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Poly[μ_2-acetato-diacetonitrile[μ_2-*N*,*N*′-bis(2-hydroxyphenyl)pyridine-2,6-dicarboxamide]potassium(I)]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.062
wR factor = 0.183
Data-to-parameter ratio = 13.1

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

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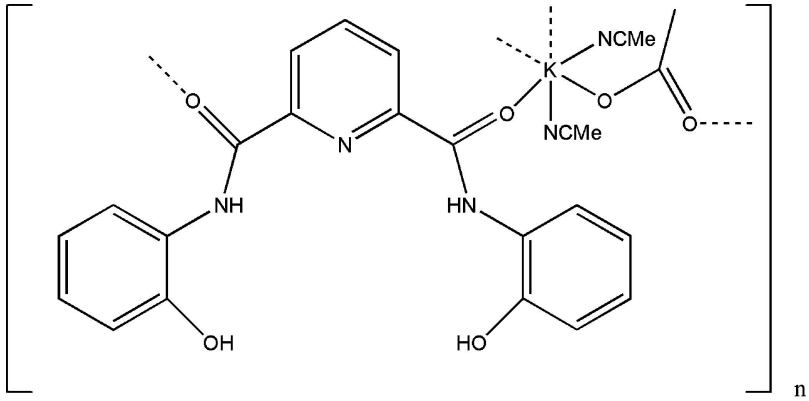
The title compound, $[K(C_2H_3O_2)(C_2H_3N)_2(C_{19}H_{15}N_3O_4)]$, is polymeric. Each K^+ ion adopts a distorted octahedral geometry, being linked to two 2,6-pyridinedicarboxamide ligands through carbonyl O atoms and to two acetate groups also through O atoms. The potassium coordination is completed by the N atoms of two acetonitrile ligands. The 2,6-pyridinedicarboxamide ligands are also involved in O—H···O hydrogen bonds.

Comment

Many derivatives of 2,6-pyridinecarboxamide show anti-inflammatory, antipyretic and analgesic activities (Singha & Sathyanarayana, 1997, and references therein). A series of Cu^{II}, Fe^{III}, Co^{III} and Ni^{II} (Chavez *et al.*, 1996, 1998; Marlin *et al.*, 1999; Hiratani & Taguchi, 1990) complexes containing 2,6-pyridinedicarboxamide ligands has been synthesized during the last two decades. The derivative *N*,*N*′-bis(2-hydroxyphenyl)pyridine-2,6-dicarboxamide has been synthesized previously (Marlin *et al.*, 2000). Several complexes of this ligand have been reported, two of which consist of the fully deprotonated pentadentate ligand with an Fe^{III} atom in the equatorial plane of the ligand, the coordination being completed by two monodentate ligands in axial sites (Marlin *et al.*, 2000). We are continuing to investigate the coordination properties of this ligand with a range of heavier main group and transition metals.



(I)

In this paper, we report the synthesis and crystal structure of the title polymeric potassium complex of *N*,*N*′-bis(2-hydroxyphenyl)pyridine-2,6-dicarboxamide, (I) (Fig. 1), where the ligand links two K^+ ions through the two carbonyl groups. The coordination environment of each K^+ centre is completed by coordination of one O atom of each of two acetate groups, and two terminal acetonitrile ligands. The

acetate groups act as linker groups between adjacent K^+ centres.

The K^+ centres display a distorted octahedral coordination geometry. The two K—O(carbonyl) distances (average 2.75 Å) are similar in length to the two K—O(acetate) distances (average 2.72 Å), and these four distances are significantly shorter than the two K—N(acetonitrile) distances (average 3.11 Å). The K—O(ethoxy) distances are similar to the average value of 2.67 Å previously reported for an ethanol-solvated dimeric potassium calixarene complex (Clague *et al.*, 1999).

In addition, the *N*,*N*′-bis(2-hydroxyphenyl)pyridine-2,6-dicarboxamide ligands are involved in O—H···O hydrogen bonds from the two hydroxyl groups to the linking acetate groups (Table 1).

Experimental

Compound (I) was synthesized by mixing solutions of *N*,*N*′-bis(2-hydroxyphenyl)pyridine-2,6-dicarboxamide and KO^{t}Bu, in the presence of potassium acetate (as a potential deprotonation reagent), in acetonitrile, in a 1:2 molar ratio, and stirring the resulting mixture for 2 h. Orange needles of (I) suitable for X-ray analysis were grown by slow diffusion of ethyl acetate into a dimethylformamide solution of (I).

Crystal data

$[K(C_2H_3O_2)(C_2H_3N)_2(C_{19}H_{15}N_3O_4)]$
M_r = 529.59
Orthorhombic, *Pbca*
a = 13.6100 (2) Å
b = 21.1060 (3) Å
c = 17.5730 (3) Å
V = 5047.89 (13) Å^3
Z = 8
D_x = 1.394 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 150 (2) K
Block, orange
0.17 × 0.13 × 0.1 mm

Data collection

Bruker NoniusKappa CCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.919, T_{max} = 0.974
30810 measured reflections
4445 independent reflections
3751 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.183
S = 1.05
4445 reflections
339 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 9.313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −1.04 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4O···O6^{i}	0.84	1.8	2.638 (3)	174
O1—H1O···O5^{i}	0.84	1.78	2.618 (3)	175

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

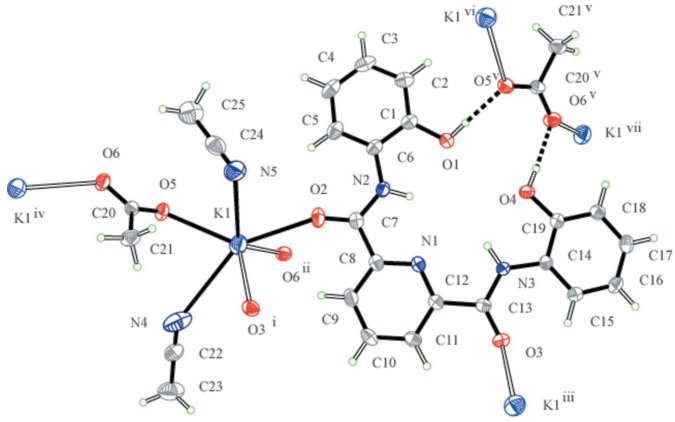


Figure 1
A view of (I), with 50% probability displacement ellipsoids, showing the polymeric nature of the structure. [Symmetry codes: (i) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (ii) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (iii) $\frac{1}{2}-x, \frac{3}{2}-y, 1-z$; (iv) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (v) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (vi) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (vii) $-x, 1-y, 1-z$.]

H atoms were constrained as riding atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H, and C—H =0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H, and with N—H = 0.88 Å and U_{iso}(H) = 1.2U_{eq}(N), and O—H = 0.84 Å and U_{iso}(H) = 1.2U_{eq}(O). There was no residual electron density above 0.5 e Å^3. The deepest hole is located 0.45 Å from atom K1.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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