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# 5,6-Dimethoxy-1-(4-tolylsulfonyl)benzo[*c*]-isoxazol-3(1*H*)-one

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.041
*wR* factor = 0.126
Data-to-parameter ratio = 13.2

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

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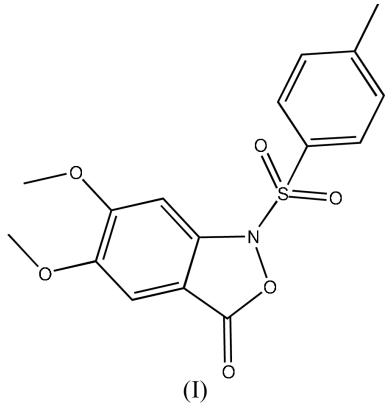
The title compound, $C_{16}H_{15}N_1O_6S$, was synthesized by reacting 5,6-dimethoxybenzo[*c*]isoxazol-3(1*H*)-one with 4-methylbenzenesulfonyl chloride in dichloromethane chloride, using triethylamine as the base and 4-(dimethylamino)pyridine as catalyst.

## Comment

In this paper, the structure of the title compound (Wierenga *et al.*, 1984), (I), is reported. It was synthesized by reacting 5,6-dimethoxybenzo[*c*]isoxazol-3(1*H*)-one (Cohen *et al.*, 1972) with 4-methylbenzenesulfonyl chloride in dichloromethane, using triethylamine as the base and 4-(dimethylamino)pyridine as catalyst. The molecular structure of (I) is illustrated in Fig. 1. The benzo[*c*]isoxazolone system is planar.



(I)

## Experimental

A mixture of 5,6-dimethoxy-1*H*-benzo[*c*]isoxazol-3-one (1 mmol), triethylamine (1 mmol), 4-(dimethylamino)pyridine (50 mg) and dichloromethane (5 ml) was cooled with stirring under $N_2$ in an ice bath. To this cooled mixture was added 4-methylbenzenesulfonyl chloride (1 mmol) in dichloromethane (5 ml) dropwise and monitored by TLC. After the reaction was over, water (5 ml) was added to the reaction mixture, which was then diluted with dichoromethane (60 ml) and shaken successively with water (30 ml), aqueous $NaHCO_3$ and brine. The organic layer was dried over anhydrous sodium sulfate, the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate 5:1) to give (I). M.p. 543–544 K. $^1$H NMR (p.p.m.): 2.37 (*s*, 3H, $CH_3$), 3.86 (*s*, 3H, O—$CH_3$), 4.08 (*s*, 3H, O—$CH_3$), 7.01–7.61 (*m*, 6H, ArH); $^{13}$C NMR (p.p.m.): 21.2, 56.4, 56.9, 99.2, 104.8, 106.1, 127.9, 130.0, 130.5, 146.1, 147.8, 150.9, 157.7, 165.9%. Compound (I) (20 mg) was dissolved in acetone (15 ml); the solution was kept at room temperature for 15 d. By natural evaporation it gave colorless single crystals of (I), suitable for X-ray analysis.

*Crystal data*

| | |
|---|---|
| $C_{16}H_{15}NO_6S$ | $Z = 2$ |
| $M_r = 349.35$ | $D_x = 1.413$ Mg m$^{-3}$ |
| Triclinic, $P\overline{1}$ | Mo $K\alpha$ radiation |
| $a = 8.595$ (2) Å | Cell parameters from 850 reflections |
| $b = 8.868$ (2) Å | $\theta = 2.9$–$25.8°$ |
| $c = 11.628$ (3) Å | $\mu = 0.23$ mm$^{-1}$ |
| $\alpha = 77.197$ (4)° | $T = 293$ (2) K |
| $\beta = 79.270$ (4)° | Block, colorless |
| $\gamma = 73.478$ (4)° | 0.36 × 0.34 × 0.18 mm |
| $V = 821.4$ (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2903 independent reflections |
| $\varphi$ and $\omega$ scans | 2000 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1997) | $R_{int} = 0.016$ |
| $T_{min} = 0.889$, $T_{max} = 1.000$ | $\theta_{max} = 25.0°$ |
| 4330 measured reflections | $h = -10 \rightarrow 10$ |
| | $k = -5 \rightarrow 10$ |
| | $l = -13 \rightarrow 13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.1843P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.041$ | |
| $wR(F^2) = 0.126$ | |
| $S = 1.05$ | $(\Delta/\sigma)_{max} = 0.004$ |
| 2903 reflections | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |
| 220 parameters | $\Delta\rho_{min} = -0.27$ e Å$^{-3}$ |
| H-atom parameters constrained | |

H atoms were positioned geometrically, with C—H = 0.93–0.96 Å, and refined in a riding model, with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

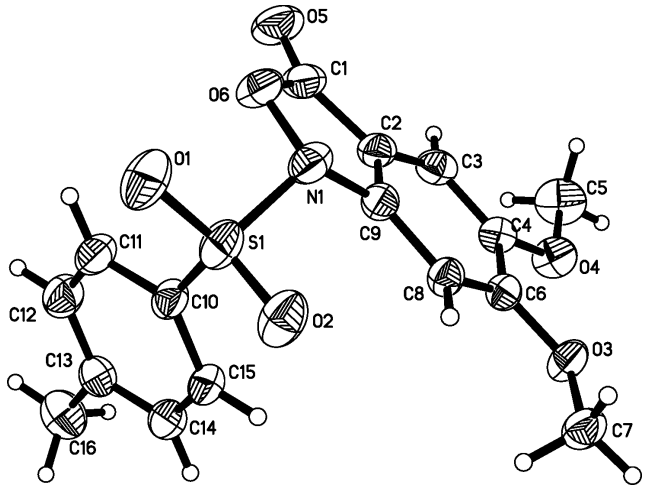



**Figure 1**
The molecular structure of (I), drawn with 30% probability ellipsoids.

## References

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