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Key indicators

Single-crystal X-ray study
 $T = 273\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.057
 wR factor = 0.176
Data-to-parameter ratio = 12.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Piperazinium *fac*-tris(salicylato- $\kappa^2\text{O},\text{O}'$)-
titanate(IV) dimethylformamide solvate

In the title compound, $(\text{C}_4\text{H}_{12}\text{N}_2)[\text{Ti}(\text{C}_7\text{H}_4\text{O}_3)_3]\cdot\text{C}_3\text{H}_7\text{NO}$, the Ti^{IV} ion is chelated by three bidentate salicylate ligands in an octahedral geometry. The arrangement of the hydroxyl O atoms about Ti corresponds to the *fac* geometrical isomer. The component species are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a layered structure.

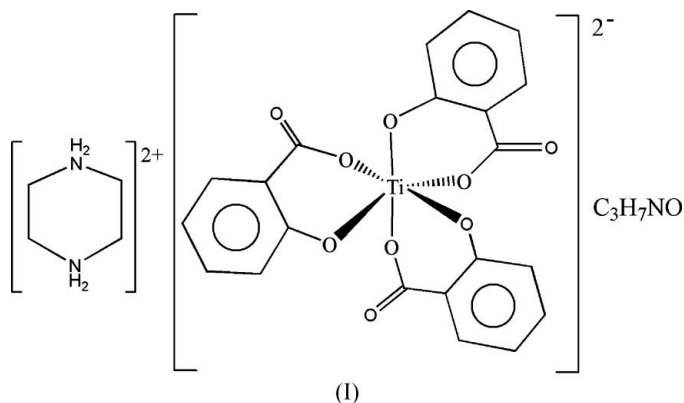
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Comment

The title compound, (I), arose from our continuing studies (Fu *et al.*, 2005*a,b*) of the reactions of tetrabutyltitanium(IV) and salicylic acid in dimethylformamide (DMF) solution, which have previously yielded crystals of bis(dimethylammonium) *mer*-tris(salicylato- $\kappa^2\text{O},\text{O}'$)titanate(IV), (II), and piperazinium(2+) *fac*-tris(salicylato- $\kappa^2\text{O},\text{O}'$)titanate(IV) monohydrate dimethylformamide solvate, (III). Piperazine was added to the reaction to hasten the formation of the corresponding piperazinium salts, (I) and (III). Whereas (III) was isolated with water and DMF solvent molecules of crystallization, (I) contains only DMF.



In (I), the Ti atom has the three bidentate salicylate groups in an octahedral arrangement, and the $[\text{Ti}(\text{C}_7\text{H}_4\text{O}_3)_3]^{2-}$ charge is balanced by a doubly protonated piperazinium cation (Fig. 1). For each chelating molecule, the $\text{Ti}-\text{O}_h$ (h = hydroxy) bond is shorter than the $\text{Ti}-\text{O}_c$ (c = carboxylate) bond (Table 1). The carboxylate O atoms engage the cations in $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions (Table 2), giving rise to a layered structure.

Experimental

Tetrabutyltitanium(IV) (0.34 ml, 1 mmol), salicylic acid (0.4 g, 3 mmol) and piperazine (0.09 g, 1 mmol) were dissolved in dimethylformamide (10 ml). The resulting clear yellow solution was set aside for 10 d for crystals of (I) to separate out.

Crystal data

(C₄H₁₂N₂)[Ti(C₇H₄O₃)₃].C₃H₇NO
M_r = 617.46
 Triclinic, *P* $\bar{1}$
a = 10.552 (2) Å
b = 10.622 (2) Å
c = 14.881 (3) Å
 α = 92.088 (4)°
 β = 104.987 (3)°
 γ = 118.429 (3)°
V = 1391.7 (5) Å³

Z = 2
D_x = 1.474 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 35 reflections
 θ = 2.2–25.0°
 μ = 0.37 mm⁻¹
T = 273 (2) K
 Block, yellow
 0.22 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.923, *T_{max}* = 0.946
 6808 measured reflections

4772 independent reflections
 3425 reflections with *I* > 2σ(*I*)
R_{int} = 0.027
 θ_{\max} = 25.0°
h = -12 → 12
k = -12 → 12
l = -17 → 8

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.057
wR (*F*²) = 0.177
S = 1.07
 4772 reflections
 381 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2 + 0.038P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.006$
 $\Delta\rho_{\max} = 0.47 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.36 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

Ti1—O1	1.868 (3)	Ti1—O6	1.881 (2)
Ti1—O2	1.983 (2)	Ti1—O7	2.041 (3)
Ti1—O5	2.001 (2)	Ti1—O8	1.865 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3B...O9	0.90	1.83	2.722 (4)	170
N3—H3A...O3 ⁱ	0.90	1.82	2.715 (4)	172
N2—H2B...O10 ⁱⁱ	0.90	1.90	2.665 (5)	142
N2—H2A...O4	0.90	1.92	2.782 (4)	159

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

H atoms were positioned geometrically (C—H = 0.93–0.96 Å and N—H = 0.90 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.2U_{\text{eq}}(\text{methyl carrier})$.

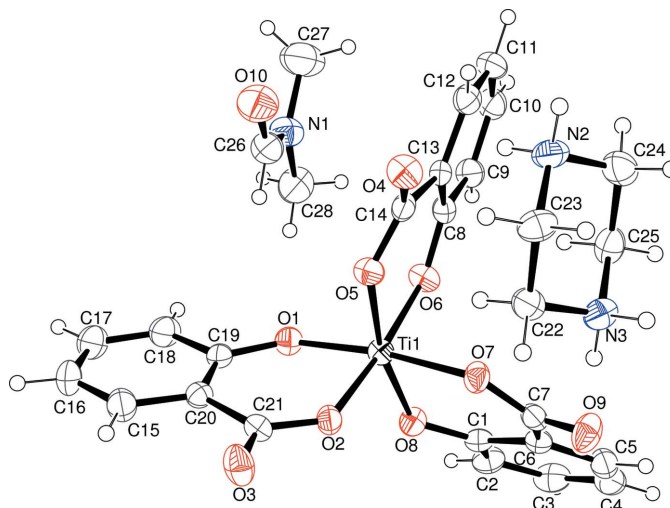


Figure 1

A view of (I), shown with 40% displacement ellipsoids (arbitrary spheres for H atoms).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2002); 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: *SHELXTL*.

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