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Low-temperature phase of hexaguanidinium heptamolybdate monohydrate

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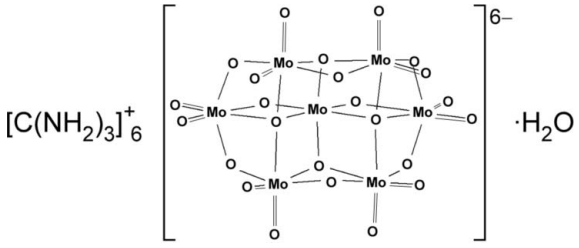
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(N–C) = 0.007 Å; R factor = 0.054; wR factor = 0.165; data-to-parameter ratio = 31.3.

The crystal structure of the title compound, $[C(NH_2)_3]_6[Mo_7O_{24}]\cdot H_2O$, previously determined at room temperature in the monoclinic space group $C2/c$ from Weissenberg techniques [Don & Weakley (1981). *Acta Cryst.* B**37**, 451–453], has been redetermined from low-temperature single-crystal data in the monoclinic space group $P2_1/c$. The asymmetric unit contains one heptamolybdate anion, six guanidinium cations and one water molecule of hydration. The anions and cations are linked by an extensive network of N—H···O hydrogen bonds.

Related literature

For the previous determination of the title compound in the monoclinic space group $C2/c$, see: Don & Weakley (1981). For an example of a structurally characterized $[Mo_7O_{24}]^{6-}$ anion, see: Kortz & Pope (1995). For more information about isopolymolybdates and polyoxometalates in general, see: Pope (1983).



Experimental

Crystal data

$(CH_6N_3)_6[Mo_7O_{24}].H_2O$
M_r = 1434.12
Monoclinic, $P2_1/c$
a = 11.9402 (6) Å
b = 15.9131 (9) Å
c = 19.8223 (13) Å
β = 92.312 (4)°
V = 3763.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.37 mm^{-1}
T = 173 (2) K
0.17 × 0.17 × 0.08 mm

Data collection

Bruker X8 APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.689, T_{max} = 0.833
153838 measured reflections
15842 independent reflections
9689 reflections with $I > 2s(I)$
R_{int} = 0.150

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.164
S = 1.05
15842 reflections
506 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.77 e Å^{-3}
$\Delta\rho_{min}$ = −2.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11A···O1W	0.88	1.90	2.765 (6)	168
N11—H11B···O3A	0.88	2.35	3.091 (6)	142
N12—H12A···O2A^{i}	0.88	2.00	2.845 (5)	160
N12—H12B···O5B	0.88	2.14	2.903 (5)	145
N13—H13A···O6A^{ii}	0.88	1.96	2.828 (5)	169
N13—H13B···O3B	0.88	2.23	2.930 (5)	136
N14—H14B···O13	0.88	1.93	2.774 (4)	159
N14—H14A···O57^{iii}	0.88	1.98	2.814 (4)	159
N15—H15A···O2B^{iv}	0.88	2.03	2.843 (5)	153
N15—H15B···O3A^{v}	0.88	2.13	2.866 (5)	141
N15—H15B···O34^{v}	0.88	2.62	3.302 (5)	135
N16—H16B···O1A^{ii}	0.88	2.13	2.967 (5)	159
N16—H16A···O45^{vi}	0.88	2.18	2.977 (5)	151
N21—H21A···O1B^{vi}	0.88	2.18	3.006 (5)	156
N21—H21A···O2B^{vi}	0.88	2.40	2.926 (5)	118
N21—H21B···O3A	0.88	2.22	2.992 (5)	147
N22—H22A···O36^{vii}	0.88	2.32	3.087 (5)	145
N22—H22A···O467^{vii}	0.88	2.50	3.216 (5)	139
N22—H22A···O6B^{vii}	0.88	2.64	3.290 (5)	131
N22—H22B···O25	0.88	2.10	2.977 (5)	177
N23—H23A···O36	0.88	2.07	2.937 (5)	169
N23—H23B···O25^{vi}	0.88	2.26	3.023 (5)	145
N23—H23B···O124^{vi}	0.88	2.50	3.219 (5)	140
N24—H24A···O13	0.88	2.45	3.154 (5)	137
N24—H24A···O3A	0.88	2.50	3.186 (5)	135
N24—H24B···O12^{viii}	0.88	2.09	2.855 (5)	145
N25—H25A···O34^{v}	0.88	2.22	2.990 (5)	146
N25—H25B···O7A^{iii}	0.88	2.08	2.931 (5)	162
N26—H26A···O3B^{ii}	0.88	1.98	2.859 (5)	175
N26—H26B···O1W^{ii}	0.88	2.50	3.084 (6)	124
N26—H26B···O6A	0.88	2.57	3.197 (5)	129
N31—H31A···O1B^{vi}	0.88	2.50	3.248 (6)	143
N31—H31B···O5A^{ix}	0.88	2.38	3.188 (6)	152
N32—H32A···N34^{x}	0.88	2.50	3.242 (6)	143
N32—H32B···O467^{vii}	0.88	2.02	2.864 (5)	160
N33—H33A···N24^{vi}	0.88	2.60	3.334 (6)	142
N33—H33B···O124^{vi}	0.88	2.02	2.867 (5)	160
N34—H34B···O67^{viii}	0.88	1.94	2.776 (5)	157
N34—H34A···O57^{iii}	0.88	2.44	3.154 (5)	139
N34—H34A···O5A^{iii}	0.88	2.55	3.195 (5)	131
N35—H35A···O2A^{iv}	0.88	2.29	3.040 (5)	144
N35—H35B···O5B^{iii}	0.88	1.98	2.849 (5)	170
N36—H36A···O5A^{vi}	0.88	2.16	2.913 (5)	144
N36—H36B···O6B	0.88	2.13	2.868 (5)	141
O1W—H1OW···O6B^{iii}	0.86	2.22	2.948 (5)	142
O1W—H1OW···O7B^{iii}	0.86	2.44	3.086 (5)	133
O1W—H2OW···O5A^{ix}	0.86	2.35	2.973 (5)	130

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure:

SHELXTL (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *WINGX* Farrugia (1999)..

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2049).

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