# organic compounds

# 4-[2,3-Dibromo-3-(4-bromophenyl)-propanoyl]-2-phenyl-1,2,3-oxadiazol-2-ium-5-olate

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In the title compound, $C_{17}H_{11}Br_3N_2O_3$, the whole molecule is disordered over two positions with a refined occupancy ratio of 0.770 (5):0.230 (5). In the major component, the 1,2,3-oxadiazolidine ring is essentially planar [maximum deviation = 0.017 (6) Å] and makes dihedral angles of 22.5 (3) and 70.2 (3)° with the 4-bromophenyl and phenyl rings, respectively. In the minor component, the corresponding values are 18.9 (11) and 84.9 (12)°. In the crystal, intermolecular C—H···Br hydrogen bonds link the molecules into ribbons along [010]. There is a short O···N contact [2.83 (3) Å] in the minor component. In the major component, the molecular structure is stabilized by an intramolecular C—H···O hydrogen bond, which forms an $S(6)$ ring motif.

## Related literature

For biological activity of sydnones, mesoionic compounds having a 1,2,3-oxadiazole skeleton and bearing an oxygen atom attached to the 5-position, see: Jyothi *et al.* (2008); Rai *et al.* (2007; 2008). For a related structure, see: Goh *et al.* (2010). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986). For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).
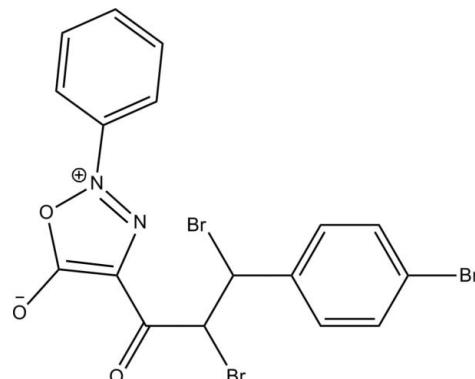


## Experimental

### Crystal data

| | |
|---|---|
| $C_{17}H_{11}Br_3N_2O_3$ | $V = 1810.62$ (5) Å$^3$ |
| $M_r = 531.01$ | $Z = 4$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a = 17.6996$ (3) Å | $\mu = 6.70$ mm$^{-1}$ |
| $b = 5.8322$ (1) Å | $T = 100$ K |
| $c = 18.2445$ (3) Å | $0.43 \times 0.38 \times 0.12$ mm |
| $\beta = 105.973$ (1)° | |

### Data collection

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 20140 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2009) | 5243 independent reflections |
| | 4218 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.159$, $T_{max} = 0.505$ | $R_{int} = 0.028$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | 207 restraints |
| $wR(F^2) = 0.055$ | H-atom parameters constrained |
| $S = 1.02$ | $\Delta\rho_{max} = 0.50$ e Å$^{-3}$ |
| 5243 reflections | $\Delta\rho_{min} = -0.37$ e Å$^{-3}$ |
| 352 parameters | |

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10$A$—H10$A$···O2$A$ | 0.98 | 2.40 | 3.168 (4) | 135 |
| C14$A$—H14$A$···Br3$A^i$ | 0.93 | 2.91 | 3.809 (5) | 163 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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# supporting information

## 4-[2,3-Dibromo-3-(4-bromophenyl)propanoyl]-2-phenyl-1,2,3-oxadiazol-2-ium-5-olate

### Hoong-Kun Fun, Tara Shahani, Nithinchandra and Balakrishna Kalluraya

### S1. Comment

Sydnones are mesoionic heterocyclic aromatic chemical compounds. The study of sydnones still remains a field of interests because of their electronic structures and also because of the varied types of biological activities displayed by some of them (Rai *et al.*, 2008). Recently sydnone derivatives were found to exhibit promising antimicrobial properties (Jyothi *et al.*, 2008). Since their discovery, sydnones have shown diverse biological activities and it is thought that the *meso*-ionic nature of the sydnone ring promotes significant interactions with biological systems. Because of wide variety of properties displayed by sydnones we were prompted to synthesize a new chalcone containing a sydnone type ring. Propenones are prepared by the condensation of 4-acetyl-3-arylsydnones with appropriately substituted aromatic aldehydes in an ethanol medium employing sodium hydroxide as catalyst. Bromination of these propenones were carried out using bromine in glacial acetic acid medium to give dibromochalcones (Rai *et al.*, 2007).

In the title compound (Fig. 1), the whole molecule is disordered over two positions with a refined occupancy ratio of 0.770 (5):0.230 (5). This molecule consists of three rings, namely phenyl (C1–C6), 1,2,3-oxadiazolidine (N1/N2/O1/C7/C8) and bromophenyl (C12–C17/Br3) rings. In the major component, the 1,2,3-oxadiazolidine ring is essentially planar (maximum deviation of 0.017 (6) Å at atom N1A) and makes dihedral angles of 22.5 (3) and 70.2 (3)° with 4-bromophenyl and phenyl rings, respectively. In the minor component, the corresponding values are 18.9 (11) and 84.9 (12)° between the 1,2,3-oxadiazolidine ring (maximum deviation of 0.020 (16) Å at atom O1B) and with the 4-bromophenyl and phenyl ring. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to the closely related structure (Goh *et al.*, 2010). The molecular structure is stabilized by an intramolecular C10A–H10A···O2A hydrogen bond, which forms an S(6) ring motif.

In the crystal packing (Fig. 2 & Fig. 3), intermolecular C14A—H14A···Br3A hydrogen bonds (Table 1), link the molecules into one-dimensional ribbons along the [010] direction. There is a short contact [O2B···N2B = 2.83 (3) Å, symmetry code 1/2 - *x*, -1/2 + *y*, 1/2 - *z*] in the minor component.

### S2. Experimental

1-(3-Phenylsydnon-4yl)-3-(*p*-bromophenyl)-propen-1-one (0.01 mol) was dissolved in glacial acetic acid (25–30 ml) by gentle warming. A solution of bromine in glacial acetic acid (30% *w/v*) was added to it with constant stirring till the yellow colour of the bromine persisted. The reaction mixture was stirred at room temperature for 1–2 h. The separated solid was filtered, washed with methanol and dried. It was then recrystallized from ethanol. Crystals suitable for X-ray analysis were obtained from 1:2 mixtures of DMF and ethanol by slow evaporation.

### S3. Refinement

All the H atoms were positioned geometrically [C–H = 0.93 to 0.98 Å] and were refined using a riding model, with $U_{iso}(H) = 1.2\ U_{eq}$ (C). The whole molecule is disordered over two positions with a refined ratio of 0.770 (5):0.230 (5). Rigidity, similarity and simulation restraints were applied. The possibility of a supercell in which the whole-molecule disorder would be no longer exit was addressed by examining the $h0l$, $0kl$, $hk0$ precession layers to ensure there are no rows of weak reflections between the rows that represent the current unit cell. No such supercell reflections were found. This finding is consistent with the fact that if such a supercell exists, the occupanies of the major and minor components would be the same. However the refined occupanies are 0.770 (5): 0.230 (5) disproving the existence of a supercell.
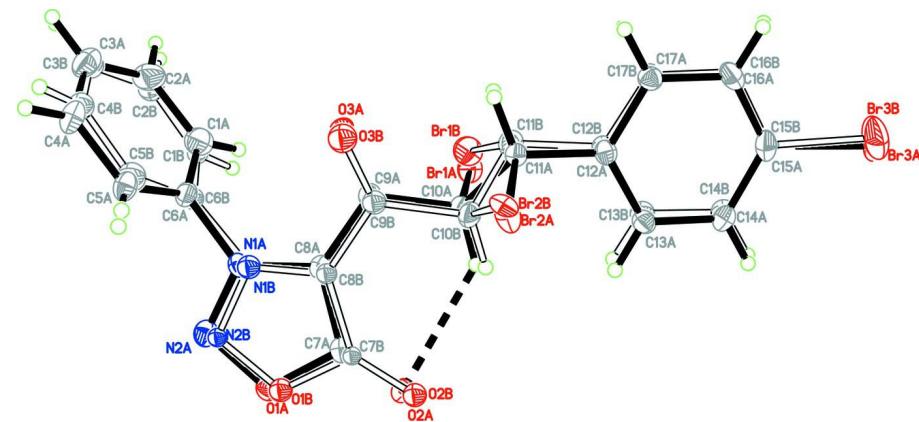


**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Both major and minor components are shown. Intramolecular interaction is shown in dashed line.
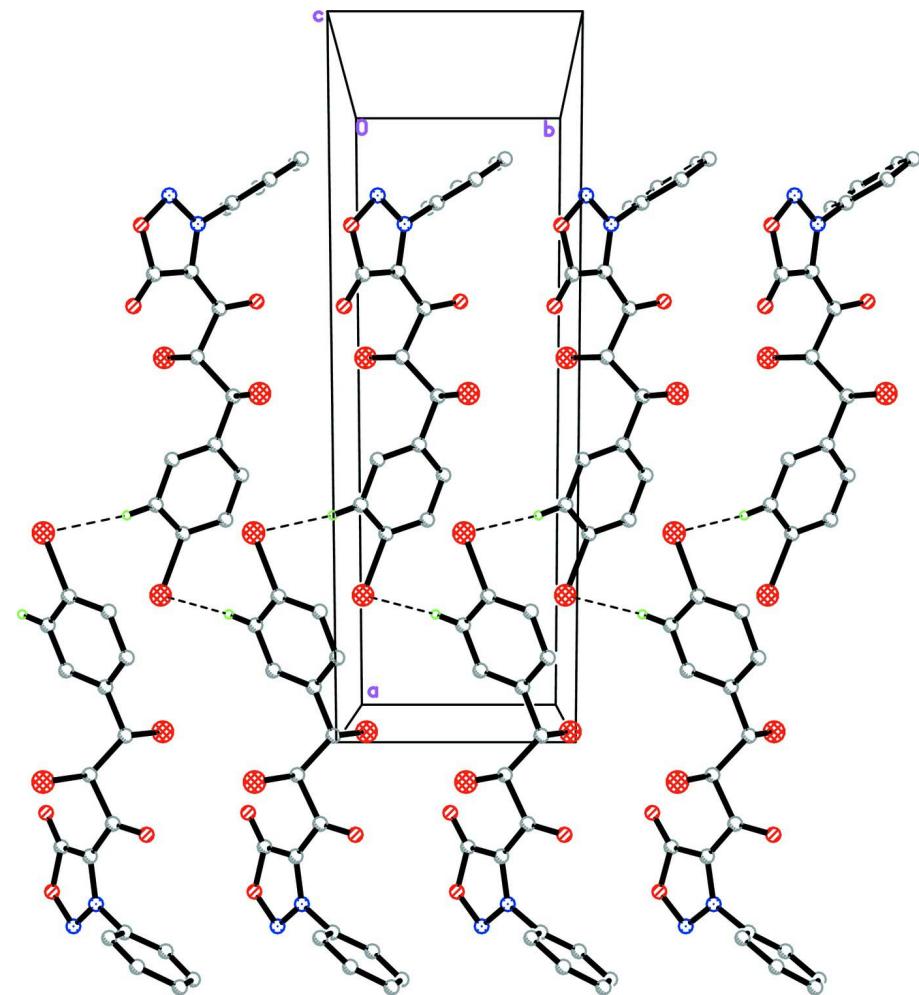
**Figure 2**

The crystal packing of the title compound, viewed along *c* axis. Only the major disordered component is shown. Hydrogen atoms not involved in intermolecular hydrogen bonding (dashed lines) are omitted for clarity.

**4-[2,3-Dibromo-3-(4-bromophenyl)propanoyl]-2-phenyl-1,2,3-oxadiazol-2- ium-5-olate**

*Crystal data*

| | |
|---|---|
| $C_{17}H_{11}Br_3N_2O_3$ | $F(000) = 1024$ |
| $M_r = 531.01$ | $D_x = 1.948$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 9198 reflections |
| $a = 17.6996$ (3) Å | $\theta = 2.4$–29.8° |
| $b = 5.8322$ (1) Å | $\mu = 6.70$ mm$^{-1}$ |
| $c = 18.2445$ (3) Å | $T = 100$ K |
| $\beta = 105.973$ (1)° | Block, yellow |
| $V = 1810.62$ (5) Å$^3$ | $0.43 \times 0.38 \times 0.12$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 20140 measured reflections |
| Radiation source: fine-focus sealed tube | 5243 independent reflections |
| Graphite monochromator | 4218 reflections with $I > 2\sigma(I)$ |
| $\varphi$ and $\omega$ scans | $R_{int} = 0.028$ |
| Absorption correction: multi-scan ($SADABS$; Bruker, 2009) | $\theta_{max} = 30.0°$, $\theta_{min} = 1.9°$ |
| | $h = -24 \rightarrow 24$ |
| | $k = -8 \rightarrow 7$ |
| $T_{min} = 0.159$, $T_{max} = 0.505$ | $l = -24 \rightarrow 25$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.055$ | |
| $S = 1.02$ | H-atom parameters constrained |
| 5243 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0238P)^2 + 0.3541P]$ |
| 352 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 207 restraints | $(\Delta/\sigma)_{max} = 0.002$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.50$ e Å⁻³ |
| | $\Delta\rho_{min} = -0.37$ e Å⁻³ |

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Br1A | 0.39809 (10) | 0.0392 (3) | 0.03443 (10) | 0.03273 (19) | 0.770 (5) |
| Br2A | 0.46821 (7) | 0.55027 (18) | 0.23460 (8) | 0.0428 (3) | 0.770 (5) |
| Br3A | 0.80250 (8) | 0.0292 (2) | 0.16887 (6) | 0.03402 (19) | 0.770 (5) |
| O1A | 0.1949 (3) | −0.0068 (9) | 0.1943 (3) | 0.0282 (8) | 0.770 (5) |
| O2A | 0.32784 (19) | −0.0399 (6) | 0.2397 (2) | 0.0293 (6) | 0.770 (5) |
| O3A | 0.30678 (12) | 0.5098 (4) | 0.06838 (17) | 0.0339 (6) | 0.770 (5) |
| N1A | 0.1841 (3) | 0.2416 (10) | 0.1078 (3) | 0.0233 (9) | 0.770 (5) |
| N2A | 0.1416 (3) | 0.1153 (13) | 0.1392 (4) | 0.0299 (11) | 0.770 (5) |
| C1A | 0.1437 (4) | 0.3491 (9) | −0.0250 (3) | 0.0435 (13) | 0.770 (5) |
| H1AA | 0.1703 | 0.2222 | −0.0363 | 0.052* | 0.770 (5) |
| C2A | 0.1033 (3) | 0.4963 (9) | −0.0814 (3) | 0.0473 (11) | 0.770 (5) |
| H2AA | 0.1010 | 0.4676 | −0.1321 | 0.057* | 0.770 (5) |
| C3A | 0.0659 (3) | 0.6880 (8) | −0.0620 (3) | 0.0416 (10) | 0.770 (5) |
| H3AA | 0.0396 | 0.7889 | −0.1000 | 0.050* | 0.770 (5) |

| | | | | | |
|---|---|---|---|---|---|
| C4A | 0.0676 (4) | 0.7296 (9) | 0.0128 (3) | 0.0381 (10) | 0.770 (5) |
| H4AA | 0.0414 | 0.8564 | 0.0248 | 0.046* | 0.770 (5) |
| C5A | 0.1078 (5) | 0.5851 (12) | 0.0702 (4) | 0.0301 (11) | 0.770 (5) |
| H5AA | 0.1109 | 0.6131 | 0.1211 | 0.036* | 0.770 (5) |
| C6A | 0.1432 (6) | 0.3969 (14) | 0.0479 (3) | 0.0292 (16) | 0.770 (5) |
| C7A | 0.2730 (2) | 0.0478 (7) | 0.1932 (3) | 0.0261 (8) | 0.770 (5) |
| C8A | 0.26327 (18) | 0.2104 (6) | 0.1337 (2) | 0.0231 (7) | 0.770 (5) |
| C12A | 0.54873 (13) | 0.3090 (5) | 0.14317 (17) | 0.0243 (6) | 0.770 (5) |
| C13A | 0.57473 (14) | 0.1083 (5) | 0.18318 (18) | 0.0274 (6) | 0.770 (5) |
| H13A | 0.5415 | 0.0287 | 0.2057 | 0.033* | 0.770 (5) |
| C14A | 0.6501 (2) | 0.0248 (8) | 0.1899 (3) | 0.0292 (8) | 0.770 (5) |
| H14A | 0.6677 | −0.1086 | 0.2172 | 0.035* | 0.770 (5) |
| C15A | 0.6983 (4) | 0.1445 (18) | 0.1551 (9) | 0.033 (2) | 0.770 (5) |
| C16A | 0.6744 (3) | 0.3462 (14) | 0.1168 (5) | 0.0332 (15) | 0.770 (5) |
| H16A | 0.7085 | 0.4278 | 0.0958 | 0.040* | 0.770 (5) |
| C17A | 0.59879 (19) | 0.4272 (7) | 0.1096 (2) | 0.0267 (7) | 0.770 (5) |
| H17A | 0.5817 | 0.5609 | 0.0823 | 0.032* | 0.770 (5) |
| C9A | 0.32136 (14) | 0.3360 (5) | 0.10637 (19) | 0.0266 (6) | 0.770 (5) |
| C10A | 0.40246 (13) | 0.2259 (5) | 0.12510 (16) | 0.0245 (6) | 0.770 (5) |
| H10A | 0.4109 | 0.1284 | 0.1704 | 0.029* | 0.770 (5) |
| C11A | 0.46785 (13) | 0.4009 (4) | 0.13558 (15) | 0.0241 (6) | 0.770 (5) |
| H11A | 0.4542 | 0.5140 | 0.0943 | 0.029* | 0.770 (5) |
| Br1B | 0.3986 (4) | 0.0778 (11) | 0.0224 (4) | 0.0432 (10) | 0.230 (5) |
| Br2B | 0.4661 (2) | 0.5668 (4) | 0.2298 (2) | 0.0207 (6) | 0.230 (5) |
| Br3B | 0.8041 (3) | 0.0491 (10) | 0.1649 (3) | 0.0645 (14) | 0.230 (5) |
| O1B | 0.2081 (8) | −0.011 (3) | 0.1999 (11) | 0.025 (3)* | 0.230 (5) |
| O2B | 0.3419 (6) | −0.015 (2) | 0.2543 (7) | 0.025 (3)* | 0.230 (5) |
| O3B | 0.3133 (5) | 0.5587 (14) | 0.0938 (5) | 0.035 (2)* | 0.230 (5) |
| N1B | 0.1956 (8) | 0.248 (4) | 0.1157 (11) | 0.025 (4)* | 0.230 (5) |
| N2B | 0.1529 (8) | 0.109 (4) | 0.1467 (12) | 0.016 (3)* | 0.230 (5) |
| C1B | 0.1377 (11) | 0.305 (3) | −0.0245 (9) | 0.021 (3)* | 0.230 (5) |
| H1BA | 0.1580 | 0.1639 | −0.0332 | 0.026* | 0.230 (5) |
| C2B | 0.0980 (11) | 0.437 (2) | −0.0865 (9) | 0.035 (4)* | 0.230 (5) |
| H2BA | 0.0960 | 0.3898 | −0.1357 | 0.042* | 0.230 (5) |
| C3B | 0.0620 (10) | 0.635 (2) | −0.0751 (9) | 0.031 (3)* | 0.230 (5) |
| H3BA | 0.0310 | 0.7161 | −0.1164 | 0.037* | 0.230 (5) |
| C4B | 0.0718 (13) | 0.714 (3) | −0.0023 (10) | 0.031 (4)* | 0.230 (5) |
| H4BA | 0.0516 | 0.8565 | 0.0055 | 0.037* | 0.230 (5) |
| C5B | 0.112 (2) | 0.581 (5) | 0.0605 (12) | 0.038 (6)* | 0.230 (5) |
| H5BA | 0.1144 | 0.6306 | 0.1095 | 0.045* | 0.230 (5) |
| C6B | 0.1480 (19) | 0.378 (5) | 0.0502 (9) | 0.022 (5)* | 0.230 (5) |
| C7B | 0.2854 (7) | 0.064 (3) | 0.2074 (7) | 0.015 (3)* | 0.230 (5) |
| C8B | 0.2729 (6) | 0.237 (2) | 0.1508 (6) | 0.019 (3)* | 0.230 (5) |
| C12B | 0.5443 (5) | 0.2546 (16) | 0.1212 (5) | 0.023 (2)* | 0.230 (5) |
| C13B | 0.5719 (6) | 0.0563 (17) | 0.1606 (6) | 0.029 (3)* | 0.230 (5) |
| H13B | 0.5374 | −0.0375 | 0.1771 | 0.035* | 0.230 (5) |
| C14B | 0.6499 (8) | −0.005 (3) | 0.1759 (10) | 0.032 (4)* | 0.230 (5) |
| H14B | 0.6676 | −0.1432 | 0.2000 | 0.038* | 0.230 (5) |

| C15B | 0.7008 (10) | 0.142 (5) | 0.155 (3) | 0.019 (6)* | 0.230 (5) |
|---|---|---|---|---|---|
| C16B | 0.6757 (10) | 0.331 (4) | 0.1083 (16) | 0.018 (3)* | 0.230 (5) |
| H16B | 0.7096 | 0.4148 | 0.0875 | 0.022* | 0.230 (5) |
| C17B | 0.5971 (7) | 0.388 (2) | 0.0948 (8) | 0.026 (3)* | 0.230 (5) |
| H17B | 0.5786 | 0.5199 | 0.0670 | 0.031* | 0.230 (5) |
| C9B | 0.3306 (5) | 0.3857 (16) | 0.1293 (6) | 0.025 (2)* | 0.230 (5) |
| C10B | 0.4170 (4) | 0.3055 (17) | 0.1585 (5) | 0.029 (2) | 0.230 (5) |
| H10B | 0.4223 | 0.1566 | 0.1843 | 0.035* | 0.230 (5) |
| C11B | 0.4576 (4) | 0.3139 (16) | 0.0966 (5) | 0.032 (2) | 0.230 (5) |
| H11B | 0.4503 | 0.4655 | 0.0725 | 0.038* | 0.230 (5) |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1A | 0.0304 (2) | 0.0336 (3) | 0.0315 (5) | −0.0036 (2) | 0.0040 (3) | −0.0067 (3) |
| Br2A | 0.0259 (3) | 0.0618 (6) | 0.0411 (4) | −0.0048 (3) | 0.0101 (3) | −0.0172 (4) |
| Br3A | 0.0247 (4) | 0.0442 (3) | 0.0324 (3) | 0.0101 (2) | 0.0065 (2) | −0.0044 (2) |
| O1A | 0.0256 (15) | 0.0239 (12) | 0.0370 (18) | 0.0000 (14) | 0.0119 (15) | 0.0039 (9) |
| O2A | 0.0259 (13) | 0.0293 (13) | 0.0317 (16) | 0.0041 (11) | 0.0061 (11) | 0.0065 (12) |
| O3A | 0.0259 (10) | 0.0315 (12) | 0.0420 (15) | 0.0016 (8) | 0.0054 (10) | 0.0170 (11) |
| N1A | 0.0199 (14) | 0.0208 (13) | 0.0299 (18) | −0.0014 (13) | 0.0080 (12) | −0.0024 (12) |
| N2A | 0.0267 (19) | 0.0269 (15) | 0.034 (2) | −0.0022 (17) | 0.0050 (17) | −0.0016 (12) |
| C1A | 0.047 (2) | 0.040 (3) | 0.048 (2) | 0.004 (2) | 0.0208 (15) | 0.0047 (19) |
| C2A | 0.054 (2) | 0.056 (3) | 0.0329 (18) | 0.000 (2) | 0.0135 (15) | 0.0065 (19) |
| C3A | 0.0350 (17) | 0.035 (2) | 0.046 (3) | −0.0005 (16) | −0.0032 (15) | 0.0080 (18) |
| C4A | 0.0311 (17) | 0.0349 (19) | 0.043 (3) | 0.0083 (11) | 0.001 (2) | −0.0001 (18) |
| C5A | 0.0250 (17) | 0.0277 (19) | 0.034 (2) | 0.0012 (10) | 0.0011 (19) | −0.0035 (15) |
| C6A | 0.022 (2) | 0.028 (3) | 0.036 (2) | 0.0023 (12) | 0.0047 (11) | 0.0061 (12) |
| C7A | 0.0251 (17) | 0.0225 (14) | 0.030 (2) | −0.0001 (12) | 0.0070 (14) | −0.0037 (15) |
| C8A | 0.0212 (14) | 0.0225 (14) | 0.0239 (16) | 0.0007 (9) | 0.0035 (12) | −0.0004 (13) |
| C12A | 0.0218 (11) | 0.0239 (13) | 0.0265 (14) | −0.0024 (9) | 0.0057 (10) | −0.0035 (11) |
| C13A | 0.0254 (12) | 0.0295 (14) | 0.0277 (15) | −0.0032 (10) | 0.0079 (10) | 0.0007 (12) |
| C14A | 0.0269 (15) | 0.0287 (17) | 0.030 (2) | 0.0057 (11) | 0.0041 (12) | 0.0021 (16) |
| C15A | 0.021 (2) | 0.045 (3) | 0.030 (2) | 0.0018 (11) | 0.0034 (11) | −0.0086 (12) |
| C16A | 0.0256 (16) | 0.045 (3) | 0.032 (3) | −0.0077 (12) | 0.0123 (13) | −0.005 (2) |
| C17A | 0.0276 (14) | 0.0241 (16) | 0.0286 (18) | −0.0012 (10) | 0.0083 (12) | 0.0004 (15) |
| C9A | 0.0225 (12) | 0.0277 (14) | 0.0279 (15) | −0.0012 (10) | 0.0042 (10) | 0.0012 (12) |
| C10A | 0.0218 (11) | 0.0253 (14) | 0.0247 (14) | −0.0001 (9) | 0.0035 (10) | 0.0024 (10) |
| C11A | 0.0217 (11) | 0.0252 (13) | 0.0250 (14) | −0.0001 (9) | 0.0054 (9) | 0.0019 (10) |
| Br1B | 0.0388 (8) | 0.057 (2) | 0.0294 (14) | −0.0112 (12) | 0.0026 (8) | −0.0042 (12) |
| Br2B | 0.0323 (12) | 0.0050 (8) | 0.0308 (10) | −0.0039 (6) | 0.0186 (8) | −0.0053 (6) |
| Br3B | 0.0315 (16) | 0.096 (3) | 0.070 (2) | 0.0027 (15) | 0.0214 (14) | −0.0156 (16) |
| C10B | 0.022 (4) | 0.029 (5) | 0.033 (5) | −0.002 (3) | 0.001 (4) | 0.004 (4) |
| C11B | 0.027 (4) | 0.032 (5) | 0.032 (5) | −0.005 (3) | 0.001 (3) | 0.001 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1A—C10A | 1.964 (4) | Br1B—C11B | 2.010 (12) |
| Br2A—C11A | 2.004 (3) | Br2B—C10B | 2.036 (11) |
| Br3A—C15A | 1.913 (5) | Br3B—C15B | 1.867 (13) |
| O1A—N2A | 1.374 (5) | O1B—N2B | 1.367 (13) |
| O1A—C7A | 1.423 (5) | O1B—C7B | 1.407 (13) |
| O2A—C7A | 1.212 (4) | O2B—C7B | 1.214 (12) |
| O3A—C9A | 1.215 (3) | O3B—C9B | 1.193 (11) |
| N1A—N2A | 1.293 (4) | N1B—N2B | 1.334 (14) |
| N1A—C8A | 1.363 (4) | N1B—C8B | 1.344 (12) |
| N1A—C6A | 1.450 (5) | N1B—C6B | 1.469 (13) |
| C1A—C6A | 1.362 (5) | C1B—C2B | 1.386 (14) |
| C1A—C2A | 1.379 (5) | C1B—C6B | 1.392 (13) |
| C1A—H1AA | 0.9300 | C1B—H1BA | 0.9300 |
| C2A—C3A | 1.393 (5) | C2B—C3B | 1.365 (14) |
| C2A—H2AA | 0.9300 | C2B—H2BA | 0.9300 |
| C3A—C4A | 1.380 (6) | C3B—C4B | 1.370 (14) |
| C3A—H3AA | 0.9300 | C3B—H3BA | 0.9300 |
| C4A—C5A | 1.379 (5) | C4B—C5B | 1.403 (15) |
| C4A—H4AA | 0.9300 | C4B—H4BA | 0.9300 |
| C5A—C6A | 1.379 (6) | C5B—C6B | 1.382 (14) |
| C5A—H5AA | 0.9300 | C5B—H5BA | 0.9300 |
| C7A—C8A | 1.415 (4) | C7B—C8B | 1.419 (12) |
| C8A—C9A | 1.458 (3) | C8B—C9B | 1.472 (11) |
| C12A—C13A | 1.390 (3) | C12B—C13B | 1.378 (11) |
| C12A—C17A | 1.391 (3) | C12B—C17B | 1.400 (12) |
| C12A—C11A | 1.499 (3) | C12B—C11B | 1.515 (10) |
| C13A—C14A | 1.393 (4) | C13B—C14B | 1.378 (13) |
| C13A—H13A | 0.9300 | C13B—H13B | 0.9300 |
| C14A—C15A | 1.385 (6) | C14B—C15B | 1.374 (14) |
| C14A—H14A | 0.9300 | C14B—H14B | 0.9300 |
| C15A—C16A | 1.374 (6) | C15B—C16B | 1.389 (14) |
| C16A—C17A | 1.391 (5) | C16B—C17B | 1.384 (14) |
| C16A—H16A | 0.9300 | C16B—H16B | 0.9300 |
| C17A—H17A | 0.9300 | C17B—H17B | 0.9300 |
| C9A—C10A | 1.523 (3) | C9B—C10B | 1.547 (10) |
| C10A—C11A | 1.515 (3) | C10B—C11B | 1.497 (11) |
| C10A—H10A | 0.9800 | C10B—H10B | 0.9800 |
| C11A—H11A | 0.9800 | C11B—H11B | 0.9800 |
| | | | |
| N2A—O1A—C7A | 110.4 (4) | N2B—O1B—C7B | 113.3 (12) |
| N2A—N1A—C8A | 116.0 (4) | N2B—N1B—C8B | 113.0 (11) |
| N2A—N1A—C6A | 117.4 (5) | N2B—N1B—C6B | 113.1 (17) |
| C8A—N1A—C6A | 126.6 (5) | C8B—N1B—C6B | 133.9 (18) |
| N1A—N2A—O1A | 104.6 (4) | N1B—N2B—O1B | 103.6 (12) |
| C6A—C1A—C2A | 117.4 (5) | C2B—C1B—C6B | 122.0 (14) |
| C6A—C1A—H1AA | 121.3 | C2B—C1B—H1BA | 119.0 |

| | | | |
|---|---|---|---|
| C2A—C1A—H1AA | 121.3 | C6B—C1B—H1BA | 119.0 |
| C1A—C2A—C3A | 119.7 (4) | C3B—C2B—C1B | 119.9 (14) |
| C1A—C2A—H2AA | 120.1 | C3B—C2B—H2BA | 120.1 |
| C3A—C2A—H2AA | 120.1 | C1B—C2B—H2BA | 120.1 |
| C4A—C3A—C2A | 120.6 (4) | C2B—C3B—C4B | 119.5 (14) |
| C4A—C3A—H3AA | 119.7 | C2B—C3B—H3BA | 120.2 |
| C2A—C3A—H3AA | 119.7 | C4B—C3B—H3BA | 120.2 |
| C5A—C4A—C3A | 120.6 (4) | C3B—C4B—C5B | 120.4 (15) |
| C5A—C4A—H4AA | 119.7 | C3B—C4B—H4BA | 119.8 |
| C3A—C4A—H4AA | 119.7 | C5B—C4B—H4BA | 119.8 |
| C4A—C5A—C6A | 116.4 (5) | C6B—C5B—C4B | 120.8 (16) |
| C4A—C5A—H5AA | 121.8 | C6B—C5B—H5BA | 119.6 |
| C6A—C5A—H5AA | 121.8 | C4B—C5B—H5BA | 119.6 |
| C1A—C6A—C5A | 125.1 (5) | C5B—C6B—C1B | 117.0 (13) |
| C1A—C6A—N1A | 118.2 (5) | C5B—C6B—N1B | 121.1 (16) |
| C5A—C6A—N1A | 116.6 (5) | C1B—C6B—N1B | 122.0 (16) |
| O2A—C7A—C8A | 136.4 (4) | O2B—C7B—O1B | 122.8 (12) |
| O2A—C7A—O1A | 119.3 (4) | O2B—C7B—C8B | 135.8 (12) |
| C8A—C7A—O1A | 104.3 (3) | O1B—C7B—C8B | 101.4 (10) |
| N1A—C8A—C7A | 104.6 (3) | N1B—C8B—C7B | 108.4 (10) |
| N1A—C8A—C9A | 124.7 (3) | N1B—C8B—C9B | 122.3 (10) |
| C7A—C8A—C9A | 130.6 (3) | C7B—C8B—C9B | 129.3 (10) |
| C13A—C12A—C17A | 119.4 (2) | C13B—C12B—C17B | 118.1 (9) |
| C13A—C12A—C11A | 121.3 (2) | C13B—C12B—C11B | 122.1 (8) |
| C17A—C12A—C11A | 119.4 (2) | C17B—C12B—C11B | 119.3 (9) |
| C12A—C13A—C14A | 120.7 (3) | C14B—C13B—C12B | 121.0 (11) |
| C12A—C13A—H13A | 119.6 | C14B—C13B—H13B | 119.5 |
| C14A—C13A—H13A | 119.6 | C12B—C13B—H13B | 119.5 |
| C15A—C14A—C13A | 118.7 (4) | C15B—C14B—C13B | 118.6 (13) |
| C15A—C14A—H14A | 120.7 | C15B—C14B—H14B | 120.7 |
| C13A—C14A—H14A | 120.7 | C13B—C14B—H14B | 120.7 |
| C16A—C15A—C14A | 121.5 (5) | C14B—C15B—C16B | 122.9 (15) |
| C16A—C15A—Br3A | 121.3 (4) | C14B—C15B—Br3B | 119.4 (12) |
| C14A—C15A—Br3A | 117.1 (4) | C16B—C15B—Br3B | 115.5 (12) |
| C15A—C16A—C17A | 119.5 (5) | C17B—C16B—C15B | 115.8 (14) |
| C15A—C16A—H16A | 120.2 | C17B—C16B—H16B | 122.1 |
| C17A—C16A—H16A | 120.2 | C15B—C16B—H16B | 122.1 |
| C12A—C17A—C16A | 120.2 (4) | C16B—C17B—C12B | 122.7 (13) |
| C12A—C17A—H17A | 119.9 | C16B—C17B—H17B | 118.7 |
| C16A—C17A—H17A | 119.9 | C12B—C17B—H17B | 118.7 |
| O3A—C9A—C8A | 123.7 (2) | O3B—C9B—C8B | 123.4 (9) |
| O3A—C9A—C10A | 121.3 (2) | O3B—C9B—C10B | 121.6 (9) |
| C8A—C9A—C10A | 114.9 (2) | C8B—C9B—C10B | 115.0 (8) |
| C11A—C10A—C9A | 112.6 (2) | C11B—C10B—C9B | 111.4 (7) |
| C11A—C10A—Br1A | 109.35 (19) | C11B—C10B—Br2B | 104.3 (7) |
| C9A—C10A—Br1A | 103.21 (18) | C9B—C10B—Br2B | 101.7 (6) |
| C11A—C10A—H10A | 110.5 | C11B—C10B—H10B | 112.9 |
| C9A—C10A—H10A | 110.5 | C9B—C10B—H10B | 112.9 |

| | | | |
|---|---|---|---|
| Br1A—C10A—H10A | 110.5 | Br2B—C10B—H10B | 112.9 |
| C12A—C11A—C10A | 116.6 (2) | C10B—C11B—C12B | 115.3 (7) |
| C12A—C11A—Br2A | 107.83 (17) | C10B—C11B—Br1B | 102.5 (7) |
| C10A—C11A—Br2A | 102.68 (17) | C12B—C11B—Br1B | 109.8 (6) |
| C12A—C11A—H11A | 109.8 | C10B—C11B—H11B | 109.7 |
| C10A—C11A—H11A | 109.8 | C12B—C11B—H11B | 109.7 |
| Br2A—C11A—H11A | 109.8 | Br1B—C11B—H11B | 109.7 |
| | | | |
| C8A—N1A—N2A—O1A | 3.2 (9) | C8B—N1B—N2B—O1B | −6 (3) |
| C6A—N1A—N2A—O1A | −178.2 (6) | C6B—N1B—N2B—O1B | 174 (2) |
| C7A—O1A—N2A—N1A | −1.8 (9) | C7B—O1B—N2B—N1B | 5 (3) |
| C6A—C1A—C2A—C3A | 1.7 (10) | C6B—C1B—C2B—C3B | −6 (3) |
| C1A—C2A—C3A—C4A | −1.3 (8) | C1B—C2B—C3B—C4B | 6 (3) |
| C2A—C3A—C4A—C5A | 1.5 (10) | C2B—C3B—C4B—C5B | −6 (4) |
| C3A—C4A—C5A—C6A | −2.0 (13) | C3B—C4B—C5B—C6B | 5 (5) |
| C2A—C1A—C6A—C5A | −2.4 (15) | C4B—C5B—C6B—C1B | −4 (6) |
| C2A—C1A—C6A—N1A | 178.8 (7) | C4B—C5B—C6B—N1B | 175 (3) |
| C4A—C5A—C6A—C1A | 2.5 (17) | C2B—C1B—C6B—C5B | 4 (5) |
| C4A—C5A—C6A—N1A | −178.6 (8) | C2B—C1B—C6B—N1B | −175 (2) |
| N2A—N1A—C6A—C1A | −109.4 (10) | N2B—N1B—C6B—C5B | 84 (4) |
| C8A—N1A—C6A—C1A | 69.1 (12) | C8B—N1B—C6B—C5B | −96 (4) |
| N2A—N1A—C6A—C5A | 71.7 (12) | N2B—N1B—C6B—C1B | −96 (4) |
| C8A—N1A—C6A—C5A | −109.9 (10) | C8B—N1B—C6B—C1B | 83 (4) |
| N2A—O1A—C7A—O2A | 178.0 (6) | N2B—O1B—C7B—O2B | 176 (2) |
| N2A—O1A—C7A—C8A | −0.1 (7) | N2B—O1B—C7B—C8B | −2 (2) |
| N2A—N1A—C8A—C7A | −3.3 (8) | N2B—N1B—C8B—C7B | 5 (3) |
| C6A—N1A—C8A—C7A | 178.3 (6) | C6B—N1B—C8B—C7B | −175 (2) |
| N2A—N1A—C8A—C9A | −179.6 (6) | N2B—N1B—C8B—C9B | −176.1 (18) |
| C6A—N1A—C8A—C9A | 2.0 (9) | C6B—N1B—C8B—C9B | 4 (4) |
| O2A—C7A—C8A—N1A | −175.7 (6) | O2B—C7B—C8B—N1B | −180 (2) |
| O1A—C7A—C8A—N1A | 1.8 (5) | O1B—C7B—C8B—N1B | −1 (2) |
| O2A—C7A—C8A—C9A | 0.3 (9) | O2B—C7B—C8B—C9B | 1 (3) |
| O1A—C7A—C8A—C9A | 177.9 (4) | O1B—C7B—C8B—C9B | 179.6 (15) |
| C17A—C12A—C13A—C14A | 0.0 (5) | C17B—C12B—C13B—C14B | 2.2 (16) |
| C11A—C12A—C13A—C14A | −179.9 (3) | C11B—C12B—C13B—C14B | 173.7 (11) |
| C12A—C13A—C14A—C15A | −0.8 (10) | C12B—C13B—C14B—C15B | 4 (3) |
| C13A—C14A—C15A—C16A | 2.3 (18) | C13B—C14B—C15B—C16B | −11 (5) |
| C13A—C14A—C15A—Br3A | 177.9 (6) | C13B—C14B—C15B—Br3B | −173 (2) |
| C14A—C15A—C16A—C17A | −3 (2) | C14B—C15B—C16B—C17B | 11 (6) |
| Br3A—C15A—C16A—C17A | −178.4 (8) | Br3B—C15B—C16B—C17B | 174 (2) |
| C13A—C12A—C17A—C16A | −0.7 (7) | C15B—C16B—C17B—C12B | −5 (4) |
| C11A—C12A—C17A—C16A | 179.2 (5) | C13B—C12B—C17B—C16B | −2 (2) |
| C15A—C16A—C17A—C12A | 2.2 (13) | C11B—C12B—C17B—C16B | −173.4 (18) |
| N1A—C8A—C9A—O3A | 14.4 (6) | N1B—C8B—C9B—O3B | 19 (2) |
| C7A—C8A—C9A—O3A | −160.9 (4) | C7B—C8B—C9B—O3B | −162.4 (13) |
| N1A—C8A—C9A—C10A | −162.3 (4) | N1B—C8B—C9B—C10B | −163.2 (16) |
| C7A—C8A—C9A—C10A | 22.4 (6) | C7B—C8B—C9B—C10B | 15.8 (17) |
| O3A—C9A—C10A—C11A | 34.0 (4) | O3B—C9B—C10B—C11B | −48.6 (13) |

| C8A—C9A—C10A—C11A | −149.2 (3) | C8B—C9B—C10B—C11B | 133.2 (10) |
| O3A—C9A—C10A—Br1A | −83.8 (3) | O3B—C9B—C10B—Br2B | 62.0 (10) |
| C8A—C9A—C10A—Br1A | 93.0 (3) | C8B—C9B—C10B—Br2B | −116.1 (8) |
| C13A—C12A—C11A—C10A | −37.9 (4) | C9B—C10B—C11B—C12B | 175.8 (7) |
| C17A—C12A—C11A—C10A | 142.2 (3) | Br2B—C10B—C11B—C12B | 66.9 (8) |
| C13A—C12A—C11A—Br2A | 76.9 (3) | C9B—C10B—C11B—Br1B | −64.9 (8) |
| C17A—C12A—C11A—Br2A | −103.0 (3) | Br2B—C10B—C11B—Br1B | −173.8 (4) |
| C9A—C10A—C11A—C12A | −171.6 (2) | C13B—C12B—C11B—C10B | 51.9 (13) |
| Br1A—C10A—C11A—C12A | −57.5 (3) | C17B—C12B—C11B—C10B | −136.7 (10) |
| C9A—C10A—C11A—Br2A | 70.8 (2) | C13B—C12B—C11B—Br1B | −63.2 (10) |
| Br1A—C10A—C11A—Br2A | −175.09 (13) | C17B—C12B—C11B—Br1B | 108.2 (9) |

*Hydrogen-bond geometry (Å, º)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C10$A$—H10$A$⋯O2$A$ | 0.98 | 2.40 | 3.168 (4) | 135 |
| C14$A$—H14$A$⋯Br3$A$[i] | 0.93 | 2.91 | 3.809 (5) | 163 |

Symmetry code: (i) −$x$+3/2, $y$−1/2, −$z$+1/2.