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2-Aminopyrimidine–Succinic Acid (1/1) Cocrystal

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Abstract. 2-Aminopyrimidine(2AP)–succinic acid (SA) 1:1 cocrystal, $C_4H_5N_3.C_4H_6O_4$, $M_r = 213{\cdot}19$, monoclinic, $P2_1/n$, $a = 5{\cdot}045$ (4), $b = 13{\cdot}426$ (5), $c = 15{\cdot}148$ (5) Å, $\beta = 95{\cdot}45$ (5)°, $V = 1021$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}39$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}05$ cm^{-1}, $F(000) = 448$, $T = 296$ K, $R = 0{\cdot}054$ for 1567 unique observed reflections. The cocrystal is composed of infinite chains of 2AP and SA molecules associated by eight-membered hydrogen-bonded rings [NH···O = 2·981 (2) and 2·955 (3) Å, OH···N = 2·665 (2) and 2·690 (2) Å], similar to the ring pattern in cocrystals of adenine and 3-bromobenzoic acid.

Experimental. Colorless, sword-like crystals obtained from acetonitrile. Crystal of dimensions 0·60 × 0·35 × 0·10 mm. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator; lattice parameters obtained from least-squares analysis of 24 reflections in the range $21 < 2\theta < 36°$; data collected by ω scan mode up to 51·9° in 2θ; $0 \le h \le 6$, $0 \le k \le 16$, and $-18 \le l \le 18$; 2103 reflections collected; three standard reflections, 2·3% decay, corrected with a linear correction factor; empirical absorption correction applied using *DIFABS* (Walker & Stuart, 1983), transmission factor range = 0·78–1·15. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens *et al.*, 1984). The non-H atoms were refined anisotropically. H-atom positions and isotropic thermal parameters for H1, H2, H2′, and H3 were refined; other H atoms were included in the structure factor calculation in idealized positions (C—H = 0·95 Å) with fixed isotropic $B = 1{\cdot}2 \times B$ of bonded atom; $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/\sigma^2(F_o)$. Final $R = 0{\cdot}054$, $wR = 0{\cdot}068$, $S = 2{\cdot}26$ for 1567 unique observed reflections [$I > 3\sigma(I)$], 153 parameters, $(\Delta/\sigma)_{max} = 0{\cdot}07$, $(\Delta\rho)_{max} = 0{\cdot}40$ e Å^{-3}, $(\Delta\rho)_{min} = -0{\cdot}54$ e Å^{-3}. All calculations using *TEXSAN* (Molecular Structure Corporation, 1985) with scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters are listed

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Table 1. *Fractional coordinates and* B_{eq} *values* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1	0·6041 (3)	0·4531 (1)	0·64103 (9)	4·67 (6)
O2	0·8280 (3)	0·3844 (1)	0·75841 (8)	4·54 (6)
O3	0·8447 (3)	0·6130 (1)	0·97100 (9)	4·53 (6)
O4	0·9028 (3)	0·6188 (1)	0·82719 (8)	4·75 (6)
N1	0·7366 (3)	0·7515 (1)	0·4992 (1)	3·77 (6)
N2	0·8407 (3)	0·7351 (1)	0·3554 (1)	4·18 (7)
N3	1·0803 (3)	0·6420 (1)	0·4626 (1)	3·80 (6)
C2	0·8861 (3)	0·7089 (1)	0·4404 (1)	3·31 (7)
C4	1·1234 (4)	0·6175 (1)	0·5474 (1)	4·37 (8)
C5	0·9803 (4)	0·6573 (2)	0·6118 (1)	4·8 (1)
C6	0·7875 (4)	0·7248 (2)	0·5836 (1)	4·38 (8)
C7	0·6537 (3)	0·4387 (1)	0·7268 (1)	3·51 (7)
C8	0·4674 (3)	0·4952 (1)	0·7797 (1)	4·31 (8)
C9	0·5679 (3)	0·5112 (1)	0·8756 (1)	3·97 (8)
C10	0·7880 (3)	0·5860 (1)	0·8880 (1)	3·49 (7)
H1	0·710 (5)	0·414 (2)	0·611 (2)	6·7 (6)
H2	0·702 (5)	0·773 (2)	0·339 (1)	5·5 (5)
H2′	0·935 (4)	0·708 (2)	0·313 (1)	4·7 (4)
H3	0·993 (5)	0·661 (2)	0·979 (2)	8·3 (8)

Table 2. *Selected intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O1—H1	0·90 (3)	N2—H2′	0·91 (2)
O1—C7	1·314 (2)	N2—C2	1·334 (2)
O2—C7	1·206 (2)	N3—C4	1·324 (2)
O3—H3	0·99 (3)	N3—C2	1·348 (2)
O3—C10	1·314 (2)	C4—C5	1·376 (3)
O4—C10	1·216 (2)	C5—C6	1·368 (3)
N1—C6	1·329 (2)	C7—C8	1·498 (3)
N1—C2	1·348 (2)	C8—C9	1·509 (3)
N2—H2	0·88 (2)	C9—C10	1·496 (3)
H1—O1—C7	111 (2)	C6—C5—C4	116·2 (2)
H3—O3—C10	113 (2)	N1—C6—C5	123·1 (2)
C6—N1—C2	116·8 (2)	O2—C7—O1	123·0 (2)
H2—N2—H2′	119 (2)	O2—C7—C8	124·4 (2)
H2—N2—C2	119 (1)	O1—C7—C8	112·6 (2)
H2′—N2—C2	121 (1)	C7—C8—C9	114·6 (1)
C4—N3—C2	117·1 (2)	C10—C9—C8	113·2 (1)
N2—C2—N3	118·1 (2)	O4—C10—O3	123·3 (2)
N2—C2—N1	118·0 (2)	O4—C10—C9	123·4 (2)
N3—C2—N1	123·9 (2)	O3—C10—C9	113·4 (1)
N3—C4—C5	122·8 (2)		

Table 3. *Hydrogen-bonding geometry*

D—H···*A*	Symmetry	*D*···*A* (Å)	*D*—H···*A*(°)
O1—H1···N3	$2-x, 1-y, 1-z$	2·665 (2)	169 (2)
O3—H3···N1	$\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$	2·690 (2)	176 (3)
O2···H2′—N2	$2-x, 1-y, 1-z$	2·981 (2)	165 (2)
O4···H2—N2	$\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$	2·955 (3)	166 (2)

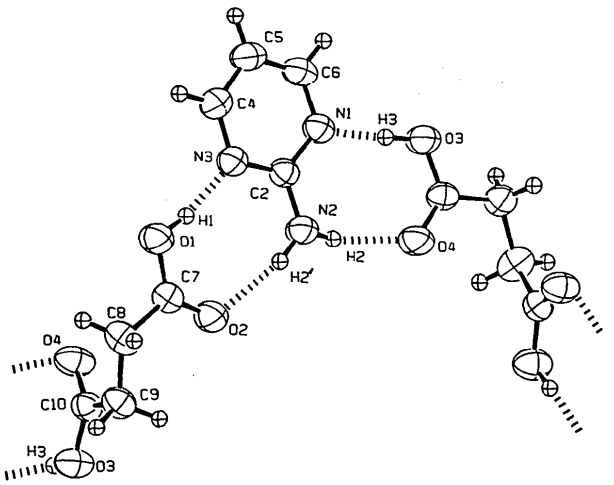

Fig. 1. *ORTEP* (Johnson, 1976) drawing (thermal ellipsoids, 50% probability level and H atoms of arbitrary size) of the hydrogen-bond pattern found in the 1:1 cocrystal of 2AP–SA. Dashed lines indicate hydrogen bonds. These three molecules are a portion of the hydrogen-bonded chain extended along the *b* axis.

in Table 1,* selected interatomic distances and angles are given in Table 2, and hydrogen-bond data in Table 3. The atomic numbering and hydrogen-bond scheme are shown in the *ORTEP* (Johnson, 1976) drawing in Fig. 1.

Related literature. 2-Aminopyrimidines typically associate *via* eight-membered hydrogen-bonded rings as seen in the crystal structures of 2-aminopyrimidine (Scheinbeim & Schempp, 1976), 2-amino-4,6-dichloropyrimidine (Clews & Cochran, 1948), and 2-amino-5-bromopyrimidine (Watton, Low, Tollin, & Howie, 1988). Succinic acid (Verweel & Macgillavry, 1939) forms typical carboxylic acid hydrogen-bonded rings. Adenine and 3-bromobenzoic acid cocrystallize to form an eight-membered hydrogen-bonded ring between the acid and aminopyrimidine moieties (Tamura, Sakurai, & Sato, 1971) similiar to the pattern in the 1:1 cocrystal of 2AP–SA described here.

* Lists of structure factors, anisotropic thermal parameters, least-squares planes, H-atom parameters and intermolecular distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52517 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Intramolecular Free-Radical Ring Closures. I. Structure of a Chiral Bicyclic Lactone*

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Abstract. $C_{13}H_{18}O_6$, $M_r = 270{\cdot}28$, monoclinic, $P2_1$, $a = 6{\cdot}6619$ (3), $b = 10{\cdot}387$ (7), $c = 19{\cdot}895$ (2) Å, $\beta = 97{\cdot}33$ (4)°, $V = 1356{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}323$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}85$ mm^{-1}, $F(000) = 576$, $T = 296$ K, $R = 0{\cdot}039$ for 1812 observed reflections. A tin-mediated intramolecular radical cyclization of a chloromethyl ester afforded the title lactone in crystalline form. Although spectroscopic assignment of the reaction

* Methyl (1*R*,2*S*,6*R*)-4,5-isopropylidenedioxy-8-oxo-*cis*-7-oxabicyclo[4.3.0]nonane-2-carboxylate.

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