

Diammonium potassium citrate, $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$

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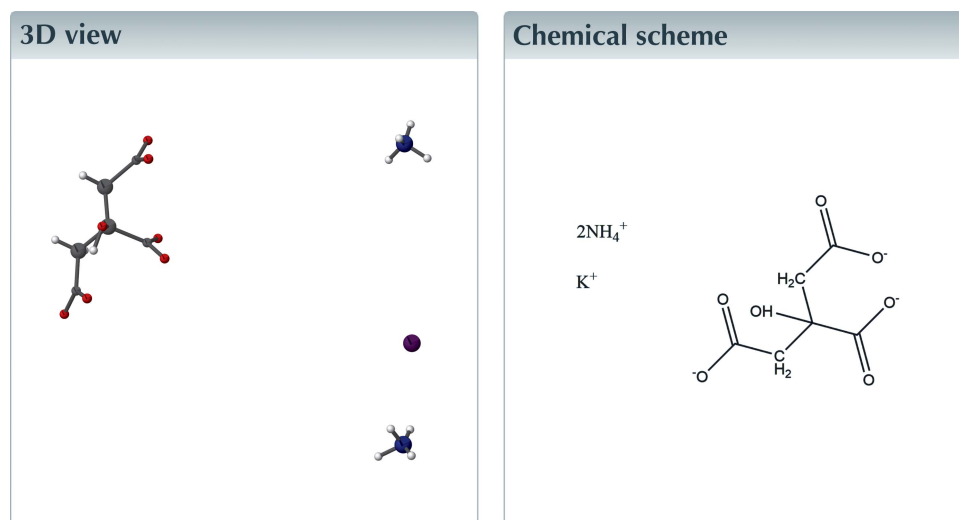
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Keywords: powder diffraction; density functional theory; citrate anion; ammonium; potassium.

Structural data: full structural data are available from iucrdata.iucr.org

The crystal structure of diammonium potassium citrate, $2\text{NH}_4^+\cdot\text{K}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}$, has been solved and refined using laboratory X-ray powder diffraction data and optimized using density functional theory. The KO_7 coordination polyhedra are isolated. The ammonium cations and the hydrophobic methylene sides of the citrate anions occupy the spaces between the coordination polyhedra. Each hydrogen atom of the ammonium ions acts as a donor in a charge-assisted $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots(\text{O},\text{O})$ or $\text{N}-\text{H}\cdots(\text{O},\text{O},\text{O})$ hydrogen bond. There is an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond in the citrate anion between the hydroxide group and one of the terminal carboxylate groups.



Structure description

A systematic study of the crystal structures of Group 1 (alkali metal) citrate salts has been reported by Rammohan & Kaduk (2018). The study was extended to ammonium citrates by Wheatley & Kaduk (2019). The title compound represents a further extension to mixed ammonium Group 1 citrates, specifically diammonium potassium citrate, $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$.

The structure of $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$ was solved and refined from powder X-ray data and optimized by density functional theory (DFT) calculations (see *Experimental* section) and is illustrated in Fig. 1. The root-mean-square Cartesian displacement of the non-hydrogen citrate atoms in the Rietveld-refined and DFT-optimized structures is 0.108 Å (Fig. 2). The maximum deviation is 0.211 Å, at O14. The r.m.s. displacement of the potassium ions is 0.054 Å. The r.m.s. displacements of the ammonium ions N19 and N20 are 0.111 and 0.151 Å respectively. The good agreement between the two structures is strong evidence that the experimental structure is correct (van de Streek & Neumann, 2014). All of the citrate bond distances, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury Mogul* Geometry Check (Macrae *et al.*, 2020). The

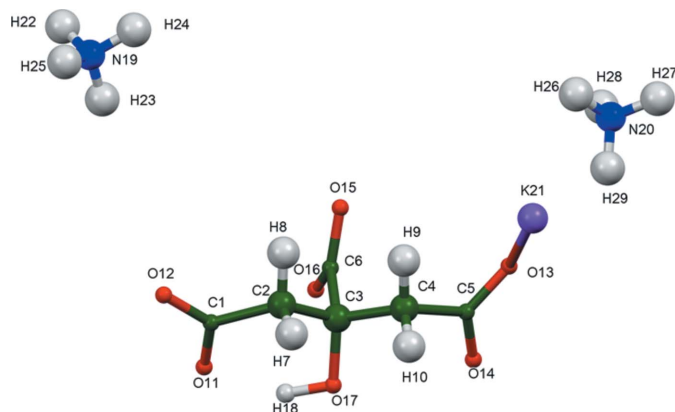


Figure 1
The asymmetric unit of $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$ with the atom numbering and 50% probability spheroids.

citrate anion occurs in the *trans,trans*-conformation (about C2–C3 and C3–C4), which is one of the two low-energy conformations of an isolated citrate anion (Rammohan & Kaduk, 2018). The central carboxylate group and the hydroxyl group exhibit a small twist [O16–C6–C3–O17 torsion angle = 7.0°] from the normal planar arrangement. The Mulliken overlap populations indicate that the K–O bonds are ionic.

The citrate anion doubly chelates to K21 through the hydroxyl group O17 and the terminal carboxylate group (atom O11). The anion doubly chelates to another potassium cation through the hydroxyl group and the other terminal carboxylate group (atom O14). Each oxygen atom bonds to a single potassium cation. As a result, K21 is seven-coordinate (capped trigonal prismatic), with a bond-valence sum of 0.98.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) method suggests that we might expect block morphology for diammonium potassium citrate. A 2nd order spherical harmonic preferred orientation model was included in the Rietveld refinement; the texture index was 1.179, indicating that preferred orientation was significant for this rotated flat sheet specimen.

The KO_7 coordination polyhedra are isolated (Fig. 3). The ammonium cations and the hydrophobic methylene sides of

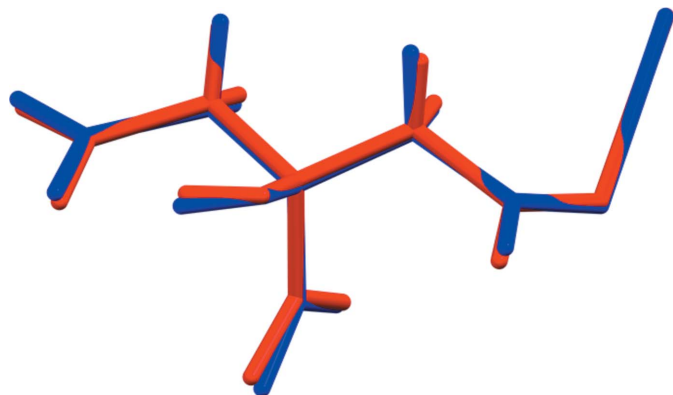


Figure 2
Comparison of the refined and optimized structures of $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$. The refined structure is in red, and the DFT-optimized structure is in blue.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O17–H18 \cdots O11	0.90	1.79	2.613 (19)	150
N19–H22 \cdots O14 ⁱ	0.77	2.18	2.88 (3)	150
N19–H22 \cdots O16 ⁱ	0.77	2.46	2.80 (3)	109
N19–H22 \cdots O17 ⁱ	0.77	2.33	2.74 (2)	114
N19–H23 \cdots O16 ⁱ	0.97	2.13	2.80 (3)	125
N19–H24 \cdots O15 ⁱⁱ	0.85	2.13	2.90 (3)	151
N19–H25 \cdots O14 ⁱⁱⁱ	1.07	1.77	2.80 (2)	162
N20–H26 \cdots O15 ^{iv}	0.94	2.58	3.26 (3)	130
N20–H26 \cdots O16 ^{iv}	0.94	1.91	2.84 (3)	173
N20–H27 \cdots O11 ^v	0.92	1.78	2.69 (3)	177
N20–H28 \cdots O13 ^{vi}	0.98	1.99	2.89 (3)	154
N20–H29 \cdots O12 ^{vii}	0.86	1.92	2.77 (3)	170

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, y, z-1$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (v) $x-1, y+1, z$; (vi) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (vii) $x, y+1, z$.

the citrate anions occupy the spaces between the coordination polyhedra. Each hydrogen atom of the ammonium ions acts as a donor in a charge-assisted N–H \cdots O hydrogen bond; there is one bifurcated M–H \cdots (O,O) bond and one trifurcated N–H \cdots (O,O,O) bond (Table 1). There is an intramolecular hydrogen bond between the hydroxide group and one of the terminal carboxylate groups. The N–H \cdots O hydrogen-bond energies were calculated by the correlation of Wheatley & Kaduk (2019), and the O–H \cdots O hydrogen bond energy was calculated by the correlation of Rammohan & Kaduk (2018).

Diammonium potassium citrate is isostructural to trimmonium citrate (Wheatley & Kaduk, 2019; Fig. 4). Comparison of the powder patterns (Fig. 5) confirms the similarity.

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2018). The

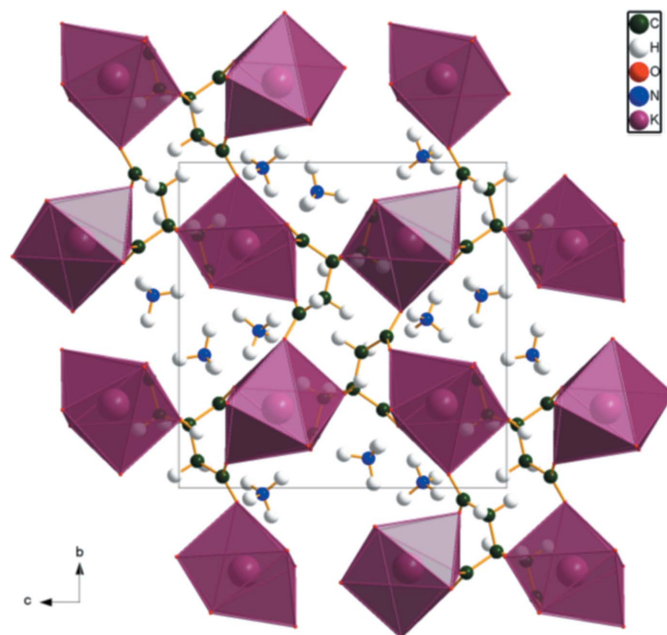


Figure 3
The crystal structure of $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$, viewed along the a axis.

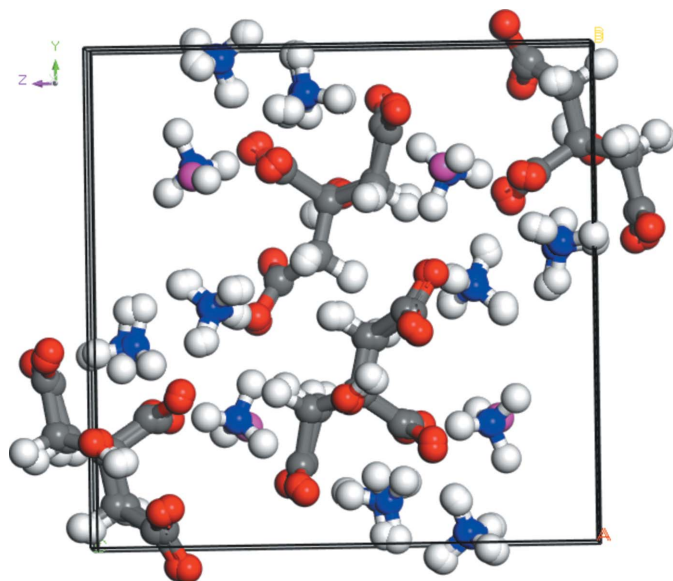


Figure 4
Overlay of the crystal structures of diammonium potassium citrate and triammonium citrate, showing that they are isostructural.

powder pattern of $(\text{NH}_4)_2\text{K}\text{C}_6\text{H}_5\text{O}_7$ was indexed using *N-TREOR* (Altomare *et al.*, 2013). A reduced-cell search of the cell of diammonium potassium citrate in the Cambridge Structural Database (Groom *et al.*, 2016) resulted in no hits.

Synthesis and crystallization

Diammonium potassium citrate was synthesized by dissolving 1.1217 g diammonium hydrogen citrate (Fisher Lot #995047) and 0.3279 g potassium carbonate (Sigma–Aldrich Lot #098 K0064) in ~5 ml of deionized water. The clear solution was dried at 363 K for two days to yield a white solid.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. A Rietveld plot is presented in Fig. 6.

The structure was solved using Monte Carlo simulated annealing techniques with *FOX* (Favre-Nicolin & Černý 2002)

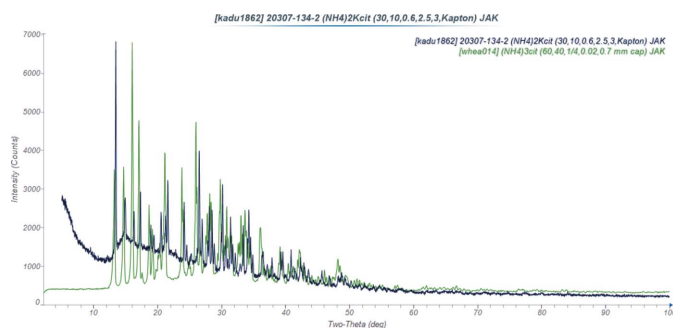


Figure 5
Comparison of the X-ray powder diffraction patterns of diammonium potassium citrate (black) and triammonium citrate (green).

Table 2
Experimental details.

Crystal data	
Chemical formula	$2\text{NH}_4^+ \cdot \text{K}^+ \cdot \text{C}_6\text{H}_5\text{O}_7^{3-}$
M_r	264.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	300
a, b, c (Å)	6.0238 (5), 13.2925 (6), 13.4155 (8)
β (°)	93.131 (4)
V (Å ³)	1072.60 (12)
Z	4
Radiation type	$K\alpha_{1,2}, \lambda = 1.54059, 1.54445$ Å
Specimen shape, size (mm)	Flat sheet, 25×25
Data collection	
Diffractometer	Bruker D2 Phaser
Specimen mounting	Standard sample holder with Kapton window
Data collection mode	Reflection
Scan method	Step
2θ values (°)	$2\theta_{\min} = 5.051, 2\theta_{\max} = 100.038,$ $2\theta_{\text{step}} = 0.020$
Refinement	
R factors and goodness of fit	$R_p = 0.056, R_{wp} = 0.072,$ $R_{\text{exp}} = 0.038, R(F^2) = 0.16190,$ $\chi^2 = 3.656$
No. of parameters	78
H-atom treatment	Only H-atom displacement parameters refined

The same symmetry and lattice parameters were used for the DFT calculations as for the powder diffraction study. Computer programs: *Data Collector* (Bruker, 2015), *FOX* (Favre-Nicolin & Černý, 2002), *GSAS-II* (Toby & Von Dreele, 2013), *DIAMOND* (Crystal Impact, 2015) and *Mercury* (Macrae *et al.*, 2020).

using a citrate anion, one K^+ cation and two ammonium cations as fragments. The structure was refined by the Rietveld method using *GSAS-II* (Toby & Von Dreele, 2013). The hydrogen atoms were included in fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systems, 2019). All C–C and C–O bond distances and all bond angles were restrained based on a *Mercury/Mogul* Geometry Check (Sykes *et al.*, 2011; Bruno *et al.*, 2004) of the molecule. The U_{iso} values of the atoms in the central and outer portions of the citrate were constrained to be equal, and the U_{iso} values of the hydrogen atoms were

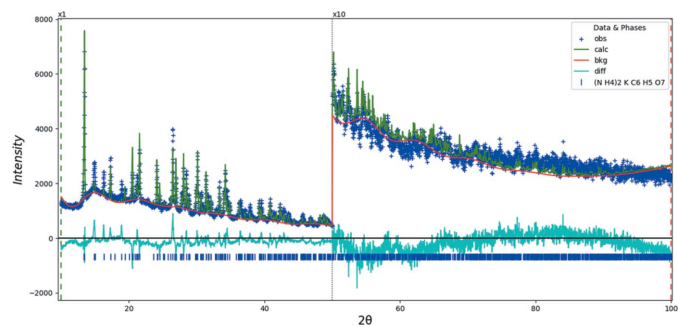


Figure 6
Rietveld plot for $(\text{NH}_4)_2\text{K}\text{C}_6\text{H}_5\text{O}_7$. The blue crosses represent the observed data points, and the green line is the calculated pattern. The cyan curve is the normalized error plot. The vertical scale has been multiplied by a factor of $10\times$ for $2\theta > 50.0^\circ$. The row of blue tick marks indicates the calculated reflection positions. The red line is the background curve.

constrained to be $1.3\times$ those of the atoms to which they are attached. A Chebyshev background function with three coefficients was used to model the background. A ten-term diffuse scattering function was used to describe the scattering from the capillary and any amorphous component. A density functional geometry optimization was carried out using *CRYSTAL14* (Dovesi *et al.*, 2014). The basis sets for the H, C, N, and O atoms were those of Gatti *et al.* (1994), and the basis set for K was that of Peintinger *et al.* (2013). The calculation was run on eight 2.1 GHz Xeon cores (each with 6 Gb RAM) of a 304-core Dell Linux cluster at IIT, using 8 *k*-points and the B3LYP functional, and took ~ 5 days.

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full crystallographic data

IUCrData (2020). 5, x200612 [https://doi.org/10.1107/S2414314620006124]

Diammonium potassium citrate, $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$

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Diammonium potassium citrate (I)

Crystal data

$2\text{NH}_4^+\cdot\text{K}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}$

$M_r = 264.27$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1bc$

$a = 6.0238\ (5)\ \text{\AA}$

$b = 13.2925\ (6)\ \text{\AA}$

$c = 13.4155\ (8)\ \text{\AA}$

$\beta = 93.131\ (4)^\circ$

$V = 1072.60\ (12)\ \text{\AA}^3$

$Z = 4$

$D_x = 1.637\ \text{Mg m}^{-3}$

$K\alpha_{1,2}$ radiation, $\lambda = 1.54059, 1.54445\ \text{\AA}$

$T = 300\ \text{K}$

Particle morphology: powder

white

flat_sheet, $25 \times 25\ \text{mm}$

Specimen preparation: Prepared at 363 K and 101 kPa

Data collection

Bruker D2 Phaser
diffractometer

Radiation source: sealed X-ray tube

Ni filter monochromator

Specimen mounting: standard sample holder
with Kapton window

Data collection mode: reflection

Scan method: step

$2\theta_{\min} = 5.051^\circ$, $2\theta_{\max} = 100.038^\circ$, $2\theta_{\text{step}} = 0.020^\circ$

Refinement

Least-squares matrix: full

$R_p = 0.056$

$R_{\text{wp}} = 0.072$

$R_{\text{exp}} = 0.038$

$R(F^2) = 0.16190$

4700 data points

Profile function: Finger-Cox-Jephcoat function

parameters U, V, W, X, Y, SH/L: peak

variance(Gauss) = $U\tan(\text{Th})^2 + V\tan(\text{Th}) + W$:

peak HW(Lorentz) = $X/\cos(\text{Th}) + Y\tan(\text{Th})$;

SH/L = S/L+H/L U, V, W in (centideg)², X & Y

in centideg 3.537, -1.411, 1.973, 2.482, 0.000,

0.048, Crystallite size in microns with

"isotropic" model: parameters: Size, G/L mix

1.000, 1.000, Microstrain, "generalized" model

$(10^6 * \Delta Q/Q)$ parameters: S400, S040, S004,

S220, S202, S022, S301, S103, S121, G/L mix

14695.026, 1.365, 571.072, 1337.970, 183.112,

-17.422, 2458.309, -70.133, 156.113, 1.000,

78 parameters

3 constraints

Only H-atom displacement parameters refined

Weighting scheme based on measured s.u.'s

$(\Delta/\sigma)_{\max} < 0.001$

Background function: Background function:

"chebyshev" function with 3 terms: 434.7(23),

-456(8), 208(14), Background Debye function

parameters: A, R, U: 11.0(8), 1.430, 0.100,

30(5), 2.270, 0.100, 95(21), 2.940, 0.100,

-4.4(4), 8.740, 0.100, -28(3), 1.970, 0.100,

-104(22), 2.880, 0.100, -8.4(18), 3.580, 0.100,

8.2(6), 14.000, 0.100, 2.5(7), 17.810, 0.100,

-0.4(8), 4.000, 0.100,

Preferred orientation correction: Simple

spherical harmonic correction Order = 0

Coefficients:

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.886 (3)	0.0374 (11)	0.8584 (14)	0.012 (2)*
C2	0.757 (2)	0.0812 (8)	0.9422 (10)	0.036 (7)*
C3	0.7931 (14)	0.1942 (7)	0.9608 (7)	0.036*
C4	0.669 (2)	0.2264 (7)	1.0527 (10)	0.036*
C5	0.656 (2)	0.3389 (7)	1.0721 (15)	0.0123*
C6	0.7043 (15)	0.2540 (13)	0.8679 (10)	0.0123*
H7	0.77670	0.04320	0.99960	0.046*
H8	0.57450	0.06940	0.92620	0.046*
H9	0.50370	0.20020	1.04630	0.046*
H10	0.71580	0.19530	1.10950	0.046*
O12	0.807 (2)	-0.0389 (9)	0.8144 (10)	0.0123*
O13	0.473 (2)	0.3730 (9)	1.0971 (13)	0.0123*
O14	0.827 (2)	0.3899 (9)	1.0640 (13)	0.0123*
O15	0.4977 (17)	0.2596 (12)	0.8525 (10)	0.0123*
O16	0.845 (2)	0.2922 (10)	0.8155 (9)	0.0123*
O17	1.0258 (16)	0.2132 (9)	0.9785 (9)	0.0123*
O11	1.061 (2)	0.0808 (9)	0.8363 (12)	0.0123*
H18	1.08650	0.16790	0.93840	0.016*
N19	0.825 (4)	0.6005 (14)	0.0743 (17)	0.040000*
N20	0.369 (4)	0.9754 (16)	0.7422 (14)	0.040*
K21	0.8415 (12)	0.7585 (6)	0.3071 (5)	0.040*
H22	0.93790	0.61560	0.05380	0.052000*
H23	0.90790	0.60060	0.13800	0.052000*
H24	0.69960	0.62600	0.08460	0.052000*
H25	0.79170	0.52180	0.07060	0.052000*
H26	0.30840	0.91370	0.72030	0.052000*
H27	0.26010	1.01030	0.77260	0.052000*
H28	0.38690	1.01050	0.67920	0.052000*
H29	0.50180	0.97670	0.76900	0.052000*

Geometric parameters (\AA , $^\circ$)

C1—C2	1.520 (3)	O15—K21 ^{iv}	2.887 (13)
C1—O12	1.254 (5)	O16—C6	1.238 (6)
C1—O11	1.249 (4)	O16—K21 ^v	2.659 (15)
C2—C1	1.520 (3)	O17—C3	1.431 (4)
C2—C3	1.536 (3)	O17—H18	0.899
C2—H7	0.923	O17—K21 ⁱⁱⁱ	3.004 (14)
C2—H8	1.119	O11—C1	1.249 (4)
C3—C2	1.536 (3)	O11—K21 ^v	2.956 (15)
C3—C4	1.537 (3)	H18—O17	0.899
C3—C6	1.549 (3)	N19—H22	0.774
C3—O17	1.431 (4)	N19—H23	0.966
C4—C3	1.537 (3)	N19—H24	0.847

C4—C5	1.520 (3)	N19—H25	1.065
C4—H9	1.055	N20—H26	0.937
C4—H10	0.899	N20—H27	0.916
C5—C4	1.520 (3)	N20—H28	0.977
C5—O13	1.251 (5)	N20—H29	0.861
C5—O14	1.244 (4)	K21—O12 ^{vi}	2.928 (14)
C6—C3	1.549 (3)	K21—O13 ^{vii}	2.799 (16)
C6—O15	1.253 (6)	K21—O14 ^{viii}	3.108 (16)
C6—O16	1.238 (6)	K21—O15 ^{iv}	2.887 (13)
H7—C2	0.923	K21—O16 ^v	2.659 (15)
H8—C2	1.119	K21—O17 ^{viii}	3.004 (14)
H9—C4	1.055	K21—O11 ^v	2.956 (15)
H9—H10	1.4957	H22—N19	0.774
H10—C4	0.899	H23—N19	0.966
O12—C1	1.254 (5)	H24—N19	0.847
O12—K21 ⁱ	2.928 (14)	H25—N19	1.065
O13—C5	1.251 (5)	H26—N20	0.937
O13—K21 ⁱⁱ	2.799 (16)	H27—N20	0.916
O14—C5	1.244 (4)	H28—N20	0.977
O14—K21 ⁱⁱⁱ	3.108 (16)	H29—N20	0.861
O15—C6	1.253 (6)		
C2—C1—O12	117.5 (5)	C4—C5—O13	117.3 (5)
C2—C1—O11	118.1 (4)	C4—C5—O14	118.0 (4)
O12—C1—O11	124.4 (4)	O13—C5—O14	124.7 (4)
C1—C2—C3	114.9 (4)	C3—C6—O15	117.4 (3)
C1—C2—H7	111.1	C3—C6—O16	116.8 (3)
C3—C2—H7	112.9	O15—C6—O16	125.9 (4)
C1—C2—H8	110.0	C6—O16—K21 ^v	141.1 (13)
C3—C2—H8	107.3	C3—O17—H18	102.1
H7—C2—H8	99.4	H22—N19—H23	83.8
C2—C3—C4	109.4 (4)	H22—N19—H24	139.7
C2—C3—C6	109.4 (4)	H23—N19—H24	106.1
C4—C3—C6	109.9 (4)	H22—N19—H25	113.9
C2—C3—O17	109.2 (4)	H23—N19—H25	97.5
C4—C3—O17	109.4 (4)	H24—N19—H25	103.5
C6—C3—O17	109.6 (4)	H26—N20—H27	108.0
C3—C4—C5	116.4 (5)	H26—N20—H28	102.0
C3—C4—H9	110.0	H27—N20—H28	105.1
C5—C4—H9	106.2	H26—N20—H29	119.0
C3—C4—H10	114.1	H27—N20—H29	118.4
C5—C4—H10	108.9	H28—N20—H29	101.9
H9—C4—H10	99.6		

Symmetry codes: (i) $x, -y+1/2, z+1/2$; (ii) $-x+1, y-1/2, -z+3/2$; (iii) $-x+2, y-1/2, -z+3/2$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+2, -y+1, -z+1$; (vi) $x, -y+1/2, z-1/2$; (vii) $-x+1, y+1/2, -z+3/2$; (viii) $-x+2, y+1/2, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O17—H18 \cdots O11	0.90	1.79	2.613 (19)	150
N19—H22 \cdots O14 ^v	0.77	2.18	2.88 (3)	150
N19—H22 \cdots O16 ^v	0.77	2.46	2.80 (3)	109
N19—H22 \cdots O17 ^v	0.77	2.33	2.74 (2)	114
N19—H23 \cdots O16 ^v	0.97	2.13	2.80 (3)	125
N19—H24 \cdots O15 ^{iv}	0.85	2.13	2.90 (3)	151
N19—H25 \cdots O14 ^{ix}	1.07	1.77	2.80 (2)	162
N20—H26 \cdots O15 ^{vii}	0.94	2.58	3.26 (3)	130
N20—H26 \cdots O16 ^{vii}	0.94	1.91	2.84 (3)	173
N20—H27 \cdots O11 ^x	0.92	1.78	2.69 (3)	177
N20—H28 \cdots O13 ^{xi}	0.98	1.99	2.89 (3)	154
N20—H29 \cdots O12 ^{xii}	0.86	1.92	2.77 (3)	170

Symmetry codes: (iv) $-x+1, -y+1, -z+1$; (v) $-x+2, -y+1, -z+1$; (vii) $-x+1, y+1/2, -z+3/2$; (ix) $x, y, z-1$; (x) $x-1, y+1, z$; (xi) $x, -y+3/2, z-1/2$; (xii) $x, y+1, z$.

(I_DFT)

Crystal data

C₆H₁₃KN₂O₇ $M_r = 264.27$ Monoclinic, $P2_1/c$ Hall symbol: $-P\ 2ybc$ $a = 6.0238\ \text{\AA}$ $b = 13.2926\ \text{\AA}$ $c = 13.4156\ \text{\AA}$ $\beta = 93.1310^\circ$ $V = 1072.60\ \text{\AA}^3$ $Z = 4$ $D_x = 1.637\ \text{Mg m}^{-3}$

Data collection

 $h = \rightarrow$ $l = \rightarrow$ $k = \rightarrow$ Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.87318	0.03565	0.86687	0.01230*
C2	0.72599	0.08314	0.94344	0.03600*
C3	0.76903	0.19496	0.96828	0.03600*
C4	0.63480	0.22315	1.05833	0.03600*
C5	0.63310	0.33416	1.08863	0.01230*
C6	0.68765	0.25946	0.87689	0.01230*
H7	0.75607	0.03931	1.01180	0.04600*
H8	0.55188	0.07301	0.91973	0.04600*
H9	0.46410	0.19855	1.04385	0.04600*
H10	0.70341	0.18054	1.12249	0.04600*
O12	0.80347	-0.04262	0.82266	0.01230*
O13	0.44391	0.37143	1.10579	0.01230*
O14	0.81343	0.38194	1.09793	0.01230*
O15	0.48252	0.26029	0.85377	0.01230*
O16	0.83050	0.30870	0.83032	0.01230*

O17	0.99968	0.21047	0.99448	0.01230*
O11	1.06265	0.07558	0.85523	0.01230*
H18	1.07924	0.17143	0.94538	0.01600*
N20	0.37168	0.98204	0.74458	0.04000*
K21	0.86217	0.75540	0.30100	0.04000*
H22	0.78800	0.60364	0.00308	0.05200*
H23	0.95796	0.62600	0.10404	0.05200*
N19	0.81121	0.59136	1.07827	0.04000*
H24	0.68536	0.63002	0.11046	0.05200*
H25	0.80797	0.51452	0.09426	0.05200*
H26	0.30306	0.91502	0.71752	0.05200*
H27	0.25619	1.01594	0.78942	0.05200*
H28	0.40466	1.03125	0.68656	0.05200*
H29	0.52233	0.96762	0.78341	0.05200*

Bond lengths (Å)

C1—C2	1.530	C6—O16	1.272
C1—O12	1.258	O17—H18	0.984
C1—O11	1.276	N20—H26	1.039
C2—C3	1.542	N20—H27	1.046
C2—H7	1.093	N20—H28	1.044
C2—H8	1.088	N20—H29	1.039
C3—C4	1.536	H22—N19 ⁱ	1.024
C3—C6	1.553	H23—N19 ⁱ	1.039
C3—O17	1.429	N19—H23 ⁱⁱ	1.039
C4—C5	1.531	N19—H22 ⁱⁱ	1.024
C4—H9	1.086	N19—H24 ⁱⁱ	1.030
C4—H10	1.093	N19—H25 ⁱⁱ	1.044
C5—O13	1.275	H24—N19 ⁱ	1.030
C5—O14	1.259	H25—N19 ⁱ	1.044
C6—O15	1.257		

Symmetry codes: (i) $x, y, z-1$; (ii) $x, y, z+1$.*Hydrogen-bond geometry (Å, °)*

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N20—H29 \cdots O12	1.039	1.751	2.770	165.64
N20—H28 \cdots O13	1.044	1.712	2.746	169.62
N20—H27 \cdots O11	1.046	1.697	2.742	176.08
N20—H26 \cdots O16	1.039	1.732	2.769	175.39
N19—H25 \cdots O14	1.044	1.763	2.796	159.41
N19—H24 \cdots O15	1.030	1.853	2.833	157.81
N19—H23 \cdots O16	1.039	1.741	2.764	167.24
N19—H22 \cdots O13	1.024	1.993	2.879	143.23
O17—H18 \cdots O11	0.984	1.756	2.632	146.38