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## Structure Reports

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**(Z)-1,3,4a-Trimethyl-5,5-diphenyl-6-oxa-1,3-diazabicyclo[4.2.0]octane-2,4-dione**

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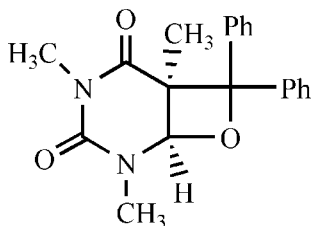
Received 23 January 2008; accepted 8 March 2008

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.117; data-to-parameter ratio = 13.4.

The title compound,  $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_3$ , is a head-to-tail oxetane, one of the regioisomers obtained by the the Paternó-Büchi reaction of 1,3-dimethylthymine with benzophenone. The oxetane ring is folded, the dihedral angle between the  $\text{C}-\text{O}-\text{C}$  and  $\text{C}-\text{C}-\text{C}$  planes being  $14.4(2)^\circ$ . The dihedral angle between the two phenyl rings is  $64.3(2)^\circ$ . The pyrimidine ring adopts a boat conformation. The crystal structure involves weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For related literature, see: Hei *et al.* (2005); Prakash *et al.* (1997).



## Experimental

## Crystal data

$\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_3$   
 $M_r = 336.38$   
 Monoclinic,  $P2_1/n$

$a = 8.1341(7)$  Å  
 $b = 9.1004(13)$  Å  
 $c = 23.485(2)$  Å

$\beta = 97.334(2)^\circ$   
 $V = 1724.2(3)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.09$  mm<sup>-1</sup>  
 $T = 298(2)$  K  
 $0.45 \times 0.41 \times 0.18$  mm

## Data collection

Bruker SMART diffractometer  
 Absorption correction: multi-scan  
 (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.962$ ,  $T_{\max} = 0.984$

8774 measured reflections  
 3039 independent reflections  
 1818 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.036$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.117$   
 $S = 1.01$   
 3039 reflections

226 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.18$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                             | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C3}-\text{H3}\cdots\text{O2}^{\text{i}}$  | 0.98         | 2.53               | 3.431 (3)   | 153                  |
| $\text{C7}-\text{H7}\cdots\text{O1}$             | 0.93         | 2.37               | 2.745 (3)   | 104                  |
| $\text{C8}-\text{H8}\cdots\text{O1}^{\text{ii}}$ | 0.93         | 2.59               | 3.513 (3)   | 171                  |
| $\text{C13}-\text{H13}\cdots\text{O1}$           | 0.93         | 2.41               | 2.752 (3)   | 101                  |
| $\text{C17}-\text{H17}\cdots\text{O3}$           | 0.93         | 2.55               | 3.040 (3)   | 113                  |
| $\text{C20}-\text{H20B}\cdots\text{O2}$          | 0.96         | 2.29               | 2.682 (4)   | 104                  |

Symmetry codes: (i)  $-x + \frac{5}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ ; (ii)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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: *SHELXTL*.

This work was supported by the Grants for Scientific Research of BSKY (grant No. XJ200712) from Anhui Medical University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2239).

## References

- Hei, X. M., Song, Q. H., Tang, W. J., Wang, H. B. & Guo, Q. X. (2005). *J. Org. Chem.* **70**, 2522–2527.  
 Prakash, G., Fettinger, J. C. & Falvey, D. E. (1997). *Acta Cryst.* **C53**, 979–981.  
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**supplementary materials**

*Acta Cryst.* (2008). E64, o796 [ doi:10.1107/S1600536808006557 ]

## (Z)-1,3,4a-Trimethyl-5,5-diphenyl-6-oxa-1,3-diazabicyclo[4.2.0]octane-2,4-dione

Z.-C. Lin, J.-B. Shi, W.-J. Tang and J. Li

### Comment

A [2 + 2] photocycloaddition (Paternò-Büchi reaction) of the 5–6 double bond of 1,3-dimethylthymine (DMT) with the carbonyl of benzophenone generates two regioisomers, head-to-head and head-to-tail oxetanes (Fig. 1). We have observed the temperature dependence of the regioselectivity (Hei *et al.*, 2005). The crystal structure of the head-to-head oxetane has already been published (Prakash *et al.*, 1997). In the present study, an X-ray crystallographic analysis of the head-to-tail oxetane has been undertaken to establish its structure and configuration.

The structure is similar to that observed in the head-to-head oxetane (Prakash *et al.*, 1997). The bond lengths and angles in the title compound are in good agreement with expected values. The oxetane ring is folded and the dihedral angle between the C1—O1—C3 and C1—C2—C3 planes is 14.4 (2)° (Fig. 2). The dihedral angle between the two phenyl rings is 64.3 (2)° and the pyrimidine ring adopts a boat conformation. The crystal structure involves weak C—H···O hydrogen bonds.

### Experimental

The title compound was prepared by first dissolving DMT (0.77 g, 5.0 mmol) and benzophenone (1.82 g, 10.0 mmol) in CH<sub>3</sub>CN (100 ml). The resulting solution was placed in a photochemical apparatus (Pyrex), purged with nitrogen, degassed for 30 min, and irradiated with a 300 W mercury high-pressure lamp for 10 h. The solvent was then rotary evaporated and the oxetane was purified by silica gel chromatography using a 4:1 petroleum ether/ethyl acetate solvent mixture. The fractions containing the h-t oxetane were combined, and the solvent removed by rotary evaporation. The purified oxetane was subsequently crystallized by dissolving the residue in 10 ml of ethyl acetate and adding n-hexane to the solution until it turned cloudy. Upon standing at room temperature, a colorless block appeared and was separated from the solvent by decanting.

### Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.98 Å (methine), 0.96 Å (methyl) and 0.93 Å (aromatic);  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{O})$  or  $1.5 U_{\text{eq}}(\text{C})$  for the methyl group.

### Figures

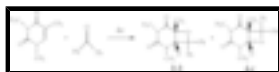


Fig. 1. Formation of the two regioisomers of the Paternò-Büchi reaction.

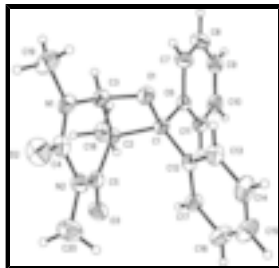


Fig. 2. Molecular structure of the title compound, with the atomic numbering scheme, and displacement ellipsoids drawn at the 30% probability level.

## (Z)-1,3,4a-Trimethyl-5,5-diphenyl-6-oxa-1,3-diazabicyclo[4.2.0]octane- 2,4-dione

### Crystal data

$C_{20}H_{20}N_2O_3$

$M_r = 336.38$

Monoclinic,  $P2_1/n$

$a = 8.1341 (7) \text{ \AA}$

$b = 9.1004 (13) \text{ \AA}$

$c = 23.485 (2) \text{ \AA}$

$\beta = 97.334 (2)^\circ$

$V = 1724.2 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 712$

$D_x = 1.296 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

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

Cell parameters from 1952 reflections

$\theta = 2.4\text{--}22.1^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

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

Block, colorless

$0.45 \times 0.41 \times 0.18 \text{ mm}$

### Data collection

Bruker SMART  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

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

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.962$ ,  $T_{\max} = 0.984$

8774 measured reflections

3039 independent reflections

1818 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 25.0^\circ$

$\theta_{\min} = 1.8^\circ$

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -24 \rightarrow 27$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.117$

$S = 1.01$

3039 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.7238P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.20 \text{ e \AA}^{-3}$

226 parameters

$$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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

|      | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| N1   | 1.2692 (2)   | 0.5663 (2)   | 0.21196 (8)  | 0.0491 (5)                       |
| N2   | 1.2831 (2)   | 0.6885 (2)   | 0.12510 (8)  | 0.0492 (5)                       |
| O1   | 0.97763 (19) | 0.60800 (17) | 0.19685 (6)  | 0.0469 (4)                       |
| O2   | 1.4490 (2)   | 0.7527 (2)   | 0.20642 (8)  | 0.0750 (6)                       |
| O3   | 1.1732 (2)   | 0.5833 (2)   | 0.04218 (7)  | 0.0625 (5)                       |
| C1   | 0.9036 (3)   | 0.5849 (2)   | 0.13794 (8)  | 0.0379 (5)                       |
| C2   | 1.0673 (3)   | 0.4991 (2)   | 0.12589 (9)  | 0.0380 (5)                       |
| C3   | 1.1126 (3)   | 0.5076 (3)   | 0.19078 (9)  | 0.0427 (6)                       |
| H3   | 1.0949       | 0.4127       | 0.2088       | 0.051*                           |
| C4   | 1.3391 (3)   | 0.6743 (3)   | 0.18356 (11) | 0.0503 (6)                       |
| C5   | 1.1782 (3)   | 0.5906 (3)   | 0.09400 (10) | 0.0440 (6)                       |
| C6   | 0.7544 (3)   | 0.4834 (2)   | 0.13563 (9)  | 0.0398 (6)                       |
| C7   | 0.7166 (3)   | 0.4145 (3)   | 0.18451 (11) | 0.0555 (7)                       |
| H7   | 0.7781       | 0.4358       | 0.2197       | 0.067*                           |
| C8   | 0.5890 (4)   | 0.3147 (3)   | 0.18183 (13) | 0.0654 (8)                       |
| H8   | 0.5646       | 0.2695       | 0.2153       | 0.079*                           |
| C9   | 0.4977 (3)   | 0.2812 (3)   | 0.13056 (13) | 0.0617 (8)                       |
| H9   | 0.4128       | 0.2124       | 0.1289       | 0.074*                           |
| C10  | 0.5325 (3)   | 0.3502 (3)   | 0.08148 (12) | 0.0580 (7)                       |
| H10  | 0.4701       | 0.3289       | 0.0465       | 0.070*                           |
| C11  | 0.6592 (3)   | 0.4506 (3)   | 0.08398 (10) | 0.0481 (6)                       |
| H11  | 0.6815       | 0.4971       | 0.0506       | 0.058*                           |
| C12  | 0.8626 (3)   | 0.7299 (2)   | 0.10882 (9)  | 0.0385 (6)                       |
| C13  | 0.8285 (3)   | 0.8494 (3)   | 0.14167 (11) | 0.0505 (6)                       |
| H13  | 0.8336       | 0.8392       | 0.1813       | 0.061*                           |
| C14  | 0.7872 (4)   | 0.9835 (3)   | 0.11655 (14) | 0.0673 (8)                       |
| H14  | 0.7645       | 1.0629       | 0.1392       | 0.081*                           |
| C15  | 0.7794 (4)   | 0.9999 (3)   | 0.05856 (14) | 0.0727 (9)                       |
| H15  | 0.7523       | 1.0906       | 0.0417       | 0.087*                           |
| C16  | 0.8116 (4)   | 0.8830 (3)   | 0.02531 (12) | 0.0666 (8)                       |
| H16  | 0.8061       | 0.8942       | -0.0142      | 0.080*                           |
| C17  | 0.8520 (3)   | 0.7485 (3)   | 0.05001 (10) | 0.0504 (6)                       |
| H17  | 0.8724       | 0.6693       | 0.0269       | 0.060*                           |
| C18  | 1.0451 (3)   | 0.3473 (3)   | 0.09951 (10) | 0.0503 (6)                       |
| H18A | 0.9886       | 0.3552       | 0.0612       | 0.075*                           |
| H18B | 0.9809       | 0.2877       | 0.1222       | 0.075*                           |
| H18C | 1.1517       | 0.3029       | 0.0985       | 0.075*                           |
| C19  | 1.3263 (4)   | 0.5493 (3)   | 0.27297 (11) | 0.0783 (9)                       |
| H19A | 1.4432       | 0.5683       | 0.2799       | 0.117*                           |
| H19B | 1.3046       | 0.4508       | 0.2847       | 0.117*                           |
| H19C | 1.2686       | 0.6175       | 0.2945       | 0.117*                           |

## supplementary materials

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|      |            |            |              |             |
|------|------------|------------|--------------|-------------|
| C20  | 1.3705 (4) | 0.7944 (3) | 0.09317 (13) | 0.0803 (10) |
| H20A | 1.4539     | 0.7445     | 0.0751       | 0.121*      |
| H20B | 1.4218     | 0.8676     | 0.1190       | 0.121*      |
| H20C | 1.2931     | 0.8408     | 0.0644       | 0.121*      |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| N1  | 0.0490 (13) | 0.0549 (13) | 0.0392 (12) | 0.0034 (11)  | -0.0110 (9)  | -0.0031 (10) |
| N2  | 0.0448 (12) | 0.0524 (13) | 0.0499 (13) | -0.0048 (10) | 0.0035 (10)  | -0.0005 (10) |
| O1  | 0.0509 (10) | 0.0554 (11) | 0.0329 (9)  | 0.0088 (8)   | -0.0009 (7)  | -0.0072 (7)  |
| O2  | 0.0666 (13) | 0.0819 (14) | 0.0730 (13) | -0.0225 (12) | -0.0044 (11) | -0.0256 (11) |
| O3  | 0.0560 (12) | 0.0921 (14) | 0.0402 (11) | -0.0015 (10) | 0.0092 (8)   | -0.0015 (9)  |
| C1  | 0.0403 (14) | 0.0433 (14) | 0.0290 (12) | 0.0050 (11)  | 0.0004 (10)  | -0.0041 (10) |
| C2  | 0.0387 (13) | 0.0403 (13) | 0.0336 (12) | 0.0042 (11)  | -0.0008 (10) | -0.0043 (10) |
| C3  | 0.0467 (15) | 0.0395 (14) | 0.0402 (14) | 0.0043 (12)  | -0.0011 (11) | -0.0030 (11) |
| C4  | 0.0450 (15) | 0.0526 (17) | 0.0519 (16) | 0.0041 (13)  | 0.0012 (13)  | -0.0122 (13) |
| C5  | 0.0377 (14) | 0.0522 (16) | 0.0414 (15) | 0.0069 (12)  | 0.0024 (11)  | -0.0021 (12) |
| C6  | 0.0379 (13) | 0.0414 (14) | 0.0405 (14) | 0.0063 (11)  | 0.0067 (11)  | 0.0006 (11)  |
| C7  | 0.0573 (17) | 0.0665 (18) | 0.0442 (15) | -0.0029 (15) | 0.0119 (12)  | 0.0020 (13)  |
| C8  | 0.0656 (19) | 0.070 (2)   | 0.065 (2)   | -0.0033 (16) | 0.0253 (16)  | 0.0127 (15)  |
| C9  | 0.0438 (16) | 0.0573 (18) | 0.085 (2)   | -0.0021 (14) | 0.0134 (15)  | 0.0074 (16)  |
| C10 | 0.0402 (15) | 0.0623 (18) | 0.0680 (19) | -0.0026 (14) | -0.0062 (13) | 0.0037 (15)  |
| C11 | 0.0447 (15) | 0.0519 (16) | 0.0467 (15) | -0.0002 (13) | 0.0015 (12)  | 0.0076 (12)  |
| C12 | 0.0324 (13) | 0.0391 (14) | 0.0434 (14) | 0.0010 (10)  | 0.0027 (10)  | -0.0035 (11) |
| C13 | 0.0477 (15) | 0.0505 (16) | 0.0512 (15) | 0.0059 (13)  | -0.0016 (12) | -0.0098 (13) |
| C14 | 0.073 (2)   | 0.0418 (16) | 0.083 (2)   | 0.0119 (14)  | -0.0031 (16) | -0.0150 (15) |
| C15 | 0.087 (2)   | 0.0434 (17) | 0.083 (2)   | 0.0064 (15)  | -0.0078 (18) | 0.0103 (16)  |
| C16 | 0.089 (2)   | 0.0519 (18) | 0.0564 (17) | 0.0058 (16)  | -0.0001 (15) | 0.0085 (14)  |
| C17 | 0.0587 (17) | 0.0438 (15) | 0.0483 (16) | 0.0068 (13)  | 0.0059 (12)  | 0.0010 (12)  |
| C18 | 0.0462 (15) | 0.0501 (15) | 0.0524 (15) | 0.0065 (12)  | -0.0026 (12) | -0.0129 (12) |
| C19 | 0.082 (2)   | 0.095 (2)   | 0.0493 (17) | -0.0024 (19) | -0.0221 (15) | 0.0001 (16)  |
| C20 | 0.078 (2)   | 0.082 (2)   | 0.082 (2)   | -0.0240 (18) | 0.0150 (18)  | 0.0105 (17)  |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|        |           |         |           |
|--------|-----------|---------|-----------|
| N1—C4  | 1.353 (3) | C9—H9   | 0.9300    |
| N1—C3  | 1.412 (3) | C10—C11 | 1.373 (3) |
| N1—C19 | 1.457 (3) | C10—H10 | 0.9300    |
| N2—C5  | 1.377 (3) | C11—H11 | 0.9300    |
| N2—C4  | 1.396 (3) | C12—C13 | 1.381 (3) |
| N2—C20 | 1.460 (3) | C12—C17 | 1.383 (3) |
| O1—C3  | 1.450 (3) | C13—C14 | 1.379 (4) |
| O1—C1  | 1.452 (2) | C13—H13 | 0.9300    |
| O2—C4  | 1.214 (3) | C14—C15 | 1.364 (4) |
| O3—C5  | 1.214 (3) | C14—H14 | 0.9300    |
| C1—C12 | 1.504 (3) | C15—C16 | 1.365 (4) |
| C1—C6  | 1.521 (3) | C15—H15 | 0.9300    |
| C1—C2  | 1.600 (3) | C16—C17 | 1.376 (3) |

|           |             |               |           |
|-----------|-------------|---------------|-----------|
| C2—C5     | 1.497 (3)   | C16—H16       | 0.9300    |
| C2—C18    | 1.515 (3)   | C17—H17       | 0.9300    |
| C2—C3     | 1.523 (3)   | C18—H18A      | 0.9600    |
| C3—H3     | 0.9800      | C18—H18B      | 0.9600    |
| C6—C7     | 1.376 (3)   | C18—H18C      | 0.9600    |
| C6—C11    | 1.386 (3)   | C19—H19A      | 0.9600    |
| C7—C8     | 1.375 (4)   | C19—H19B      | 0.9600    |
| C7—H7     | 0.9300      | C19—H19C      | 0.9600    |
| C8—C9     | 1.366 (4)   | C20—H20A      | 0.9600    |
| C8—H8     | 0.9300      | C20—H20B      | 0.9600    |
| C9—C10    | 1.373 (4)   | C20—H20C      | 0.9600    |
| C4—N1—C3  | 121.2 (2)   | C11—C10—C9    | 120.2 (3) |
| C4—N1—C19 | 117.4 (2)   | C11—C10—H10   | 119.9     |
| C3—N1—C19 | 117.8 (2)   | C9—C10—H10    | 119.9     |
| C5—N2—C4  | 124.3 (2)   | C10—C11—C6    | 120.9 (2) |
| C5—N2—C20 | 117.6 (2)   | C10—C11—H11   | 119.6     |
| C4—N2—C20 | 116.6 (2)   | C6—C11—H11    | 119.6     |
| C3—O1—C1  | 92.41 (14)  | C13—C12—C17   | 118.0 (2) |
| O1—C1—C12 | 110.28 (17) | C13—C12—C1    | 119.0 (2) |
| O1—C1—C6  | 110.74 (17) | C17—C12—C1    | 122.9 (2) |
| C12—C1—C6 | 112.78 (18) | C14—C13—C12   | 120.9 (2) |
| O1—C1—C2  | 89.20 (14)  | C14—C13—H13   | 119.6     |
| C12—C1—C2 | 119.28 (18) | C12—C13—H13   | 119.6     |
| C6—C1—C2  | 112.08 (17) | C15—C14—C13   | 120.1 (3) |
| C5—C2—C18 | 110.54 (19) | C15—C14—H14   | 119.9     |
| C5—C2—C3  | 112.83 (19) | C13—C14—H14   | 119.9     |
| C18—C2—C3 | 117.16 (19) | C14—C15—C16   | 119.9 (3) |
| C5—C2—C1  | 112.63 (18) | C14—C15—H15   | 120.1     |
| C18—C2—C1 | 117.30 (18) | C16—C15—H15   | 120.1     |
| C3—C2—C1  | 84.20 (15)  | C15—C16—C17   | 120.3 (3) |
| N1—C3—O1  | 112.77 (18) | C15—C16—H16   | 119.8     |
| N1—C3—C2  | 117.63 (19) | C17—C16—H16   | 119.8     |
| O1—C3—C2  | 92.34 (15)  | C16—C17—C12   | 120.7 (2) |
| N1—C3—H3  | 110.9       | C16—C17—H17   | 119.6     |
| O1—C3—H3  | 110.9       | C12—C17—H17   | 119.6     |
| C2—C3—H3  | 110.9       | C2—C18—H18A   | 109.5     |
| O2—C4—N1  | 122.7 (2)   | C2—C18—H18B   | 109.5     |
| O2—C4—N2  | 120.6 (3)   | H18A—C18—H18B | 109.5     |
| N1—C4—N2  | 116.6 (2)   | C2—C18—H18C   | 109.5     |
| O3—C5—N2  | 120.4 (2)   | H18A—C18—H18C | 109.5     |
| O3—C5—C2  | 121.7 (2)   | H18B—C18—H18C | 109.5     |
| N2—C5—C2  | 117.8 (2)   | N1—C19—H19A   | 109.5     |
| C7—C6—C11 | 118.2 (2)   | N1—C19—H19B   | 109.5     |
| C7—C6—C1  | 120.6 (2)   | H19A—C19—H19B | 109.5     |
| C11—C6—C1 | 121.07 (19) | N1—C19—H19C   | 109.5     |
| C8—C7—C6  | 120.7 (2)   | H19A—C19—H19C | 109.5     |
| C8—C7—H7  | 119.7       | H19B—C19—H19C | 109.5     |
| C6—C7—H7  | 119.7       | N2—C20—H20A   | 109.5     |
| C9—C8—C7  | 120.7 (3)   | N2—C20—H20B   | 109.5     |

## supplementary materials

|               |              |                 |             |
|---------------|--------------|-----------------|-------------|
| C9—C8—H8      | 119.7        | H20A—C20—H20B   | 109.5       |
| C7—C8—H8      | 119.7        | N2—C20—H20C     | 109.5       |
| C8—C9—C10     | 119.4 (3)    | H20A—C20—H20C   | 109.5       |
| C8—C9—H9      | 120.3        | H20B—C20—H20C   | 109.5       |
| C10—C9—H9     | 120.3        |                 |             |
| C3—O1—C1—C12  | -131.48 (18) | C20—N2—C5—C2    | -170.0 (2)  |
| C3—O1—C1—C6   | 102.98 (19)  | C18—C2—C5—O3    | 37.6 (3)    |
| C3—O1—C1—C2   | -10.39 (16)  | C3—C2—C5—O3     | 170.9 (2)   |
| O1—C1—C2—C5   | -102.41 (19) | C1—C2—C5—O3     | -95.8 (3)   |
| C12—C1—C2—C5  | 10.5 (3)     | C18—C2—C5—N2    | -144.8 (2)  |
| C6—C1—C2—C5   | 145.47 (19)  | C3—C2—C5—N2     | -11.4 (3)   |
| O1—C1—C2—C18  | 127.57 (19)  | C1—C2—C5—N2     | 81.9 (2)    |
| C12—C1—C2—C18 | -119.5 (2)   | O1—C1—C6—C7     | -6.9 (3)    |
| C6—C1—C2—C18  | 15.5 (3)     | C12—C1—C6—C7    | -131.1 (2)  |
| O1—C1—C2—C3   | 9.93 (15)    | C2—C1—C6—C7     | 91.0 (2)    |
| C12—C1—C2—C3  | 122.9 (2)    | O1—C1—C6—C11    | 177.12 (19) |
| C6—C1—C2—C3   | -102.19 (18) | C12—C1—C6—C11   | 53.0 (3)    |
| C4—N1—C3—O1   | -72.4 (3)    | C2—C1—C6—C11    | -85.0 (2)   |
| C19—N1—C3—O1  | 85.8 (3)     | C11—C6—C7—C8    | 0.7 (4)     |
| C4—N1—C3—C2   | 33.3 (3)     | C1—C6—C7—C8     | -175.3 (2)  |
| C19—N1—C3—C2  | -168.6 (2)   | C6—C7—C8—C9     | 0.3 (4)     |
| C1—O1—C3—N1   | 132.30 (19)  | C7—C8—C9—C10    | -1.1 (4)    |
| C1—O1—C3—C2   | 10.93 (17)   | C8—C9—C10—C11   | 0.7 (4)     |
| C5—C2—C3—N1   | -15.1 (3)    | C9—C10—C11—C6   | 0.4 (4)     |
| C18—C2—C3—N1  | 115.0 (2)    | C7—C6—C11—C10   | -1.1 (3)    |
| C1—C2—C3—N1   | -127.2 (2)   | C1—C6—C11—C10   | 174.9 (2)   |
| C5—C2—C3—O1   | 102.19 (19)  | O1—C1—C12—C13   | -27.7 (3)   |
| C18—C2—C3—O1  | -127.7 (2)   | C6—C1—C12—C13   | 96.7 (2)    |
| C1—C2—C3—O1   | -9.95 (15)   | C2—C1—C12—C13   | -128.7 (2)  |
| C3—N1—C4—O2   | 160.5 (2)    | O1—C1—C12—C17   | 154.5 (2)   |
| C19—N1—C4—O2  | 2.3 (4)      | C6—C1—C12—C17   | -81.1 (3)   |
| C3—N1—C4—N2   | -22.0 (3)    | C2—C1—C12—C17   | 53.5 (3)    |
| C19—N1—C4—N2  | 179.8 (2)    | C17—C12—C13—C14 | -0.8 (4)    |
| C5—N2—C4—O2   | 169.6 (2)    | C1—C12—C13—C14  | -178.7 (2)  |
| C20—N2—C4—O2  | 3.8 (3)      | C12—C13—C14—C15 | 0.0 (4)     |
| C5—N2—C4—N1   | -7.9 (3)     | C13—C14—C15—C16 | 0.5 (5)     |
| C20—N2—C4—N1  | -173.7 (2)   | C14—C15—C16—C17 | -0.1 (5)    |
| C4—N2—C5—O3   | -157.9 (2)   | C15—C16—C17—C12 | -0.7 (4)    |
| C20—N2—C5—O3  | 7.7 (3)      | C13—C12—C17—C16 | 1.2 (4)     |
| C4—N2—C5—C2   | 24.4 (3)     | C1—C12—C17—C16  | 179.0 (2)   |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                   | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|-------|-------------|-------------|---------------|
| C3—H3 $\cdots$ O2 <sup>i</sup>  | 0.98  | 2.53        | 3.431 (3)   | 153           |
| C7—H7 $\cdots$ O1               | 0.93  | 2.37        | 2.745 (3)   | 104           |
| C8—H8 $\cdots$ O1 <sup>ii</sup> | 0.93  | 2.59        | 3.513 (3)   | 171           |
| C13—H13 $\cdots$ O1             | 0.93  | 2.41        | 2.752 (3)   | 101           |

|             |      |      |           |     |
|-------------|------|------|-----------|-----|
| C17—H17…O3  | 0.93 | 2.55 | 3.040 (3) | 113 |
| C20—H20B…O2 | 0.96 | 2.29 | 2.682 (4) | 104 |

Symmetry codes: (i)  $-x+5/2, y-1/2, -z+1/2$ ; (ii)  $-x+3/2, y-1/2, -z+1/2$ .

Fig. 1

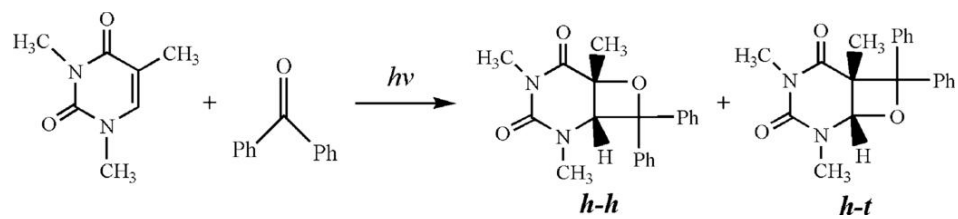


Fig. 2

