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2,6-Diamino-4-chloropyrimidine–succinic acid (2/1)

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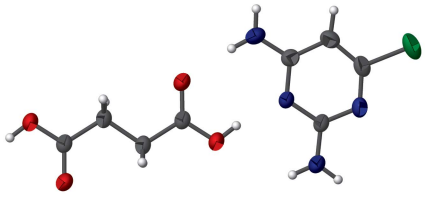
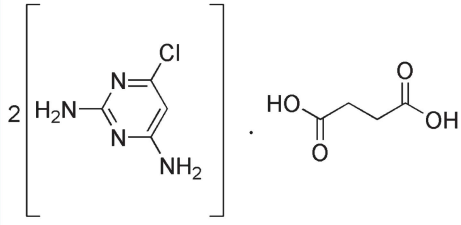
In the title 2:1 co-crystal, $2C_4H_5ClN_4\cdot C_4H_6O_4$ the complete succinic acid molecule is generated by a crystallographic centre of symmetry. In the crystal, pairwise O—H···N and N—H···O hydrogen bonds link the pyrimidine and succinic acid molecules, generating $R_2^2(8)$ loops. The pyrimidine molecules are linked by pairwise N—H···N hydrogen bonds, again generating $R_2^2(8)$ loops. Collectively, the hydrogen bonds link the components into corrugated (100) sheets. The Hirshfeld surface is presented.

3D view



Chemical scheme



Structure description

Some aminopyrimidine derivatives are used as antifolate drugs (Hunt *et al.*, 1980). The crystal structures of various aminopyrimidine derivatives (Schwalbe & Williams, 1982; Edison *et al.*, 2014; Thanigaimani *et al.*, 2012) have been reported. In the present study, the synthesis and structure of the title 2:1 co-crystal are described.

The complete succinic acid molecule is generated by a crystallographic centre of symmetry (Fig. 1), with key torsion angles O1—C5—C6—C6^{i} = −2.5 (3)° and O2—C5—C6—C6^{i} = 178.28 (18)° [symmetry code: (i) 2 − *x*, 3 − *y*, 1 − *z*].

In the crystal, pairwise O2—H2···N2 and N4—H4*A*··O1 hydrogen bonds (for symmetry codes, see Table 1) link the pyrimidine and succinic acid molecules, generating $R_2^2(8)$ loops. The mean planes of the succinic acid and linked pyrimidine molecules are close to parallel [dihedral angle = 8.67 (6)°]. The pyrimidine molecules are linked by pairwise N3—H3*A*···N1^{i} hydrogen bonds, again generating $R_2^2(8)$ loops. An N4—H4*B*···O1^{ii} hydrogen bond also links the pyrimidine and succinic acid species. Collectively, the hydrogen bonds link the components into corrugated (100) sheets (Fig. 2).

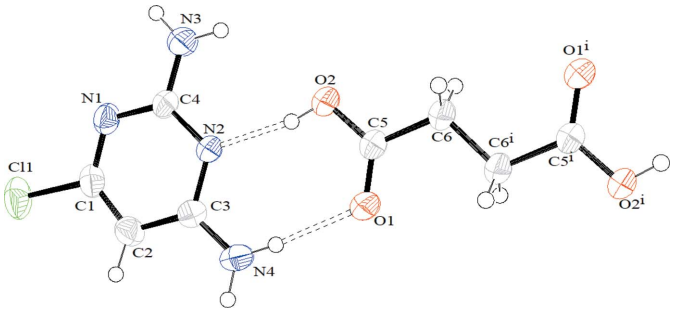


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids and hydrogen bonds indicated by dashed lines. Symmetry code: (i) $2-x, 3-y, 1-z$.

The Hirshfeld surface (Turner *et al.*, 2017) of the pyrimidine–succinic acid grouping is shown in Fig. 3, where red spots represent short intermolecular contacts associated with the various hydrogen bonds. The most significant contact percentages arising from two-dimensional fingerprint plots are: H···H = 32.5%, O···H/H···O = 19.7%, N···H/H···N = 13.6%, Cl···H/H···Cl = 7.9%, H···C/C···H = 5.5% and O···C/C···O = 4.8%. Other contact types contribute a negligible amount.

Synthesis and crystallization

A 10 ml methanolic solution (hot) of 2,6-diamino-4-chloropyrimidine (32 mg) and a 10 ml aqueous solution (hot) of succinic acid (29 mg) were mixed and heated for 10 min and then cooled to room temperature. Colourless blocks grew over the course of a few days as the solvents evaporated.

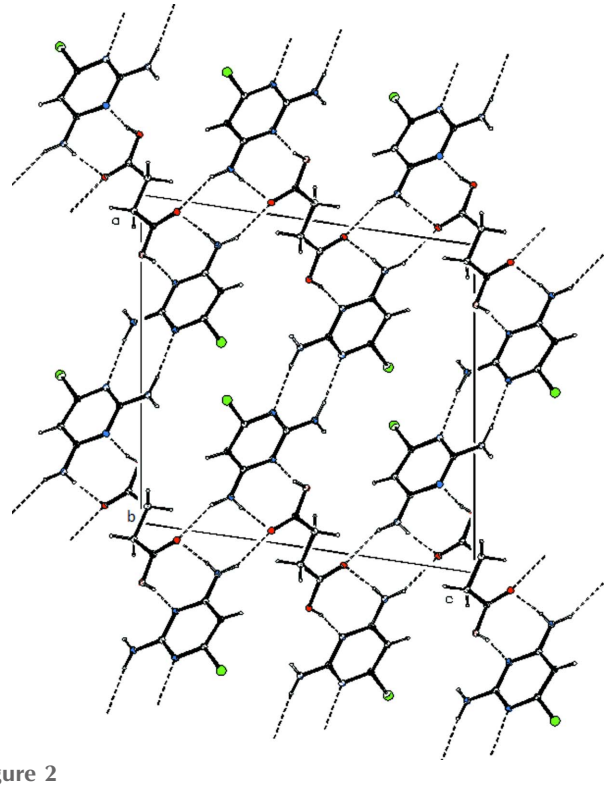


Figure 2
Partial packing diagram of the title compound showing hydrogen bonds as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N2	0.82	1.81	2.622 (2)	169
N3—H3A···N1^{i}	0.86	2.19	3.048 (2)	173
N4—H4A···O1	0.86	1.94	2.794 (2)	169
N4—H4B···O1^{ii}	0.86	2.04	2.8510 (19)	156

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_4H_5ClN_4\cdot C_4H_6O_4$
M_r	407.23
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	13.2096 (14), 4.9765 (5), 13.5673 (14)
β (°)	98.603 (2)
V (Å^3)	881.85 (16)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.41
Crystal size (mm)	0.72 × 0.34 × 0.13
Data collection	
Diffractometer	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.631, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15031, 2599, 2102
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.707
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.128, 1.04
No. of reflections	2599
No. of parameters	118
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.44

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

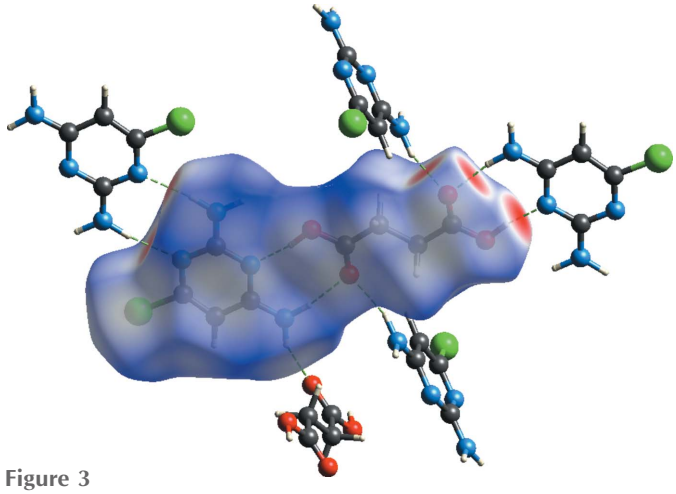


Figure 3
The Hirshfeld surface mapped over d_{norm} for the title compound.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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full crystallographic data

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2,6-Diamino-4-chloropyrimidine–succinic acid (2/1)

Crystal data

$2C_4H_5ClN_4·C_4H_6O_4$
M_r = 407.23
Monoclinic, $P2_1/c$
a = 13.2096 (14) Å
b = 4.9765 (5) Å
c = 13.5673 (14) Å
β = 98.603 (2)°
V = 881.85 (16) Å^3
Z = 2
$F(000)$ = 420
D_x = 1.534 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3778 reflections
θ = 2.6–29.9°
μ = 0.41 mm^{-1}
T = 296 K
Plate, colourless
0.72 × 0.34 × 0.13 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scan
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.631, T_{max} = 0.746
15031 measured reflections
2599 independent reflections
2102 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 30.2°, θ_{min} = 1.6°
h = −18→18
k = −7→7
l = −18→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.128
S = 1.04
2599 reflections
118 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.3195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 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. The hydrogen atoms were positioned geometrically (C—H = 0.93–0.97 Å, N—H = 0.86) Å, O—H =0.82 Å) and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(carrier).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.58724 (4)	0.10526 (9)	0.74117 (4)	0.06071 (19)
N2	0.74496 (9)	0.7549 (3)	0.60431 (9)	0.0345 (3)
O2	0.81782 (9)	1.1416 (3)	0.50401 (9)	0.0504 (3)
H2	0.803150	1.017333	0.538975	0.076*
O1	0.96386 (9)	1.1222 (3)	0.60813 (9)	0.0523 (3)
N1	0.60385 (10)	0.4544 (3)	0.60332 (10)	0.0400 (3)
C5	0.90949 (11)	1.2237 (3)	0.53630 (11)	0.0367 (3)
C2	0.74276 (13)	0.4470 (3)	0.73964 (12)	0.0420 (4)
H2A	0.771513	0.374017	0.800544	0.050*
C4	0.65260 (11)	0.6548 (3)	0.56340 (11)	0.0358 (3)
C6	0.94673 (11)	1.4522 (3)	0.47896 (12)	0.0391 (3)
H6A	0.899454	1.601474	0.478469	0.047*
H6B	0.946393	1.395885	0.410457	0.047*
N4	0.88038 (11)	0.7585 (3)	0.73071 (11)	0.0514 (4)
H4A	0.906878	0.884453	0.699449	0.062*
H4B	0.912066	0.700416	0.786646	0.062*
C1	0.65147 (12)	0.3614 (3)	0.68959 (12)	0.0392 (3)
C3	0.79074 (11)	0.6540 (3)	0.69271 (11)	0.0364 (3)
N3	0.60778 (11)	0.7630 (3)	0.47853 (11)	0.0532 (4)
H3A	0.549166	0.704581	0.450636	0.064*
H3B	0.637362	0.891492	0.451444	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0568 (3)	0.0454 (3)	0.0867 (4)	0.00090 (19)	0.0328 (3)	0.0224 (2)
N2	0.0290 (6)	0.0381 (6)	0.0350 (6)	−0.0071 (5)	0.0006 (4)	0.0029 (5)
O2	0.0348 (6)	0.0595 (8)	0.0529 (7)	−0.0178 (5)	−0.0068 (5)	0.0173 (6)
O1	0.0417 (6)	0.0602 (8)	0.0495 (7)	−0.0200 (6)	−0.0111 (5)	0.0144 (6)
N1	0.0340 (6)	0.0392 (7)	0.0473 (7)	−0.0080 (5)	0.0078 (5)	0.0021 (5)
C5	0.0322 (7)	0.0381 (7)	0.0390 (7)	−0.0070 (6)	0.0028 (5)	−0.0001 (6)
C2	0.0424 (8)	0.0426 (8)	0.0415 (8)	0.0049 (6)	0.0077 (6)	0.0102 (6)
C4	0.0304 (6)	0.0397 (7)	0.0366 (7)	−0.0074 (6)	0.0028 (5)	−0.0005 (6)
C6	0.0324 (7)	0.0383 (8)	0.0456 (8)	−0.0076 (6)	0.0026 (6)	0.0048 (6)
N4	0.0412 (7)	0.0614 (10)	0.0462 (8)	−0.0086 (7)	−0.0113 (6)	0.0121 (7)
C1	0.0379 (7)	0.0333 (7)	0.0495 (8)	0.0011 (6)	0.0170 (6)	0.0052 (6)
C3	0.0336 (7)	0.0379 (7)	0.0372 (7)	0.0017 (6)	0.0030 (5)	0.0007 (6)
N3	0.0432 (7)	0.0651 (10)	0.0460 (8)	−0.0230 (7)	−0.0102 (6)	0.0136 (7)

Geometric parameters (Å, º)

Cl1—C1	1.7356 (16)	C2—H2A	0.9300
N2—C3	1.3561 (18)	C4—N3	1.327 (2)
N2—C4	1.3572 (18)	C6—C6^{i}	1.514 (3)
O2—C5	1.2911 (17)	C6—H6A	0.9700
O2—H2	0.8200	C6—H6B	0.9700
O1—C5	1.2294 (19)	N4—C3	1.325 (2)
N1—C1	1.327 (2)	N4—H4A	0.8600
N1—C4	1.3443 (19)	N4—H4B	0.8600
C5—C6	1.501 (2)	N3—H3A	0.8600
C2—C1	1.361 (2)	N3—H3B	0.8600
C2—C3	1.410 (2)		
C3—N2—C4	118.71 (13)	C5—C6—H6B	108.8
C5—O2—H2	109.5	C6^{i}—C6—H6B	108.8
C1—N1—C4	114.95 (13)	H6A—C6—H6B	107.7
O1—C5—O2	123.08 (14)	C3—N4—H4A	120.0
O1—C5—C6	121.62 (13)	C3—N4—H4B	120.0
O2—C5—C6	115.30 (13)	H4A—N4—H4B	120.0
C1—C2—C3	115.32 (14)	N1—C1—C2	126.81 (14)
C1—C2—H2A	122.3	N1—C1—Cl1	114.59 (12)
C3—C2—H2A	122.3	C2—C1—Cl1	118.61 (13)
N3—C4—N1	118.16 (13)	N4—C3—N2	116.94 (14)
N3—C4—N2	117.58 (14)	N4—C3—C2	123.14 (14)
N1—C4—N2	124.26 (14)	N2—C3—C2	119.91 (14)
C5—C6—C6^{i}	113.62 (16)	C4—N3—H3A	120.0
C5—C6—H6A	108.8	C4—N3—H3B	120.0
C6^{i}—C6—H6A	108.8	H3A—N3—H3B	120.0
C1—N1—C4—N3	−178.01 (15)	C4—N1—C1—Cl1	179.33 (11)
C1—N1—C4—N2	1.9 (2)	C3—C2—C1—N1	−0.8 (3)
C3—N2—C4—N3	177.81 (15)	C3—C2—C1—Cl1	179.50 (12)
C3—N2—C4—N1	−2.1 (2)	C4—N2—C3—N4	−179.40 (14)
O1—C5—C6—C6^{i}	−2.5 (3)	C4—N2—C3—C2	0.7 (2)
O2—C5—C6—C6^{i}	178.28 (18)	C1—C2—C3—N4	−179.27 (16)
C4—N1—C1—C2	−0.4 (2)	C1—C2—C3—N2	0.6 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N2	0.82	1.81	2.622 (2)	169
N3—H3*A*···N1^{ii}	0.86	2.19	3.048 (2)	173
N4—H4*A*···O1	0.86	1.94	2.794 (2)	169
N4—H4*B*···O1^{iii}	0.86	2.04	2.8510 (19)	156

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