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Tetraethylammonium bis(benzene-1,2-dithiolato)-(2-disulfanylbenzenethiolato)niobate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.082
Data-to-parameter ratio = 16.7

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

The title compound, $(C_8H_{20}N)[Nb(C_6H_4S_2)_2(C_6H_4S_3)]$, is the major product of the reaction of $(Et_4N)_4[Nb_2S_4(NCS)_8]$ and disodium benzene-1,2-dithiolate. The coordination environment of the Nb atom contains seven S atoms, *viz*. four S atoms from two benzene-1,2-dithiolate ligands and three S atoms from 2-disulfanylbenzenethiolate. The Nb—S distances are in the range 2.4391 (6)–2.5932 (7) Å.

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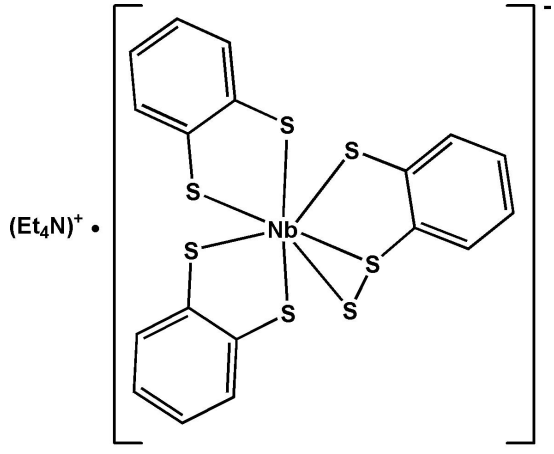
Comment

A ligand exchange in $[Nb_2S_4(NCS)_8]^{4-}$ was shown to be a useful approach to the synthesis of $Nb_2S_4L_4$ complexes, where L is a bidentate ligand such as dialkyldithiocarbamate, dialkyldithiophosphate or alkyldithiocarbonate (Sokolov *et al.*, 1994, 1996; Virovets *et al.*, 1993). However, in the case of L = benzene-1,2-dithiolate, the ligand exchange was found to be accomplished by $[Nb_2S_4(NCS)_8]^{4-}$ cluster decomposition to yield several products, among which the title Nb complex, (I), is the major one. The complex anion contains two benzene-1,2-dithiolates and one 2-disulfanylbenzenethiolate coordinated to the Nb atom (Fig. 1). 2-Disulfanylbenzenethiolate may be described as an η^3-ligand, with one Nb—S bond [2.593 (1) Å] longer than the other two [2.448 (1) and 2.439 (1) Å]. An $S_3C_6H_4^{2-}$ ligand formation may indicate sulfur elimination from S_2^{2-} as one of the intermediate steps.



(I)

Experimental

A mixture of $(Et_4N)_4[Nb_2S_4(NCS)_8]$ (200 mg, 0.154 mmol) and $Na_2S_2C_6H_4$ (115 mg, 0.618 mmol) was refluxed in dry acetonitrile (15 ml) for 3 h. The solvent was removed *in vacuo*; the residue was extracted with 10 ml of CH_2Cl_2 and purified on a chromatography column (1 × 10 cm, SiO_2, CH_2Cl_2). The first brown fraction was collected. Crystals were grown by evaporation of a solution of (I) in a $CHCl_3$–heptane mixture at room temperature (yield *ca* 5%).

Crystal data

$(C_8H_{20}N)[Nb(C_6H_4S_2)_2(C_6H_4S_3)]$
M_r = 675.86
Monoclinic, $P2_1/c$
a = 10.8255 (3) Å
b = 17.9119 (5) Å
c = 15.0647 (4) Å
β = 90.423 (1)°
V = 2921.05 (14) Å^3
Z = 4
D_x = 1.537 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6454 reflections
θ = 2.6–30.7°
μ = 0.93 mm^{-1}
T = 150 (2) K
Prism, black
0.30 × 0.20 × 0.15 mm

Data collection

Bruker–Nonius X8APEX CCD area-detector diffractometer
φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.768, T_{max} = 0.873
17 313 measured reflections
5338 independent reflections
4345 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.4°
$h = -12 \rightarrow 13$
$k = -21 \rightarrow 20$
$l = -18 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.04
5338 reflections
320 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.018P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Nb1—S11	2.4522 (6)	Nb1—S31	2.4479 (6)
Nb1—S12	2.5331 (6)	Nb1—S32	2.5932 (7)
Nb1—S21	2.4688 (6)	Nb1—S33	2.4391 (6)
Nb1—S22	2.4748 (6)	S32—S33	2.0561 (9)
S11—Nb1—S12	79.38 (2)	S31—Nb1—S33	93.80 (2)
S21—Nb1—S22	79.18 (2)	S32—Nb1—S33	48.11 (2)
S31—Nb1—S32	80.36 (2)		

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95–0.98 Å and U_{iso} = 1.2–1.5U_{eq}(parent atom). **Location of $\Delta\rho_{max}$?**

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2004); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

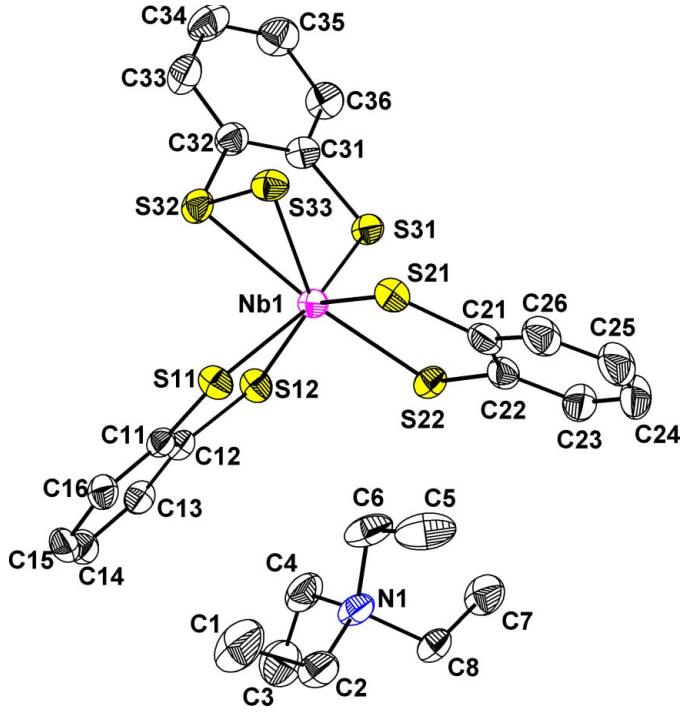


Figure 1
View of the title compound, with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

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