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The dipotassium salt of *p*-nitrocatechol sulfate

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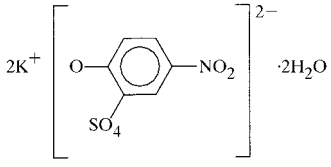
The structure of *p*-nitrocatechol sulfate dipotassium salt dihydrate (dipotassium 2-oxido-5-nitrophenyl sulfate dihydrate), $K_2(C_6H_3NO_7S)\cdot 2H_2O$, is reported. An accurate structural determination was needed to derive reliable restraints for use in macromolecular refinement at medium resolution of a protein–substrate complex.

Comment

The structure of the title compound, (I), was investigated to determine the structure of the chromogenic sulfatase substrate, which should be used to generate accurate restraints in the refinement of an enzyme–substrate complex. Experimentally determined bond lengths and angles to be used as restraints in macromolecular refinement should whenever possible be preferred to models derived from databases.



(I)

Sulfatases are enzymes involved in the degradation of sulfated substrates. The physiological importance of these enzymes is illustrated by seven distinct human disorders (Mehl & Jatzkewitz, 1964). Arylsulfatase A belongs to the sulfatase family and is a human lysosomal enzyme required for desulfatation of cerebroside 3-sulfate, a major constituent of myelin sheets. Naturally occurring mutants determine two genetic defects, namely metachromatic leukodystrophy (MLD), a fatal lysosomal storage disorder associated with severe neurological symptoms, and multiple sulfatase deficiency (MSD). There is no medical treatment known for patients suffering from either of these defects (von Figura *et al.*, 1998).

Compound (I) is the synthetic substrate for sulfatases and it is universally used as the standard substrate for *in vitro* tests. A substrate enzyme complex of sulfatases with *p*-nitrocatechol sulfate is of interest to explain the way in which it is bound to the enzyme. This previous knowledge is a key to understanding the catalytic mechanism (von Bülow *et al.*, 2000).

The packing of the *p*-nitrocatechol sulfate in the crystal structure shows planes that are connected by the potassium ions and the sulfate groups. Stacking of the aromatic rings and the interaction of an O atom from the nitro group with the cations form an additional contact between the layers.

The phenyl ring, the nitro group and the substituting O atoms all lie in a plane with an r.m.s. deviation of 0.0713 Å from planarity. The angle between the aromatic plane and the substituting sulfate (C1—O4—S1) is 118.9 (1)°. The distances between the S and three of the surrounding O atoms lie in the range of 1.441 (2) to 1.451 (2) Å, which are shorter than the values expected and which underscore Kálmán's bond-length rule (Kálmán, 1971).

The K1 and K2 atoms are coordinated by nine and eight O atoms, respectively (Table 1). In the case of K1, four of the nine O atoms belong to three sulfate groups, one belongs to the nitro group, one is a phenolic group and three of them are water molecules. In the coordination sphere of K2, there are five O atoms belonging to three sulfate groups, one oxygen belonging to the nitro group and two from water molecules (Fig. 1).

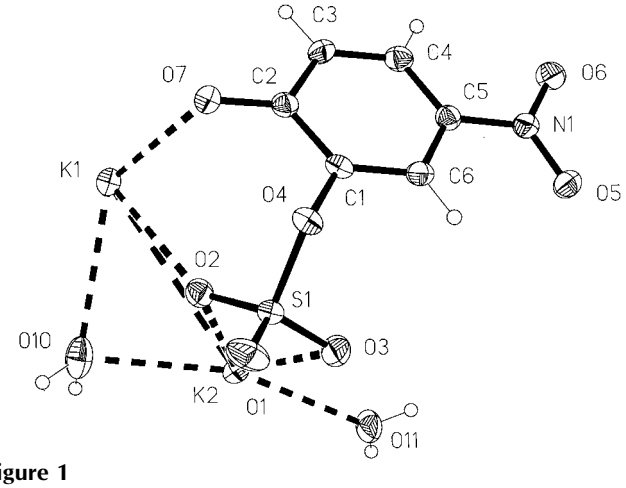


Figure 1
The asymmetric unit of the title compound showing 50% probability displacement ellipsoids.

Experimental

p-Nitrocatechol sulfate was purchased from Sigma. It was dissolved in H_2O and crystallized from a 1:1 mixture of ethanol/water by vapour diffusion at 277 K.

Crystal data

$K_2(C_6H_3NO_7S)\cdot 2H_2O$
M_r = 347.39
Triclinic, $P\overline{1}$
a = 6.9997 (12) Å
b = 7.4134 (14) Å
c = 12.348 (2) Å
α = 105.627 (3)°
β = 90.456 (4)°
γ = 95.905 (3)°
V = 613.37 (19) Å^3
Z = 2
D_x = 1.881 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
θ = 2.87–29.45°
μ = 0.984 mm^{-1}
T = 133 (2) K
Block, yellow
0.3 × 0.2 × 0.2 mm

Data collection

Stoe–Siemens–Huber four-circle diffractometer coupled to a CCD area detector
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.757, T_{max} = 0.828
9928 measured reflections
2993 independent reflections
2457 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.28°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 16$

Refinement

Refinement on F^2
$R(F)$ = 0.035
$wR(F^2)$ = 0.097
S = 1.075
3098 reflections
192 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0490P)^2 + 0.3697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.03
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.447 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.027 (3)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1345). Services for accessing these data are described at the back of the journal.

Table 1
Selected geometric parameters (Å).

K1···O7	2.7376 (16)	K2···O11	2.6731 (18)
K1···O3^{i}	2.7575 (17)	K2···O1^{iv}	2.6777 (16)
K1···O2^{ii}	2.8105 (16)	K2···O10	2.7603 (18)
K1···O6^{iii}	2.8617 (16)	K2···O4^{v}	2.7794 (15)
K1···O2	2.8630 (16)	K2···O2	2.7795 (15)
K1···O11^{i}	3.032 (32)	K2···O6^{iii}	2.7845 (16)
K1···O1^{ii}	3.0899 (18)	K2···O3	2.9834 (19)
K1···O10	3.141 (2)	K2···O1^{v}	3.0139 (17)
K1···O10^{ii}	3.3778 (19)		

Symmetry codes: (i) $x, y-1, z$; (ii) $1-x, -y, 1-z$; (iii) $1-x, -y, -z$; (iv) $1-x, 1-y, 1-z$; (v) $1+x, y, z$

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