Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Sodium 1-hydroxypyridine-2(1*H*)-thiolate dihydrate

Andrew D. Bond

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, England

Correspondence e-mail: adb29@cam.ac.uk

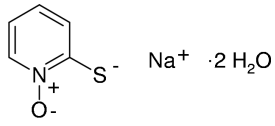
Received 6 August 2002
Accepted 19 August 2002
Online 23 August 2002

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.113
Data-to-parameter ratio = 8.9

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

The title compound, $Na^+\cdot C_5H_4NOS^-\cdot 2H_2O$, (I), contains extended cationic chains of empirical formula $[Na_3(C_5H_4NOS)_2(H_2O)_6]^+$, linked by $C_5H_4NOS^-$ moieties *via* an extensive hydrogen-bond network.



(I)

Experimental

Sodium pyrithione hydrate was obtained from the Aldrich Company and a single-crystal of (I) was grown by slow evaporation of an ethanolic solution. The crystal structure of (I) was shown to be representative of the bulk sample by comparison of simulated and measured powder XRD profiles (available from the author on request). Powder XRD measurements also show that an additional anhydrous sodium pyrithione phase with stoichiometry $Na(C_5H_4NOS)$ is present in the bulk sample (as supplied by Aldrich). Powder XRD at 373 K shows that the hydrate (I) reverts to this anhydrous phase at elevated temperatures. On standing in air, spontaneous rehydration is observed to regenerate polycrystalline (I).

Crystal data

$Na^+\cdot C_5H_4NOS^-\cdot 2H_2O$
M_r = 185.17
Triclinic, $P\overline{1}$
a = 7.916 (4) Å
b = 12.114 (6) Å
c = 13.280 (7) Å
α = 106.19 (2)°
β = 98.18 (1)°
γ = 95.43 (2)°
V = 1198.4 (11) Å^3
Z = 6
D_x = 1.540 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 40–50°
μ = 3.83 mm^{-1}
T = 296 (2) K
Block, colourless
0.30 × 0.15 × 0.15 mm

Data collection

Stoe Stadi-4 diffractometer
ω–θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.333, T_{max} = 0.563
2997 measured reflections
2997 independent reflections
2387 reflections with $I > 2\sigma(I)$
θ_{max} = 54.9°
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 14$
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.05
2997 reflections
337 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.4893P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0206 (10)

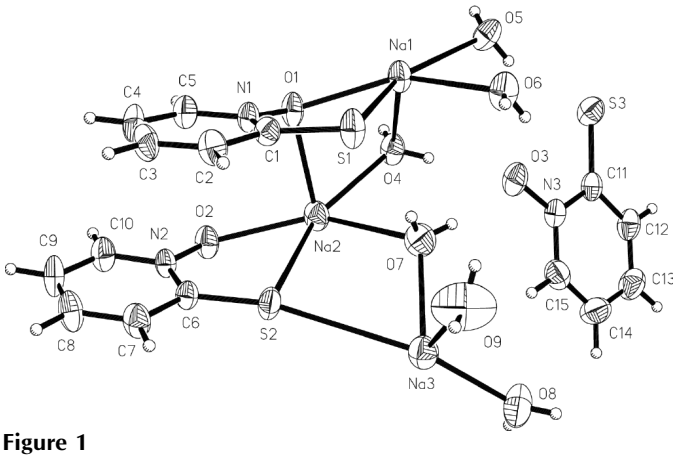


Figure 1
The asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms.

Exactly one hemisphere of unique data was collected, with no Friedel pairs measured; a merging R factor cannot therefore be calculated. Attempts were made at a later date to re-collect data using Mo $K\alpha$ radiation, but crystals were observed to become opaque and cease diffracting in the X-ray beam. H atoms bound to C atoms were placed geometrically and allowed to ride with $U_{iso}(H) = 1.2U_{eq}(C)$. Water H atoms were located in difference Fourier maps and refined with a common isotropic displacement parameter. The O—H distances were restrained to be equivalent, with s.u. values of 0.01 Å, with the distance refined as a single variable [final value: 0.81 (2) Å]. The H···H distances were also restrained to be 1.633 times this variable to ensure chemically reasonable H—O—H bond angles.

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL*97.

The author is grateful to the EPSRC for financial assistance.

References

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1993). *XP*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Stoe & Cie (1992). *DIF*4 (Version 7.09/DOS) and *REDU*4 (Version 7.03). Stoe & Cie, Darmstadt, Germany.