

Potassium *trans*-diaquabis(malonato)chromium(III) trihydrateMarkus Lemmer,^a Hans-Wolfram Lerner^a and Michael Bolte^{b*}^aInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and ^bInstitut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

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Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.025

wR factor = 0.066

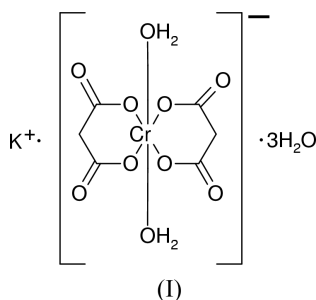
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

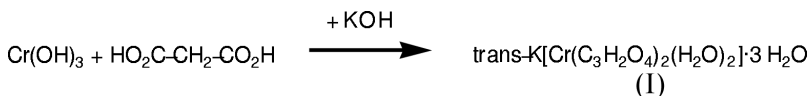
The title compound, *trans*-K[Cr(C₃H₂O₄)₂(H₂O)₂]·3H₂O, comprises an octahedral coordinated Cr atom, bonded to two malonato ions and two water molecules and a K ion surrounded by nine O atoms.

Comment

Octahedral complexes featuring paramagnetic Cr³⁺ ions are of great importance for the development of new molecule-based magnets and electronic materials. Therefore, we became interested in the magnetic properties of the title compound, (I). The X-ray structure determination of (I) was carried out in order to establish unambiguously the structure of the reaction product.



The synthesis of (I) was carried out according to a literature procedure, as indicated in the reaction *Scheme* below (Randall & Perkins, 1955). A perspective view of (I) is shown in Fig. 1. The Cr atom shows an octahedral coordination and is bonded to two malonate ions and two water molecules. The K ion is surrounded by nine O atoms (Table 1).



Experimental

The title complex, (I), has been synthesized by heating a slurry of Cr(OH)₃, KOH, HO₂C-CH₂-CO₂H and water at 353 K for 12 h. Purple crystals of (I) were grown by storing this solution at ambient temperature for 2 d.

Crystal data

K[Cr(C₃H₂O₄)₂(H₂O)₂](H₂O)₃*M_r* = 385.27Monoclinic, *P*2₁/*n**a* = 7.0279 (5) Å*b* = 11.6943 (5) Å*c* = 16.5623 (12) Å

β = 96.791 (6)°

V = 1351.65 (15) Å³*Z* = 4*D_x* = 1.893 Mg m⁻³Mo *K*α radiation

Cell parameters from 34274

reflections

θ = 3.7–29.7°

μ = 1.22 mm⁻¹*T* = 173 (2) K

Block, purple

0.48 × 0.24 × 0.22 mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: empirical (MULABS; Spek, 1990; Blessing, 1995)
 $T_{\min} = 0.583$, $T_{\max} = 0.764$
 25 347 measured reflections

3803 independent reflections
 3536 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$
 $\theta_{\text{max}} = 29.7^\circ$
 $h = -9 \rightarrow 9$
 $k = -16 \rightarrow 16$
 $l = -23 \rightarrow 23$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.066$
 $S = 1.03$
 3803 reflections
 230 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.4185P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.44 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.70 \text{ e } \text{\AA}^{-3}$

Table 1

Selected interatomic distances (Å).

Cr1—O61	1.9417 (8)	K1—O5 ⁱ	2.8014 (10)
Cr1—O11	1.9474 (8)	K1—O4	2.8069 (11)
Cr1—O31	1.9512 (8)	K1—O32 ⁱⁱ	2.8488 (9)
Cr1—O41	1.9630 (8)	K1—O42 ⁱⁱⁱ	3.0811 (11)
Cr1—O1	2.0018 (8)	K1—O3	3.1394 (13)
Cr1—O2	2.0034 (8)	K1—O62	3.1752 (10)
K1—O61	2.7428 (9)	K1—O11	3.2408 (9)
K1—O5	2.7723 (10)		

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1A \cdots O12 ⁱ	0.81 (2)	1.80 (2)	2.6054 (12)	175 (2)
O1—H1B \cdots O62 ⁱⁱ	0.83 (2)	1.81 (2)	2.6262 (12)	170 (2)
O2—H2C \cdots O42 ⁱⁱⁱ	0.85 (2)	1.82 (2)	2.6614 (13)	171.0 (19)
O2—H2D \cdots O3	0.83 (2)	1.81 (2)	2.6321 (14)	169 (2)
O3—H3A \cdots O4 ^{iv}	0.87 (3)	1.94 (3)	2.7710 (15)	160 (2)
O3—H3B \cdots O41 ⁱⁱⁱ	0.87 (3)	2.38 (3)	3.1280 (14)	145 (2)
O3—H3B \cdots O31 ⁱⁱⁱ	0.87 (3)	2.48 (3)	3.2361 (14)	146 (2)
O4—H4A \cdots O32 ^v	0.86 (2)	1.95 (2)	2.7968 (14)	165 (2)
O4—H4B \cdots O32 ^{vi}	0.80 (3)	2.14 (3)	2.9271 (15)	169 (2)
O5—H5C \cdots O31 ⁱⁱⁱ	0.80 (3)	2.37 (3)	3.1675 (14)	174 (3)
O5—H5D \cdots O12	0.87 (2)	2.04 (2)	2.9011 (13)	170 (2)

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

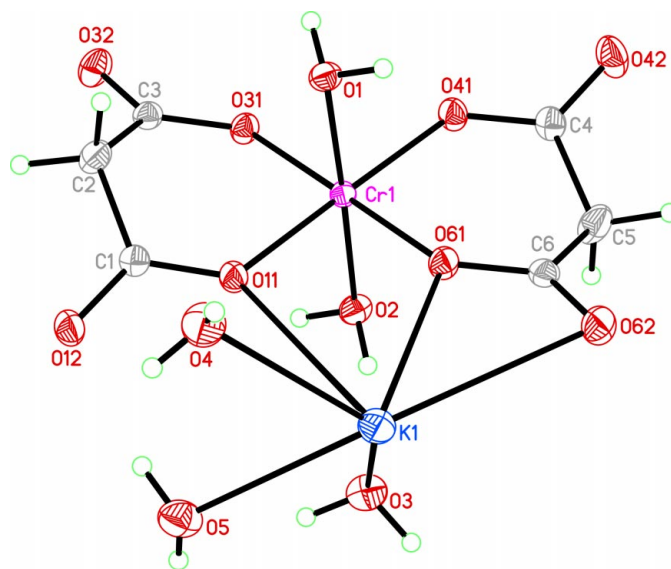


Figure 1

Perspective view of (I), with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level.

All H atoms were located by difference Fourier syntheses. H atoms bonded to C atoms were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$], using a riding model with $C-H_{\text{methyl}} = 0.99 \text{ \AA}$. H atoms bonded to O atoms were refined isotropically. The O—H bond lengths are 0.80 (3)–0.87 (3) Å.

Data collection: X-AREA (Stoe & Cie, 2001); cell refinement: X-AREA; data reduction: X-AREA; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL-Plus (Sheldrick, 1991).

References

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