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Bis(2-aminopyrimidin-1-ium) dichromate(VI)

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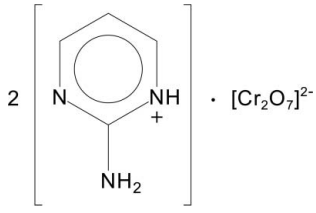
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.070; data-to-parameter ratio = 14.3.

The asymmetric unit of the title salt, $(C_4H_6N_3)_2[Cr_2O_7]$, consists of two 2-aminopyrimidinium cations and one dichromate dianion linked together by N—H···O hydrogen bonds to form a ribbon structure lying parallel to the (013) plane and running along the *a* axis. Pairs of symmetry-related cations are connected into centrosymmetric dimers *via* N—H···N hydrogen bonds, forming eight-membered $R_2^2(8)$ rings.

Related literature

For related literature, see: Allen *et al.* (1987); Etter *et al.* (1990); Jeffrey & Saenger (1991); Sieroń (2007).



Experimental

Crystal data

$(C_4H_6N_3)_2[Cr_2O_7]$
M_r = 408.24
Triclinic, $P\overline{1}$
a = 5.4576 (2) Å
b = 7.6077 (2) Å
c = 18.684 (1) Å
α = 99.429 (1)°
β = 91.277 (1)°
γ = 108.319 (2)°
V = 724.23 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.55 mm^{-1}
T = 293 K
0.35 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.699, T_{max} = 0.861
18234 measured reflections
3308 independent reflections
3215 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.070
S = 1.06
3308 reflections
232 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—O1	1.6040 (14)	Cr2—O4	1.6090 (13)
Cr1—O2	1.6063 (17)	Cr2—O5	1.6175 (12)
Cr1—O3	1.6212 (15)	Cr2—O6	1.6251 (13)
Cr1—O7	1.7739 (13)	Cr2—O7	1.7710 (13)
O1—Cr1—O2	108.35 (8)	O4—Cr2—O7	107.95 (7)
O1—Cr1—O3	111.84 (8)	O5—Cr2—O6	108.76 (7)
O1—Cr1—O7	110.24 (7)	O5—Cr2—O7	109.96 (7)
O2—Cr1—O3	110.62 (8)	O6—Cr2—O7	111.42 (7)
O2—Cr1—O7	110.37 (8)	O4—Cr2—O6	109.11 (7)
O3—Cr1—O7	105.43 (7)	Cr1—O7—Cr2	131.99 (8)
O4—Cr2—O5	109.62 (8)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11···O6^{i}	0.74 (2)	2.14 (2)	2.841 (2)	158 (3)
N21—H21···O3^{ii}	0.75 (3)	2.00 (3)	2.693 (2)	155 (3)
N12—H121···O5^{iii}	0.78 (3)	2.18 (3)	2.930 (2)	162 (3)
N12—H122···N13^{iv}	0.87 (3)	2.19 (3)	3.053 (2)	176 (2)
N22—H221···O2^{v}	0.76 (3)	2.41 (2)	3.042 (2)	141 (2)
N22—H221···O3^{ii}	0.76 (3)	2.59 (2)	3.144 (2)	131 (2)
N22—H222···N23^{vi}	0.81 (3)	2.22 (3)	3.030 (2)	176 (2)
C14—H14···O4^{vii}	0.93	2.38	3.161 (2)	142
C16—H16···O3^{viii}	0.93	2.40	3.312 (2)	165
C24—H24···O1^{ix}	0.93	2.37	3.287 (2)	170
C25—H25···O1^{viii}	0.93	2.46	3.085 (3)	125

Symmetry codes: (i) $x-1, y-1, z$; (ii) $x, y+1, z$; (iii) $x, y-1, z$; (iv) $-x+1, -y, -z$; (v) $x+1, y+1, z$; (vi) $-x+2, -y+2, -z+1$; (vii) $-x+1, -y+1, -z$; (viii) $x-1, y, z$; (ix) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2074).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2002). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SAINT-Plus* (Version 6.45) and *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*. New York: Springer-Verlag.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
Sieroń, L. (2007). *Acta Cryst.* E**63**, m2068.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

Acta Cryst. (2007). E**63**, m2336 [**doi:10.1107/S1600536807039347**]

Bis(2-aminopyrimidin-1-ium) dichromate(VI)

L. Sieron

Comment

The title compound, (I), has been synthetized and investigated as a continuation of the structural study on hydrogen bonding in *N*-heterocyclic dichromate salts (Sieroń, 2007). In (I), the asymmetric unit is composed of two monoprotonated 2-aminopyrimidinium cations and one dichromate dianion (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the crystal structure, pairs of symmetry-related cations are connected into centrosymmetric dimers *via* N–H···N hydrogen bonds, forming eight-membered rings described by the $R_2^2(8)$ graph-set motif (Etter *et al.*, 1990). Cations and anions are linked together by N–H···O hydrogen bonds, forming a ribbon structure lying parallel to the (013) plane and running along the *a* axis (Fig. 2). The hydrogen-bond arrangements around the two independent cations are not identical. One of the H atoms of one 2-aminopyrimidinium ion is engaged in a bifurcated unsymmetrical hydrogen bond [N22···O2^{v} = 3.042 (2) and N22···O3^{ii} = 3.144 (2) Å] to two dichromate O atoms. A bifurcation is confirmed by the sums of angles about atom H221, which is 356° (Jeffrey & Saenger, 1991). The corresponding H121 atom is involved only in one hydrogen-bond [N12···O5^{iii} = 2.930 (2) Å]. A three-dimensional network is built up *via* N–H···O hydrogen bonds, together with weak C–H···O interactions.

Experimental

The title compound was prepared by dissolving 2-aminopirimidine (1 mmol) and chromic anhydride (1 mmol) in hot water (25 ml). The resulting solid was recrystallized from water.

Refinement

All H atoms were initially located in a difference Fourier map. N-bonded hydrogen atoms were refined isotropically. Remaining H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

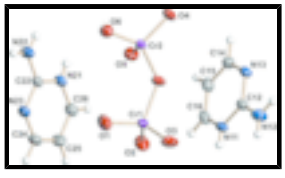

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 40% probability level.

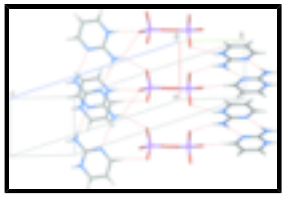

Fig. 2. A packing view of (I), showing a ribbon of hydrogen-bonded (dashed lines) cations and anions running along the *a* axis.

Bis(2-aminopyrimidin-1-ium) dichromate(VI)

Crystal data

$(C_4H_6N_3)_2[Cr_2O_7]$	$Z = 2$
$M_r = 408.24$	$F_{000} = 412$
Triclinic, $P\overline{1}$	$D_x = 1.872$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.4576$ (2) Å	Cell parameters from 7381 reflections
$b = 7.6077$ (2) Å	$\theta = 2.9$–27.9°
$c = 18.684$ (1) Å	$\mu = 1.55$ mm^{-1}
$\alpha = 99.429$ (1)°	$T = 293$ K
$\beta = 91.277$ (1)°	Prism, orange
$\gamma = 108.319$ (2)°	0.35 × 0.20 × 0.10 mm
$V = 724.23$ (5) Å^3	

Data collection

Bruker SMART APEX II CCD diffractometer	3308 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	3215 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
$T = 293$ K	$\theta_{max} = 27.5^\circ$
ω scans	$\theta_{min} = 1.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -7 \rightarrow 7$
$T_{min} = 0.699$, $T_{max} = 0.861$	$k = -9 \rightarrow 9$
18234 measured reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.023$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.382P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
3308 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}

232 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *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}
N11	0.0915 (3)	0.0379 (2)	0.14285 (7)	0.0307 (4)
N12	0.3540 (3)	−0.1153 (2)	0.08361 (10)	0.0403 (5)
N13	0.2962 (3)	0.1365 (2)	0.04052 (7)	0.0288 (4)
C12	0.2490 (3)	0.0192 (2)	0.08896 (8)	0.0269 (4)
C14	0.1811 (3)	0.2672 (2)	0.04731 (9)	0.0326 (5)
C15	0.0130 (3)	0.2883 (3)	0.10054 (9)	0.0362 (5)
C16	−0.0280 (3)	0.1690 (3)	0.14863 (9)	0.0351 (5)
N21	0.4818 (3)	0.8505 (2)	0.36313 (7)	0.0309 (4)
N22	0.8983 (3)	1.0428 (2)	0.40391 (9)	0.0415 (5)
N23	0.6664 (3)	0.8431 (2)	0.47701 (7)	0.0318 (4)
C22	0.6843 (3)	0.9118 (2)	0.41445 (8)	0.0283 (4)
C24	0.4469 (4)	0.7125 (3)	0.48464 (9)	0.0366 (5)
C25	0.2358 (4)	0.6438 (3)	0.43309 (10)	0.0395 (5)
C26	0.2601 (3)	0.7179 (3)	0.37136 (9)	0.0347 (5)
Cr1	0.51287 (5)	0.31598 (3)	0.29255 (1)	0.0250 (1)
Cr2	0.63801 (5)	0.63913 (3)	0.19279 (1)	0.0249 (1)
O1	0.6767 (3)	0.4541 (2)	0.36377 (7)	0.0514 (5)
O2	0.2111 (3)	0.2848 (2)	0.30192 (8)	0.0485 (4)
O3	0.5676 (3)	0.1161 (2)	0.27901 (8)	0.0521 (5)
O4	0.7031 (3)	0.63515 (19)	0.10909 (7)	0.0432 (4)
O5	0.3696 (2)	0.68446 (19)	0.20400 (7)	0.0380 (4)
O6	0.8691 (2)	0.80393 (18)	0.24414 (7)	0.0376 (4)
O7	0.6073 (3)	0.41487 (17)	0.21384 (7)	0.0381 (4)
H11	0.072 (5)	−0.024 (3)	0.1705 (13)	0.045 (6)*
H14	0.21470	0.35000	0.01450	0.0390*
H15	−0.06740	0.38000	0.10290	0.0430*
H16	−0.13800	0.17760	0.18540	0.0420*
H121	0.329 (5)	−0.186 (4)	0.1107 (15)	0.054 (7)*
H122	0.449 (5)	−0.127 (3)	0.0477 (13)	0.051 (7)*

H21	0.496 (5)	0.897 (4)	0.3306 (14)	0.057 (8)*
H24	0.43260	0.66340	0.52740	0.0440*
H25	0.08520	0.55130	0.44060	0.0470*
H26	0.12460	0.67720	0.33520	0.0420*
H221	0.910 (4)	1.076 (3)	0.3673 (14)	0.043 (6)*
H222	1.018 (5)	1.079 (3)	0.4351 (14)	0.047 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0316 (7)	0.0364 (7)	0.0222 (6)	0.0054 (5)	0.0053 (5)	0.0108 (5)
N12	0.0470 (9)	0.0463 (9)	0.0401 (8)	0.0250 (7)	0.0122 (7)	0.0221 (7)
N13	0.0329 (7)	0.0333 (7)	0.0233 (6)	0.0125 (5)	0.0075 (5)	0.0095 (5)
C12	0.0257 (7)	0.0318 (7)	0.0228 (7)	0.0077 (6)	0.0009 (5)	0.0067 (6)
C14	0.0400 (9)	0.0321 (8)	0.0288 (8)	0.0136 (7)	0.0040 (6)	0.0097 (6)
C15	0.0392 (9)	0.0384 (9)	0.0341 (8)	0.0193 (7)	0.0041 (7)	0.0016 (7)
C16	0.0296 (8)	0.0446 (9)	0.0273 (8)	0.0100 (7)	0.0064 (6)	−0.0008 (7)
N21	0.0378 (7)	0.0367 (7)	0.0216 (6)	0.0139 (6)	0.0030 (5)	0.0106 (5)
N22	0.0424 (9)	0.0463 (9)	0.0289 (8)	0.0006 (7)	0.0006 (7)	0.0154 (7)
N23	0.0391 (7)	0.0337 (7)	0.0225 (6)	0.0101 (6)	0.0005 (5)	0.0082 (5)
C22	0.0352 (8)	0.0293 (7)	0.0217 (7)	0.0116 (6)	0.0035 (6)	0.0054 (6)
C24	0.0451 (9)	0.0389 (9)	0.0274 (8)	0.0116 (7)	0.0058 (7)	0.0138 (7)
C25	0.0359 (9)	0.0431 (10)	0.0358 (9)	0.0046 (7)	0.0063 (7)	0.0124 (7)
C26	0.0322 (8)	0.0413 (9)	0.0305 (8)	0.0123 (7)	0.0007 (6)	0.0056 (7)
Cr1	0.0286 (1)	0.0253 (1)	0.0221 (1)	0.0073 (1)	0.0059 (1)	0.0092 (1)
Cr2	0.0336 (1)	0.0248 (1)	0.0210 (1)	0.0124 (1)	0.0071 (1)	0.0105 (1)
O1	0.0426 (7)	0.0661 (10)	0.0323 (7)	0.0037 (7)	−0.0022 (5)	0.0005 (6)
O2	0.0304 (6)	0.0633 (9)	0.0449 (8)	0.0080 (6)	0.0087 (5)	0.0039 (7)
O3	0.0810 (11)	0.0379 (7)	0.0548 (8)	0.0317 (7)	0.0293 (8)	0.0286 (6)
O4	0.0676 (9)	0.0423 (7)	0.0255 (6)	0.0204 (6)	0.0160 (6)	0.0154 (5)
O5	0.0359 (6)	0.0434 (7)	0.0406 (7)	0.0184 (5)	0.0059 (5)	0.0128 (5)
O6	0.0370 (6)	0.0387 (7)	0.0354 (6)	0.0086 (5)	0.0011 (5)	0.0090 (5)
O7	0.0600 (8)	0.0315 (6)	0.0334 (6)	0.0223 (6)	0.0186 (5)	0.0186 (5)

Geometric parameters (Å, °)

Cr1—O1	1.6040 (14)	N21—C26	1.341 (3)
Cr1—O2	1.6063 (17)	N21—H21	0.75 (3)
Cr1—O3	1.6212 (15)	N22—C22	1.319 (2)
Cr1—O7	1.7739 (13)	N22—H221	0.76 (3)
Cr2—O4	1.6090 (13)	N22—H222	0.81 (3)
Cr2—O5	1.6175 (12)	N23—C22	1.350 (2)
Cr2—O6	1.6251 (13)	N23—C24	1.322 (3)
Cr2—O7	1.7710 (13)	C14—C15	1.392 (2)
N11—H11	0.74 (2)	C15—C16	1.353 (3)
N11—C12	1.357 (2)	C14—H14	0.93
N11—C16	1.345 (3)	C15—H15	0.93
N12—C12	1.313 (2)	C16—H16	0.93
N12—H121	0.78 (3)	C24—C25	1.391 (3)

N12—H122	0.87 (3)	C25—C26	1.356 (3)
N13—C12	1.346 (2)	C24—H24	0.93
N13—C14	1.325 (2)	C25—H25	0.93
N21—C22	1.357 (2)	C26—H26	0.93
O1—Cr1—O2	108.35 (8)	C22—N22—H221	118.8 (17)
O1—Cr1—O3	111.84 (8)	N11—C12—N13	120.44 (15)
O1—Cr1—O7	110.24 (7)	N12—C12—N13	119.79 (16)
O2—Cr1—O3	110.62 (8)	N11—C12—N12	119.77 (15)
O2—Cr1—O7	110.37 (8)	N13—C14—C15	124.03 (16)
O3—Cr1—O7	105.43 (7)	C14—C15—C16	116.85 (17)
O4—Cr2—O5	109.62 (8)	N11—C16—C15	119.48 (16)
O4—Cr2—O7	107.95 (7)	N13—C14—H14	118
O5—Cr2—O6	108.76 (7)	C15—C14—H14	118
O5—Cr2—O7	109.96 (7)	C14—C15—H15	122
O6—Cr2—O7	111.42 (7)	C16—C15—H15	122
O4—Cr2—O6	109.11 (7)	N11—C16—H16	120
Cr1—O7—Cr2	131.99 (8)	C15—C16—H16	120
C12—N11—C16	121.72 (14)	N21—C22—N23	120.56 (15)
C12—N13—C14	117.45 (15)	N21—C22—N22	119.75 (14)
C16—N11—H11	119 (2)	N22—C22—N23	119.68 (15)
C12—N11—H11	120 (2)	N23—C24—C25	124.39 (17)
C12—N12—H122	118.0 (16)	C24—C25—C26	116.9 (2)
C12—N12—H121	122 (2)	N21—C26—C25	119.19 (17)
H121—N12—H122	120 (3)	N23—C24—H24	118
C22—N21—C26	122.00 (14)	C25—C24—H24	118
C22—N23—C24	116.98 (15)	C26—C25—H25	122
C26—N21—H21	121 (2)	C24—C25—H25	122
C22—N21—H21	117 (2)	C25—C26—H26	120
C22—N22—H222	119.0 (17)	N21—C26—H26	120
H221—N22—H222	122 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O6^{i}	0.74 (2)	2.14 (2)	2.841 (2)	158 (3)
N21—H21···O3^{ii}	0.75 (3)	2.00 (3)	2.693 (2)	155 (3)
N12—H121···O5^{iii}	0.78 (3)	2.18 (3)	2.930 (2)	162 (3)
N12—H122···N13^{iv}	0.87 (3)	2.19 (3)	3.053 (2)	176 (2)
N22—H221···O2^{v}	0.76 (3)	2.41 (2)	3.042 (2)	141 (2)
N22—H221···O3^{ii}	0.76 (3)	2.59 (2)	3.144 (2)	131 (2)
N22—H222···N23^{vi}	0.81 (3)	2.22 (3)	3.030 (2)	176 (2)
C14—H14···O4^{vii}	0.93	2.38	3.161 (2)	142
C16—H16···O3^{viii}	0.93	2.40	3.312 (2)	165
C24—H24···O1^{ix}	0.93	2.37	3.287 (2)	170
C25—H25···O1^{viii}	0.93	2.46	3.085 (3)	125

Symmetry codes: (i) *x*−1, *y*−1, *z*; (ii) *x*, *y*+1, *z*; (iii) *x*, *y*−1, *z*; (iv) −*x*+1, −*y*, −*z*; (v) *x*+1, *y*+1, *z*; (vi) −*x*+2, −*y*+2, −*z*+1; (vii) −*x*+1, −*y*+1, −*z*; (viii) *x*−1, *y*, *z*; (ix) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

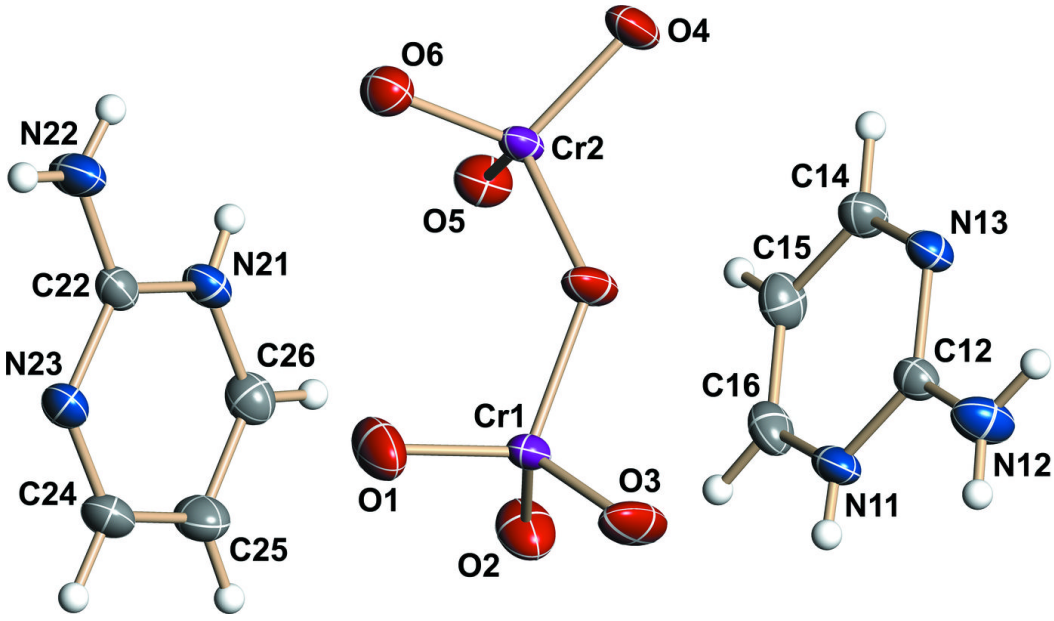
N22
C22
N21
N23
C26
C24
C25
O6
O4
Cr2
O5
Cr1
O1
O3
O2
C14
N13
C15
C12
C16
N12
N11

Fig. 2