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$K_2[HCr_2AsO_{10}]$: redetermination of phase II and the predicted structure of phase I

T. J. R. Weakley,[a] E. R. Ylvisaker,[b] R. J. Yager,[b] P. Wu,[b] P. Photinos[b] and S. C. Abrahams[b]*

[a]Department of Chemistry, University of Oregon, Eugene, OR 97403, USA, and [b]Physics Department, Southern Oregon University, Ashland, OR 97520, USA
Correspondence e-mail: sca@mind.net

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Our prediction that phase II of dipotassium hydrogen chromatoarsenate, $K_2[HCr_2AsO_{10}]$, is ferroelectric, based on the analysis of the atomic coordinates by Averbuch-Pouchot, Durif & Guitel [*Acta Cryst.* (1978), B**34**, 3725–3727], led to an independent redetermination of the structure using two separate crystals. The resulting improved accuracy allows the inference that the H atom is located in the hydrogen bonds of length 2.555 (5) Å which form between the terminal O atoms of shared AsO_3OH tetrahedra in adjacent $HCr_2AsO_{10}{}^{2-}$ ions. The largest atomic displacement of 0.586 Å between phase II and the predicted paraelectric phase I is by these two O atoms. The H atoms form helices of radius ~0.60 Å about the 3_1 or 3_2 axes. Normal probability analysis reveals systematic error in seven or more of the earlier atomic coordinates.

Comment

The possibility that the potential barrier between the structure of $K_2[HCr_2AsO_{10}]$ phase II (Averbuch-Pouchot *et al.*, 1978; hereinafter A-P) and that of paraelectric phase I is surmountable below the Curie temperature led to the prediction that phase II is ferroelectric (Abrahams, 2003). Crystals grown for associated physical property measurements were also used for an independent structural redetermination on two different crystals. Unsurprisingly, crystal 1 in space group $P3_1$ has a small enantiomorphic component, whereas crystal 2 in space group $P3_2$ is single-component (see the *Refinement* section for further discussion of this aspect).

The asymmetric unit of phase II contains one independent AsO_3OH and two CrO_4 tetrahedra, sharing O atoms to form individual $HAsCr_2O_{10}{}^{2-}$ ions (Fig. 1). The average Cr—O distance for the terminal O atoms in both crystals is 1.600 (4) Å (Table 1) and this does not differ significantly from the value of 1.612 (7) Å in II-$K_2Cr_2O_7$ (Weakley *et al.*, 2004), whereas the mean bridging Cr—O distance of 1.844 (4) Å in $K_2[HCr_2AsO_{10}]$ significantly exceeds the value of 1.785 (4) Å in II-$K_2Cr_2O_7$.

Unlike the bond-length distribution in the CrO_4 tetrahedra, the As—O distance of 1.632 (3) Å for the terminal O5 atom is significantly less than the mean As—O distance of 1.699 (9) Å for the two bridging atoms and the terminal O6 atom in the AsO_3OH tetrahedron. The short inter-anionic contact $O5\cdots O6^{i}$ [2.550 (11) and 2.555 (5) Å for crystals 1 and 2, respectively; symmetry code: (i) $y-x, -x, z-\frac{1}{3}$, for crystal 1, and $-y, x-y, z-\frac{1}{3}$, for crystal 2] results from the formation of an $O5\cdots H\cdots O6^{i}$ bond between $HAsCr_2O_{10}{}^{2-}$ ions. The arsenate distances given in Supplementary Table S1 show that the mean bridging and terminal As—O distances generally do not differ significantly [1.708 (20) and 1.682 (12) Å], and so the mean As—O distance may be taken as 1.69 (2) Å.

The bond-valence (BV) sums (Brown & Altermatt, 1985) for each atom in $K_2[HCr_2AsO_{10}]$ are 5.03 (3) for As, 5.98 (4) for Cr1 and 5.96 (4) for Cr2, 1.327 (8) for K1 and 1.157 (6) for K2, 1.612 (14) for O5 and 1.400 (14) for O6, and 1.94–2.20 (3) for all other O atoms in crystal 2, with comparable values in

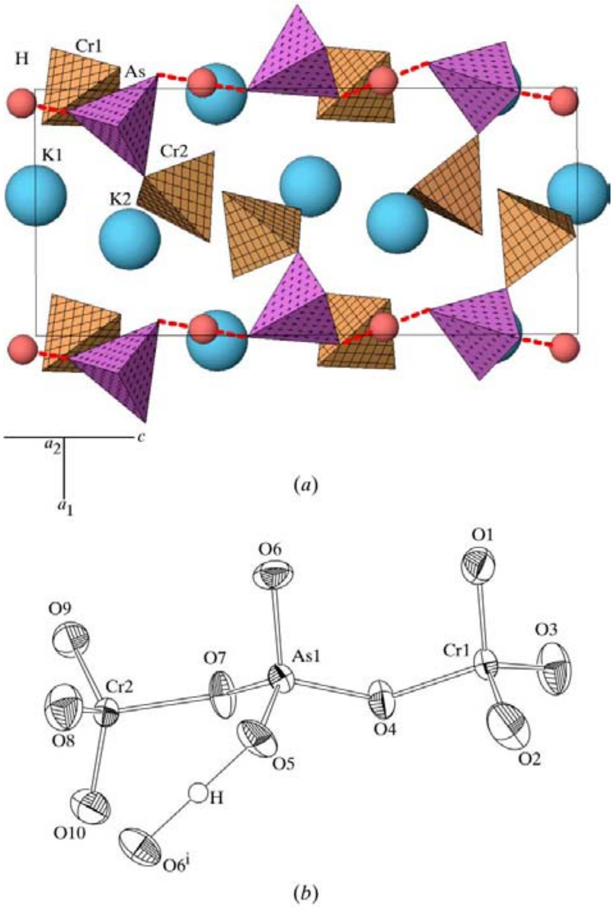

Figure 1
(*a*). The $K_2[HCr_2AsO_{10}]$ structure at 295 K. The $O6—H\cdots O5$ hydrogen bonds are shown as dashed lines. The labelling of all atoms in the unit cell is given in Supplementary Fig. S1. (*b*) The $HCr_2AsO_{10}{}^{2-}$ ion of crystal 1, showing the interionic hydrogen bond, with displacement ellipsoids drawn at the 50% probability level. The H atom is shown as a sphere and the hydrogen bond as single lines. [Symmetry code: (i) $y-x, -x, z-\frac{1}{3}$.]

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crystal 1. The connectivity within the asymmetric unit is shown in Fig. 1(*b*), and the labelling for all atoms in the unit cell is given in Supplementary Fig. S1. The BV sum for As agrees with the expected value, while those for Cr, K and O atoms other than O5 and O6 agree with the values observed in II-$K_2Cr_2O_7$ (Weakley *et al.*, 2004). The deficit of 0.99 in the joint valence of atoms O5 and O6 is clearly equivalent to an H atom which, while not located directly, results in the bond revealed in both crystals by the short O5···O6 distance. The H atoms form helices of radius ~0.60 Å about the 3_1 or 3_2 axes (Fig. 2), as they link $HCr_2AsO_{10}{}^{2-}$ anions. H-atom location was not considered in the report by A-P; if taken as midway between O5 and O6^{i} (Table 2), then the H atom in crystal 2 is at (0.0555, 0.0307, −0.0245). Since the BV sum for As—O6 is 1.23 (1), this bond is close to single, hence the H atom is most likely to be nearer to O6.

Atomic coordinates (x',y',z') in the predicted supergroup $P3_121$ or $P3_221$ are derived from the coordinates of related pairs of atoms in $P3_1$ or $P3_2$ under the higher-symmetry constraint (Table 2). The maximum differences between the positions of independent atoms in phase II and those predicted in paraelectric phase I, following the necessary symmetry conversions, are 0.41 Å for H and 0.586 Å for O5 and O6, confirming the basis for the original prediction of ferroelectricity (Abrahams, 2003). [Atoms O9 and O10 in the designation of Averbuch-Puchot *et al.* (1978) are equivalent to atoms O5 and O6 in Table 2.] The predicted atomic arrangement in phase I is shown in Supplementary Figs. S2 and S3.

The formation of $K_2[CrO_3AsO_3OHCrO_3]$ from its constituent ions (see *Experimental*) is possible only by fission of an O—Cr—O bond in the $Cr_2O_7{}^{2-}$ anion and the presence of an AsO_3OH^{2-} ion. The two resulting As—O—Cr bonds form by elimination of O^{2-} as H_2O. The protonation of a terminal O atom on As results in chains of $CrO_3AsO_3OHCrO_3{}^{2-}$ ions linked through hydrogen bonds, as shown in Figs. 1(*a*) and 2. The potential barrier to these rearrangements appears to be surmounted only at aqueous reaction solution temperatures close to boiling.

Comparison of the three independent sets of atomic coordinates is made by plotting the ordered experimental quantiles $Q_{\rm exp} = |\xi_i(1) - \xi_i(2)|/\{\sigma^2[\xi_i(1)] + \sigma^2[\xi_i(2)]\}^{1/2}$ against the corresponding normal quantiles $Q_{\rm norm}$, where $\xi_i(1)$, $\xi_i(2)$ are the *i*th parameters from the first and second independent determinations with the same setting, and $\sigma[\xi_i(1)]$, $\sigma[\xi_i(2)]$ are the corresponding standard uncertainties (s.u.) of each parameter. In the absence of error, a linear array of unit slope and zero intercept results (Abrahams & Keve, 1971). The $Q_{\rm norm}$ magnitudes are conveniently calculated by the program *NORMPA* (Ross, 2003).

The 45 atomic coordinate magnitudes determined with crystals 1 and 2 are compared in Fig. 3, the straight line giving the fit obtained by linear regression. The absence of outliers and the small departure of the slope from unity or linearity is indicative of minor systematic error in either coordinate set, but with joint s.u. (j.s.u.) values that are underestimated by a factor of ~1.3. The corresponding normal probability comparison of each set with the A-P atomic coordinates (Supplementary Figs. S4 and S5) contrasts strongly with that in Fig. 3. Seven $Q_{\rm exp} - Q_{\rm norm}$ terms in each of the latter two cases depart strongly from linearity (Table 3). These may hence be rejected as outliers, since major departures from a normal distribution can be due only to major systematic error. The strong possibility of uncompensated enantiomorphous twinning giving rise to the strong parameter correlation in the least-squares refinement noted by A-P is one of several likely sources of error. The remaining 38 terms exhibit a slightly S-shaped distribution, with j.s.u. values underestimated by a factor of ~1.7 in the comparison of crystal 1 and A-P, and of ~2.0 for the comparison of crystal 2 and A-P. The correlation coefficient in all fits made by linear regression is 0.987–0.992. The experimental uncertainties should be corrected by factors

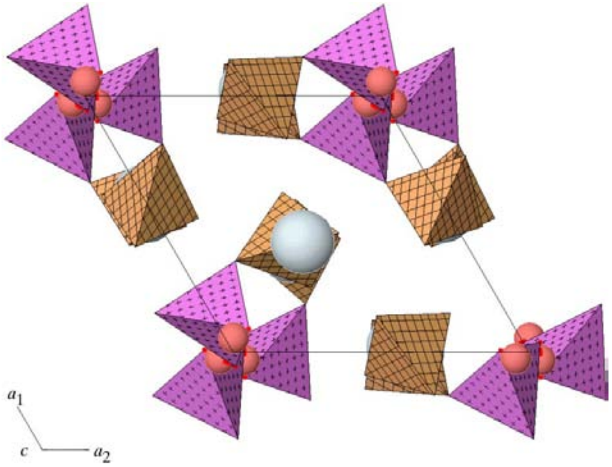

Figure 2
The $K_2[HCr_2AsO_{10}]$ structure at 298 K, with H atoms shown as small spheres and K atoms as large spheres. The AsO_4 tetrahedra are plus-hatched and the CrO_4 tetrahedra are cross-hatched.

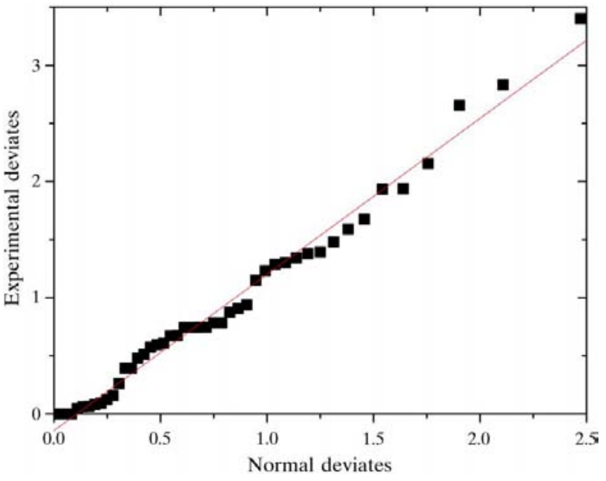

Figure 3
A normal probability $Q_{\rm exp} - Q_{\rm norm}$ plot for the atomic coordinates determined for $K_2[HCr_2AsO_{10}]$, with data for crystal 1 plotted *versus* those for crystal 2.

not less than those derived from the magnitude of the slopes listed in Table 3; inclusion of the outliers would clearly increase these factors.

The recommended phase-transition nomenclature (Tolédano *et al.*, 1998, 2001) for phases I and II of $K_2[HCr_2AsO_{10}]$, using the thermal values of Ylvisaker *et al.* (2001), is I | ~590–540 K | $P3_221$ (152) | $Z = 3$ | non-ferroic | decomposes above ~590 K, II | 540–270 K | $P3_2$ (145) | $Z = 3$ | ferroelectric | 2 variants, lower thermal limit not known.

Experimental

Averbuch-Pouchot *et al.* (1978) prepared the title chromatoarsenate by the reaction

$$K_2Cr_2O_7 + H_3AsO_4 \rightarrow K_2[HCr_2AsO_{10}] + H_2O$$

Substitution of either $0.5[As_2O_5{\cdot}xH_2O]$ with $x \simeq 3$ or KH_2AsO_4 for H_3AsO_4 also yields $K_2HCr_2AsO_{10}$ (CAS Registry No. 69107-38-6). Initial crystal growth in either reaction using 99.9% pure starting products with 0.1 mol of each reactant dissolved in about 10 mol H_2O, on heating to boiling for several minutes and then cooling naturally to room temperature, commonly gives two crops of strong red–orange [ISCC–NBS Color (Suppl. to NBS Circular 553); a complete list with RGB values is given at http://swiss.csail.mit.edu/~jaffer/Color/nbs-iscc.pdf and also at http://tx4.us/nbs-iscc.htm] crystals (Weakley *et al.*, 2004). Two crystals from first-growth crops were selected for the present study. The solubility at 298 K was determined as 207 g l^{-1}.

Crystal 1

Crystal data

$K_2[HCr_2AsO_{10}]$
$M_r = 418.13$
Trigonal, $P3_1$
$a_1 = 7.6931$ (8) Å
$a_2 = 7.6931$ (8) Å
$c = 14.623$ (3) Å
$V = 749.50$ (19) Å^3
$Z = 3$
$D_x = 2.779$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.1$–$16.4°$
$\mu = 6.33$ mm^{-1}
$T = 296$ (2) K
Hexagonal prism, strong red–orange
0.40 × 0.22 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
$T_{min} = 0.205$, $T_{max} = 0.320$
3208 measured reflections
2051 independent reflections
2040 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 30.0°$
$h = -8 \rightarrow 10$
$k = 0 \rightarrow 10$
$l = -20 \rightarrow 20$
3 standard reflections every 300 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.129$
$S = 1.29$
2051 reflections
137 parameters
H atom treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0086P)^2 + 7.6027P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 1.31$ e Å^{-3}
$\Delta\rho_{min} = -1.08$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 7.2 (11) × 10^{-6}
Absolute structure: Flack (1983), with 1157 Bijvoet pairs
Flack parameter = 0.17 (3)

Crystal 2

Crystal data

$K_2[HCr_2AsO_{10}]$
$M_r = 418.13$
Trigonal, $P3_2$
$a_1 = 7.6963$ (9) Å
$a_2 = 7.6963$ (9) Å
$c = 14.6171$ (11) Å
$V = 749.82$ (14) Å^3
$Z = 3$
$D_x = 2.778$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 13.5$–$14.9°$
$\mu = 6.33$ mm^{-1}
$T = 294$ (2) K
Hexagonal prism, strong red–orange
0.29 × 0.13 × 0.11 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none (neither analytical corrections nor azimuthal scans for crystal 2 resulted in significantly improved residuals or standard uncertainties, hence uncorrected intensities were used)
2560 measured reflections
1224 independent reflections
1182 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 27.5°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 9$
$l = -18 \rightarrow 18$
3 standard reflections every 300 reflections frequency: 120 min intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.053$
$S = 1.08$
1224 reflections
137 parameters
H atom treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0181P)^2 + 0.0502P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 9.6 (12) × 10^{-6}
Absolute structure: Flack (1983), with 1224 Bijvoet pairs
Flack parameter = 0.030 (16)

Table 1
Selected distances (Å) in $K_2[HCr_2AsO_{10}]$ phase II.

Only K—O distances less than 3.08 Å are given. The uncertainty in the averaged K—O distances was calculated using Bessel's method.

Bond	Crystal 1	Crystal 2
Cr1—O1	1.609 (12)	1.599 (4)
Cr1—O2	1.602 (10)	1.597 (4)
Cr1—O3	1.594 (13)	1.599 (4)
Cr1—O4	1.848 (8)	1.840 (4)
Cr2—O7	1.842 (8)	1.846 (3)
Cr2—O8	1.599 (13)	1.596 (4)
Cr2—O9	1.600 (9)	1.595 (4)
Cr2—O10	1.606 (11)	1.600 (4)
As—O4	1.707 (9)	1.707 (4)
As—O5	1.633 (8)	1.632 (3)
As—O6	1.686 (11)	1.692 (4)
As—O7	1.695 (8)	1.704 (4)
K1—O × 7	2.720–2.854	2.724–2.871
$\langle d(K—O)\rangle$	2.78 (5)	2.78 (6)
K2—O × 8	2.760–3.075	2.765–3.076
$\langle d(K—O)\rangle$	2.88 (12)	2.89 (12)

The general structure solution made use of a *SIR*92 electron-density map (Altomare *et al.*, 1994). The Flack (1983) parameter shows that the space group for crystal 1 is primarily $P3_1$ but with a small enantiomorphic component, whereas the space group for crystal 2 does not differ significantly from $P3_2$. The H atom could not

Table 2
Atomic coordinates of $K_2[HCr_2AsO_{10}]$ in phase II and predicted coordinates in phase I, with component [$\Delta(x)$, $\Delta(y)$ and $\Delta(z)$] and total [$\Delta(xyz)$] differences between the phases (Å).

a = 7.6963 (9) and c = 14.6171 (11) Å; $z^* = z - 0.34541$; $\Delta x = (x - x')a$, $\Delta y = (y - y')a$, $\Delta z = (z^* - z')c$ Å.

	Wyckoff position† $P3_2$, $P3_221$	x	y	z^*	x'	y'	z'	$\Delta(x)$	$\Delta(y)$	$\Delta(z)$	$\Delta(xyz)$
As	3(a), 3(b)	0.12958 (6)	0.02179 (6)	0.15463 (5)	0.12958	0	0.16667	0	0.168	−0.176	0.243
Cr1	3(a)	0.00117 (11)	−0.41910 (11)	0.08497 (5)	0.00411	−0.42195	0.07858	−0.023	0.022	0.093	0.098
	6(c)										
Cr2	3(a)	0.42899 (11)	0.42481 (11)	0.26114 (5)	0.42606	0.42195	0.25475	−0.022	−0.022	0.093	0.098
K1	3(a), 3(a)	0.43080 (16)	0.45187 (16)	0.00095 (8)	0.44134	0.44134	0	−0.081	0.081	0.014	0.115
K2	3(a), 3(b)	0.60914 (17)	0.00606 (17)	0.17366 (8)	0.60914	0	0.16667	0	−0.047	0.102	0.112
H‡	3(a), 3(a)	0.0555	0.0307	−0.0245	0.0532	0.0532	0	0.012	−0.173	−0.364	0.405
O1	3(a)	0.1424 (6)	−0.4423 (7)	0.0116 (3)	0.1531	−0.4608	0.0123	−0.081	0.142	−0.010	0.164
	6(c)										
O8	3(a)	0.6242 (6)	0.4793 (7)	0.3203 (4)	0.6139	0.4608	0.3210	0.080	0.142	−0.010	0.164
O2	3(a)	−0.0883 (8)	−0.5988 (6)	0.1570 (3)	−0.1084	−0.5949	0.1522	0.155	0.030	0.070	0.173
	6(c)										
O9	3(a)	0.4665 (9)	0.5909 (6)	0.1860 (3)	0.4866	0.5949	0.1812	−0.155	−0.031	0.070	0.173
O3	3(a)	−0.1692 (6)	−0.4012 (7)	0.0304 (3)	−0.1567	−0.3968	0.0171	−0.096	−0.034	0.196	0.221
	6(c)										
O10	3(a)	0.2526 (7)	0.3923 (7)	0.3295 (3)	0.2401	0.3968	0.3162	0.096	−0.035	0.194	0.219
O4	3(a)	0.1520 (6)	−0.1879 (6)	0.1512 (3)	0.1602	−0.1874	0.1416	−0.063	−0.004	0.140	0.154
	6(c)										
O7	3(a)	0.3559 (5)	0.1869 (5)	0.2014 (3)	0.3477	0.1874	0.1918	0.063	−0.004	0.140	0.154
O5	3(a)	0.0914 (6)	0.1001 (5)	0.0570 (2)	0.0465	0.0598	0.0815	0.346	0.310	−0.358	0.586
	6(c)										
O6	3(a)	−0.0582 (5)	−0.0195 (7)	0.2274 (2)	−0.0133	−0.0598	0.2518	−0.346	0.310	−0.357	0.586

† Each atom occupies the general 3(a) Wyckoff position of subgroup $P3_2$ with coordinates $x, y, z; \overline{y}, x - y, z + \frac{2}{3}; \overline{x} + y, \overline{x}, z + \frac{1}{3}$. In supergroup $P3_221$, the special 3(a) positions are $x, 0, \frac{2}{3}; 0, x, \frac{1}{3}; \overline{x}, \overline{x}, 0$, and the 3($b$) positions are $x, 0, \frac{1}{6}; 0, x, \frac{5}{6}; \overline{x}, \overline{x}, \frac{1}{2}$, with the 6($c$) positions as in $P3_2$ + ($y, x, \overline{z}; \overline{x}, \overline{x} + y, \overline{z} + \frac{2}{3}; x - y, \overline{y}, \overline{z} + \frac{1}{3}$). ‡ H-atom coordinates in both phases are taken as midway between those of O5(x, y, z) and O6($\overline{y}, x - y, \frac{2}{3} + z$).

Table 3
Linear-regression indicators for the $K_2[HCr_2AsO_{10}]$ $Q_{\rm exp} - Q_{\rm norm}$ plots.

$\xi(1)$	$\xi(2)$	Slope	Intercept	Correlation coefficient	Ordered rejected outliers
Crystal 1	Crystal 2	1.34 (2)	−0.14 (2)	0.992	None
Crystal 1	Averbuch-Puchot *et al.* (1978)	1.72 (5)	−0.15 (5)	0.987	51.0 > O7z, K1x, K1y, Cr2z, K1z, K2z, Asx > 4.6
Crystal 2	Averbuch-Puchot *et al.* (1978)	2.02 (5)	−0.22 (5)	0.990	32.7 > K1y, K1x, Cr2z, Asy, K1z, Asx, K2z > 6.6

be located directly with confidence in either crystal but was later inferred (see *Comment*).

For both crystals, data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4-*PC Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *TEXSAN*; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORIGINS* (OriginLab Corporation, 1994) and *ATOMS* (Dowty, 2003).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1472). Five additional figures and one further table are also available. Services for accessing these data are described at the back of the journal.

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