

References

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Acta Cryst. (1993). **C49**, 1031–1032

Structure of 2-(2'-Thienyl)pyridine at 193 K

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(Received 15 September 1992; accepted 10 November 1992)

Abstract

The molecular geometry of the thiophene and pyridine rings is very similar to other derivatives. Both rings are planar and almost in the same plane. The slight twist between the two planes arises from the intramolecular short contact between S and N. The C4'—C5' bond between the thiophene and pyridine rings is a partial double bond as a result of conjugation.

Comment

2-(2'-Thienyl)pyridine (Figs. 1 and 2) (Aldrich) crystallizes from methanol solution in the non-centrosymmetric space group $P2_12_12_1$. The rotation of 4.48° around the C2—C2' bond joining the thiophene and pyridine rings results in a conformationally chiral molecule. The twist between the two rings may be attributed to the short intramolecular contact between S and N ($N \cdots S = 2.933 \text{ \AA}$; the sum of the radii = 3.35 \AA). The bond C2—C2' [$1.469(3) \text{ \AA}$] between the thiophene and pyridine rings is shorter than a C—C single bond (1.54 \AA), implying a resonance between the rings. The C5'—C4' and C3'—C2' distances of $1.360(5)$ and $1.369(4) \text{ \AA}$, respectively, are slightly longer than a double bond, and the C4'—C3' distance of $1.407(4) \text{ \AA}$ is shorter than a single bond. The C—C and C—S bonds in the thiophene ring are in good agreement with those in 6-phenyl-4-(2-thienyl)-1H-pyrimidin-2-one (Tenon, Ebby, Degny, Viani & Bodot, 1988), 2-(5-methyl-3-thienyl)-2H,5H-pyrazolo[4,3-c]quinolin-3-one (Nakai, 1990) and 2-(5-methyl-2-thienyl)-2,5-dihydro-3H-pyrazolo[4,3-c]quinolin-3-one (Shiro, 1990). The molecular geometry of the six-membered ring is very similar to that found in 4-aminopyridine (Chao & Schempp, 1977), 2-amino-4-

methylpyridine (Kvick & Noordik, 1977), 2-amino-3-nitropyridine (Destro, Pilati & Simonetta, 1975) and 2-amino-3-(5-methyl-1,2,4-oxadiazol-3-yl)pyridine (Golič, Leban, Stanovnik & Tišler, 1979). However, the N—C6 bond length of $1.294(4) \text{ \AA}$ in the title compound is very close to a pure double bond (1.25 \AA). The C2—N—C6 angle [$116.3(2)^\circ$] is, as expected for a non-protonated ring N atom, smaller than 120° , and the N—C2—C3 [$123.0(2)^\circ$] and N—C6—C5 [$126.5(3)^\circ$] angles are enlarged (Kvick & Noordik, 1977). Both the rings are planar (maximum deviations from the planes of the thiophene and pyridine rings are 0.003 and 0.007 \AA , respectively).

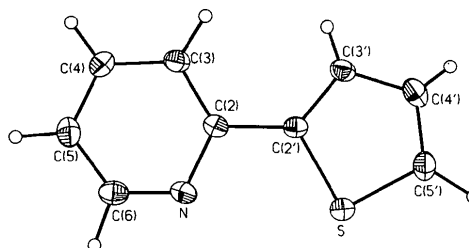


Fig. 1. View of C_9H_7NS showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 30% probability levels; H atoms are drawn as small circles of arbitrary radii.

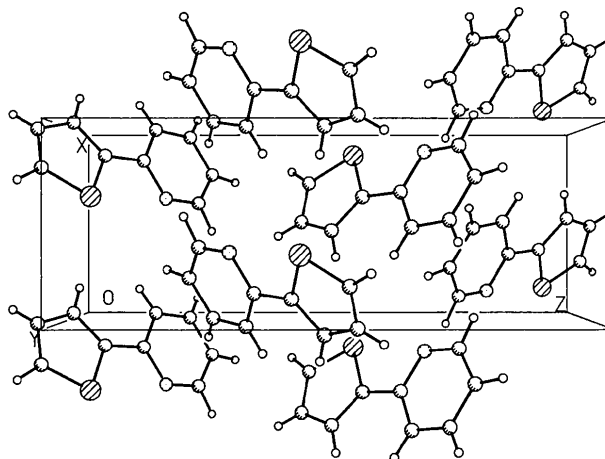


Fig. 2. Packing diagram viewed down the b axis.

Experimental

Crystal data

C_9H_7NS
 $M_r = 161.22$
 Orthorhombic
 $P2_12_12_1$
 $a = 5.769(3) \text{ \AA}$
 $b = 8.793(3) \text{ \AA}$
 $c = 15.695(6) \text{ \AA}$
 $V = 796.2(6) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.345 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 30 reflections
 $\theta = 18.66\text{--}30.06^\circ$
 $\mu = 0.32 \text{ mm}^{-1}$
 $T = 193 \text{ K}$
 Rectangular block
 $0.64 \times 0.29 \times 0.24 \text{ mm}$
 White

Data collection

| | |
|------------------------------------|---|
| Nicolet P3 diffractometer | $h = 0 \rightarrow 8; 0 \rightarrow 8$ |
| ω scans | $k = -12 \rightarrow 0; 0 \rightarrow 12$ |
| 2237 measured reflections | $l = -21 \rightarrow 0; 0 \rightarrow 21$ |
| 1099 independent reflections | 4 standard reflections |
| 1023 observed reflections | monitored every 96 reflections |
| $[F > 4.0\sigma(F)]$ | intensity variation: 1.28% |
| $R_{\text{int}} = 0.0086$ | |
| $\theta_{\text{max}} = 55.0^\circ$ | |

Refinement

| | |
|--|--|
| Refinement on F | $\Delta\rho_{\text{max}} = 0.38 \text{ e } \text{\AA}^{-3}$ |
| Final $R = 0.0349$ | $\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$ |
| $wR = 0.0499$ | Extinction correction: |
| $S = 1.9613$ | $F^* = F[1 + 0.002\chi F^2$ |
| 1023 reflections | $/\sin(2\theta)]^{-1/4}$ |
| 129 parameters | Extinction coefficient: |
| All H-atom parameters refined | 0.0012 |
| Calculated weights $w =$ | Atomic scattering factors |
| $1/[\sigma^2(F) + 0.0004F^2]$ | from <i>International Tables</i> |
| $(\Delta/\sigma)_{\text{max}} = 0.065$ | for <i>X-ray Crystallography</i> |
| | (1974, Vol. IV, Table 2.3.1) |

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *PLATON* (Spek, 1990).

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

| | x | y | z | U_{eq} |
|-----|-------------|-------------|-------------|-----------------|
| S | 0.3566 (1) | 0.04553 (8) | 0.04855 (4) | 0.0411 (2) |
| N | 0.3627 (4) | -0.1937 (3) | 0.1787 (1) | 0.0355 (6) |
| C2' | 0.1411 (4) | -0.0868 (3) | 0.0655 (1) | 0.0279 (6) |
| C3' | -0.0322 (5) | -0.0699 (3) | 0.0066 (2) | 0.0361 (7) |
| C4' | 0.0111 (5) | 0.0481 (3) | -0.0518 (2) | 0.0420 (8) |
| C5' | 0.2145 (6) | 0.1218 (3) | -0.0372 (2) | 0.0423 (8) |
| C2 | 0.1616 (4) | -0.1991 (3) | 0.1344 (1) | 0.0286 (6) |
| C3 | -0.0133 (5) | -0.3025 (4) | 0.1520 (2) | 0.0391 (8) |
| C4 | 0.0202 (6) | -0.4077 (4) | 0.2165 (2) | 0.0465 (9) |
| C5 | 0.2279 (5) | -0.4045 (4) | 0.2618 (2) | 0.0434 (8) |
| C6 | 0.3867 (5) | -0.2933 (4) | 0.2388 (2) | 0.0397 (8) |

Table 2. Geometric parameters (\AA , $^\circ$)

| | | | |
|-------------|-----------|-----------|-----------|
| S—C5' | 1.712 (3) | C2—C3 | 1.386 (4) |
| S—C2' | 1.723 (3) | C2—N | 1.353 (3) |
| C5'—C4' | 1.360 (5) | N—C6 | 1.294 (4) |
| C4'—C3' | 1.407 (4) | C3—C4 | 1.385 (4) |
| C3'—C2' | 1.369 (4) | C4—C5 | 1.393 (4) |
| C2'—C2 | 1.469 (3) | C5—C6 | 1.387 (4) |
| C5'—S—C2' | 92.3 (1) | C2'—C2—C3 | 121.9 (2) |
| S—C5'—C4' | 111.0 (2) | N—C2—C3 | 123.0 (2) |
| C5'—C4'—C3' | 113.3 (3) | C2—C3—C4 | 118.8 (3) |
| C4'—C3'—C2' | 112.9 (2) | C3—C4—C5 | 118.6 (3) |
| C3'—C2'—C2 | 128.9 (2) | C4—C5—C6 | 116.7 (3) |
| C3'—C2'—S | 110.5 (2) | C5—C6—N | 126.5 (3) |
| S—C2'—C2 | 120.6 (2) | C6—N—C2 | 116.3 (2) |
| C2'—C2—N | 115.1 (2) | | |

SHS acknowledges the support of the Robert A. Welch Foundation (grant F-0017).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55767 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1039]

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Acta Cryst. (1993). C49, 1032–1035

Structures of Tetradecyltrimethylammonium Salicylate Monohydrate (1) and Hexadecyltrimethylammonium Salicylate Monohydrate (2)

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(Received 10 August 1992; accepted 9 December 1992)

Abstract

(1) $[(C_{14}H_{29})(CH_3)_3N]^+ \cdot C_7H_5O_3^- \cdot H_2O$: the tetradecyl chain is fully extended in a zigzag form; the cation, anion and the water molecule are packed in layers with the tetradecyl chains parallel within a layer and antiparallel in alternate layers; the water molecule is hydrogen bonded to one of the carboxylate O atoms of the anion. (2) $[(C_{16}H_{33})(CH_3)_3N]^+ \cdot C_7H_5O_3^- \cdot H_2O$: the crystal packing, bond lengths and bond angles are all very similar to those of (1).

Comment

It has been reported recently that aqueous solutions of (1) and (2) show interesting viscoelastic and spinable properties (Imae, Hashimoto & Ikeda, 1990;