

## Methyl 2-[[3-(4,6-dimethoxypyrimidin-2-yl)ureido]sulfonylmethyl]benzoate

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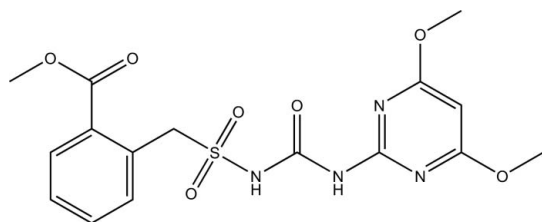
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.056;  $wR$  factor = 0.156; data-to-parameter ratio = 12.9.

In the title compound,  $\text{C}_{16}\text{H}_{18}\text{N}_4\text{O}_7\text{S}$ , a synthetic sulfonylurea herbicide, there are intramolecular  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds. Intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds form centrosymmetric dimers. The dihedral angle between the two rings is  $50.00$  ( $15$ )°.

### Related literature

For related literature, see: Kong *et al.* (1990); Lee *et al.* (2002); Sabadie (1996).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{18}\text{N}_4\text{O}_7\text{S}$   
 $M_r = 410.41$   
Monoclinic,  $C2/c$   
 $a = 33.831$  (7) Å  
 $b = 6.9020$  (14) Å

$c = 16.021$  (3) Å  
 $\beta = 104.48$  (3)°  
 $V = 3622.1$  (13) Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation

$\mu = 0.23$  mm<sup>-1</sup>  
 $T = 298$  (2) K

$0.40 \times 0.20 \times 0.10$  mm

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.914$ ,  $T_{\max} = 0.978$   
3265 measured reflections

3265 independent reflections  
2421 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$   
3 standard reflections every 200 reflections  
intensity decay: none

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.156$   
 $S = 1.03$   
3265 reflections

253 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                             | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{N1}-\text{H1A}\cdots\text{N3}$     | 0.86  | 1.94        | 2.648 (4)   | 138           |
| $\text{N2}-\text{H2A}\cdots\text{O5}^i$   | 0.86  | 2.10        | 2.951 (3)   | 170           |
| $\text{C9}-\text{H9B}\cdots\text{O1}$     | 0.97  | 2.36        | 2.970 (4)   | 120           |
| $\text{C15}-\text{H15C}\cdots\text{O1}^i$ | 0.96  | 2.43        | 3.068 (4)   | 124           |

Symmetry code: (i)  $-x, y, -z + \frac{1}{2}$ .

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2185).

### References

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Kong, F., Hu, X., Wang, L., Tao, Z., Wang, X. & Cao, W. (1990). *Huaxue Shijie*, **31**, 211–213.  
Lee, J. K., Ahn, K. C., Park, O. S., Ko, Y. K. & Kim, D.-W. (2002). *J. Agric. Food Chem.* **50**, 1791–1803.  
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.  
Sabadie, J. (1996). *Weed Res.* **36**, 441–448.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

*Acta Cryst.* (2008). E64, o632 [ doi:10.1107/S1600536808005011 ]

## Methyl 2-[[3-(4,6-dimethoxypyrimidin-2-yl)ureido]sulfonylmethyl]benzoate

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### Comment

The title compound, bensulfuron-methyl, belongs to the class of systemic sulfonylurea herbicides inhibiting acetolactate synthase, a key enzyme in the biosynthesis of the branched-chain amino acids of target plants (Lee *et al.*, 2002). It is widely used in transplanted and direct-seeded rice fields to control most annual and perennial broadleaved weeds selectively (Sabadie, 1996).

We report here the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the C3–C8 and C11/N3/C12/C13/C14/N4 rings is 50.00 (15)°. There are intramolecular N1—H1A···N3 and C9—H9B···O1 hydrogen bonds (Fig. 1), and intermolecular N—H···O and C—H···O hydrogen bonds form centrosymmetric dimers (Fig. 2).

### Experimental

The title compound, (I), was prepared according to the literature method (Kong *et al.*, 1990).

Crystals suitable for X-ray analysis were obtained by dissolving (I) (0.2 g) in ethyl acetate (25 ml) and evaporating the solvent slowly at room temperature for about 15 d.

### Refinement

All H atoms were positioned geometrically with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and were included in the refinement in a riding model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5U_{\text{eq}}(\text{N}, \text{C})$ .

### Figures

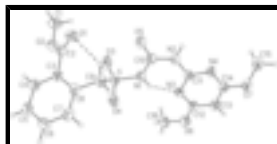


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular hydrogen bonds.

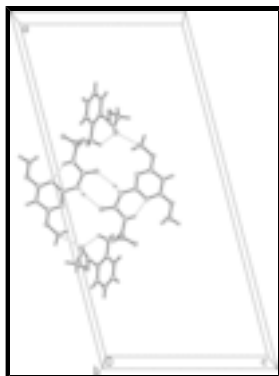


Fig. 2. A hydrogen-bonded dimer. Dashed lines indicate hydrogen bonds.

**Methyl 2-[[3-(4,6-dimethoxypyrimidin-2-yl)ureido]sulfonylmethyl]benzoate**

*Crystal data*

$C_{16}H_{18}N_4O_7S$

$M_r = 410.41$

Monoclinic,  $C2/c$

Hall symbol:  $-C\ 2yc$

$a = 33.831\ (7)\ \text{\AA}$

$b = 6.9020\ (14)\ \text{\AA}$

$c = 16.021\ (3)\ \text{\AA}$

$\beta = 104.48\ (3)^\circ$

$V = 3622.1\ (13)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1712$

$D_x = 1.505\ \text{Mg m}^{-3}$

Melting point: 450–451 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 10\text{--}13^\circ$

$\mu = 0.23\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Needle, colorless

$0.40 \times 0.20 \times 0.10\ \text{mm}$

*Data collection*

Enraf–Nonius CAD-4  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)\ \text{K}$

$\omega/2\theta$  scans

Absorption correction:  $\psi$  scan  
(North *et al.*, 1968)

$T_{\min} = 0.914$ ,  $T_{\max} = 0.978$

3325 measured reflections

3265 independent reflections

2421 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.2^\circ$

$\theta_{\min} = 1.2^\circ$

$h = -40 \rightarrow 38$

$k = 0 \rightarrow 8$

$l = 0 \rightarrow 19$

3 standard reflections

every 200 reflections

intensity decay: none

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.056$$

$$wR(F^2) = 0.156$$

$$S = 1.03$$

3265 reflections

253 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 5P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| S   | 0.12664 (2)  | 0.14338 (13) | 0.69392 (5)  | 0.0401 (2)                       |
| N1  | 0.07804 (7)  | 0.1705 (4)   | 0.64572 (16) | 0.0424 (7)                       |
| H1A | 0.0719       | 0.1836       | 0.5906       | 0.051*                           |
| O1  | 0.15578 (7)  | 0.0798 (4)   | 0.92720 (16) | 0.0557 (7)                       |
| C1  | 0.19376 (12) | -0.2563 (5)  | 0.9629 (3)   | 0.0570 (10)                      |
| H1B | 0.2121       | -0.3646      | 0.9704       | 0.085*                           |
| H1C | 0.1676       | -0.2946      | 0.9278       | 0.085*                           |
| H1D | 0.1909       | -0.2139      | 1.0182       | 0.085*                           |
| O2  | 0.20987 (7)  | -0.0991 (3)  | 0.92109 (16) | 0.0484 (6)                       |
| N2  | 0.00822 (7)  | 0.1970 (4)   | 0.63164 (16) | 0.0388 (6)                       |
| H2A | -0.0114      | 0.1857       | 0.6565       | 0.047*                           |
| C2  | 0.18727 (9)  | 0.0615 (5)   | 0.90655 (19) | 0.0379 (7)                       |
| N3  | 0.02372 (7)  | 0.2300 (4)   | 0.49733 (16) | 0.0363 (6)                       |
| O3  | 0.13329 (8)  | -0.0390 (4)  | 0.73670 (16) | 0.0576 (7)                       |
| C3  | 0.20669 (9)  | 0.2172 (5)   | 0.86550 (18) | 0.0356 (7)                       |
| O4  | 0.14650 (7)  | 0.1838 (4)   | 0.62677 (15) | 0.0542 (7)                       |
| N4  | -0.04352 (7) | 0.2765 (4)   | 0.51521 (16) | 0.0375 (6)                       |
| C4  | 0.24906 (9)  | 0.2405 (5)   | 0.8922 (2)   | 0.0432 (8)                       |
| H4C | 0.2647       | 0.1513       | 0.9300       | 0.052*                           |
| O5  | 0.05149 (7)  | 0.1570 (4)   | 0.76305 (14) | 0.0509 (6)                       |
| C5  | 0.26803 (10) | 0.3949 (5)   | 0.8631 (2)   | 0.0470 (8)                       |
| H5A | 0.2962       | 0.4101       | 0.8819       | 0.056*                           |
| O6  | 0.03580 (7)  | 0.2645 (4)   | 0.36258 (15) | 0.0531 (6)                       |
| C6  | 0.24518 (11) | 0.5258 (6)   | 0.8063 (2)   | 0.0505 (9)                       |

## supplementary materials

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|      |               |            |              |             |
|------|---------------|------------|--------------|-------------|
| H6A  | 0.2578        | 0.6302     | 0.7870       | 0.061*      |
| O7   | -0.09468 (7)  | 0.3699 (4) | 0.39861 (15) | 0.0508 (6)  |
| C7   | 0.20337 (10)  | 0.5023 (5) | 0.7779 (2)   | 0.0468 (8)  |
| H7A  | 0.1883        | 0.5896     | 0.7382       | 0.056*      |
| C8   | 0.18338 (9)   | 0.3511 (5) | 0.80731 (19) | 0.0363 (7)  |
| C9   | 0.13757 (9)   | 0.3323 (5) | 0.7722 (2)   | 0.0406 (8)  |
| H9A  | 0.1264        | 0.4537     | 0.7460       | 0.049*      |
| H9B  | 0.1249        | 0.3034     | 0.8188       | 0.049*      |
| C10  | 0.04638 (9)   | 0.1743 (5) | 0.6850 (2)   | 0.0369 (7)  |
| C11  | -0.00401 (9)  | 0.2356 (4) | 0.54321 (19) | 0.0341 (7)  |
| C12  | 0.00998 (10)  | 0.2704 (5) | 0.4133 (2)   | 0.0388 (7)  |
| C13  | -0.03018 (10) | 0.3179 (5) | 0.3764 (2)   | 0.0438 (8)  |
| H13A | -0.0395       | 0.3469     | 0.3181       | 0.053*      |
| C14  | -0.05548 (9)  | 0.3198 (5) | 0.4314 (2)   | 0.0391 (7)  |
| C15  | -0.12294 (10) | 0.3408 (6) | 0.4504 (2)   | 0.0573 (10) |
| H15A | -0.1496       | 0.3834     | 0.4193       | 0.086*      |
| H15B | -0.1143       | 0.4138     | 0.5028       | 0.086*      |
| H15C | -0.1239       | 0.2056     | 0.4639       | 0.086*      |
| C16  | 0.07680 (10)  | 0.1963 (6) | 0.3980 (2)   | 0.0597 (10) |
| H16A | 0.0918        | 0.2013     | 0.3545       | 0.090*      |
| H16B | 0.0760        | 0.0652     | 0.4175       | 0.090*      |
| H16C | 0.0899        | 0.2770     | 0.4457       | 0.090*      |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| S   | 0.0287 (4)  | 0.0485 (5)  | 0.0398 (4)  | 0.0025 (3)   | 0.0022 (3)   | -0.0034 (4)  |
| N1  | 0.0293 (13) | 0.0636 (19) | 0.0332 (14) | -0.0023 (13) | 0.0057 (11)  | 0.0033 (13)  |
| O1  | 0.0422 (14) | 0.0684 (17) | 0.0634 (15) | 0.0099 (12)  | 0.0260 (12)  | 0.0167 (13)  |
| C1  | 0.067 (2)   | 0.041 (2)   | 0.065 (2)   | -0.0069 (18) | 0.0199 (19)  | 0.0080 (19)  |
| O2  | 0.0434 (13) | 0.0434 (14) | 0.0610 (15) | 0.0055 (10)  | 0.0178 (11)  | 0.0100 (11)  |
| N2  | 0.0292 (13) | 0.0450 (16) | 0.0394 (14) | 0.0004 (11)  | 0.0035 (11)  | 0.0083 (12)  |
| C2  | 0.0350 (17) | 0.0447 (18) | 0.0313 (15) | 0.0025 (14)  | 0.0032 (13)  | -0.0006 (14) |
| N3  | 0.0344 (14) | 0.0381 (15) | 0.0353 (14) | -0.0032 (11) | 0.0069 (11)  | -0.0003 (11) |
| O3  | 0.0556 (16) | 0.0440 (15) | 0.0620 (16) | 0.0068 (12)  | -0.0061 (12) | 0.0009 (12)  |
| C3  | 0.0359 (16) | 0.0420 (18) | 0.0281 (14) | -0.0003 (14) | 0.0067 (12)  | -0.0035 (13) |
| O4  | 0.0377 (13) | 0.0835 (19) | 0.0434 (13) | -0.0003 (12) | 0.0140 (10)  | -0.0076 (12) |
| N4  | 0.0319 (13) | 0.0339 (14) | 0.0419 (15) | -0.0018 (11) | 0.0001 (11)  | 0.0019 (12)  |
| C4  | 0.0339 (16) | 0.055 (2)   | 0.0373 (17) | 0.0008 (15)  | 0.0018 (13)  | -0.0002 (16) |
| O5  | 0.0351 (12) | 0.0769 (18) | 0.0383 (13) | 0.0013 (12)  | 0.0046 (10)  | 0.0061 (12)  |
| C5  | 0.0336 (17) | 0.062 (2)   | 0.0423 (18) | -0.0094 (16) | 0.0041 (14)  | -0.0014 (17) |
| O6  | 0.0460 (14) | 0.0707 (17) | 0.0428 (13) | 0.0023 (12)  | 0.0117 (11)  | 0.0010 (12)  |
| C6  | 0.049 (2)   | 0.057 (2)   | 0.0463 (19) | -0.0161 (17) | 0.0120 (16)  | 0.0005 (17)  |
| O7  | 0.0359 (12) | 0.0585 (15) | 0.0513 (14) | 0.0048 (11)  | -0.0016 (10) | 0.0134 (12)  |
| C7  | 0.0469 (19) | 0.050 (2)   | 0.0393 (17) | 0.0003 (16)  | 0.0035 (15)  | 0.0036 (16)  |
| C8  | 0.0306 (15) | 0.0397 (17) | 0.0360 (16) | -0.0009 (13) | 0.0035 (12)  | -0.0060 (14) |
| C9  | 0.0344 (16) | 0.0445 (19) | 0.0406 (17) | 0.0049 (14)  | 0.0050 (13)  | -0.0014 (15) |
| C10 | 0.0304 (15) | 0.0371 (17) | 0.0412 (18) | -0.0007 (13) | 0.0052 (13)  | 0.0015 (14)  |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0312 (15) | 0.0291 (16) | 0.0389 (16) | -0.0024 (12) | 0.0028 (13)  | 0.0013 (13)  |
| C12 | 0.0439 (18) | 0.0330 (17) | 0.0385 (17) | -0.0033 (14) | 0.0083 (14)  | -0.0030 (14) |
| C13 | 0.0460 (19) | 0.045 (2)   | 0.0340 (16) | -0.0047 (15) | -0.0020 (14) | 0.0006 (14)  |
| C14 | 0.0363 (17) | 0.0323 (17) | 0.0433 (18) | -0.0011 (13) | -0.0001 (14) | 0.0054 (14)  |
| C15 | 0.0384 (19) | 0.068 (3)   | 0.062 (2)   | 0.0021 (18)  | 0.0057 (17)  | 0.011 (2)    |
| C16 | 0.0396 (19) | 0.081 (3)   | 0.057 (2)   | -0.0040 (19) | 0.0105 (16)  | -0.009 (2)   |

*Geometric parameters (Å, °)*

|            |             |            |           |
|------------|-------------|------------|-----------|
| S—O3       | 1.424 (3)   | O5—C10     | 1.224 (4) |
| S—O4       | 1.431 (2)   | C5—C6      | 1.374 (5) |
| S—N1       | 1.643 (3)   | C5—H5A     | 0.930     |
| S—C9       | 1.782 (3)   | O6—C12     | 1.334 (4) |
| N1—C10     | 1.372 (4)   | O6—C16     | 1.440 (4) |
| N1—H1A     | 0.860       | C6—C7      | 1.383 (5) |
| O1—C2      | 1.199 (4)   | C6—H6A     | 0.930     |
| C1—O2      | 1.451 (4)   | O7—C14     | 1.344 (4) |
| C1—H1B     | 0.960       | O7—C15     | 1.428 (4) |
| C1—H1C     | 0.960       | C7—C8      | 1.388 (5) |
| C1—H1D     | 0.960       | C7—H7A     | 0.930     |
| O2—C2      | 1.333 (4)   | C8—C9      | 1.516 (4) |
| N2—C10     | 1.368 (4)   | C9—H9A     | 0.970     |
| N2—C11     | 1.399 (4)   | C9—H9B     | 0.970     |
| N2—H2A     | 0.860       | C12—C13    | 1.379 (4) |
| C2—C3      | 1.495 (5)   | C13—C14    | 1.373 (5) |
| N3—C11     | 1.329 (4)   | C13—H13A   | 0.930     |
| N3—C12     | 1.339 (4)   | C15—H15A   | 0.960     |
| C3—C4      | 1.399 (4)   | C15—H15B   | 0.960     |
| C3—C8      | 1.406 (4)   | C15—H15C   | 0.960     |
| N4—C11     | 1.330 (4)   | C16—H16A   | 0.960     |
| N4—C14     | 1.336 (4)   | C16—H16B   | 0.960     |
| C4—C5      | 1.384 (5)   | C16—H16C   | 0.960     |
| C4—H4C     | 0.930       |            |           |
| O3—S—O4    | 119.13 (17) | C6—C7—H7A  | 119.3     |
| O3—S—N1    | 110.25 (15) | C8—C7—H7A  | 119.3     |
| O4—S—N1    | 103.13 (14) | C7—C8—C3   | 118.6 (3) |
| O3—S—C9    | 109.17 (16) | C7—C8—C9   | 118.5 (3) |
| O4—S—C9    | 109.40 (16) | C3—C8—C9   | 122.8 (3) |
| N1—S—C9    | 104.73 (15) | C8—C9—S    | 109.7 (2) |
| C10—N1—S   | 126.2 (2)   | C8—C9—H9A  | 109.7     |
| C10—N1—H1A | 116.9       | S—C9—H9A   | 109.7     |
| S—N1—H1A   | 116.9       | C8—C9—H9B  | 109.7     |
| O2—C1—H1B  | 109.5       | S—C9—H9B   | 109.7     |
| O2—C1—H1C  | 109.5       | H9A—C9—H9B | 108.2     |
| H1B—C1—H1C | 109.5       | O5—C10—N2  | 121.3 (3) |
| O2—C1—H1D  | 109.5       | O5—C10—N1  | 122.7 (3) |
| H1B—C1—H1D | 109.5       | N2—C10—N1  | 116.0 (3) |
| H1C—C1—H1D | 109.5       | N3—C11—N4  | 127.5 (3) |
| C2—O2—C1   | 115.9 (3)   | N3—C11—N2  | 118.9 (3) |

## supplementary materials

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|             |            |                |            |
|-------------|------------|----------------|------------|
| C10—N2—C11  | 130.5 (3)  | N4—C11—N2      | 113.6 (3)  |
| C10—N2—H2A  | 114.8      | O6—C12—N3      | 119.4 (3)  |
| C11—N2—H2A  | 114.8      | O6—C12—C13     | 118.1 (3)  |
| O1—C2—O2    | 123.4 (3)  | N3—C12—C13     | 122.5 (3)  |
| O1—C2—C3    | 124.3 (3)  | C14—C13—C12    | 115.6 (3)  |
| O2—C2—C3    | 112.2 (3)  | C14—C13—H13A   | 122.2      |
| C11—N3—C12  | 115.7 (3)  | C12—C13—H13A   | 122.2      |
| C4—C3—C8    | 119.3 (3)  | N4—C14—O7      | 118.1 (3)  |
| C4—C3—C2    | 118.5 (3)  | N4—C14—C13     | 124.2 (3)  |
| C8—C3—C2    | 121.9 (3)  | O7—C14—C13     | 117.7 (3)  |
| C11—N4—C14  | 114.4 (3)  | O7—C15—H15A    | 109.5      |
| C5—C4—C3    | 120.8 (3)  | O7—C15—H15B    | 109.5      |
| C5—C4—H4C   | 119.6      | H15A—C15—H15B  | 109.5      |
| C3—C4—H4C   | 119.6      | O7—C15—H15C    | 109.5      |
| C6—C5—C4    | 119.8 (3)  | H15A—C15—H15C  | 109.5      |
| C6—C5—H5A   | 120.1      | H15B—C15—H15C  | 109.5      |
| C4—C5—H5A   | 120.1      | O6—C16—H16A    | 109.5      |
| C12—O6—C16  | 118.8 (3)  | O6—C16—H16B    | 109.5      |
| C5—C6—C7    | 120.1 (3)  | H16A—C16—H16B  | 109.5      |
| C5—C6—H6A   | 120.0      | O6—C16—H16C    | 109.5      |
| C7—C6—H6A   | 120.0      | H16A—C16—H16C  | 109.5      |
| C14—O7—C15  | 118.4 (3)  | H16B—C16—H16C  | 109.5      |
| C6—C7—C8    | 121.4 (3)  |                |            |
| O3—S—N1—C10 | 61.5 (3)   | N1—S—C9—C8     | -167.4 (2) |
| O4—S—N1—C10 | -170.2 (3) | C11—N2—C10—O5  | 173.7 (3)  |
| C9—S—N1—C10 | -55.8 (3)  | C11—N2—C10—N1  | -6.9 (5)   |
| C1—O2—C2—O1 | 1.1 (5)    | S—N1—C10—O5    | -0.1 (5)   |
| C1—O2—C2—C3 | 178.5 (3)  | S—N1—C10—N2    | -179.5 (2) |
| O1—C2—C3—C4 | 139.0 (3)  | C12—N3—C11—N4  | 0.0 (5)    |
| O2—C2—C3—C4 | -38.4 (4)  | C12—N3—C11—N2  | -179.3 (3) |
| O1—C2—C3—C8 | -34.9 (5)  | C14—N4—C11—N3  | -1.4 (5)   |
| O2—C2—C3—C8 | 147.7 (3)  | C14—N4—C11—N2  | 178.0 (3)  |
| C8—C3—C4—C5 | 1.0 (5)    | C10—N2—C11—N3  | 8.8 (5)    |
| C2—C3—C4—C5 | -173.1 (3) | C10—N2—C11—N4  | -170.6 (3) |
| C3—C4—C5—C6 | -0.9 (5)   | C16—O6—C12—N3  | 6.0 (5)    |
| C4—C5—C6—C7 | -0.5 (5)   | C16—O6—C12—C13 | -173.8 (3) |
| C5—C6—C7—C8 | 1.9 (5)    | C11—N3—C12—O6  | -178.8 (3) |
| C6—C7—C8—C3 | -1.7 (5)   | C11—N3—C12—C13 | 1.0 (5)    |
| C6—C7—C8—C9 | -179.0 (3) | O6—C12—C13—C14 | 179.3 (3)  |
| C4—C3—C8—C7 | 0.3 (4)    | N3—C12—C13—C14 | -0.5 (5)   |
| C2—C3—C8—C7 | 174.2 (3)  | C11—N4—C14—O7  | -177.5 (3) |
| C4—C3—C8—C9 | 177.5 (3)  | C11—N4—C14—C13 | 1.9 (5)    |
| C2—C3—C8—C9 | -8.7 (5)   | C15—O7—C14—N4  | -11.2 (4)  |
| C7—C8—C9—S  | 102.8 (3)  | C15—O7—C14—C13 | 169.4 (3)  |
| C3—C8—C9—S  | -74.3 (3)  | C12—C13—C14—N4 | -1.0 (5)   |
| O3—S—C9—C8  | 74.5 (3)   | C12—C13—C14—O7 | 178.4 (3)  |
| O4—S—C9—C8  | -57.5 (3)  |                |            |



Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i>    | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1A···N3                | 0.86        | 1.94          | 2.648 (4)             | 138                     |
| N2—H2A···O5 <sup>i</sup>   | 0.86        | 2.10          | 2.951 (3)             | 170                     |
| C9—H9B···O1                | 0.97        | 2.36          | 2.970 (4)             | 120                     |
| C15—H15C···O1 <sup>i</sup> | 0.96        | 2.43          | 3.068 (4)             | 124                     |

Symmetry codes: (i)  $-x, y, -z+3/2$ .

Fig. 1

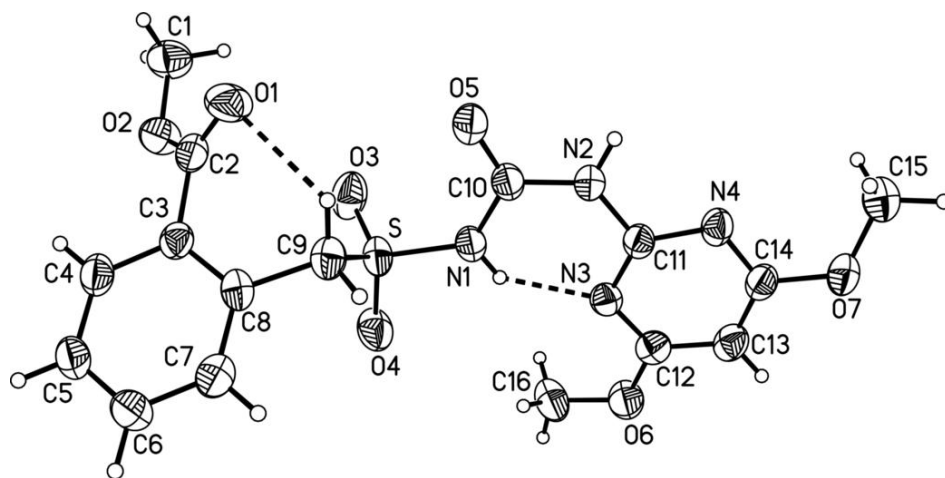


Fig. 2

