Instrumental Achievements

X-Ray Analysis

# Crystal Structure of Diquinolyl-8,8′-disulfide

**Akio YUCHI*, Motoo SHIRO** and Takashi YASUI***

**Nagoya Institute of Technology, Gokiso, Showa, Nagoya 466, Japan*
***Shionogi Research Laboratory, Shionogi & Co., Ltd., Fukushima, Osaka 553, Japan*

8-Quinolinethiol, a selective chelating reagent towards soft metal ions[1], is usually stored in its oxidized form, diquinolyl-8,8′-disulfide, for easy handling. In this study, we carried out an X-ray structural analysis for this compound. The molecule adopts an approximate $C_2$ symmetry: the atoms in one of the halves, quinolyl-8-sulfide, are represented by the primed numbers of the corresponding ones in the other. The H atoms were located on a difference Fourier map and refined with isotropic temperature factors set equal to the $B_{eq}$ values of the bonded atoms. The corresponding bond distances and angles in the two halves agree within experimental error. The dihedral angle between the ring planes is 78.8(1)°.

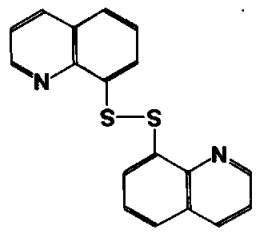


Fig. 1 Chemical structure.

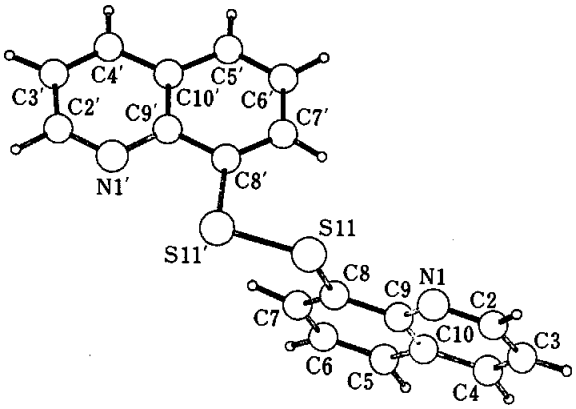


Fig. 2 Perspective view with atom-numbering.

Table 1 Crystal and experimental data

| | |
|---|---|
| Formula: $C_{18}H_{12}N_2S_2$ | |
| Formula weight=320.43 | |
| Crystal system: monoclinic | |
| Space group: $P2_1/c$ | $Z$=4 |
| $a$=13.602(2) Å | |
| $b$=9.540(1) | $\beta$=116.37(1)° |
| $c$=13.112(2) | |
| $V$=1524.4(3) Å$^3$ | |
| $D_x$=1.396 g/cm$^3$ | |
| $R$=0.048 | |
| No. of reflections used=2134 | |
| Measurement: Rigaku AFC-5R | |
| Program system: private (XPACK86 SHIONOGI) | |
| Structure determination: MULTAN87 | |
| Refinement:block-diagonal least-squares | |

Table 2 Final coordinates (×10$^4$) of non-H atoms

| Atom | $x$ | $y$ | $z$ | $B_{eq}$/10$^2$ Å$^2$ |
|---|---|---|---|---|
| N1 | 8310(2) | 1913(3) | -297(2) | 508(9) |
| C2 | 8824(3) | 2672(4) | -756(3) | 659(15) |
| C3 | 9969(3) | 2741(4) | -331(3) | 675(16) |
| C4 | 10619(3) | 2012(4) | 620(3) | 610(13) |
| C5 | 10718(3) | 357(4) | 2132(3) | 589(12) |
| C6 | 10185(3) | -429(4) | 2586(3) | 597(12) |
| C7 | 9037(3) | -451(3) | 2112(3) | 495(10) |
| C8 | 8425(2) | 334(3) | 1162(2) | 423(9) |
| C9 | 8959(2) | 1172(3) | 659(2) | 419(9) |
| C10 | 10119(2) | 1177(3) | 1149(3) | 479(10) |
| S11 | 6966(1) | 395(1) | 454(1) | 471(3) |
| N1′ | 5884(2) | -3429(3) | 2100(2) | 458(8) |
| C2′ | 5569(3) | -4477(3) | 2537(3) | 529(11) |
| C3′ | 5617(3) | -5891(3) | 2275(3) | 571(12) |
| C4′ | 6008(2) | -6224(3) | 1512(3) | 519(11) |
| C5′ | 6768(2) | -5386(3) | 200(3) | 489(10) |
| C6′ | 7083(3) | -4284(3) | -243(3) | 508(10) |
| C7′ | 7014(2) | -2890(3) | 83(3) | 475(10) |
| C8′ | 6617(2) | -2628(3) | 855(2) | 410(9) |
| C9′ | 6268(2) | -3748(3) | 1329(2) | 385(8) |
| C10′ | 6354(2) | -5146(3) | 1004(2) | 422(8) |
| S11′ | 6480(1) | -937(1) | 1359(1) | 487(3) |

$B_{eq}=(4/3)\Sigma_i\Sigma_j\beta_{ij}(\boldsymbol{a}_i\cdot\boldsymbol{a}_j)$.

Table 3 Bond distances (Å) and angles (°)

| | | | |
|---|---|---|---|
| N1 - C2 | 1.323(5) | N1 - C9 | 1.366(4) |
| C2 - C3 | 1.403(6) | C3 - C4 | 1.357(6) |
| C4 - C10 | 1.413(6) | C5 - C6 | 1.351(6) |
| C5 - C10 | 1.416(6) | C6 - C7 | 1.401(6) |
| C7 - C8 | 1.373(5) | C8 - C9 | 1.424(4) |
| C8 - S11 | 1.780(3) | C9 - C10 | 1.415(5) |
| N1' - C2' | 1.315(5) | N1' - C9' | 1.363(4) |
| C2' - C3' | 1.401(6) | C3' - C4' | 1.363(6) |
| C4' - C10' | 1.415(5) | C5' - C6' | 1.359(6) |
| C5' - C10' | 1.417(5) | C6' - C7' | 1.413(6) |
| C7' - C8' | 1.365(5) | C8' - C9' | 1.420(4) |
| C8' - S11' | 1.784(3) | C9' - C10' | 1.421(4) |
| S11 - S11' | 2.039(2) | | |
| | | | |
| C2 -N1 -C9 | 116.4(3) | N1 -C2 -C3 | 124.2(4) |
| C2 -C3 -C4 | 119.7(4) | C3 -C4 -C10 | 118.7(4) |
| C6 -C5 -C10 | 120.2(4) | C5 -C6 -C7 | 121.6(4) |
| C6 -C7 -C8 | 120.1(4) | C7 -C8 -C9 | 119.9(3) |
| C7 -C8 -S11 | 125.3(3) | C9 -C8 -S11 | 114.8(2) |
| N1 -C9 -C8 | 117.4(3) | N1 -C9 -C10 | 123.4(3) |
| C8 -C9 -C10 | 119.1(3) | C4 -C10 -C5 | 123.4(4) |
| C4 -C10 -C9 | 117.5(3) | C5 -C10 -C9 | 119.1(3) |
| C2'-N1' -C9' | 117.4(3) | N1'-C2' -C3' | 124.4(4) |
| C2'-C3' -C4' | 118.7(4) | C3'-C4' -C10' | 119.7(3) |
| C6'-C5' -C10' | 119.9(3) | C5'-C6' -C7' | 121.5(4) |
| C6'-C7' -C8' | 119.9(3) | C7'-C8' -C9' | 120.4(3) |
| C7'-C8' -S11' | 125.5(3) | C9'-C8' -S11' | 114.1(2) |
| N1'-C9' -C8' | 118.0(3) | N1'-C9' -C10' | 122.8(3) |
| C8'-C9' -C10' | 119.2(3) | C4'-C10'-C5' | 123.9(3) |
| C4'-C10'-C9' | 116.9(3) | C5'-C10'-C9' | 119.2(3) |
| C8 -S11 -S11' | 104.5(2) | C8'-S11'-S11 | 103.5(2) |

## Reference

1. A. Yuchi, K. Sugiura, H. Wada and G. Nakagawa, *Bull. Chem. Soc. Jpn.*, **60**, 4291 (1987) and references cited therein.

(Received July 28, 1990)
(Accepted September 14, 1990)