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Bis(creatininium) 2,5-dicarboxybenzene-1,4-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.113
Data-to-parameter ratio = 16.1

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

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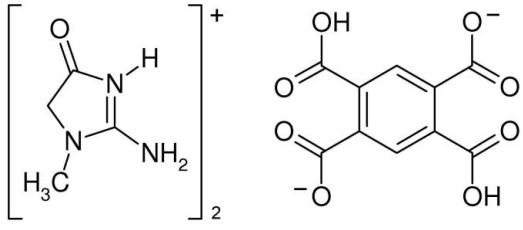
In the title compound, $2C_4H_7N_5O^+\cdot C_{10}H_6O_8^{2-}$, the benzene-1,2,4,5-tetracarboxylate anion lies on a centre of inversion, the two carboxyl H atoms being transferred to the endocyclic imine N atoms of two creatinine molecules. Hydrogen bonds link the ions into a two-dimensional network.

Comment

Proton-transfer reactions, involving intra- or intermolecular transfer of a proton from an acidic group to a basic group, are important in many chemical and biological processes. Investigations involving hydrogen-bonded clusters provide insight into the process of proton transfer (Cleland & Kreevoy, 1994; Chojnacki, 2003). Over the last decade, there have been an increasing number of reports of proton-transfer compounds including carboxylic acids and amines (Aghabozorg *et al.*, 2006; Aghabozorg *et al.*, 2005; Moghimi, Aghabozorg, Sheshmani, Kickelbick & Soleimannejad, 2005; Moghimi, Aghabozorg, Sheshmani & Soleimannejad, 2005; Moghimi, Aghabozorg, Soleimannejad & Ramezanipour, 2005; Moghimi, Sheshmani *et al.*, 2004). Creatinine has previously been used as a proton acceptor in the synthesis of some 1:1 proton-transfer compounds (Moghimi, Sharif & Aghabozorg, 2004; Soleimannejad *et al.*, 2005).



(I)

The title compound, $(C_4H_7N_5O)_2(C_{10}H_6O_8)$, (I), is a new proton-transfer system obtained from creatinine and benzene-1,2,4,5-tetracarboxylic acid. The crystal structure of (I) (Fig. 1) shows that protons from two of the carboxyl groups are transferred to the double-bonded N atoms of two creatinine molecules. This results in localization of the exocyclic C2═N2 double bond [1.3108 (18) Å] and the adjacent C2—N3 single bond [1.3612 (17) Å]. These values may be compared with the intermediate, delocalized, values in the parent neutral creatinine molecule [1.320 (3) and 1.349 (3) Å, respectively] (Smith & White, 2001). The anion lies on an inversion centre.

A number of N—H···O and O—H···O hydrogen bonds are observed in the structure (Table 1), linking the ions into two-dimensional networks parallel to the (100) plane (Fig. 2).

Experimental

A solution of benzene-1,2,4,5-tetracarboxylic acid (0.254 g, 1 mmol) and creatinine (0.113 g, 1 mol) was refluxed in water (50 ml) for 3 h. The solid residue was filtered and the filtrate was kept at 293 K. Colourless crystals of the title compound were obtained after a few days.

Crystal data

$2C_4H_7N_5O^+\cdot C_{10}H_6O_8^{2-}$
M_r = 480.40
Monoclinic, $P2_1/c$
a = 7.4863 (16) Å
b = 12.825 (2) Å
c = 10.846 (2) Å
β = 97.418 (16)°
V = 1032.6 (3) Å^3
Z = 2
D_x = 1.545 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
Prism, colourless
0.40 × 0.13 × 0.13 mm

Data collection

Stoe IPDS II diffractometer
φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.980, T_{max} = 0.985
8191 measured reflections
2757 independent reflections
2450 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 29.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.11
2757 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.3677P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O3	0.93 (2)	1.76 (2)	2.6965 (15)	176 (2)
N2—H2A···O2	0.88 (2)	1.99 (2)	2.8672 (17)	173.7 (18)
N2—H2B···O4^{i}	0.89 (2)	2.02 (2)	2.9004 (17)	170 (2)
O5—H5···O2^{ii}	0.86 (2)	1.75 (3)	2.5743 (15)	159 (2)

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

H atoms bound to C atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and allowed to ride during refinement, with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl group. The remainder of the H atoms were located in difference Fourier maps and refined isotropically without restraint. The range of Y—H distances is 0.87 (2)–0.99 (3) Å.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-RED* (Stoe & Cie, 2005); data reduction: *X-RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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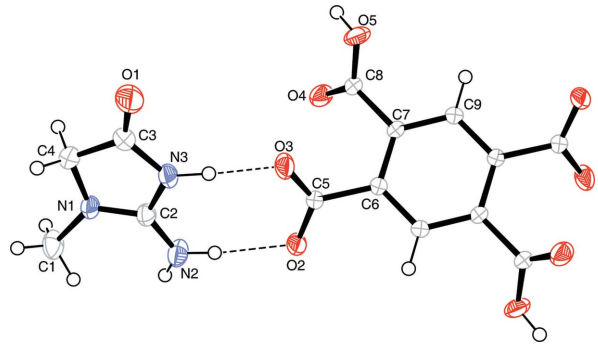

Figure 1
The structure of the ions of (I), showing displacement ellipsoids at the 50% probability level. Dashed lines denote hydrogen bonds. Unlabelled atoms are related to labelled atoms by the symmetry operator $(1-x, 1-y, 2-z)$.

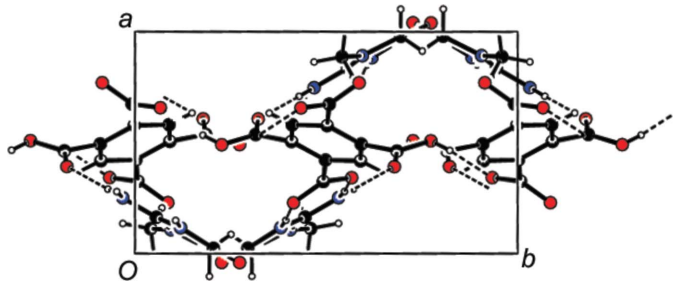

Figure 2
A view of (I) along the c direction, showing ions linked by hydrogen bonds (dashed lines) into a two-dimensional network.

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