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Piperazinium(2+) *fac*-tris(salicylato-κ^2O,O')-titanate(IV) monohydrate dimethylformamide solvate

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Key indicators

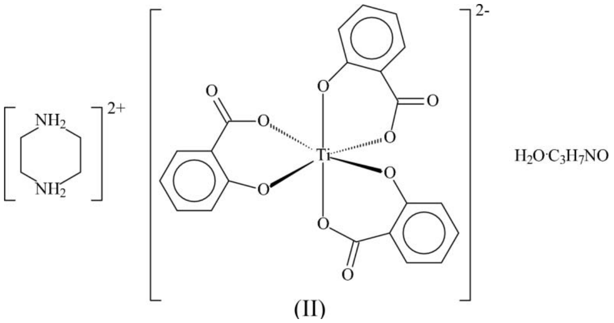
Single-crystal X-ray study
T = 295 K
Mean *σ*(C–C) = 0.007 Å
R factor = 0.076
wR factor = 0.234
Data-to-parameter ratio = 16.2

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

In the title compound, $(C_4H_{12}N_2)[Ti(C_7H_4O_3)_3]\cdot H_2O\cdot C_3H_7NO$, the Ti^{IV} atom is chelated by three salicylate ligands in an octahedral geometry, the complex anion being the *fac* isomer. The cations, complex anions and solvent molecules are linked by hydrogen bonds into a layer structure.

Comment

In the preceding study (Fu *et al.*, 2005), tetrabutyl titanate and salicylic acid were allowed to react in DMF solution for several months to yield bis(dimethylammonium) *mer*-tris-(salicylato)titanate(IV), (I). In the present study, piperazine was deliberately added to the reaction to hasten the formation of the corresponding piperazinium salt, (II), which has been isolated in a crystalline form as a monohydrated DMF solvate (Fig. 1). In (II), the Ti atom has the three salicylate groups in a facial–octahedral arrangement. Whether a meridional compound existed in the product was not investigated. The anion, cation and solvent molecules interact by hydrogen bonds (Table 2) to give rise to a layer structure.



(II)

Experimental

Tetrabutyl titanate (0.34 ml, 1 mmol), salicylic acid (0.40 g, 3 mmol) and piperazine (0.09 g, 1 mmol) were dissolved in DMF (10 ml). The clear yellow solution was set aside for 3 d for crytals of (II) to separate out.

Crystal data

$(C_4H_{12}N_2)[Ti(C_7H_4O_3)_3]\cdot H_2O\cdot C_3H_7NO$
M_r = 635.47
Triclinic, $P\overline{1}$
a = 9.021 (1) Å
b = 11.072 (1) Å
c = 15.067 (2) Å
α = 75.545 (2)°
β = 84.830 (2)°
γ = 87.146 (3)°
V = 1450.8 (3) Å^3

Z = 2
D_x = 1.455 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1049 reflections
θ = 2.3–20.1°
μ = 0.36 mm^{-1}
T = 295 (2) K
Rod, yellow
0.21 × 0.13 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
$T_{\rm min} = 0.383$, $T_{\rm max} = 0.972$
8209 measured reflections

5700 independent reflections
3379 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\rm max} = 26.3°$
$h = -9 \rightarrow 11$
$k = -13 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.234$
$S = 1.01$
5700 reflections
351 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1201P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—O1	1.864 (3)	Ti1—O5	2.008 (4)
Ti1—O2	2.007 (3)	Ti1—O7	1.878 (3)
Ti1—O4	1.863 (3)	Ti1—O8	2.024 (3)
O1—Ti1—O2	84.7 (1)	O2—Ti1—O8	84.5 (1)
O1—Ti1—O4	97.0 (1)	O4—Ti1—O5	86.0 (1)
O1—Ti1—O5	168.3 (1)	O4—Ti1—O7	93.9 (1)
O1—Ti1—O7	96.9 (2)	O4—Ti1—O8	170.4 (2)
O1—Ti1—O8	92.6 (2)	O5—Ti1—O7	94.2 (2)
O2—Ti1—O4	96.2 (1)	O5—Ti1—O8	84.6 (1)
O2—Ti1—O5	83.7 (1)	O7—Ti1—O8	85.1 (1)
O2—Ti1—O7	169.5 (1)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1w—H1w1$\cdots$O9	0.85	2.05	2.855 (5)	161
O1w—H1w2$\cdots$O8$^{\rm i}$	0.86	2.21	2.939 (5)	144
N1—H1$a\cdots$O3	0.90	1.91	2.808 (5)	177
N1—H1$b\cdots$O10$^{\rm ii}$	0.90	1.96	2.728 (6)	142
N2—H2$b\cdots$O6	0.90	1.83	2.726 (5)	174
N2—H2$a\cdots$O9$^{\rm iii}$	0.90	2.04	2.840 (6)	147

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+1$.

The three aromatic rings were refined as rigid hexagons of 1.39 Å sides so as to increase the data-to-parameters ratio to nearly 10. The $2\theta_{\rm max}$ was set to 52.5°; omitting high-angle data reduced the R values but reduced the data-to-parameters ratio unacceptably. The carbon-bound H atoms were positioned geometrically [C—H = 0.93 Å for the aromatic H atoms and C—H = 0.96 Å for the methyl H atoms; $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for the aromatic H atoms and 1.5$U_{\rm eq}$(C) for the methyl H atoms]. The nitrogen-bound H atoms were similarly allowed to ride on their parent atoms [N—H = 0.90 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(N)]. The water H atoms were placed at chemically sensible positions on the basis of hydrogen bonds [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(O)] and were fixed in position.

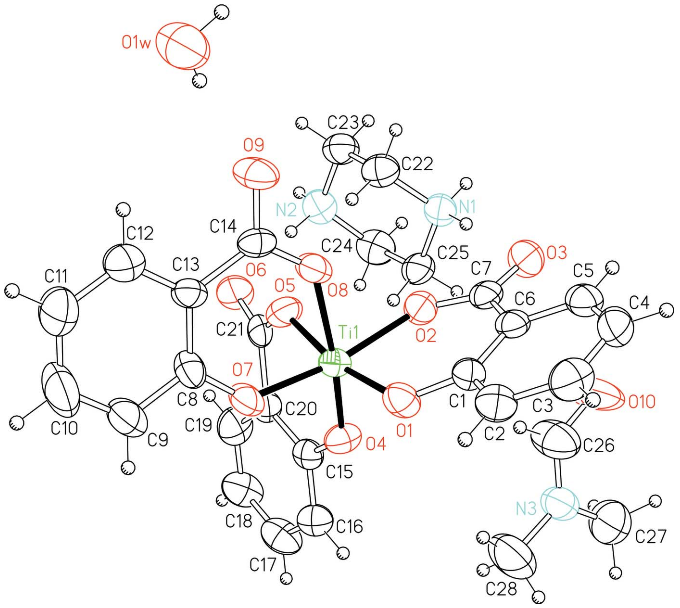


Figure 1
ORTEPII plot (Johnson, 1976), showing the numbering scheme of (II). Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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