Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Poly[μ_4-acetylacetonato-di-μ_3-acetylacetonato-pyridinetrisodium(I)]

Lionel Salmon, Pierre Thuéry* and Michel Ephritikhine

CEA/Saclay, DSM/DRECAM/SCM (CNRS URA 331), Bâtiment 125, 91191 Gif-sur-Yvette, France

Correspondence e-mail: pierre.thuery@cea.fr

Received 26 April 2006
Accepted 5 May 2006

Key indicators

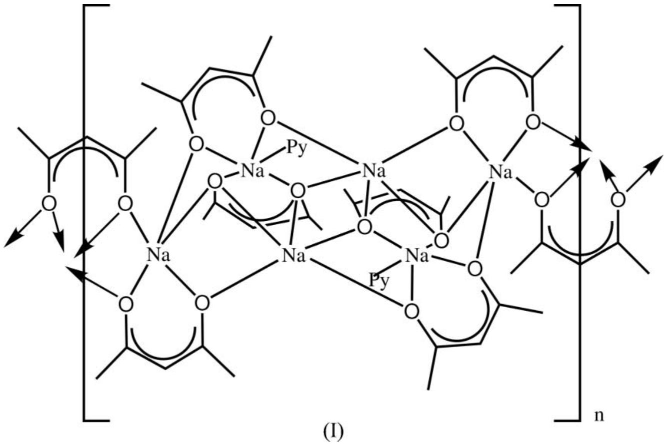
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.125
Data-to-parameter ratio = 15.7

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

In the title compound, $[Na_3(C_5H_7O_2)_3(C_5H_5N)]_n$, the two O atoms of one acetylacetonate (acac) ligand bridge four Na atoms in a μ_3 fashion, whereas the two other independent acac ligands bridge three Na atoms each, in a μ_2-fashion. All three Na atoms are in distorted square-pyramidal edge-sharing coordination polyhedra. The resulting assemblage is a polymeric chain directed along the *c* axis. This is the first example of Na—μ_3-O(acac) bonds to be reported.

Comment

In the course of our investigations of the synthesis and crystal structure of calix[*n*]arene complexes containing uranium ions (Salmon *et al.*, 2006), we fortuitously obtained crystals of the pyridine adduct of sodium acetylacetonate, (I). The structure of the corresponding monohydrate salt (II) has previously been described (Sahbari & Olmstead, 1983). Compound (I) is the first anhydrous sodium acetylacetonate to be structurally characterized.



(I)

The asymmetric unit of (I) contains three Na atoms, all of them pentacoordinate, three acetylacetonate ligands and one pyridine molecule (Fig. 1). Atom Na1 is bound to two μ_3-O atoms (O1 and O2) and two μ_2-O atoms (O3 and O4) which belong to two acetylacetonate ligands, and also to the pyridine molecule. Na2 is bound to three μ_3-O atoms [O1, O1^{i}, O2^{i}; symmetry code: (i) $1 - x, -y, 1 - z$] and two μ_2-O atoms [O4 and O6^{ii}; symmetry code: (ii) $x, y, z - 1$], whereas Na3 is bound to one μ_3-O (O2) and four μ_2-O atoms [O3, O5, O5^{iii}, O6^{iii}; symmetry code: (iii) $1 - x, -y, 2 - z$]. All Na atoms are in distorted square-pyramidal environments, with N1, O4 and O3 in apical positions and the metal atom located at 0.8514 (14), 0.7947 (12) or 0.3680 (11) Å from the basal plane (r.m.s. deviations 0.031, 0.115 and 0.177 Å, respectively). One

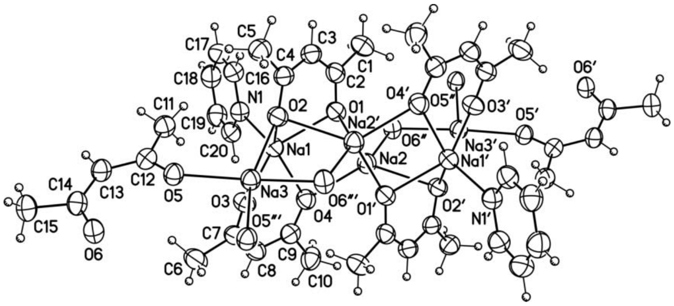


Figure 1
A view of compound (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (′) $1 - x, -y, 1 - z$; (″) $x, y, z - 1$; (‴) $1 - x, -y, 2 - z$.]

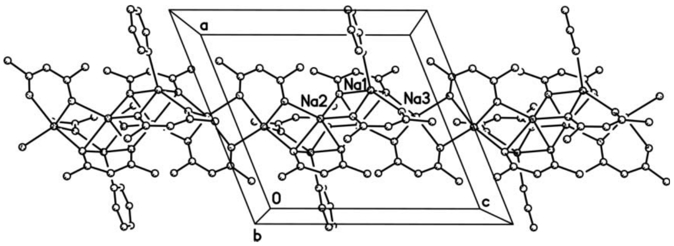


Figure 2
A view of the one-dimensional polymeric arrangement. Hydrogen atoms are omitted.

acac ligand thus has its two O atoms bonded in a μ_3-fashion (O1 and O2) and is bridging four Na atoms, whereas the two others correspond to μ_2-bridging, with the acac groups bound to three Na atoms only. The mean value of the Na—μ_2-O bond length of 2.30 (3) Å is slightly shorter than the average value of 2.35 (3) Å in (II), which may be due to the hexacoordinate environment of Na in that case. The μ_3-O atoms are at an average distance of 2.37 (7) Å from the Na atoms. A search of the Cambridge Structural Database (Version 5.27; Allen, 2002) shows that this is the first example of Na—μ_3-O(acac) bonds.

The structure of (I) consists of polymeric one-dimensional chains directed along the *c* axis (Fig. 2), with the acac group containing O5 and O6 and its equivalent through the inversion centre bridging tightly packed hexanuclear subunits through the Na3 atoms. All the contiguous Na coordination polyhedra are edge-sharing. No significant weak interactions are present between the chains, apart from van der Waals ones, and the packing corresponds to a space-filling value of 67.6% (Spek, 2003).

Experimental

NaH and calix[6]arene were purchased from the Aldrich Chemical Co. and Acros Organic Co., respectively, and used as supplied. $U(acac)_4$ was prepared as previously reported (Vallat *et al.*, 1990). An NMR tube was charged with calix[6]arene (7 mg, 0.011 mmol) and NaH (1.5 mg, 0.066 mmol) in pyridine (0.4 ml). $U(acac)_4$ (14.0 mg, 0.022 mmol) was added and the orange solution was refluxed overnight, affording few colourless crystals of (I) as a by-product.

Crystal data

$[Na_3(C_5H_7O_2)_3(C_5H_5N)]$
$M_r = 445.39$
Monoclinic, $P2_1/n$
$a = 10.8858$ (13) Å
$b = 18.969$ (2) Å
$c = 12.1338$ (12) Å
$\beta = 111.941$ (7)°
$V = 2324.1$ (5) Å^3

$Z = 4$
$D_x = 1.273$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 100$ (2) K
Irregular fragment, colourless
$0.14 \times 0.12 \times 0.09$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
Wide-frame φ scans
Absorption correction: none
16242 measured reflections

4353 independent reflections
2999 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.065$
$\theta_{\rm max} = 25.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.126$
$S = 1.04$
4353 reflections
277 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.6412P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Na1—O1	2.307 (2)	Na2—O4	2.332 (2)
Na1—O2	2.354 (2)	Na2—O6$^{\rm ii}$	2.286 (2)
Na1—O3	2.229 (2)	Na3—O2	2.336 (2)
Na1—O4	2.302 (2)	Na3—O3	2.305 (2)
Na1—N1	2.423 (2)	Na3—O5	2.321 (2)
Na2—O1	2.311 (2)	Na3—O5$^{\rm iii}$	2.291 (2)
Na2—O1$^{\rm i}$	2.447 (2)	Na3—O6$^{\rm iii}$	2.314 (2)
Na2—O2$^{\rm i}$	2.481 (2)		

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

H atoms were introduced at calculated positions as riding atoms, with C—H bond lengths of 0.93 (CH) or 0.96 Å (CH_3), and isotropic displacement parameters equal to $1.2U_{\rm eq}$(CH) or $1.5U_{\rm eq}$(CH_3) of the parent atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL2000* (Otwinowski & Minor, 1997); data reduction: *HKL2000*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1999). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sahbari, J. J. & Olmstead, M. M. (1983). *Acta Cryst.* C**39**, 1037–1038.
Salmon, L., Thuéry, P. & Ephritikhine, M. (2006). *Chem. Commun.*, 856–858.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Vallat, A., Laviron, E. & Dormond, A. (1990). *J. Chem. Soc. Dalton Trans.*, pp. 921–924.

Acta Cryst. (2006). E**62**, m1250–m1251 Salmon *et al.* • $[Na_3(C_5H_7O_2)_3(C_5H_5N)]$ **m1251**