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# Crystal engineering strategies in alkane-bis(ammonium) 2,5-diaza-3,6-dioxobicyclo[2.2.2]octane-1,4-dicarboxylates

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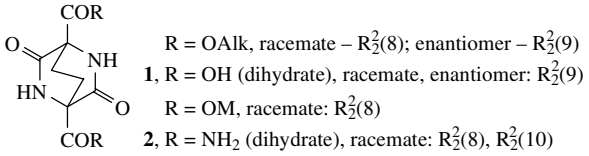
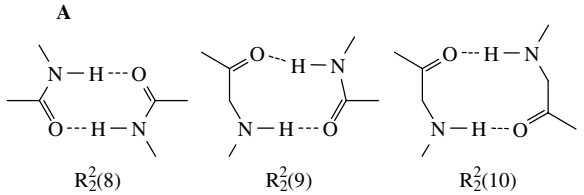
**The crystal structures of (+)-3, (±)-4 and *P*-(–)-5 were studied; the competitive H-bonding of carboxylic groups in (+)-3 and (±)-4 leads to disruption of crystal structure elements, used previously in crystal engineering of chiral bis(lactams); the new structure elements may be described as 1D homochiral $R_2^2(10)$ tapes; the diastereomeric resolution in *P*-(–)-5 is rationalised using an analogy between the H-bonded structure of the salt and 2,5-diaza-3,6-dioxobicyclo[2.2.2]octane-1,4-dicarboxylic acid dihydrate.**

The detailed knowledge of the crystal structures of racemic compounds *vs.* pure enantiomers or racemic conglomerates of organic molecules[1] is important for understanding the phenomena of enantiomeric self-assembly on three-dimensional crystal,[2] 2D interface,[3] 1D helicate[4] and 0D cluster level[5] and for directed construction of chiral quasi-racemates[6] and pre-determined self-resolution.[7] On the level of the 'crystal as a supermolecule', the racemic compound should be treated as achiral meso-diastereomorph, whereas enantiomeric crystals, as the *d*,*l*-diastereomorphic form.[2(b)] The spontaneous resolution of a racemic conglomerate thus is analogous to Pasteur's classical diastereomeric resolutions. We may therefore await similar mechanisms for both phenomena.[8]




R = OAlk, racemate – $R_2^2(8)$; enantiomer – $R_2^2(9)$
**1**, R = OH (dihydrate), racemate, enantiomer: $R_2^2(9)$
R = OM, racemate: $R_2^2(8)$
**2**, R = $NH_2$ (dihydrate), racemate: $R_2^2(8)$, $R_2^2(10)$

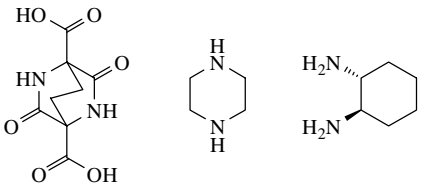
**A**




**Scheme 1** H-bonding graphs[9] in the crystal structures of racemic and enantiopure bis(lactams) **A**.

Previously, we have used racemic and enantiopure bis(lactams) **A** for crystal engineering of organic[10(a),(b)] and metal-organic[10(c)] substances (Scheme 1). The racemic and quasi-racemic compounds tend to form heterochiral zig-zag H-bonded tapes of $R_2^2(8)$ H-bonding graph,[9] and the stacking of such tapes leads to 'molecular brick walls with organic[10] or inorganic[10(c)] coating'. Competitive H-bonding in the case of racemic and enantiopure diacid dihydrates **1** and enantiopure diester (–)-**A** (R = OEt)[10(b)] leads to the formation of homochiral H-bonded 'double zig-zag' tapes of $R_2^2(9)$ graph, united in **1** by $H_2O$ molecules in homochiral layers, with the stacking of such layers in 3D by either a screw axis (**1**, enantiomer) or a glide plane (**1**, racemate).[10(b),11] In the case of primary bis(amide) **2**, the participation of amidic groups in H-bonding leads to alternation of $R_2^2(8)$ and $R_2^2(10)$ graphs in the 'crinkled' tapes, forming unexpected hydrated nano-channels.[12]

In order to use this extensive knowledge in the interpretation of the mechanisms of diastereomeric resolutions and further explore the limits of our crystal engineering strategies, we studied the crystalline properties of racemic and enantiopure salts of acid **1** with organic amines. (1*S*,4*S*)-(+)- and (±)-**1** were crystallised in the presence of an equimolar quantity of piperazine (Pip), whereas (±)-**1**, in the presence of enantiopure (1*R*,2*R*)-(–)- and racemic (±)-*trans*-1,2-diaminocyclohexane (DACH) to give piperazinium (+)-**3**, (±)-**4**, and cyclohexane-*trans*-1,2-diammonium 2,5-diaza-3,6-dioxobicyclo[2.2.2]octane-1,4-dicarboxylates (–)-**5** and (±)-**6** (racemic compound), respectively. (–)-(1*R*,4*R*;1*R*,2*R*)-**5** monohydrate (*P*-salt[2(a)]) was obtained by diastereomeric resolution of racemic **1** (Table 1).† The yield of the diastereomeric crystallization from $H_2O$ was 50% (from pure enantiomer) (*cf.* ref. 11).



**1** Pip DACH

The crystal structures of (+)-**3**, (±)-**4** and (–)-**5** were studied (crystals of **6** were of insufficient quality).‡ In the enantiopure and racemic structures of (+)-**3** and (±)-**4** (Figures 1–3) the formation of zig-zag tapes characteristic of bis(lactams) **A** (see above) is lost. Instead, both in (+)-**3** and (±)-**4**, translation-generated diagonal tapes composed of anions of the same chirality interconnected with $R_2^2(10)$ H-bonding graph[9] (*cf.* bis-amide **2**[12]) are observed (Figure 3). The main difference in the two crystal structures is that the structure of (+)-**3** is a 3D H-bonded framework with the herringbone disposition of anions (Figure 1), whereas in racemic compound (±)-**4**, tapes of different chirality alternate and are joined in centrosymmetric H-bonded layers (Figure 2). The occurrence of similar structural elements in the crystal structures of both enantiopure and racemic compounds is not uncommon,[1,10(*b*)] and it is in accordance with the Kitaigorodsky Aufbau principle.[13]

**Table 1** Compositions of enantiopure and racemic ammonium salts **3**–**6**.

| Compound | Constitution |
|---|---|
| (+)-**3** | (1*S*,4*S*)-(+)-**1**·Pip |
| (±)-**4** | *rac*-(±)-**1**·Pip |
| (–)-**5** | (1*R*,4*R*)-(–)-**1**·(1*R*,2*R*)-(–)-DACH·$H_2O$ |
| (±)-**6** | *rac*-(±)-**1**·*rac*-(±)-DACH |

† The described salts were obtained as white precipitates from MeOH in high yields. The diffraction quality crystals were grown from $H_2O$. Specific rotations were measured on a Polamat A instrument; CD spectra were measured with a Jasco J-500A dichrograph.

(+)-(1*S*,4*S*)-**3**, colourless blocks, mp > 400 °C, specific rotations [$\alpha$] (*c* 1, $H_2O$), $\lambda$/nm: +6.3 (578), +7.2 (546), +12.5 (436), +16 (406), +25 (366).

(±)-**4**, colourless blocks, mp > 400 °C.

*P*-(–)-(1*R*,4*R*;1*R*,2*R*)-**5** monohydrate, colourless prisms, mp 297–299 °C (decomp.); specific rotations [$\alpha$] (*c* 1, $H_2O$): –14 (578), –16 (546), –29 (436), –31 (406), –40 (366); CD spectrum ($3.1\times10^{-3}$ M, $H_2O$), $\Delta\varepsilon$/dm$^3$ mol$^{-1}$ cm$^{-1}$ ($\lambda_{max}$/nm): –0.2 (247), 0.4 (237), –4.9 (221), 1.2 (206).

(±)-**6**, thin needles, mp 329–331 °C, no observable optical rotation. Found (%): C 49.31, H 6.42 N 16.25. Calc. for $C_{14}H_{22}N_4O_6$ (%): C, 49.12; H, 6.48; N, 16.37.

‡ *X-ray diffraction data*. At 298 K crystals of (+)-**3** ($C_{12}H_{18}N_4O_6$) are orthorhombic, space group $P2_12_12_1$, $a$ = 6.811(1), $b$ = 10.891(2) and $c$ = 18.519(4) Å, $V$ = 1373.8(5) Å$^3$, $Z$ = 4 ($Z'$ = 1), $M$ = 314.30, $d_{calc}$ = = 1.520 g cm$^{-3}$, $\mu$(MoK$\alpha$) = 1.23 cm$^{-1}$, $F$(000) = 664.

At 120 K crystals of (±)-**4** ($C_{12}H_{18}N_4O_6$) are monoclinic, space group $C_2/c$, $a$ = 7.037(1), $b$ = 10.558(2) and $c$ = 18.365(3) Å, $\beta$ = 91.723(4)°, $V$ = 1363.8(4) Å$^3$, $Z$ = 4 ($Z'$ = 0.5), $M$ = 314.30, $d_{calc}$ = 1.531 g cm$^{-3}$, $\mu$(MoK$\alpha$) = 1.24 cm$^{-1}$, $F$(000) = 664.

At 120 K crystals of (–)-**5** ($C_{14}H_{24}N_4O_7$) are orthorhombic, space group $P2_12_12_1$, $a$ = 10.3802(9), $b$ = 12.110(1) and $c$ = 12.999(1) Å, $V$ = 1634.0(2) Å$^3$, $Z$ = 4 ($Z'$ = 1), $M$ = 360.37, $d_{calc}$ = 1.465 g cm$^{-3}$, $\mu$(MoK$\alpha$) = 1.18 cm$^{-1}$, $F$(000) = 768.

Intensities of 1100 [(+)-**3**], 3904 [(±)-**4**] and 11145 [(–)-**5**] reflections were measured with a Siemens P3/PC diffractometer at 298 K [$\lambda$(MoK$\alpha$) = = 0.71072 Å, $2\theta < 52°$ (+)-**3**] and a Smart 1000 CCD diffractometer at 120 K [$\lambda$(MoK$\alpha$) = 0.71072 Å, $2\theta < 56$ for (±)-**4** and $2\theta < 58$ for (–)-**5**)]. The structures were solved by a direct method and refined by the full-matrix least-squares technique against $F^2$ in the anisotropic-isotropic approximation. The hydrogen atoms were located from the Fourier density synthesis.

The refinement converged to $wR_2$ = 0.0810 and GOF = 0.991 for all 1100 independent reflections [$R_1$ = 0.0301 for 987 observed reflections with $I > 2\sigma(I)$] for (+)-**3**; $wR_2$ = 0.1581 and GOF = 1.086 for 1654 independent reflections [$R_1$ = 0.0559 for 1288 observed reflections with $I > 2\sigma(I)$] for (±)-**4**; $wR_2$ = 0.0809 and GOF = 1.049 for 4304 independent reflections [$R_1$ = 0.0344 for 4060 observed reflections with $I > 2\sigma(I)$] for (–)-**5**. All calculations were performed using SHELXTL PLUS 5.0.

Atomic coordinates, bond lengths, bond angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre (CCDC). These data can be obtained free of charge *via* www.ccdc.cam.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or deposit@ccdc.cam.ac.uk). Any request to the CCDC for data should quote the full literature citation and CCDC reference numbers 256801–256803. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2004.

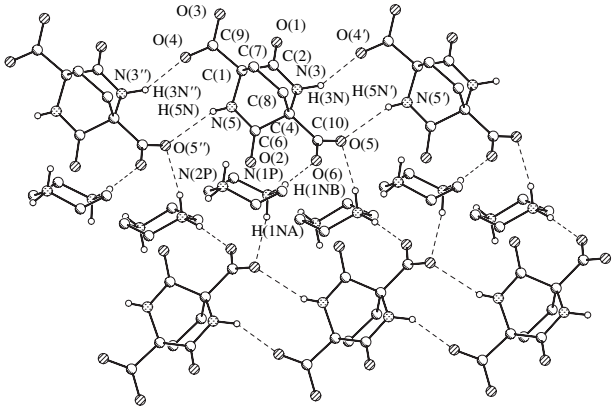



**Figure 1** Projection of the 3D H-bonded network in the crystal structure of (+)-**3**. The parameters of H-bonds: N(3)–H(3N)···O(4′) ($x-1$, $y$, $z$) [N(3)···O(4′) 2.857 Å, H(3N)···O(4′) 2.01 Å, N(3)H(3N)O(4′) 149°], N(5)–H(5N)···O(5″) ($x+1$, $y$, $z$) [N(5)···O(5″) 2.986 Å, H(5N)···O(5″) 2.28 Å, N(5)H(5N)O(5″) 154°], N(1P)–H(1NB)···O(6) [N(1P)···O(6) 2.716 Å, H(1NB)···O(6) 1.741 Å, N(1P)H(1NB)O(6) 169°], N(2P)–H(2NA)···O(3) ($-x+3/2$, $-y$, $z+1/2$) [N(2P)···O(3) 2.675 Å, H(2NA)···O(3) 1.768 Å, N(2P)H(2NA)O(3) 156°], N(2P)–H(2NB)···O(1) ($-x+1$, $y-1/2$, $-z+3/2$) [N(2P)···O(1) 2.791 Å, H(2NB) 2.119 Å, N(2P)H(2NB)O(1) 133°], N(2P)–H(2NB)···O(3) ($-x+1$, $y-1/2$, $-z+3/2$) [N(2P)···O(3) 3.028 Å, H(2NB)···O(3) 2.263 Å, N(2P)H(2NB)O(3) 145°], N(1P)–H(1NA)···O(5) ($x+1/2$, $-y+1/2$, $-z+2$) [N(1P)···O(5) 2.850 Å, H(1NA)···O(5) 2.017 Å, N(1P)H(1NA)O(5) 158°], N(1P)–H(1NA)···O(6) ($x+1/2$, $-y+1/2$, $-z+2$) [N(1P)···O(6) 3.164 Å, H(1NA)···O(6) 2.517 Å, N(1P)H(1NA)O(6) 131°].

For (–)-**5**, the anions form obviously homochiral double zig-zag tapes with $R_2^2(9)$ H-bonding graph, which are united by ammonium groups in the layers with the same H-bonding topology, as previously found for (±)- and (–)-**1** dihydrates[10(*b*),11] (Figure 4). In this case, the diamine molecules play the role of two water molecules in their H-bonding capabilities, and proton transfer to N atoms does not change the overall topology. Moreover, the removal of the possibility of glide plane symmetry of the adjacent H-bonded layers by introduction of enantiopure (–)-DACH leads to the screw axis relationship [compare (–)-**1** and (±)-**1**] and 3D diastereomeric resolution. The effect of hydrating $H_2O$ molecule is demonstrated, most of all, by a weak-

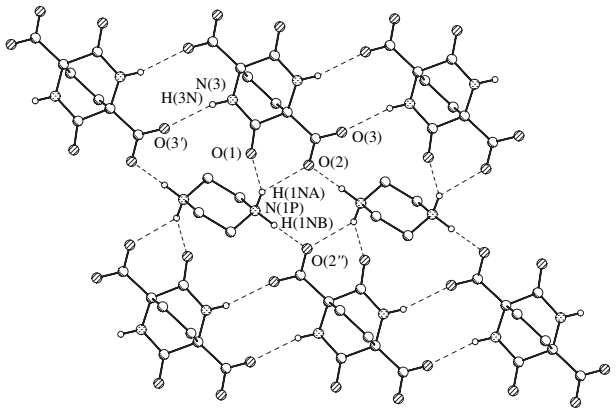



**Figure 2** Alternating homochiral tapes of $R_2^2(10)$ graph in (±)-**4**. The parameters of H-bonds: N(1P)–H(1NA)···O(1) [N(1P)···O(1) 2.912(2) Å, H(1NA)···O(1) 2.10 Å, N(1P)H(1NA)O(1) 150°], N(1P)–H(1NA)···O(2) [N(1P)···O(3) 2.793(2) Å, H(1NA)···O(1) 2.19 Å, N(1P)H(1NA)O(1) 124°]; N(1P)–H(1NB)···O(2″) ($x-1/2$, $y+1/2$, $z$) [N(1P)···O(2′) 2.642(2), H(1NB)···O(2) 1.68, N(1P)H(1NA)O(1) 180°], N(3)–H(3N)···O(3′) [N(3)···O(3′) 2.934(2) Å, H(3N)···O(3′) 2.13 Å, N(3)H(3N)O(3′) 158°].

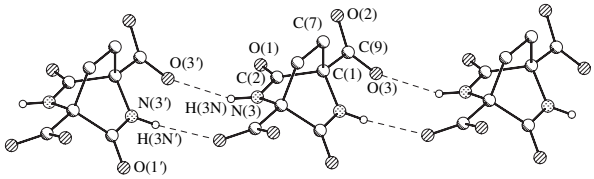



**Figure 3** Homochiral tapes found in the crystal structures of racemic and enantiomeric (±)-**4** and (+)-**3**.

ening of the N(5)H(5N)···O(1′) H-bond (3.315 Å *vs.* 3.1–3.2 Å in **1**).

In conclusion, note the strongest intermolecular interaction in the the crystals is ionic H-bonding, which defines their peculiarities; for example, the presence of bifurcated H-bonds. Moreover, in the crystal structures of (+)-**3** and (±)-**4** the strongly competitive H-bonding of carboxylic groups leads to disruption of crystal structure elements, used previously in crystal engineering of chiral bis(lactams). New structure elements may be described as 1D homochiral $R_2^2(10)$ tapes.

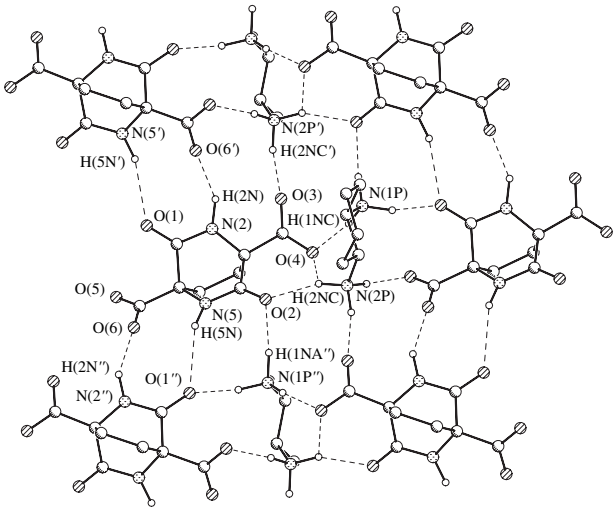



**Figure 4** Projection of the H-bonded layers in the crystal structure of (–)-**5**. The parameters of H-bonds: N(2)–H(2N)···O(6′) (–*x* + 1, *y* – 1/2, –*z* + 1/2) [N(2)···O(6) 2.802(2) Å, H(2N)···O(6′) 2.04 Å, N(2)H(2N)O(6′) 154°], N(5)–H(5N)···O(1′) (–*x* + 1, *y* + 1/2, –*z* + 1/2) [N(5)···O(1′) 3.315(2) Å, H(5N)···O(1) 2.58 Å, N(5)H(5N)O(1′) 151°], N(1P)–H(1NA)···O(2′) (–*x* + 1, *y* – 1/2, –*z* + 1/2) [N(1P)···O(2′) 2.951(2) Å, H(1NA)···O(2′) 2.11 Å, N(1P)H(1NA)O(2′) 170°], N(1P)–H(1NB)···O(1) [N(1P)···O(1) 2.775(2) Å, H(1NB)···O(1) 1.93 Å, N(1P)H(1NB)O(1) 162°], N(1P)–H(1NC)···O(4′) (*x* – 1, *y*, *z*) [N(1P)···O(4′) 2.798(2) Å, H(1NC)···O(4′) 1.89 Å, N(1P)-H(1NC)O(4′) 161°], N(2P)–H(2NA)···O(3′) (–*x* + 1, *y* + 1/2, –*z* + 1/2) [N(2P)···O(3′) 2.753(2) Å, H(2NA)···O(3′) 1.85 Å, N(2P)H(2NA)O(3′) 163°], N(2P)–H(2NB)···O(5) [N(2P)···O(5) 2.740(2) Å, H(2NB)···O(5) 1.89 Å, N(2P)H(2NB)O(5) 159°], N(2P)–H(2NC)···O(2′) (*x* – 1, *y*, *z*) [N(2P)···O(2′) 2.919(2) Å, H(2NC)···O(2′) 2.08 Å, N(2P)H(2NC)O(2′) 157°], N(2P)–H(2NC)···O(4′) (*x* – 1, *y*, *z*) [N(2P)···O(2′) 2.862(2) Å, H(2NC)···O(4′) 2.34 Å, N(2P)H(2NC)O(4′) 118°].

The crystal structure of (–)-**5** is hydrated; however, the previously found homochiral $R_2^2(9)$ H-bonding pattern is not significantly perturbed. Moreover, DACH molecules play the same role in (–)-**5** as the hydrating $H_2O$ molecules in the previously studied crystal structures of (–)-**1** and (±)-**1** in the formation of H-bonded homochiral layers. The screw axis relationship of H-bonded layers is the mechanism of the overall diastereomeric resolution. The homochiral 3D-H-bonded structure of (+)-**3** is non-centrosymmetric, non-solvated, high-melting (> 400 °C) and offers perspectives for use as a non-linear optical, pyro- or piezoelectric material.

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