	- 58
0	12	6	69	- 67
0	13	2	146	- 149
0	13	4	30	- 24
0	14	0	111	- 100
0	14	3	39	- 44
0	14	4	109	105
0	15	1	80	- 80
0	15	2	34	- 25
0	15	3	46	49
0	16	1	70	61
1	0	-10	73	89
1	0	-6	320	- 319
1	0	-4	249	- 255
1	0	-2	881	885
1	0	0	356	301
1	0	2	1491	-1505
1	0	4	17	9
1	0	6	275	272
1	0	8	108	112
1	1	-9	29	35
1	1	-8	95	- 91
1	1	-7	77	- 78
1	1	-5	150	- 151
1	1	-4	193	192
1	1	-3	363	351
1	1	-2	510	498
1	1	0	132	134
1	1	1	250	250
1	1	2	797	- 790
1	1	3	155	- 155
1	1	4	468	- 462
1	1	5	124	- 122
1	1	6	199	201
1	1	7	35	36
1	1	8	152	159
1	2	-10	42	- 44
1	2	-9	57	- 54
1	2	-8	25	- 27
1	2	-7	148	- 151
1	2	-6	70	70
1	2	-5	20	- 19
1	2	-4	204	200
1	2	-3	547	535
1	2	-2	54	53
1	2	-1	44	41
1	2	0	22	21
1	2	1	389	- 383
1	2	2	57	58
1	2	3	214	- 216
1	2	4	209	212
1	2	5	263	264
1	2	6	180	- 165
1	2	7	74	74
1	2	8	83	- 85
1	2	9	32	- 31
1	3	-8	36	45
1	3	-7	115	118
1	3	-6	42	- 43
1	3	-5	128	128
1	3	-4	310	- 309
1	3	-3	169	- 167
1	3	-2	224	218
1	3	-1	591	- 598
1	3	0	131	133
1	3	1	202	- 199
1	3	2	252	245
1	3	3	243	244
1	3	4	120	121
1	3	5	258	263
1	3	6	19	- 18
1	3	8	40	- 32
1	3	9	95	- 94
1	4	-9	52	47
1	4	-7	198	195
1	4	-6	158	158
1	4	-4	42	- 39
1	4	-3	486	- 483
1	4	-2	127	- 130
1	4	-1	291	- 276
1	4	0	357	- 350
1	4	1	678	678
1	4	2	192	- 184
1	4	3	191	- 193
1	4	4	220	217
1	4	5	175	- 176
1	4	6	101	100
1	4	7	57	- 55
1	4	8	28	- 28
1	4	9	35	36
1	5	-9	36	27
1	5	-8	69	67
1	5	-7	114	- 114
1	5	-6	23	28
1	5	-5	17	- 15
1	5	-4	224	- 232
1	5	-3	201	201
1	5	-2	44	35
1	5	-1	200	203
1	5	0	271	257
1	5	1	236	232
1	5	2	174	179
1	5	3	60	- 62
1	5	4	152	- 152
1	5	5	257	- 261
1	5	6	147	- 144
1	5	9	59	59
1	6	-9	29	- 24
1	6	-7	53	- 55
1	6	-6	105	- 107
1	6	-5	33	- 35
1	6	-4	291	- 293
1	6	-3	98	100
1	6	-2	423	431
1	6	-1	38	- 33
1	6	0	430	424
1	6	1	36	- 33
1	6	2	92	- 92
1	6	3	117	113
1	6	4	283	- 267
1	6	5	145	149
1	6	7	37	- 42
1	6	8	45	42
1	6	9	38	- 39
1	7	-9	46	43
1	7	-8	36	- 36
1	7	-7	40	- 36
1	7	-5	75	- 78
1	7	-4	82	81
1	7	-3	80	- 60
1	7	-2	107	108
1	7	-1	126	128
1	7	0	74	- 76
1	7	1	135	- 135
1	7	2	196	- 198
1	7	3	111	- 112
1	7	5	52	51
1	7	6	125	124
1	7	7	79	78
1	7	8	34	33
1	8	-7	47	- 49
1	8	-6	153	158
1	8	-5	167	171
1	8	-4	137	135
1	8	-3	108	110
1	8	-2	117	- 118
1	8	0	318	- 318
1	8	1	215	- 211
1	8	2	110	113
1	8	3	209	- 212
1	8	4	152	146
1	8	5	54	55
1	8	7	90	88
1	8	8	53	- 52
1	9	-8	43	35
1	9	-7	31	- 37
1	9	-6	86	- 85
1	9	-5	137	136
1	9	-4	60	- 61
1	9	-3	149	155
1	9	-2	141	133
1	9	-1	163	- 161
1	9	0	110	110
1	9	1	134	- 143
1	9	2	20	- 17
1	9	3	117	118
1	9	5	87	87
1	9	6	38	- 42
1	10	-8	30	21
1	10	-7	64	67
1	10	-6	46	47
1	10	-5	139	- 141
1	10	-4	115	- 114
1	10	-3	294	- 296
1	10	-2	90	- 92
1	10	-1	165	176
1	10	1	281	286
1	10	2	29	36
1	10	4	46	42
1	10	5	115	- 113
1	11	-6	39	41
1	11	-5	53	- 54
1	11	-4	77	- 78
1	11	-3	29	- 37
1	11	-2	128	- 132
1	11	-1	27	33
1	11	1	86	88
1	11	2	84	86
1	11	3	60	- 59
1	11	5	85	- 83
1	11	6	54	- 57
1	11	7	40	34
1	12	-7	30	- 30
1	12	-6	56	- 57
1	12	-5	51	54
1	12	-3	46	39
1	12	-2	64	71
1	12	-1	39	37
1	12	1	63	- 64
1	12	3	53	- 55
1	12	4	101	- 104
1	12	5	27	27
1	12	6	40	- 37
1	13	-6	44	- 42
1	13	-5	36	- 29
1	13	-4	62	61
1	13	-2	130	130
1	13	-1	28	34
1	13	1	38	41
1	13	2	97	- 97
1	13	3	30	- 31
1	13	4	34	37
1	13	6	55	47
1	14	-2	135	- 130
1	14	2	65	58
1	14	3	68	- 62
1	14	4	64	63
1	15	-2	34	- 30
1	15	-1	112	- 108
1	15	0	81	- 59
1	15	2	32	39
1	15	3	64	66
1	16	-2	38	25
1	16	-1	27	6
1	16	0	32	- 8
1	16	1	85	79
1	16	2	30	- 27
2	0	-8	67	- 75
2	0	-6	110	- 107
2	0	-4	184	188
2	0	-2	508	513
2	0	0	302	- 293
2	0	2	413	- 414
2	0	4	61	55
2	0	6	104	- 109
2	1	-10	47	- 40
2	1	-8	161	- 160
2	1	-7	87	- 86
2	1	-6	69	69
2	1	-5	45	- 46
2	1	-4	359	359
2	1	-3	184	177
2	1	-2	365	348
2	1	-1	165	- 150
2	1	0	444	- 452
2	1	1	401	397
2	1	2	294	- 297
2	1	3	208	- 201
2	1	4	240	239
2	1	5	35	- 36
2	1	6	254	258
2	1	7	62	65
2	1	8	80	- 86
2	2	-10	35	- 33
2	2	-9	95	- 98
2	2	-7	59	- 59
2	2	-6	121	120
2	2	-5	255	253
2	2	-4	56	57
2	2	-3	365	355
2	2	-2	552	- 542
2	2	-1	550	- 543
2	2	0	433	- 421
2	2	1	609	- 601
2	2	2	372	366
2	2	3	87	85
2	2	4	241	242
2	2	5	239	244
2	2	6	35	- 35
2	2	9	37	- 30
2	3	-7	109	107
2	3	-6	62	- 62
2	3	-5	188	183
2	3	-4	34	35
2	3	-3	77	- 82
2	3	-2	353	351
2	3	-1	389	- 393
2	3	0	559	- 543
2	3	2	73	- 69
2	3	3	205	215
2	3	4	53	- 53
2	3	5	79	80
2	3	7	137	- 137
2	3	8	32	33
2	3	9	63	- 65
2	4	-10	34	- 35
2	4	-9	96	97
2	4	-8	64	68
2	4	-7	64	59
2	4	-6	60	61
2	4	-5	258	- 259
2	4	-4	73	- 68
2	4	-3	328	- 328
2	4	-1	559	555
2	4	0	106	- 103
2	4	1	649	633
2	4	2	111	106
2	4	3	159	- 159
2	4	4	83	85
2	4	5	125	- 119
2	4	6	96	- 97
2	4	7	63	- 60

Table 1. Continued.

h	k	l	F_o	F_c
2	4	8	56	- 55
2	5	-9	40	38
2	5	-8	51	54
2	5	-7	137	- 132
2	5	-6	37	- 38
2	5	-4	229	- 216
2	5	-3	52	- 50
2	5	-2	41	43
2	5	-1	98	101
2	5	0	84	90
2	5	1	146	144
2	5	2	273	267
2	5	3	107	- 104
2	5	4	78	- 81
2	5	5	44	- 43
2	5	6	84	- 84
2	5	7	132	136
2	6	-8	59	- 63
2	6	-7	37	37
2	6	-6	190	- 191
2	6	-5	19	19
2	6	-4	62	58
2	6	-3	24	26
2	6	-2	463	466
2	6	-1	170	- 172
2	6	0	68	- 66
2	6	1	178	- 181
2	6	2	311	- 311
2	6	3	43	43
2	6	4	115	- 118
2	6	5	32	29
2	6	6	93	95
2	6	8	77	79
2	7	-8	63	- 63
2	7	-7	48	- 55
2	7	-6	40	- 31
2	7	-5	62	62
2	7	-4	246	244
2	7	-3	51	52
2	7	-2	211	218
2	7	-1	175	179
2	7	0	235	- 236
2	7	1	140	- 142
2	7	2	251	- 253
2	7	3	90	- 89
2	7	4	42	40
2	7	5	26	- 33
2	7	6	131	130
2	8	-9	64	- 63
2	8	-8	40	50
2	8	-6	105	105
2	8	-5	78	79
2	8	-4	96	- 100
2	8	-3	52	46
2	8	-2	211	- 219
2	8	-1	56	56
2	8	1	94	- 94
2	8	2	303	303
2	8	4	51	50
2	8	5	108	108
2	8	6	94	- 91
2	8	8	40	- 38
2	9	-7	98	91
2	9	-6	65	- 66
2	9	-5	190	193
2	9	-4	67	67
2	9	-3	214	- 217
2	9	-2	42	- 43
2	9	-1	381	- 390
2	9	0	145	- 144
2	9	1	75	75
2	9	3	296	304
2	9	4	75	73
2	9	5	65	63
2	9	7	79	- 80
2	10	-6	26	16
2	10	-5	86	- 80
2	10	-4	35	- 39
2	10	-2	97	95
2	10	-1	128	127
2	10	0	23	- 19
2	10	1	38	- 48
2	10	2	100	- 100
2	10	3	44	- 46
2	10	5	24	- 20
2	11	-7	65	- 69
2	11	-6	41	- 47
2	11	-5	84	- 80
2	11	-4	56	- 54
2	11	-3	98	99
2	11	-1	173	184
2	11	0	169	171
2	11	2	71	71
2	11	3	178	- 180
2	11	4	70	- 70
2	11	5	31	- 35
2	11	6	59	- 63
2	11	7	64	63
2	12	-6	89	- 96
2	12	-4	29	- 20
2	12	-2	126	125
2	12	0	91	91
2	12	1	58	- 54
2	12	2	74	- 71
2	12	3	36	33
2	12	4	69	- 66
2	12	5	40	42
2	13	-4	105	108
2	13	0	170	- 171
2	13	2	50	- 51
2	13	3	39	- 32
2	13	4	68	68
2	14	-5	77	77
2	14	-4	34	- 30
2	14	-3	49	49
2	14	-2	37	- 36
2	14	-1	83	- 87
2	14	3	42	- 45
2	15	-4	57	- 54
2	15	-1	49	- 43
2	15	0	60	63
2	15	1	36	43
2	15	2	85	80
2	15	3	50	46
2	16	-2	44	- 43
2	16	-1	49	50
2	16	1	53	52
3	0	-10	30	16
3	0	-8	37	- 34
3	0	-6	174	- 176
3	0	-4	61	- 59
3	0	-2	616	601
3	0	0	198	183
3	0	2	138	- 134
3	0	4	107	- 104
3	0	6	53	54
3	0	8	113	118
3	1	-10	92	- 95
3	1	-9	47	- 39
3	1	-8	54	- 51
3	1	-7	61	- 62
3	1	-6	270	265
3	1	-5	20	- 15
3	1	-4	103	97
3	1	-3	130	128
3	1	-2	405	- 391
3	1	-1	96	98
3	1	0	506	- 520
3	1	1	46	44
3	1	2	77	77
3	1	3	391	- 398
3	1	4	262	265
3	1	5	72	74
3	1	6	80	76
3	1	7	77	76
3	1	8	77	- 78
3	2	-9	49	- 54
3	2	-8	85	80
3	2	-7	55	56
3	2	-6	135	135
3	2	-5	275	273
3	2	-4	150	- 154
3	2	-3	31	31
3	2	-2	455	- 438
3	2	-1	374	- 372
3	2	0	96	98
3	2	1	101	- 106
3	2	2	352	350
3	2	3	204	207
3	2	4	93	- 91
3	2	5	53	55
3	2	6	111	- 111
3	2	7	41	- 39
3	2	9	40	- 27
3	3	-8	34	- 40
3	3	-7	195	194
3	3	-6	50	50
3	3	-5	54	51
3	3	-4	99	98
3	3	-3	432	- 427
3	3	-2	42	- 40
3	3	-1	237	- 232
3	3	0	278	- 264
3	3	1	386	383
3	3	2	285	286
3	3	3	317	316
3	3	4	29	- 35
3	3	5	19	- 7
3	3	6	34	- 31
3	3	7	155	- 158
3	4	-9	51	52
3	4	-8	79	79
3	4	-7	65	- 63
3	4	-6	26	- 28
3	4	-5	238	- 242
3	4	-4	54	- 61
3	4	-3	126	- 128
3	4	-2	55	- 62
3	4	-1	434	- 429
3	4	0	36	- 36
3	4	1	95	98
3	4	2	175	178
3	4	3	198	- 164
3	4	4	67	67
3	4	5	64	- 63
3	4	6	32	- 35
3	4	7	23	18
3	5	-9	40	- 39
3	5	-8	52	51
3	5	-7	118	- 123
3	5	-6	47	- 47
3	5	-5	97	103
3	5	-4	202	- 203
3	5	-3	38	37
3	5	-2	27	30
3	5	-1	146	147
3	5	0	211	210
3	5	1	180	- 186
3	5	2	118	121
3	5	3	250	- 253
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7	0	2	179	173

Table 1. Continued.

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7	2	-5	174	- 176
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7	2	1	212	216
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7	4	-5	209	207
7	4	-4	103	- 107
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7	4	5	76	75
7	5	-8	34	- 48
7	5	-7	68	71
7	5	-6	51	52
7	5	-5	24	20
7	5	-4	191	190
7	5	-3	38	41
7	5	-1	23	- 30
7	5	0	160	- 159
7	5	1	20	16
7	5	2	98	- 91
7	5	3	35	37
7	5	4	28	27
7	5	6	55	56
7	6	-6	146	141
7	6	-4	25	19
7	6	-3	99	- 99
7	6	-2	149	- 149
7	6	-1	103	101
7	6	0	53	- 53
7	6	1	72	74
7	6	2	75	78
7	6	4	61	62
7	7	-8	80	82
7	7	-4	142	- 139
7	7	-3	51	- 50
7	7	-2	91	- 94
7	7	-1	110	- 115
7	7	0	81	79
7	7	1	38	38
7	7	2	175	176
7	8	-7	65	62
7	8	-6	79	- 81
7	8	-4	31	30
7	8	-3	119	- 115
7	8	-2	146	146
7	8	1	91	91

h	k	l	F_o	F_c
7	8	2	120	- 119
7	8	4	27	29
7	8	5	42	- 54
7	9	-7	76	- 79
7	9	-5	66	- 69
7	9	-4	30	- 19
7	9	-3	130	128
7	9	-2	24	35
7	9	-1	137	141
7	9	3	90	- 98
7	10	-5	53	56
7	10	-3	75	76
7	10	1	67	- 63
7	11	-4	53	54
7	11	-3	71	- 67
7	11	-2	45	46
7	11	-1	35	- 46
7	11	0	68	- 71
7	11	3	45	43
7	12	-3	90	- 81
7	12	-2	82	- 86
7	12	0	64	- 67
7	12	1	66	63
7	12	2	70	65
7	13	-1	41	- 34
A	0	-8	104	102
A	0	-6	151	159
A	0	-4	47	- 48
A	0	-2	146	- 145
A	0	0	44	42
A	0	2	119	118
A	0	4	42	- 38
A	1	-7	42	41
A	1	-6	102	- 101
A	1	-5	52	54
A	1	-4	71	- 73
A	1	-3	145	- 138
A	1	-1	77	- 78
A	1	0	203	201
A	1	1	52	49
A	1	2	134	148
A	1	3	94	93
A	1	4	72	- 66
A	2	-8	59	- 63
A	2	-7	27	13
A	2	-6	138	- 136
A	2	-5	146	- 148
A	2	-4	86	85
A	2	-3	55	- 50
A	2	-2	131	146
A	2	-1	123	125
A	2	0	175	- 177
A	2	2	56	- 57
A	2	3	90	- 59
A	2	4	92	88
A	3	-7	90	- 97
A	3	-6	36	- 37
A	3	-5	25	21
A	3	-3	123	120
A	3	-2	30	37
A	3	-1	194	169
A	3	0	25	- 32
A	3	1	50	- 52
A	3	2	127	- 125
A	3	3	189	- 189
A	4	-6	61	- 63
A	4	-5	187	188
A	4	-4	27	27
A	4	-3	75	74
A	4	-2	163	168
A	4	-1	143	- 147
A	4	0	23	26
A	4	1	44	- 41
A	4	2	85	- 88
A	4	3	102	94
A	4	4	65	- 63
A	4	5	34	31
A	5	-5	55	- 55
A	5	-4	77	77
A	5	-3	62	- 62
A	5	-1	78	- 82
A	5	0	23	- 32

h	k	l	F_o	F_c
A	5	1	47	42
A	5	3	90	87
A	5	4	44	39
A	6	-7	75	73
A	6	-6	104	105
A	6	-5	37	- 41
A	6	-4	103	- 96
A	6	-3	93	- 86
A	6	-2	156	- 162
A	6	-1	46	43
A	6	1	56	- 48
A	6	2	121	120
A	6	3	41	- 38
A	6	4	44	46
A	7	-7	41	42
A	7	-6	79	- 82
A	7	-5	107	102
A	7	-4	93	- 92
A	7	-2	30	41
A	7	-1	61	- 59
A	7	0	137	138
A	7	2	35	33
A	7	3	29	- 20
A	7	4	98	- 98
A	8	-6	61	- 63
A	8	-5	90	- 87
A	8	-4	30	35
A	8	-2	67	76
A	8	-1	127	129
A	8	1	80	80
A	8	2	32	- 26
A	9	-5	45	- 52
A	9	-3	72	73
A	9	-2	30	- 1
A	9	-1	46	46
A	9	0	37	- 45
A	9	1	34	- 28
A	9	3	35	- 45
A	10	-5	102	102
A	10	-2	29	- 10
A	10	-1	96	- 94
A	10	1	100	- 97
A	11	-4	99	98
A	11	0	73	- 73
9	0	-4	166	- 165
9	0	-2	114	- 119
9	0	0	134	140
9	0	2	118	118
9	1	-7	51	45
9	1	-6	103	- 101
9	1	-4	96	- 92
9	1	-3	111	- 108
9	1	-1	39	33
9	1	0	117	120
9	1	1	52	53
9	1	4	97	- 97
9	2	-7	34	- 32
9	2	-5	116	- 117
9	2	-4	147	143
9	2	-3	42	48
9	2	-1	126	129
9	2	0	119	- 116
9	2	1	37	33
9	2	3	36	- 40
9	3	-7	96	- 98
9	3	-4	66	66
9	3	-3	112	111
9	3	-1	79	80
9	3	1	112	- 111
9	3	3	55	- 55
9	3	4	36	34
9	4	-7	93	90
9	4	-6	29	- 18
9	4	-5	79	84
9	4	-4	68	69
9	4	-3	104	- 97
9	4	-2	35	29
9	4	-1	171	- 172
9	4	0	57	- 59
9	4	3	74	69
9	5	-7	61	52
9	5	-6	52	51
9	5	-4	116	109

h	k	l	F_o	F_c
9	5	-3	53	- 48
9	5	-2	67	- 70
9	5	-1	71	- 68
9	5	0	113	- 118
9	5	1	36	27
9	5	2	32	- 28
9	5	3	37	28
9	6	-5	72	- 65
9	6	-4	112	- 113
9	6	-3	83	83
9	6	-2	41	- 52
9	6	-1	110	104
9	6	0	67	70
9	6	1	29	- 37
9	6	2	94	81
9	6	3	29	- 20
9	7	-6	93	- 98
9	7	-5	33	- 34
9	7	-3	120	- 111
9	7	-2	73	68
9	7	0	83	82
9	7	1	92	87
9	8	-4	66	63
9	8	0	60	- 57
9	8	2	43	- 37
9	9	-3	124	122
9	9	1	95	- 98
9	10	-2	39	33
9	10	-1	42	- 38
10	0	-4	131	- 133
10	0	-2	58	- 55
10	0	0	88	93
10	1	-6	61	- 66
10	1	-5	44	- 48
10	1	-4	51	44
10	1	-2	122	125
10	1	-1	52	51
10	1	0	55	57
10	1	2	104	- 102
10	2	-5	92	- 97
10	2	-4	77	77
10	2	-3	83	84
10	2	-1	91	91
10	2	3	53	- 54
10	3	-5	97	93
10	3	-4	57	- 54
10	3	-3	70	70
10	3	-2	44	- 38
10	3	-1	49	- 55
10	3	1	136	- 135
10	4	-6	52	51
10	4	-5	38	45
10	4	-3	43	- 43
10	4	-1	65	- 64
10	4	0	31	- 22
10	5	-5	68	- 66
10	5	-4	32	31
10	5	-3	50	- 57
10	5	-2	132	- 133
10	5	-1	57	54
10	5	1	101	96
10	5	2	58	63
10	6	-4	90	- 84
10	6	-3	43	43
10	6	0	66	68
10	7	-2	49	56
10	7	-1	35	34
10	8	-1	61	63
11	0	-4	102	- 90
11	0	-2	67	62
11	0	0	126	122
11	1	-4	42	42
11	1	-2	52	59
11	2	-4	89	82
11	2	-3	106	108
11	2	-2	46	- 50
11	2	0	68	- 60
11	3	-3	29	26
11	3	-2	34	35
11	3	-1	69	- 71
11	4	-3	72	- 72
11	4	-2	33	34
11	4	-1	53	- 49

About 2000 reflections with $2\theta < 60°$ were measured on an automatic four circle diffractometer ($\omega/2\theta$ scan) using Mo$K\alpha$-radiation (0.002″ Nb filter). 1575 reflections were recorded as observed, their intensities being greater than twice the standard deviation from counter statistics, including a 2 % uncertainty in the measurements. These were corrected for absorption effects, and used in the structure determination. A crystal of dimensions $0.33 \times 0.32 \times 0.13$ mm^3 has been used for all X-ray measurements.

All programs used are included in Ref. 5.

Table 2. Fractional atomic coordinates and thermal parameters with estimated standard deviations ($\times 10^5$). The temperature factor is given by $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$. Isotropic temperature factors (Å^2) are given for the hydrogen atoms. For numbering of atoms, see Fig. 1.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
K^+	9412	32875	60624	917	525	1392	−152	851	−47
	4	3	5	6	3	8	6	9	7
C1	62866	25969	39451	837	420	1222	−111	678	−30
	17	12	19	23	11	30	25	40	29
C2	74860	17921	37158	755	482	1003	−178	427	−7
	17	12	19	21	11	27	25	38	27
C3	70424	5514	38753	1179	474	2138	−30	1549	−26
	22	14	29	29	13	44	30	58	37
C4	66598	37573	37973	997	525	1476	−41	580	−59
	19	15	21	25	13	33	29	46	33
C5	47097	23043	42428	926	461	1212	112	527	−41
	19	13	20	23	12	30	27	41	29
C6	82012	−1797	31846	1260	504	1589	169	782	−65
	21	14	22	27	13	35	31	52	34
O	88620	20453	34221	848	638	1858	−252	1144	−66
	12	10	16	17	9	26	19	32	24
N4	69823	46857	36664	1931	529	2792	−368	1118	62
	21	13	23	31	13	43	32	57	36
N5	34144	20751	44679	930	711	2099	19	1091	−70
	16	12	20	21	12	35	26	43	33
N6	90903	−7292	26170	2044	828	2533	645	1604	−555
	21	15	24	35	15	43	35	62	39
H1	58598	4074	31314	5.26					
	269	169	270	.50					
H2	70383	3494	50760	5.30					
	253	167	304	.52					

STRUCTURE DETERMINATION

The structure was determined by the heavy atom method and refined by full-matrix least squares technique to an R_w-value of 3.6 % (conventional R-value 3.6 %). The 54 strongest reflections were excluded from the refinement. Secondary extinction correction and further refinement gave a final R_w-value of 3.5 % ($R=3.2$ %). The weight analysis showed no intensity dependence. A final difference Fourier map contained no larger density fluctuations than ± 0.22 e Å^{-3}. Atomic form factors of Cromer and Waber [6] have been used, except for hydrogen.[7]

Observed and calculated structure factors are listed in Table 1, and atomic parameters in Table 2. For numbering of atoms, see Fig. 1.* An analysis of the atomic vibration tensors is presented in Table 3.

The r.m.s. discrepancy between "observed" atomic anisotropic vibration tensor components and those calculated from the rigid-body parameters obtained by analysis of librational, translational, and screw motion [8] of the anion is 0.0038 Å^2, and drops to 0.0026 Å^2 if C6–N6 of the $CH_2(CN)$ group is

* A similar numbering is used for $KC_9N_5H_2$ and $C_6N_4H_4$.

Table 3. The root mean square amplitudes of vibration $(\overline{u^2})^{\frac{1}{2}}$ (Å) and B-values (Å^2) along the principal axes, given by the components of a unit vector in fractional coordinates ($\times 10^3$).

Atom	$(\overline{u^2})^{\frac{1}{2}}$	B	e_x	e_y	e_z
	.204	3.28	70	−45	99
K^+	.191	2.87	18	69	79
	.158	1.97	99	19	−52
	.188	2.78	74	−27	114
C1	.173	2.37	−11	75	60
	.154	1.87	98	29	−47
	.190	2.84	46	−78	9
C2	.165	2.15	47	12	133
	.151	1.80	104	29	−32
	.250	4.95	69	−2	126
C3	.183	2.66	−5	84	7
	.169	2.26	102	6	−53
	.201	3.19	40	−34	124
C4	.192	2.90	1	77	57
	.180	2.57	117	11	−15
	.186	2.74	70	69	10
C5	.182	2.63	52	−21	129
	.166	2.18	−87	44	47
	.213	3.58	90	−7	111
C6	.197	3.06	79	35	−69
	.187	2.77	−29	77	41
	.231	4.21	58	−32	120
O	.213	3.57	−2	76	59
	.146	1.67	109	19	−32
	.275	5.96	45	−1	135
N4	.257	5.21	−109	27	23
	.185	2.72	37	80	−7
	.240	4.56	44	−16	133
N5	.224	3.97	13	83	24
	.164	2.12	−114	4	25
	.283	6.32	95	53	9
N6	.276	6.01	50	32	123
	.186	2.75	−61	57	60

omitted. This indicates that a part of the anion may be regarded as a rigid body, although the problem of differentiating between such a motion and an internal torsional motion around the C1−C2 bond may be difficult.[9] The reduced r.m.s. translational amplitudes corresponding to the discrepancy of 0.0026 Å^2 are 0.180, 0.161, and 0.152 Å, while the r.m.s. librational amplitudes are 5.3, 3.0, and 2.6°. The eigenvectors of C2 correspond closely to those of **T**.

"Riding" motion corrections[10] show that C1, C3, and O "ride" on C2. Contrary to what might be expected, C5 and C6 do not "ride" on C1 and C3, respectively.

Bond distances, bond angles, and dihedral angles of the anion, short *intra*-molecular contacts, and non-bonded distances between anions may be found in Table 4. The C−N bond distances in parentheses are corrected for "riding" motion. Librational corrections in other bonds correspond to the rigid-body calculation with C6−N6 omitted. The standard deviations in bond lengths

Table 4. Bond distances, bond angles, and dihedral angles of the anion, short *intra*-molecular contacts, and non-bonded distances between anions (equivalent position numbers in parentheses, as defined in Table 6 or given below). Estimated standard deviations in C–C, C–O, and C–N bond lengths are 0.002 Å, in C–C–C and C–C–O angles 0.1°, and in C–C–N angles 0.2°. Distances in parentheses are corrected for anisotropic thermal motion (see text).

Bond distances (Å)		
C1–C2	1.410	(1.414)
C2–C3	1.524	(1.530)
C1–C4	1.417	(1.423)
C1–C5	1.410	(1.415)
C3–C6	1.458	(1.460)
C2–O	1.238	(1.243)
C4–N4	1.140	(1.171)
C5–N5	1.150	(1.170)
C6–N6	1.125	(1.167)
C3–H1	1.04	
C3–H2	0.92	

Intra-molecular contacts (Å)	
C4···O	2.78
C6···O	2.69
C5···H1	2.63

Other contacts (Å)	
C3···N5(7)	3.39
C6···N4(8)	3.36
C6···N5(7)	3.28
N4···H1(1)	2.61
N4···H2(8)	2.68

7:$(1-x,-y,1-z)$
8:$(x,\frac{1}{2}-y,-\frac{1}{2}+z)$

Bond angles (°)	
C4–C1–C2	118.4
C5–C1–C2	123.3
C4–C1–C5	118.2
C1–C2–C3	117.1
C1–C2–O	123.5
C3–C2–O	119.4
C2–C3–C6	111.1
C1–C4–N4	178.9
C1–C5–N5	179.1
C3–C6–N6	178.4
C2–C3–H1	109
C2–C3–H2	112
C6–C3–H1	109
C6–C3–H2	108
H1–C3–H2	107

Dihedral angles (°)	
C4–C1–C2–C3	179.7
C5–C1–C2–C3	2.3
C4–C1–C2–O	−1.3
C5–C1–C2–O	−178.7
C1–C2–C3–C6	−166.3
O–C2–C3–C6	14.6

and bond angles have been calculated from the correlation matrix of the last least squares refinement cycle, neglecting the E.S.D.'s of the cell parameters. σ(C–C), σ(C–O), and σ(C–N) are 0.002 Å, σ(C–C–C) and σ(C–C–O) are 0.1°, and σ(C–C–N) 0.2°. Bond distances (uncorrected) and angles are also given in Fig. 1.

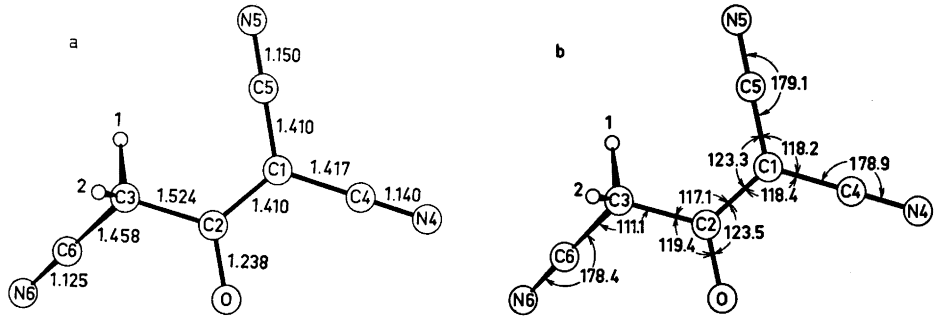

Fig. 1. Schematical drawings of the anion, showing bond distances (a) and bond angles (b). Small circles indicate hydrogen atoms.

Table 5. Deviations of atoms from some least squares planes (Å). Plane No. **1** is through all atoms except the cyano group and the hydrogen atoms of the $CH_2(CN)$ group, plane No. **2** is through the $C(CN)_2$ group, and plane No. **3** is through the carbonyl group and the adjacent carbon atoms. Deviations of atoms not defining the planes in parentheses.

Atom	1	2	3
C1	0.023	−0.003	0.002
C2	−0.002	(−0.067)	−0.005
C3	0.007		0.002
C4	0.010	0.002	(−0.010)
C5	0.008	0.005	(−0.033)
O	−0.013		0.002
N4	−0.007	−0.001	(−0.024)
N5	−0.017	−0.002	(−0.075)

Table 6. Coordination distances of the potassium ion, and the corresponding $C-N\cdots K^+$ and $C-O\cdots K^+$ angles. For numbering of equivalent positions, see Fig. 2.

Atom	Equiv. pos.	No.	(Å)	(°)
N6	$(1-x,\frac{1}{2}+y,\frac{1}{2}-z)$	1	2.97	133
N4	$(1-x,1-y,1-z)$	2	2.94	158
N5	$(x,\frac{1}{2}-y,\frac{1}{2}+z)$	3	2.96	115
N5	(x,y,z)	4	2.93	134
O	$(-1+x,y,z)$	5	2.77	118
O	$(-1+x,\frac{1}{2}-y,\frac{1}{2}+z)$	6	2.72	143

In Table 5, the deviations of atoms from some least squares planes are listed, and coordination distances of the cation are presented in Table 6. Fig. 2 is a schematical drawing of the structure viewed along the *c* axis, showing the coordination.

DISCUSSION

Apart from the cyano group and hydrogen atoms bonded to C3, the anion is nearly planar (plane **1** of Table 5), the largest deviation being 0.023 Å. The essentially planar $C(CN)_2$ group is slightly bent (and twisted) about the C1 – C2 bond, which makes an angle of 2.6° with this group (plane **2**). Similar deviations from planarity are observed for $KC_9N_5H_2$ and $C_6N_4H_4$. The degree of coplanarity of C2 and adjacent atoms is shown by plane **3** of Table 5. The differences in deviations of C4,N4 and C5,N5 from this plane are caused by the small twist of the $C(CN)_2$ group.

The cyano group attached to C3 is twisted 14° out of plane **1** (about the C2 – C3 bond); the CN group being in *anti* position with respect to the $C(CN)_2$ group.

The conformation of the anion is determined by *intra*-ionic non-bonded interactions and by coordination and packing effects. However, the observed opening of the bond angles C1 – C2 – O and C2 – C1 – C5 is probably mainly caused by coordination forces. O as well as N5 are coordinated to two potas-

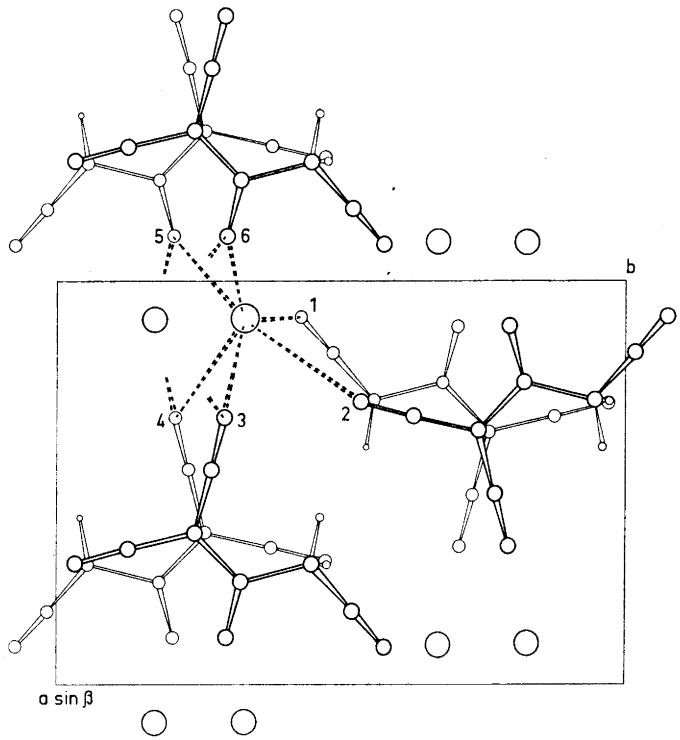

Fig. 2. Schematical drawing of the structure viewed along the *c* axis, showing the coordination. The equivalent positions of anions numbered from 1 to 6 are given in Table 6.

sium ions. Opening of C2 – C3 – C6 may be due to repulsion between C6 and O. CCN angles are essentially 180°.

The thermal corrections of the C – N bonds, assuming "riding" motion, are quite large (0.042 Å for C6 – N6) and may not be reliable, although "normal" bond lengths (1.17 Å) are obtained. The rigid body corrections of other bonds may also be questioned. The discussion will therefore be based on the uncorrected distances.

Molecular orbital calculations of π-electron densities of the anion of 2,2-dicyanoethanal and 2-amino-1,1-dicyanoethene have been carried out by Lofthus, using a method[11] based on extended HMO theory. The π-bond order of $sp - sp^2$ carbon – carbon bonds are 0.45 and 0.48, respectively; for the $sp^2 - sp^2$ C – C, the corresponding values are 0.66 and 0.58, for the C – N bonds (of the cyano groups) 0.81 and 0.82, and for the C – O bond and the C – N bond (of the amino group) 0.50 and 0.69. These values are assumed to be applicable for $KC_6N_3OH_2$ (A) and for $C_6N_4H_4$ (B) in the following discussion; similar data for $KC_9N_5H_2$ (C) are also included.

The bond lengths and bond orders (in parentheses) for the $sp - sp^2$ carbon – carbon bonds of the $C(CN)_2$ groups are: (A) 1.413 Å (0.45); (B) 1.421 Å (0.48);

(C) 1.425 Å (0.46); and for sp^2-sp^2 carbon–carbon bonds: (A) 1.410 Å (0.66); (B) 1.382 Å (0.58); (C) 1.391 Å (0.62). The correspondence between bond lengths and bond orders for (A) in particular are not convincing; both the approximations of the bond order calculation as well as the neglect of effects of the CH_2CN group may be questioned.

The C–O bond of 1.238 Å ("riding" corrected value of 1.254 Å) is close to the cyclopropanecarboxamide [9] value of 1.244 Å (uncorrected). The C2–C3 distance is somewhat large (as in the case of $KC_9N_5H_2$), while C3–C6 has a normal bond length.

The packing and coordination is shown in Fig. 2. Each cation is coordinated to six atoms (of different anions), forming a distorted trigonal prism. The oxygen atom of an anion is coordinated to two potassium ions, symmetry related by a glide plane along c (in $y=1/4$). N5, being *trans* to O, is also bonded to two cations, related to the former two by translation along a. Thus zigzag chains of coordinated atoms in the [001]-direction are linked along a by covalent bonds of the anion. The coordination bonds of N4 and N6 complete the rather complex three-dimensional network. No correlation (as found in $KC_9N_6H_2$) between $N\cdots K^+$ distances and $C-N\cdots K^+$ angles is observed.

Plane 1 of Table 5 is nearly parallel to b, and makes an angle of 69.5° with the c axis. This corresponds to an anion "thickness" of 3.47 Å. The shortest contacts between glide plane related ions in the [001]-direction are $C6\cdots N4=3.36$ Å, and $N4\cdots H1=2.61$ Å.

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