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Creatininium hydrogen oxalate monohydrate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.097
Data-to-parameter ratio = 8.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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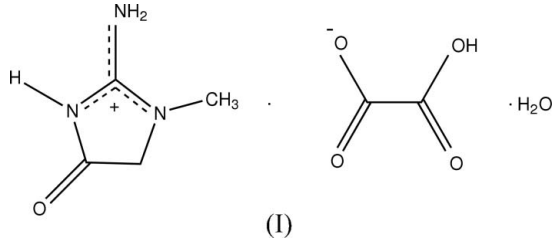
In the title compound, $C_4H_8N_3O^+\cdot C_2HO_4^-\cdot H_2O$, the asymmetric unit comprises one creatinium cation, one hydrogen oxalate anion and one water molecule. All non-H atoms lie on a crystallographic mirror plane and both the cation skeleton and the anion are therefore totally planar. In the crystal packing, an extended network of O—H···O and N—H···O hydrogen-bonding interactions results in the formation of two-dimensional sheets parallel to the *ac* plane.

Comment

Creatinine is formed by the metabolism of creatine in muscle tissue and is normally excreted in the urine as a metabolic waste. In renal physiology, creatinine clearance (CCr; Madaras & Buck, 1996) is the volume of blood plasma that is cleared of creatinine per unit time. Clinically, creatinine clearance is a useful measure for estimating the glomerular filtration rate (GFR) of the kidneys. Oxalic acid occurs naturally in quite a large number of plants. The human body also synthesizes oxalic acid from ascorbic acid (vitamin C). Oxalic acid may combine with calcium, iron, sodium, magnesium or potassium to form less soluble oxalate salts. Oxalates also occur naturally in plants. In continuation of our ongoing programme of structure determination of a series of creatinine complexes (Bahadur *et al.*, 2007), the structure of creatinine with oxalic acid, (I), was carried out and results are presented here.



(I)

The asymmetric unit of (I) contains one creatininium cation protonated at N3, one hydrogen oxalate anion and one water molecule (Fig. 1). All non-H atoms lie on a crystallographic mirror plane. As a consequence, the cation skeleton and anion are completely planar. The bond distances and angles in (I) are in normal ranges (Allen *et al.*, 1987) and are comparable to the corresponding values observed in similar structures (Bahadur *et al.*, 2007; Berrah *et al.*, 2005). The C2—N3 [1.362 (2) Å] bond length is increased, while the C2═N6 bond length is decreased [1.303 (2) Å], compared with the corresponding values of 1.349 (2) and 1.320 (3) Å, respectively, found in the neutral creatinine molecule (Smith & White, 2001).

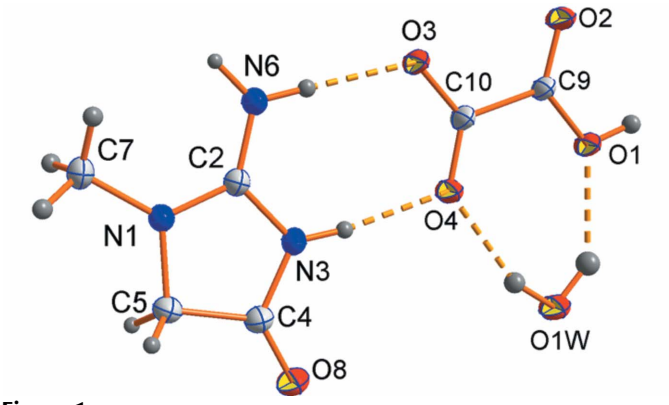


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius. Dashed lines denote hydrogen bonds. Only one component of the disordered methyl H atoms is shown.

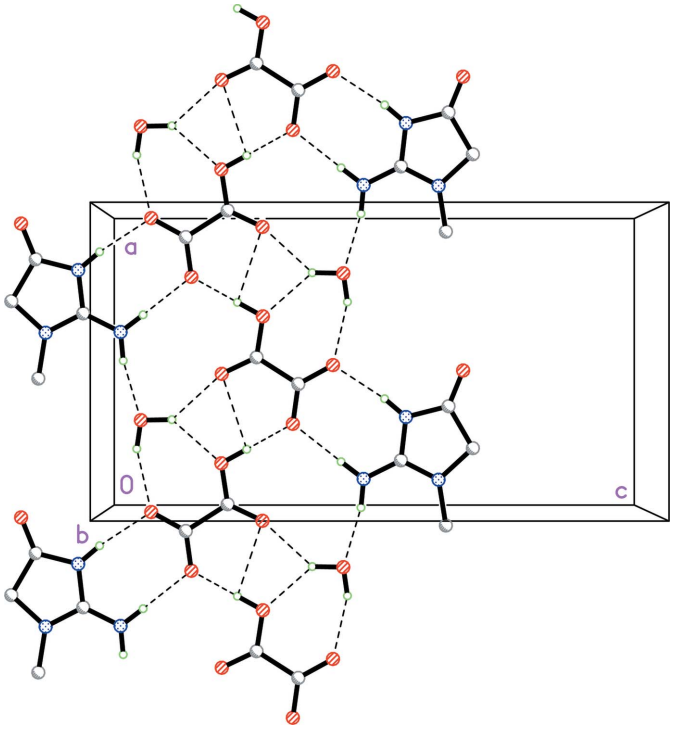


Figure 2
A partial packing diagram for (I), viewed down the *b* axis. Dashed lines indicate N—H···O and O—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity.

N—H···O hydrogen bonds link cation and anion into an ion pair, with the formation of an $R_2^2(8)$ motif (Bernstein *et al.*, 1995). These pairs are further interlinked by intermolecular O—H···O hydrogen bonds involving atoms O1 and O3 of the anions (Table 1). The water molecule plays an important role in hydrogen-bonding interactions. The water molecule as donor donates to the anion *via* intermolecular O—H···O hydrogen bonds and forms an $R_2^2(7)$ motif. In addition, the water molecule is also involved in an intermolecular O—H···O hydrogen bond with a neighbouring anion, which leads to a three-centred hydrogen-bonding pattern (Jeffrey & Saenger, 1991). As an acceptor, the water molecule interacts *via* hydrogen bonding with the creatininium cation (Fig. 2). Thus, the combination of O—H···O and N—H···O hydrogen bonds form an $R_3^3(10)$ motif. In the crystal packing, the hydrogen interactions link the ions and water molecules into a supramolecular two-dimensional sheet parallel to the *ac* plane.

Experimental

Creatinine and oxalic acid were mixed in a 1:1 stoichiometric ratio and dissolved in water. Crystals were obtained by slow evaporation.

Crystal data

$C_4H_8N_3O^+\cdot C_2HO_4^-\cdot H_2O$
M_r = 221.18
Orthorhombic, *Pnma*
a = 9.1728 (7) Å
b = 6.3520 (5) Å
c = 16.6567 (12) Å
V = 970.51 (13) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.14 mm^{-1}
T = 294 (2) K
0.21 × 0.18 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
6481 measured reflections
933 independent reflections
895 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.11
933 reflections
110 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*···O2^{i}	0.97 (4)	2.11 (4)	2.9094 (19)	138 (3)
O1*W*—H2*W*···O1	0.97 (4)	2.06 (4)	2.8484 (18)	138 (3)
O1*W*—H1*W*···O4	0.90 (3)	2.02 (3)	2.8840 (18)	159 (3)
O1—H1*O*···O3^{i}	0.91 (3)	1.64 (3)	2.5498 (16)	180 (3)
N6—H6*B*···O3	0.88 (3)	1.92 (3)	2.8012 (18)	179 (2)
N6—H6*A*···O1*W*^{ii}	0.90 (2)	1.94 (2)	2.825 (2)	168.7 (18)
N3—H3*N*···O4	0.88 (3)	1.92 (3)	2.7966 (18)	173 (2)

Symmetry codes: (i) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (ii) $x-1, y, z$.

All N-bound and O-bound H atoms were located in a difference Fourier map and their positions and isotropic displacement parameters were refined. All other H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å, and with U_{iso}(H) values of 1.5U_{eq}(C) for methyl H atoms or 1.2U_{eq}(C) for the other H atoms. The methyl group was allowed to rotate but not to tip. The H atoms of the methyl group are equally disordered over two positions across the mirror plane.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005) and *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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