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# 1-(2-Bromoethyl)-1,4-diazoniabicyclo-[2.2.2]octane bromide dihydrogen phosphate–phosphoric acid (1/1)

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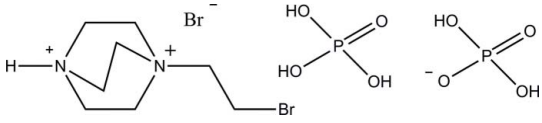
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.057; $wR$ factor = 0.147; data-to-parameter ratio = 20.1.

In the crystal structure of the title compound, $C_8H_{17}BrN_2^{2+}\cdot Br^-\cdot H_2PO_4^-\cdot H_3PO_4$, the cations, anions and phosphoric acid molecules are linked by O—H···O, N—H···O and O—H···Br hydrogen bonds into layers parallel to (101).

## Related literature

For the dielectric properties of *N*-protonated compounds, see: Szafranski & Katrusiak (2008); Katrusiak & Szafranski (1999); Chen *et al.* (2008); Fu *et al.* (2009); Zhao *et al.* (2008).



## Experimental

### Crystal data

$C_8H_{17}BrN_2^{2+}\cdot Br^-\cdot H_2PO_4^-\cdot H_3PO_4$
$M_r$ = 496.02
Monoclinic, $P2_1/c$
$a$ = 12.963 (3) Å
$b$ = 7.5959 (15) Å
$c$ = 17.721 (4) Å
$\beta$ = 92.00 (3)°
$V$ = 1743.9 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.87 mm$^{-1}$
$T$ = 293 K
0.3 × 0.25 × 0.2 mm

### Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min}$ = 0.240, $T_{max}$ = 0.379
17431 measured reflections
3991 independent reflections
3097 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.072

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
$S$ = 1.10
3991 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.03 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*C*···O2 | 0.91 | 1.79 | 2.667 (6) | 162 |
| O1—H1*D*···O6 | 0.82 | 1.71 | 2.519 (5) | 170 |
| O4—H4*C*···O3$^{i}$ | 0.82 | 1.80 | 2.560 (5) | 155 |
| O5—H5*C*···O3$^{i}$ | 0.82 | 1.73 | 2.555 (5) | 179 |
| O7—H7*C*···O2$^{ii}$ | 0.82 | 1.77 | 2.555 (5) | 159 |
| O8—H8*D*···Br1$^{iii}$ | 0.96 | 2.18 | 3.100 (4) | 160 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by a start-up grant from Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2443).

## References

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# supplementary materials

*Acta Cryst.* (2010). E**66**, o1339 [ **doi:10.1107/S1600536810017071** ]

# 1-(2-Bromoethyl)-1,4-diazoniabicyclo[2.2.2]octane bromide dihydrogen phosphate-phosphoric acid (1/1)

**J.-M. Xiao**

## Comment

The variable-temperature dielectric response, especially in relatively high frequency range, is very useful for searching phase transitions in which there is a dielectric anomaly at the transition temperature (Szafranski & Katrusiak, 2008; Katrusiak & Szafranski, 1999; Chen *et al.*, 2008; Fu *et al.*, 2009; Zhao *et al.*, 2008). Unluckily, the title compound, whose crystal structure is reported herein, has no dielectric disuniformity from 93 K to 400 K (m.p. = 410-412 K).

The asymmetric unit of the title compound (Fig. 1) consists of a 1-(2-bromoethyl)-1,4-diazabicyclo[2.2.2]octane-1,4-diium dication, a bromide anion, a dihydrogenphosphate anion and a phosphoric acid molecule. In the crystal packing (Fig. 2), intermolecular N—H···O, O—H···O and O—H···Br hydrogen interactions (Table 1) link ions and molecules forming layers parallel to the (101) plane.

## Experimental

To a solution of 1,4-diazabicyclo[2.2.2]octane (5.6 g, 0.05 mol) in chloroform (20 ml) 1,2-dibromoethane (0.048 mol) was added in one portion and the mixture was refluxed for 8 hour. On standing for about 16 hour at room temperature, colourless hygroscopic crystals of 4-diazabicyclo[2.2.2]octan-1-ium bromide were obtained. The crude product was collected and dissolved in 20 ml methanol, then 1 M $H_3PO_4$ was dropped slowly with stirring. The white precipitate formed (yield 80%) was filtered and dissolved in the $H_2O$. After a few weeks, colourless hygroscopic block crystals of the title compound were obtained on slow evaporation of the solvent.

## Refinement

All the H atoms were calculated geometrically and refined using a riding model, with N—H = 0.90 Å, C—H = 0.97 Å, O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C, N)$ or $1.5U_{eq}(O)$.

## Figures

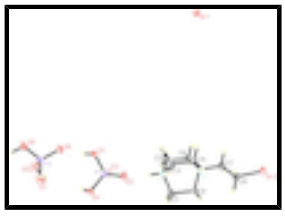

Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

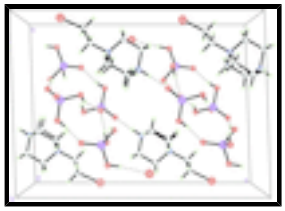

Fig. 2. A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

## 1-(2-Bromoethyl)-1,4-diazoniabicyclo[2.2.2]octane bromide dihydrogen phosphate–phosphoric acid (1/1)

*Crystal data*

| | |
|---|---|
| $C_8H_{17}BrN_2^{2+}\cdot Br^-\cdot H_2PO_4^-\cdot H_3PO_4$ | $F(000) = 992$ |
| $M_r = 496.02$ | $D_x = 1.889$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 7671 reflections |
| $a = 12.963$ (3) Å | $\theta = 3.4$–$27.5°$ |
| $b = 7.5959$ (15) Å | $\mu = 4.87$ mm$^{-1}$ |
| $c = 17.721$ (4) Å | $T = 293$ K |
| $\beta = 92.00$ (3)° | Prism, colourless |
| $V = 1743.9$ (7) Å$^3$ | 0.3 × 0.25 × 0.2 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Rigaku Mercury2 diffractometer | 3097 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.072$ |
| graphite | $\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$ |
| CCD_Profile_fitting scans | $h = -16\rightarrow16$ |
| Absorption correction: multi-scan (CrystalClear; Rigaku, 2005) | $k = -9\rightarrow9$ |
| $T_{min} = 0.240$, $T_{max} = 0.379$ | $l = -23\rightarrow22$ |
| 17431 measured reflections | 1814 standard reflections |
| 3991 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.147$ | H-atom parameters constrained |
| $S = 1.10$ | $w = 1/[\sigma^2(F_o^2) + (0.0703P)^2 + 1.8668P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 3991 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 199 parameters | $\Delta\rho_{max} = 1.03$ e Å$^{-3}$ |

0 restraints $\Delta\rho_{min} = -1.30$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.8045 (5) | 0.7487 (8) | 0.5147 (3) | 0.0471 (14) |
| H1A | 0.8221 | 0.6458 | 0.5450 | 0.057* |
| H1B | 0.8240 | 0.8527 | 0.5436 | 0.057* |
| C2 | 0.8627 (4) | 0.7443 (7) | 0.4414 (3) | 0.0352 (11) |
| H2A | 0.9045 | 0.8496 | 0.4374 | 0.042* |
| H2B | 0.9083 | 0.6431 | 0.4413 | 0.042* |
| C3 | 0.6645 (5) | 0.9084 (8) | 0.4485 (3) | 0.0458 (14) |
| H3A | 0.6851 | 1.0154 | 0.4748 | 0.055* |
| H3B | 0.5905 | 0.9126 | 0.4387 | 0.055* |
| C4 | 0.7193 (4) | 0.8955 (6) | 0.3750 (2) | 0.0283 (10) |
| H4A | 0.7610 | 0.9999 | 0.3681 | 0.034* |
| H4B | 0.6690 | 0.8887 | 0.3333 | 0.034* |
| C5 | 0.6608 (5) | 0.5869 (8) | 0.4562 (3) | 0.0491 (15) |
| H5A | 0.5875 | 0.5894 | 0.4434 | 0.059* |
| H5B | 0.6748 | 0.4858 | 0.4885 | 0.059* |
| C6 | 0.7217 (4) | 0.5720 (6) | 0.3848 (3) | 0.0323 (11) |
| H6A | 0.7654 | 0.4684 | 0.3877 | 0.039* |
| H6B | 0.6745 | 0.5591 | 0.3415 | 0.039* |
| C7 | 0.8408 (4) | 0.7146 (7) | 0.3008 (3) | 0.0378 (12) |
| H7A | 0.8807 | 0.6065 | 0.3019 | 0.045* |
| H7B | 0.7888 | 0.7047 | 0.2603 | 0.045* |
| C8 | 0.9111 (5) | 0.8654 (8) | 0.2840 (3) | 0.0492 (14) |
| H8A | 0.9638 | 0.8778 | 0.3238 | 0.059* |
| H8B | 0.8722 | 0.9743 | 0.2803 | 0.059* |
| N1 | 0.6916 (4) | 0.7512 (5) | 0.4966 (2) | 0.0344 (10) |
| H1C | 0.6570 | 0.7580 | 0.5404 | 0.041* |
| N2 | 0.7874 (3) | 0.7339 (5) | 0.3752 (2) | 0.0262 (8) |
| O1 | 0.4055 (3) | 0.7844 (4) | 0.65616 (19) | 0.0342 (8) |
| H1D | 0.3713 | 0.7503 | 0.6914 | 0.051* |
| O2 | 0.5835 (3) | 0.8397 (4) | 0.6163 (2) | 0.0407 (9) |
| O3 | 0.5392 (3) | 0.5469 (4) | 0.6708 (2) | 0.0401 (9) |

| | | | | |
|---|---|---|---|---|
| O4 | 0.5490 (3) | 0.8038 (5) | 0.7549 (2) | 0.0438 (9) |
| H4C | 0.5100 | 0.8835 | 0.7667 | 0.066* |
| O5 | 0.2784 (3) | 0.9370 (5) | 0.8525 (3) | 0.0589 (11) |
| H5C | 0.3371 | 0.9707 | 0.8448 | 0.088* |
| O6 | 0.2824 (3) | 0.6928 (6) | 0.7558 (2) | 0.0578 (12) |
| O7 | 0.3544 (4) | 0.6584 (6) | 0.8901 (2) | 0.0622 (12) |
| H7C | 0.3639 | 0.5548 | 0.8794 | 0.093* |
| O8 | 0.1613 (4) | 0.6852 (7) | 0.8600 (2) | 0.0734 (15) |
| H8D | 0.1511 | 0.7297 | 0.9099 | 0.110* |
| Br1 | 0.11078 (4) | 0.73719 (7) | 0.02823 (3) | 0.03808 (18) |
| Br2 | 0.97424 (5) | 0.81384 (9) | 0.18848 (3) | 0.0491 (2) |
| P1 | 0.52097 (10) | 0.74245 (15) | 0.67295 (7) | 0.0260 (3) |
| P2 | 0.26999 (11) | 0.73716 (17) | 0.83560 (8) | 0.0336 (3) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.053 (4) | 0.061 (4) | 0.027 (3) | −0.003 (3) | −0.015 (2) | 0.004 (2) |
| C2 | 0.027 (3) | 0.037 (3) | 0.041 (3) | −0.001 (2) | −0.010 (2) | −0.003 (2) |
| C3 | 0.054 (3) | 0.045 (3) | 0.039 (3) | 0.018 (3) | 0.011 (3) | 0.008 (2) |
| C4 | 0.034 (3) | 0.025 (2) | 0.026 (2) | 0.0043 (19) | −0.0025 (19) | 0.0012 (18) |
| C5 | 0.064 (4) | 0.044 (3) | 0.040 (3) | −0.024 (3) | 0.007 (3) | −0.005 (3) |
| C6 | 0.038 (3) | 0.023 (2) | 0.035 (3) | −0.004 (2) | −0.008 (2) | −0.0012 (19) |
| C7 | 0.040 (3) | 0.038 (3) | 0.035 (3) | 0.004 (2) | 0.004 (2) | −0.008 (2) |
| C8 | 0.048 (3) | 0.060 (4) | 0.040 (3) | −0.005 (3) | 0.017 (3) | −0.013 (3) |
| N1 | 0.045 (3) | 0.032 (2) | 0.026 (2) | −0.0003 (18) | 0.0029 (19) | 0.0015 (16) |
| N2 | 0.027 (2) | 0.0226 (18) | 0.029 (2) | 0.0039 (15) | −0.0020 (16) | −0.0014 (15) |
| O1 | 0.0329 (19) | 0.0411 (19) | 0.0284 (17) | 0.0070 (15) | −0.0029 (15) | 0.0076 (15) |
| O2 | 0.052 (2) | 0.0301 (18) | 0.042 (2) | −0.0086 (16) | 0.0173 (17) | 0.0011 (15) |
| O3 | 0.047 (2) | 0.0254 (17) | 0.048 (2) | 0.0046 (15) | 0.0133 (17) | 0.0055 (15) |
| O4 | 0.041 (2) | 0.058 (2) | 0.0319 (19) | 0.0148 (18) | −0.0095 (16) | −0.0110 (17) |
| O5 | 0.053 (2) | 0.040 (2) | 0.085 (3) | −0.0016 (19) | 0.014 (2) | −0.010 (2) |
| O6 | 0.054 (3) | 0.088 (3) | 0.033 (2) | −0.016 (2) | 0.0131 (19) | −0.014 (2) |
| O7 | 0.084 (3) | 0.047 (2) | 0.055 (3) | 0.016 (2) | −0.007 (2) | −0.005 (2) |
| O8 | 0.063 (3) | 0.113 (4) | 0.045 (2) | −0.044 (3) | 0.020 (2) | −0.022 (3) |
| Br1 | 0.0384 (3) | 0.0382 (3) | 0.0377 (3) | 0.0004 (2) | 0.0016 (2) | −0.0041 (2) |
| Br2 | 0.0453 (4) | 0.0619 (4) | 0.0406 (3) | −0.0004 (3) | 0.0118 (3) | −0.0015 (3) |
| P1 | 0.0294 (6) | 0.0237 (6) | 0.0251 (6) | 0.0022 (5) | 0.0035 (5) | 0.0006 (4) |
| P2 | 0.0386 (7) | 0.0318 (7) | 0.0308 (7) | −0.0040 (5) | 0.0064 (6) | −0.0031 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.488 (7) | C7—C8 | 1.500 (8) |
| C1—C2 | 1.526 (8) | C7—N2 | 1.517 (6) |
| C1—H1A | 0.9700 | C7—H7A | 0.9700 |
| C1—H1B | 0.9700 | C7—H7B | 0.9700 |
| C2—N2 | 1.501 (6) | C8—Br2 | 1.945 (5) |
| C2—H2A | 0.9700 | C8—H8A | 0.9700 |
| C2—H2B | 0.9700 | C8—H8B | 0.9700 |

| | | | |
|---|---|---|---|
| C3—N1 | 1.501 (6) | N1—H1C | 0.9100 |
| C3—C4 | 1.510 (7) | O1—P1 | 1.549 (4) |
| C3—H3A | 0.9700 | O1—H1D | 0.8200 |
| C3—H3B | 0.9700 | O2—P1 | 1.507 (3) |
| C4—N2 | 1.511 (6) | O3—P1 | 1.505 (3) |
| C4—H4A | 0.9700 | O4—P1 | 1.556 (4) |
| C4—H4B | 0.9700 | O4—H4C | 0.8200 |
| C5—N1 | 1.486 (6) | O5—P2 | 1.550 (4) |
| C5—C6 | 1.519 (7) | O5—H5C | 0.8200 |
| C5—H5A | 0.9700 | O6—P2 | 1.468 (4) |
| C5—H5B | 0.9700 | O7—P2 | 1.554 (4) |
| C6—N2 | 1.509 (6) | O7—H7C | 0.8200 |
| C6—H6A | 0.9700 | O8—P2 | 1.539 (4) |
| C6—H6B | 0.9700 | O8—H8D | 0.9600 |
| N1—C1—C2 | 109.1 (4) | C8—C7—H7B | 108.9 |
| N1—C1—H1A | 109.9 | N2—C7—H7B | 108.9 |
| C2—C1—H1A | 109.9 | H7A—C7—H7B | 107.7 |
| N1—C1—H1B | 109.9 | C7—C8—Br2 | 107.2 (4) |
| C2—C1—H1B | 109.9 | C7—C8—H8A | 110.3 |
| H1A—C1—H1B | 108.3 | Br2—C8—H8A | 110.3 |
| N2—C2—C1 | 109.9 (4) | C7—C8—H8B | 110.3 |
| N2—C2—H2A | 109.7 | Br2—C8—H8B | 110.3 |
| C1—C2—H2A | 109.7 | H8A—C8—H8B | 108.5 |
| N2—C2—H2B | 109.7 | C5—N1—C1 | 109.8 (4) |
| C1—C2—H2B | 109.7 | C5—N1—C3 | 110.0 (4) |
| H2A—C2—H2B | 108.2 | C1—N1—C3 | 110.0 (4) |
| N1—C3—C4 | 109.3 (4) | C5—N1—H1C | 109.0 |
| N1—C3—H3A | 109.8 | C1—N1—H1C | 109.0 |
| C4—C3—H3A | 109.8 | C3—N1—H1C | 109.0 |
| N1—C3—H3B | 109.8 | C2—N2—C6 | 108.0 (4) |
| C4—C3—H3B | 109.8 | C2—N2—C4 | 108.8 (3) |
| H3A—C3—H3B | 108.3 | C6—N2—C4 | 109.3 (4) |
| C3—C4—N2 | 110.1 (4) | C2—N2—C7 | 112.3 (4) |
| C3—C4—H4A | 109.6 | C6—N2—C7 | 107.2 (4) |
| N2—C4—H4A | 109.6 | C4—N2—C7 | 111.1 (4) |
| C3—C4—H4B | 109.6 | P1—O1—H1D | 109.5 |
| N2—C4—H4B | 109.6 | P1—O4—H4C | 109.5 |
| H4A—C4—H4B | 108.2 | P2—O5—H5C | 109.5 |
| N1—C5—C6 | 109.0 (4) | P2—O7—H7C | 109.5 |
| N1—C5—H5A | 109.9 | P2—O8—H8D | 109.0 |
| C6—C5—H5A | 109.9 | O3—P1—O2 | 112.23 (19) |
| N1—C5—H5B | 109.9 | O3—P1—O1 | 110.5 (2) |
| C6—C5—H5B | 109.9 | O2—P1—O1 | 108.1 (2) |
| H5A—C5—H5B | 108.3 | O3—P1—O4 | 106.7 (2) |
| N2—C6—C5 | 110.2 (4) | O2—P1—O4 | 111.2 (2) |
| N2—C6—H6A | 109.6 | O1—P1—O4 | 108.1 (2) |
| C5—C6—H6A | 109.6 | O6—P2—O8 | 110.1 (3) |
| N2—C6—H6B | 109.6 | O6—P2—O5 | 113.7 (3) |
| C5—C6—H6B | 109.6 | O8—P2—O5 | 104.8 (3) |

| | | | |
|---|---|---|---|
| H6A—C6—H6B | 108.1 | O6—P2—O7 | 114.4 (3) |
| C8—C7—N2 | 113.5 (4) | O8—P2—O7 | 111.0 (3) |
| C8—C7—H7A | 108.9 | O5—P2—O7 | 102.3 (2) |
| N2—C7—H7A | 108.9 | | |
| N1—C1—C2—N2 | −3.1 (6) | C1—C2—N2—C4 | 61.1 (5) |
| N1—C3—C4—N2 | −2.8 (6) | C1—C2—N2—C7 | −175.5 (4) |
| N1—C5—C6—N2 | −4.0 (6) | C5—C6—N2—C2 | 62.0 (5) |
| N2—C7—C8—Br2 | 178.6 (3) | C5—C6—N2—C4 | −56.2 (5) |
| C6—C5—N1—C1 | −58.3 (6) | C5—C6—N2—C7 | −176.8 (4) |
| C6—C5—N1—C3 | 62.8 (6) | C3—C4—N2—C2 | −57.6 (5) |
| C2—C1—N1—C5 | 62.7 (5) | C3—C4—N2—C6 | 60.1 (5) |
| C2—C1—N1—C3 | −58.4 (5) | C3—C4—N2—C7 | 178.2 (4) |
| C4—C3—N1—C5 | −59.0 (6) | C8—C7—N2—C2 | −60.4 (6) |
| C4—C3—N1—C1 | 62.0 (6) | C8—C7—N2—C6 | −178.9 (4) |
| C1—C2—N2—C6 | −57.5 (5) | C8—C7—N2—C4 | 61.8 (6) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1C···O2 | 0.91 | 1.79 | 2.667 (6) | 162. |
| O1—H1D···O6 | 0.82 | 1.71 | 2.519 (5) | 170. |
| O4—H4C···O3$^{i}$ | 0.82 | 1.80 | 2.560 (5) | 155. |
| O5—H5C···O3$^{i}$ | 0.82 | 1.73 | 2.555 (5) | 179. |
| O7—H7C···O2$^{ii}$ | 0.82 | 1.77 | 2.555 (5) | 159. |
| O8—H8D···Br1$^{iii}$ | 0.96 | 2.18 | 3.100 (4) | 160 |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) *x*, *y*, *z*+1.

**Fig. 1**

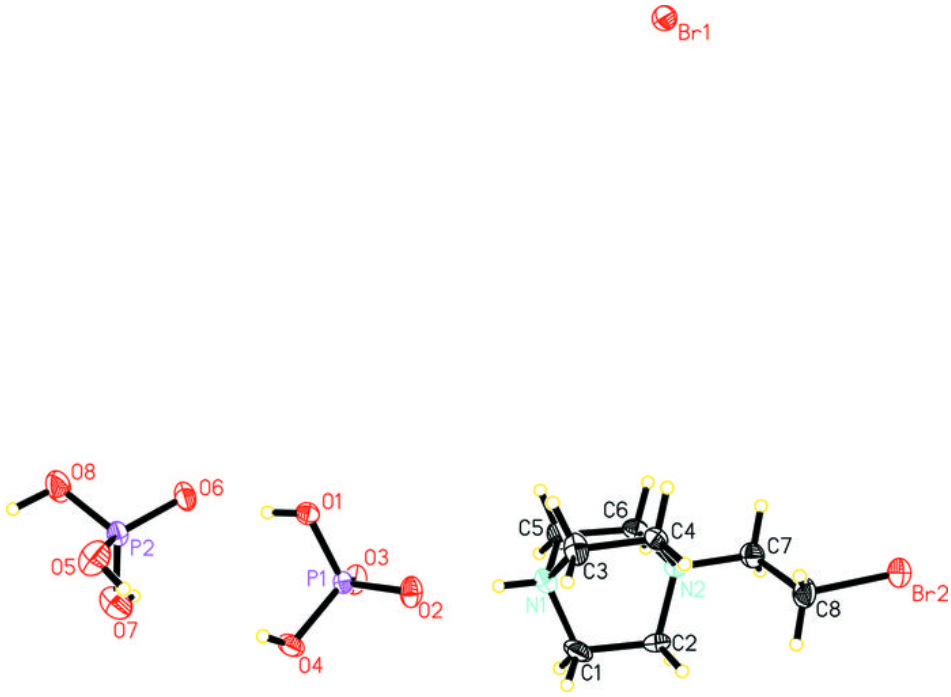

**Fig. 2**