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The Crystal Structure of the Potassium Methoxide Adduct of 4-Methoxy-5,7-Dinitrobenzofurazan, a Meisenheimer Complex

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The crystal and molecular structure of the potassium methoxide adduct of 4-methoxy-5,7-dinitrobenzofurazan, $K^+[(CH_3O)_2C_6H(NO_2)_2N_2O]^-$, has been determined by X-ray diffraction techniques. The complex crystallizes as red, monoclinic crystals with $a = 15.152 \pm 0.015$, $b = 9.307 \pm 0.003$, $c = 17.780 \pm 0.010$ Å and $\beta = 106.22 \pm 0.08^\circ$, measured at room temperature. The space group is $P2_1/c$ and with eight molecules per unit cell, $D_c = 1.712$ g·cm⁻³ compared with $D_m = 1.718$ g·cm⁻³. The structure was solved using the symbolic addition procedure and Fourier syntheses. A refinement by full-matrix least-squares methods converged to a final R of 0.068 for the 4013 observed reflections measured using the stationary-crystal stationary-counter technique with molybdenum radiation. The two methoxyl groups are covalently bonded to the same ring carbon atom with an average C-O distance of 1.415 ± 0.006 Å. The sp^3 hybridized carbon atom in the ring produces distortions throughout the entire molecule. The electron withdrawing power of the coplanar furazan ring has a pronounced effect on the benzene ring system.

Introduction

Meisenheimer complexes, the stable intermediates in aromatic nucleophilic substitution reactions, have been of interest (Fendler, Fendler & Griffin, 1969; Crampton & Gold, 1966; Foster & Fyfe, 1966). The question is whether the intermediate formed by the attack of an alkoxide ion on nitroaryl compounds is a covalently bonded species or a charge-transfer complex. The recent crystal structure determinations of 1,1'-dimethoxy-2,4,6-trinitrobenzene potassium dihydrate by Ueda, Sakabe, Tanaka & Furusaki (1968) and the ethoxide adducts of 2,4,6-trinitrophenetole by Destro, Gramaccioli & Simonetta (1968) have confirmed the covalent nature of the intermediate.

The present study was undertaken in 1965 but was not completed until recently. The benzofurazan ring alters the possible resonance structures and the molecular geometry is distinctly different from the two previous studies. A preliminary account of our results has appeared recently (Messmer & Palenik, 1969).

Experimental

Orange-red platelets of the potassium methoxide adduct of 4-methoxy-5,7-dinitrobenzofurazan (KDMDNBF) were kindly supplied by Dr W. P. Norris. Preliminary Weissenberg photographs indicate that the crystals are monoclinic with the systematic absences of $h0l$ for $l = 2n + 1$ and $0k0$ for $k = 2n + 1$, indicating that the space group is $P2_1/c$.

A crystal of dimensions $0.23 \times 0.22 \times 0.10$ mm which was dipped in liquid nitrogen to minimize extinction

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effects was used for the intensity measurements. The unit-cell dimensions determined from diffractometer measurements are given in Table 1. Diffraction data for 8660 reflections with $2\theta < 60^\circ$ [Mo $K\alpha$ radiation, $\lambda(\alpha_1) = 0.70926 \text{ \AA}$] were measured using the stationary-crystal stationary-counter method. A 20-sec count was taken for each reflection with a zirconium filter in front of the counter window. A General Electric XRD-5 diffractometer controlled by a prepunched paper tape was used for measuring the intensity data. Four standard reflections were measured after every 140 reflections and showed no significant change or trend with time. A background curve as a function of 2θ was derived from the systematically absent reflections which were not affected by a radiation

Table 1. *Crystal data*

$a = 15.152 \pm 0.015 \text{ \AA}$	$\text{K}_8\text{H}_7\text{N}_4\text{O}_7$
$b = 9.307 \pm 0.003$	M.W. 310.26
$c = 17.780 \pm 0.010$	$Z = 8$
$\beta = 106.22 \pm 0.08^\circ$	$D_m = 1.718 \text{ g.cm}^{-3}$
Space group $P2_1/c(C2^5_n)$	$D_x = 1.712 \text{ g.cm}^{-3}$

streak. The 4013 reflections greater than or equal to 1.2 times the appropriate background count were considered to be observed; the remaining 3035 reflections were treated as unobserved reflections and entered as -0.1 times the background count. Since the linear absorption coefficient is only 4.8 cm^{-1} , no absorption corrections were applied (the maximum error in an intensity is 8%). The α_1 - α_2 doublet could be contained

Table 2. *Final atomic parameters and estimated standard deviations ($\times 10^4$) for $\text{K}^+[(\text{CH}_3\text{O})_2\text{C}_6\text{H}(\text{NO}_2)_2\text{N}_2\text{O}]^-$*

The estimated standard deviations are in parentheses. The temperature factor is in the form

$$\exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)].$$

Molecule A									
	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K(1)	2170 (1)	996 (1)	946 (1)	40 (0)	122 (2)	20 (0)	-0 (1)	10 (1)	-0 (1)
C(1)	6534 (3)	1631 (6)	4116 (3)	33 (2)	85 (6)	25 (14)	-8 (5)	10 (3)	-5 (5)
C(2)	5997 (3)	279 (5)	3823 (3)	39 (2)	77 (6)	27 (1)	-7 (6)	7 (3)	-6 (5)
C(3)	5104 (3)	215 (6)	3388 (3)	41 (2)	107 (7)	23 (1)	-21 (6)	12 (3)	-16 (5)
C(4)	4555 (3)	1429 (6)	3153 (3)	28 (2)	119 (7)	24 (1)	-11 (6)	8 (2)	-14 (5)
C(5)	4947 (3)	2808 (6)	3403 (3)	34 (2)	108 (6)	23 (1)	7 (6)	16 (3)	-5 (5)
C(6)	5874 (3)	2884 (5)	3877 (3)	32 (2)	91 (6)	27 (1)	-8 (5)	18 (3)	-3 (5)
C(7)	6331 (5)	1456 (10)	5414 (4)	61 (4)	237 (14)	32 (2)	-17 (11)	29 (4)	-6 (9)
C(8)	7168 (4)	1688 (8)	3027 (3)	56 (3)	161 (9)	30 (2)	41 (9)	26 (4)	2 (7)
O(1)	7310 (2)	1859 (4)	3846 (2)	30 (1)	129 (5)	32 (1)	-4 (4)	12 (2)	-8 (4)
O(2)	6940 (2)	1620 (5)	4943 (2)	42 (2)	130 (5)	22 (1)	-15 (5)	2 (2)	6 (4)
O(3)	7322 (3)	-1054 (5)	4248 (3)	45 (2)	102 (5)	54 (2)	24 (5)	-14 (3)	-1 (5)
O(4)	6042 (3)	-2187 (5)	3906 (3)	70 (3)	81 (5)	65 (2)	-25 (6)	13 (4)	9 (5)
O(5)	3354 (3)	69 (5)	2436 (3)	44 (2)	147 (6)	43 (2)	-36 (6)	-2 (3)	-26 (5)
O(6)	3200 (3)	2382 (5)	2438 (2)	35 (2)	174 (7)	39 (2)	31 (6)	-4 (3)	-18 (5)
O(7)	5333 (3)	4996 (4)	3705 (3)	44 (2)	84 (5)	56 (2)	8 (5)	17 (3)	-5 (5)
N(1)	6477 (3)	-1061 (5)	4013 (3)	50 (2)	87 (5)	33 (2)	1 (6)	3 (3)	-4 (5)
N(2)	3664 (3)	1290 (5)	2659 (2)	30 (2)	145 (7)	26 (1)	-12 (6)	9 (2)	-14 (5)
N(3)	4604 (3)	4121 (5)	3293 (3)	33 (2)	106 (6)	42 (2)	8 (5)	18 (3)	1 (5)
N(4)	6107 (3)	4206 (5)	4054 (3)	40 (2)	84 (5)	45 (2)	-8 (5)	17 (3)	-11 (5)

Molecule B									
	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K(1)	8715 (1)	605 (1)	5124 (1)	36 (0)	128 (2)	21 (0)	6 (1)	9 (1)	-4 (1)
C(1)	1463 (3)	3589 (5)	3947 (2)	30 (2)	98 (6)	16 (1)	-4 (5)	14 (2)	8 (4)
C(2)	1238 (3)	4943 (5)	3488 (2)	35 (2)	81 (5)	18 (1)	-15 (5)	19 (2)	-3 (4)
C(3)	1005 (3)	5068 (5)	2685 (2)	30 (2)	79 (5)	19 (1)	-12 (5)	18 (2)	-6 (4)
C(4)	950 (3)	3881 (5)	2191 (2)	32 (2)	78 (5)	17 (1)	4 (5)	8 (2)	1 (4)
C(5)	1149 (3)	2486 (5)	2531 (3)	30 (2)	79 (5)	23 (1)	-2 (5)	16 (2)	-7 (4)
C(6)	1382 (3)	2360 (5)	3357 (3)	33 (2)	81 (5)	23 (1)	3 (5)	16 (2)	14 (4)
C(7)	-57 (3)	3565 (7)	4120 (3)	33 (2)	138 (8)	35 (2)	-13 (7)	30 (3)	7 (6)
C(8)	3100 (4)	3813 (9)	4178 (4)	33 (2)	271 (14)	33 (2)	-9 (9)	21 (3)	-31 (9)
O(1)	2343 (2)	3592 (5)	4495 (2)	31 (1)	163 (6)	20 (1)	13 (5)	3 (2)	1 (4)
O(2)	905 (2)	3280 (4)	4446 (2)	40 (2)	118 (5)	20 (1)	1 (4)	22 (2)	17 (4)
O(3)	1382 (3)	6181 (4)	4628 (2)	57 (2)	126 (5)	23 (1)	-17 (5)	24 (2)	-26 (4)
O(4)	1174 (3)	7417 (4)	3573 (2)	82 (3)	85 (5)	34 (1)	-18 (6)	43 (3)	-9 (4)
O(5)	498 (3)	5314 (5)	1108 (2)	53 (2)	135 (6)	20 (1)	31 (5)	16 (2)	25 (4)
O(6)	670 (3)	3030 (5)	948 (2)	54 (2)	160 (6)	19 (1)	27 (6)	8 (2)	-34 (4)
O(7)	1374 (3)	280 (4)	2855 (2)	63 (2)	77 (4)	37 (1)	5 (5)	31 (3)	-3 (4)
N(1)	1264 (3)	6262 (5)	3913 (2)	40 (2)	88 (5)	24 (1)	-23 (5)	25 (2)	-19 (4)
N(2)	698 (3)	4079 (5)	1389 (2)	31 (2)	132 (6)	18 (1)	11 (5)	12 (2)	-2 (4)
N(3)	1141 (3)	1205 (5)	2217 (2)	47 (2)	82 (5)	27 (1)	-13 (5)	21 (3)	-9 (4)
N(4)	1522 (3)	1020 (5)	3551 (3)	58 (2)	103 (6)	28 (1)	10 (6)	28 (3)	11 (5)

within the counter window under the experimental conditions employed and no correction for the splitting was applied. The intensity data were reduced to a set of observed amplitudes on an arbitrary scale in the usual manner.

Determination and refinement of the structure

The structure was solved using the symbolic addition procedure of Karle & Karle (1966). The computer programs *FAME-MAGIC-LINK-SYMP* written by Dewar, Stone & Fleischer (1966) were used to carry out the symbolic addition. The two K^+ ions were easily located in an *E*-map computed with 509 *E* values whose signs were determined by the symbolic addition. Two Fourier syntheses were used to locate the remaining 38 atoms. In retrospect 34 atoms could have been located in the *E* map. The conventional *R* value was 0.24 after all 40 atoms were located.

The least-squares refinement was carried out using the full matrix for all calculations. Atomic scattering factors for K^+ , C, N, O, H were taken from *International Tables for X-ray Crystallography* (1962). The potassium curve was corrected for the real part of the anomalous dispersion using the value given by Cromer (1965). The weighting scheme used was $1/w=1$ if $F_{obs} \leq 18.5$ and $1/w=18.5/F_{obs}$, if $F_{obs} > 18.5$. The refinement was carried out with individual isotropic thermal parameters to an *R* of 0.11 and with anisotropic thermal parameters to an *R* of 0.068. The parameter shifts were all less than 0.1 of a standard deviation in the last cycle and the refinement was terminated. The final atomic parameters are given in Table 2 with the observed and calculated structure amplitudes being given in Table 3.

A final difference Fourier synthesis was essentially featureless except for peaks that could be assigned to hydrogen atoms. However, no attempt was made to

include the hydrogen atoms in a further refinement of the structure.

Discussion

The crystal consists of K^+ and $[(CH_3O)_2C_6H(NO_2)_2N_2O]^-$ ions separated by van der Waals contacts. Since there are two molecules of KDMDNBF per unit cell, there are two measurements of the bond distances and angles in the anion. The atomic numbering and average bond distances are given in Fig. 1, with the average bond angles in Fig. 2. The individual values are tabulated in Table 4. The differences between chemically equivalent bond lengths vary from 0.000 to 0.021 Å but chemically equivalent bonds are not significantly different.

The four C(methyl)-O(methoxy) bonds are equal within the experimental error with a mean value of 1.426 ± 0.007 Å, in agreement with the values of 1.427 ± 0.007 Å found by Destro *et al.* (1968) in the trinitrophenetole complex (KDETNB) and 1.44 ± 0.02 Å found by Ueda *et al.* (1968) in the trinitrobenzene case (KDMTNB). The O(methoxy)-C(ring) distances average 1.415 ± 0.007 Å, also in agreement with the values found in KDETNB of 1.417 ± 0.007 Å and in KDMTNB of 1.42 ± 0.02 Å. Since the O(methoxy)-C(methyl) bond is required to be a single covalent bond, a comparison with the O(methoxy)-C(ring) bond distance indicates that the latter is also a single covalent bond. The twelve angles about the two C(1) atoms average 109.5° , in agreement with an sp^3 hybridized C(1) atom. The plane defined by O(1)-C(1)-O(2) is normal within experimental error to the plane of the ring (see Table 5). The angular distortions about the C(1) atom can be attributed to either crystal packing or steric effects within the anion itself. Unfortunately, the presence of the NO_2 group adjacent

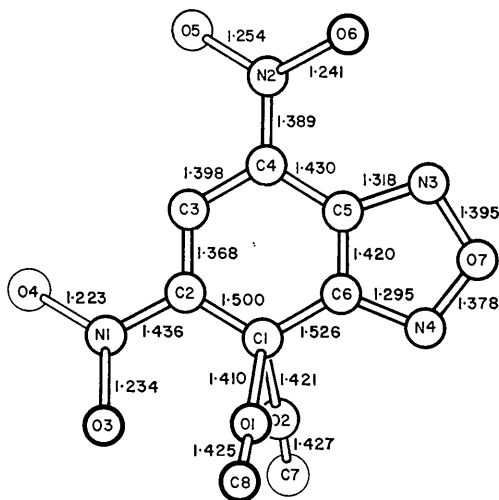


Fig. 1. The atomic numbering and average bond distance (in Å) in the $[(CH_3O)_2C_6H(NO_2)_2N_2O]^-$ anion.

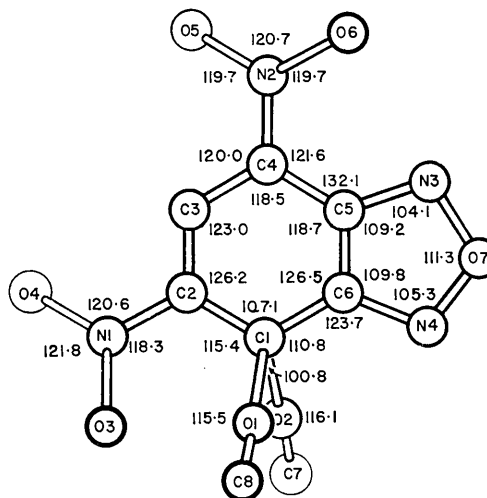


Fig. 2. The average bond angles (in degrees) in the $[(CH_3O)_2C_6H(NO_2)_2N_2O]^-$ anion.

of the sp^3 hybridized carbon atom into the ring makes the use of the term 'benzene ring' dubious. The C(1)–C(2) and C(1)–C(6) distances are close to the

values reported by Lide (1962) for a C(sp^3)–C(sp^2) single bond. Although KDETNB and KDMTNB were close to mm symmetry, the benzofurazan group intro-

Table 4. Bond distances (Å) and bond angles (°) in the two anions

The estimated standard deviations are given in parentheses.

Bond	In anion		Bond	In anion	
	A	B		A	B
C(1)–C(2)	1.510 (7)	1.489 (6)	C(1)–O(1)	1.406 (6)	1.413 (5)
C(2)–C(3)	1.359 (7)	1.377 (6)	C(1)–O(2)	1.426 (6)	1.415 (5)
C(3)–C(4)	1.397 (8)	1.399 (6)	O(1)–C(8)	1.421 (7)	1.428 (7)
C(4)–C(5)	1.432 (7)	1.428 (6)	O(2)–C(7)	1.419 (8)	1.435 (6)
C(5)–C(6)	1.424 (7)	1.416 (6)	C(2)–N(1)	1.436 (7)	1.435 (6)
C(6)–C(1)	1.518 (7)	1.534 (6)	N(1)–O(3)	1.231 (7)	1.236 (5)
C(5)–N(3)	1.321 (7)	1.315 (6)	N(1)–O(4)	1.224 (7)	1.222 (6)
C(6)–N(4)	1.295 (7)	1.295 (7)	C(4)–N(2)	1.396 (6)	1.381 (5)
N(3)–O(7)	1.402 (6)	1.388 (6)	N(2)–O(5)	1.251 (7)	1.257 (6)
N(4)–O(7)	1.377 (7)	1.376 (6)	N(2)–O(6)	1.235 (7)	1.246 (6)
Angle			Angle		
C(1)–C(2)–C(3)	126.0 (5)	126.3 (4)	C(2)–N(1)–O(3)	119.1 (5)	117.6 (4)
C(2)–C(3)–C(4)	123.4 (5)	122.6 (4)	C(2)–N(1)–O(4)	119.5 (5)	120.6 (4)
C(3)–C(4)–C(5)	118.1 (4)	118.9 (4)	O(3)–N(1)–O(4)	121.4 (5)	121.8 (4)
C(4)–C(5)–C(6)	118.9 (4)	118.5 (4)	C(3)–C(4)–N(2)	120.3 (4)	119.6 (4)
C(5)–C(6)–C(1)	126.3 (4)	126.6 (4)	C(5)–C(4)–N(2)	121.5 (4)	121.6 (4)
C(6)–C(1)–C(2)	107.1 (4)	107.1 (4)	C(4)–N(2)–O(5)	119.6 (4)	120.0 (4)
C(6)–C(1)–O(1)	109.7 (4)	110.7 (4)	C(4)–N(2)–O(6)	119.3 (4)	119.7 (4)
C(6)–C(1)–O(2)	110.9 (4)	109.2 (4)	O(5)–N(2)–O(6)	121.1 (5)	120.3 (4)
C(2)–C(1)–O(1)	115.4 (3)	113.8 (4)	C(4)–C(5)–N(3)	132.2 (5)	132.0 (4)
C(2)–C(1)–O(2)	112.7 (4)	115.4 (4)	C(5)–N(3)–O(7)	103.4 (4)	104.2 (4)
O(1)–C(1)–O(2)	101.4 (4)	100.5 (3)	N(3)–C(5)–C(6)	108.9 (4)	109.5 (4)
C(1)–O(1)–C(8)	115.6 (4)	115.8 (4)	N(3)–O(7)–N(4)	111.9 (4)	111.3 (4)
C(1)–O(2)–C(7)	116.4 (4)	115.3 (4)	O(7)–N(4)–C(6)	104.9 (4)	105.6 (4)
C(1)–C(2)–N(1)	117.0 (4)	117.9 (4)	N(4)–C(6)–C(5)	110.4 (4)	109.3 (4)
C(3)–C(2)–N(1)	117.0 (5)	115.4 (4)	N(4)–C(6)–C(1)	123.2 (5)	124.1 (4)

Table 5. Least-squares planes

Deviations (Å × 10³) in boldface type indicate atoms which were used to define the plane.

Atom	In anion		In anion		In anion		In anion		In anion	
	A	B	A	B	A	B	A	B	A	B
C(1)	-27	2	-59	5						
C(2)	9	0	-10	2			-4	0		
C(3)	11	0	12	0						
C(4)	-14	-2	-3	-3					-2	0
C(5)	-7	5	-6	5	-1	2				
C(6)	27	-5	7	-3	3	-3				
N(3)	1	-13	7	-14	-1	0				
N(4)	47	-18	21	-16	-3	3				
O(7)	40	-29	30	-28	3	-1				
N(1)	26	-42	-2	-39			16	-1		
O(3)	-274	-126	-318	-122			-6	0		
O(4)	280	133	261	135			-6	0		
N(2)	-108	-25	-78	-28					7	0
O(5)	-149	-76	-111	-79					-2	0
O(6)	-174	5	-135	2					-2	0
	Parameters of planes†									
$l \times 10^4$	-5446	9924	-5562	9924	-5611	9938	-3415	9880	-6056	9876
$m \times 10^4$	-0723	1150	-0731	1149	-0869	1010	0549	0504	-0670	1538
$n \times 10^4$	8356	0434	8278	0447	8232	0459	9383	1461	7930	0321
p (Å)	1.511	0.929	1.395	0.934	1.299	0.911	3.688	1.243	0.951	1.023
angle‡					1.5°	0.8°	15°	7°	4.3°	2.3°

† Equation of the plane is in the form: deviation (Å) = $lX + mY + nZ - p$ where X, Y, Z are the coordinates of the atom in Å referred to a, b and c^* .

‡ The angle the plane makes with the 'benzene' ring.

duces a second perturbation on the C₆ ring. The C(5)–C(6)–N(3)–O(7)–N(4) ring is planar and coplanar with the C₆ ring with a slight but not significant asymmetry. The C(1)–C(6), C(6)–C(5) and C(5)–C(4) bonds are all longer than the corresponding bonds on the opposite side of the ring. The lengthening of these bonds is a manifestation of the greater electron withdrawing power of the N–O–N system relative to two *meta* nitro groups. Unfortunately, there are no structural studies on other benzfurazan compounds for comparison with our results.

The two C–N(nitro) distances are significantly different as are the angles of twist [11° for O(3)–N(1)–O(4) and 3° for O(5)–N(2)–O(6)]. Presumably, the steric repulsion between the nitro group N(1)–O(3)–O(4) and the methoxy groups may account for the large twist relative to the nitro group O(5)–N(2)–O(6). Moreover, a similar shortening of the C–N(nitro) bond length for the NO₂ group *para* to the tetrahedral carbon atom was also noted by Destro *et al.* (1968) in KDETNB and by Ueda *et al.* (1968) in KDMTNB. The redistribution of charge introduced by the tetrahedral carbon atom in the C₆ ring must account for a large portion of the shortening.

There appears to be no regular coordination polyhedron about the K⁺ ion. The shortest contacts involving the potassium ion are listed in Table 6.

Table 6. *Potassium coordination* (< 3.0 Å)*

From K(1) to atom	Translation			Distance
O(5)				2.885 (5) Å
O(6)				2.969 (4)
O(3)	1-x	$\frac{1}{2}+y$	$\frac{1}{2}-z$	2.898 (4)
O(1')	x	$\frac{1}{2}-y$	$z-\frac{1}{2}$	2.694 (3)
O(2')	x	$\frac{1}{2}-y$	$z-\frac{1}{2}$	2.897 (3)
O(6')				2.959 (5)
From K(1') to atom				
O(1)				2.890 (4)
O(2)				2.782 (4)
O(3)				2.725 (5)
O(4')	1-x	1-y	1-z	2.929 (4)

N(4')	1-x	-y	1-z	2.906 (5)
O(5')	1+x	$\frac{1}{2}-y$	$\frac{1}{2}+z$	2.907 (4)
O(5')	1-x	$y-\frac{1}{2}$	$\frac{1}{2}-z$	2.784 (4)

* Primed atoms refer to molecule B.

All the intermolecular distances less than 3.6 Å were calculated and surveyed. Assuming the van der Waals radii for C of 1.6, for N of 1.5 and for O of 1.4 Å, there were no unusually short intermolecular contacts. In fact there are no C...C or N...N distances less than 3.6 Å and all the shorter contacts involve C...O, C...N, N...O or O...O. The only two contacts less than 3.0 Å are O(7) (in molecule A) to O(4) (in molecule A related by x, 1+y, z) of 2.818 Å and O(6) (in molecule A) to O(4) (in molecule A related by x, 1+y, z) of 2.954 Å.

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