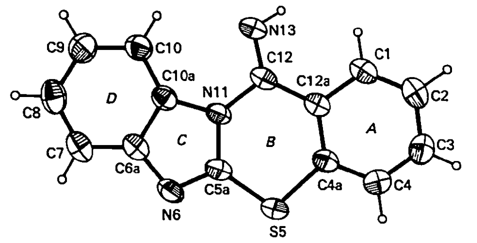

Fig. 1. Dessin de la molécule, numéros attribués à ses atomes et lettres utilisées pour désigner les cycles.

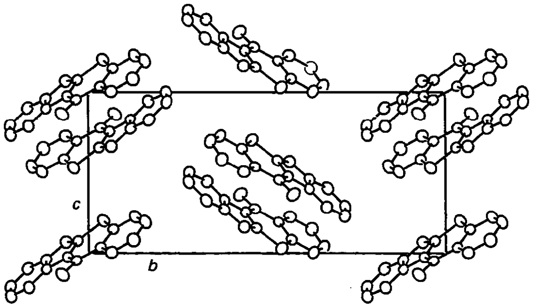

Fig. 2. Projection de la structure sur la face (100).

calculé avec tous les atomes constituant le cycle *A*. Il en est de même respectivement pour les plans moyens *P*(*B*), *P*(*C*), *P*(*D*) et *P*(*ABCD*).

Les cycles *C* et *D* sont plans et le cycle *A* pratiquement plan ($\chi^2 = 87$). Par contre, la planéité de l'hétérocycle *B* et celle de la molécule ne sont qu'approximatives. En effet, les distances à *P*(*B*) des atomes du cycle *B* atteignent 0,050 (2) Å et celles à *P*(*ABCD*) de l'ensemble des atomes des quatre cycles 0,148 (2) Å. Le distance de N(13) à *P*(*ABCD*) est égale à 0,298 (2) Å.

C(12)—N(13) [1,269 (3) Å] est une double liaison. Il en est de même, dans le cycle *C*, de C(5a)—N(6) [1,308 (3) Å]. Les longueurs des autres liaisons des cycles *B* et *C* (Tableau 2) indiquent un caractère π partiel pour S(5)—C(5a), N(6)—C(6a) et les trois liaisons issues de N(11) et un caractère π très partiel pour C(4a)—S(5) et C(12)—C(12a). Les distances C—C entre atomes voisins ont pour longueurs extrêmes 1,369 (3) et 1,405 (3) Å dans le cycle *A* [moyenne 1,386 (6) Å], 1,376 (4) et 1,399 (3) dans le cycle *D* [moyenne 1,387 (3) Å]. Les valeurs des angles valenciels endocycliques expériment l'allongement du cycle *B* dans la direction S(5)C(12) et celui du cycle *D* dans la direction C(7)C(10). Ces déformations sont dues aux tensions existant dans le cycle *C* auxquelles s'ajoute, dans le cas du cycle *B*, l'hybridation sp^3 tronquée de l'hétéroatome.

La liaison hydrogène N(13)—H(13)···N($6^{\rm i}$) [3,225 (2) Å, 153 (2)°] [(i) $1 + x$, y, z] unit deux molécules qui se déduisent l'une de l'autre par la translation **a**. Ainsi, les molécules forment des chaînes infinies parallèles à l'axe *a*. La Fig. 2 montre que la structure peut aussi être considérée comme formée de couches de molécules que s'étendent au voisinage des plans $y = 0$ et $y = \frac{1}{2}$.

Références

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package.* College Station, Texas, EU, et Enraf–Nonius, Delft, Pays-Bas.

Johnson, C. K. (1976). *ORTEP*II. Rapport ORNL-5138. Oak Ridge National Laboratory, Tennessee, EU.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. de York, Angleterre, et de Louvain, Belgique.

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Acta Cryst. (1991). C**47**, 1060–1062

Structure of Hexaaquachromium(III) Nitrate Trihydrate

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Abstract. $[Cr(H_2O)_6](NO_3)_3.3H_2O$, $M_r = 400{\cdot}1$, monoclinic, $P2_1/c$, $a = 13{\cdot}967$ (1), $b = 9{\cdot}6528$ (9), $c = 10{\cdot}981$ (1) Å, $\beta = 95{\cdot}41°$, $V = 1473{\cdot}87$ Å^3, $Z = 4$, $D_x = 1{\cdot}802$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 6{\cdot}71$ cm^{-1}, $F(000) = 828$, final $R = 0{\cdot}030$ for 2567 unique reflections. The structure comprises two crys-

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non*-H *atoms*

$U_{eq} = [U_{11}(a^*a)^2 + U_{22}(b^*b)^2 + U_{33}(c^*c)^2 + 2(U_{12}a^*b^*ab\cos\gamma + U_{13}a^*c^*ac\cos\beta + U_{23}b^*c^*bc\cos\alpha)]/3.$

	x	y	z	U_{eq}
Cr1	0	0	0	0·0169 (0)
Cr2	0·5	0	0·5	0·0180 (0)
N1	0·7138 (1)	−0·7144 (2)	0·2809 (1)	0·0280 (4)
N2	0·7987 (1)	−0·3107 (2)	0·2098 (1)	0·0263 (4)
N3	0·3077 (1)	−0·5092 (2)	0·3737 (2)	0·0282 (4)
O11	0·7418 (1)	−0·6644 (2)	0·1856 (1)	0·0413 (4)
O12	0·6269 (1)	−0·7379 (2)	0·2874 (1)	0·0464 (5)
O13	0·7733 (1)	−0·7426 (2)	0·3697 (1)	0·0366 (4)
O21	0·7469 (1)	−0·3149 (2)	0·1091 (1)	0·0336 (4)
O22	0·8856 (1)	−0·8856 (1)	0·2087 (1)	0·0388 (5)
O23	0·7630 (1)	−0·3344 (2)	0·3066 (1)	0·0391 (5)
O31	0·2355 (1)	−0·4966 (2)	0·2995 (1)	0·0421 (4)
O32	0·3006 (1)	−0·4888 (2)	0·4833 (1)	0·0517 (4)
O33	0·3870 (1)	−0·5444 (2)	0·3388 (1)	0·0429 (4)
O*w*1	0·0353 (1)	−0·1223 (2)	0·3712 (1)	0·0324 (4)
O*w*2	0·5517 (1)	−0·3837 (2)	0·3714 (1)	0·0359 (4)
O*w*3	0·8566 (1)	−0·0246 (2)	0·4513 (2)	0·0402 (4)
O*w*11	−0·0942 (1)	−0·1505 (1)	−0·0050 (1)	0·0257 (4)
O*w*12	−0·0741 (1)	0·1060 (2)	0·1112 (1)	0·0287 (4)
O*w*13	0·0708 (1)	−0·0879 (2)	0·1406 (1)	0·0296 (4)
O*w*21	0·5748 (1)	−0·1098 (2)	0·3909 (1)	0·0337 (4)
O*w*22	0·4040 (1)	−0·1487 (2)	0·4959 (2)	0·0356 (4)
O*w*23	0·4278 (1)	0·0938 (2)	0·3639 (1)	0·0319 (5)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Octahedron 1			
Cr1—O*w*11	1·956 (1)	O*w*11—Cr1—O*w*12	90·7 (1)
Cr1—O*w*12	1·962 (1)	O*w*11—Cr1—O*w*13	89·3 (1)
Cr1—O*w*13	1·947 (1)	O*w*12—Cr1—O*w*13	89·5 (1)
Octahedron 2			
Cr2—O*w*21	1·972 (2)	O*w*21—Cr2—O*w*22	89·6 (1)
Cr2—O*w*22	1·962 (2)	O*w*21—Cr2—O*w*23	92·8 (1)
Cr2—O*w*23	1·946 (1)	O*w*22—Cr2—O*w*23	91·1 (1)
Nitrate 1			
N1—O11	1·248 (2)	O11—N1—O12	120·2 (2)
N1—O12	1·243 (2)	O11—N1—O13	120·1 (2)
N1—O13	1·249 (2)	O12—N1—O13	119·7 (1)
Nitrate 2			
N2—O21	1·263 (2)	O21—N2—O22	118·5 (2)
N2—O22	1·245 (2)	O21—N2—O23	120·1 (2)
N2—O23	1·237 (2)	O22—N2—O23	121·4 (2)
Nitrate 3			
N3—O31	1·241 (2)	O31—N3—O32	119·3 (2)
N3—O32	1·233 (2)	O31—N3—O33	120·8 (2)
N3—O33	1·253 (2)	O32—N3—O33	119·8 (2)

tallographically distinct $Cr(H_2O)_6^{3+}$ octahedra, each possessing crystallographic $\bar{1}$ symmetry, connected by three-dimensional hydrogen bonding involving the nitrate anions and water molecules.

Experimental. Single crystals of Merck's hexaaquachromium(III) nitrate trihydrate are dark-red, nontransparent and highly hygroscopic. The crystal used for the data collection was sealed in a Lindemann-glass capillary in order to protect it from moisture.

The intensity data were collected on an Enraf–Nonius CAD-4 diffractometer using a crystal of size 0·48 × 0·20 × 0·24 mm and graphite-monochromated Mo *Kα* radiation. Unit-cell parameters were obtained by least-squares refinement of 2θ values of 19 reflections. 3407 reflections were collected in the $\omega/2\theta$ scan mode ($2\theta_{max}$ 54°) and corrected for Lorentz and polarization effects, but not for absorption. Standard reflections $6\bar{4}2$ and $3\bar{3}\bar{7}$ showed no significant change. Of 2926 measured reflections, 2567 with $I > 3\sigma(I)$ were used for structure solution and refinement. *hkl* range: $-17 \leq h \leq 17$, $0 \leq k \leq 12$, $0 \leq l \leq 13$.

Analysis of the data showed that reflections with either $(h + k)$ or $(k + l)$ or $(h + l)$ *even* were on average stronger than those with *odd* values for these combinations. This effect was most noticeable for the $(h + l)$ case. The pseudo-face-centered lattice, thus implied, was possible if the Cr^{3+} ions occupied two independent sets of special positions in $P2_1/c$, both of site symmetry $\bar{1}$ [(*a*) 0,0,0; $0,\frac{1}{2},\frac{1}{2}$; (*d*) $\frac{1}{2},0,\frac{1}{2}$; $\frac{1}{2},\frac{1}{2},0$]. A Patterson synthesis confirmed this assignment.

The structure was solved by the heavy-atom method with *SHELXS*86 (Sheldrick, 1986) and refined by full-matrix least squares minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976); $w = 1{\cdot}1538/[\sigma^2(F_o) + 0{\cdot}000773F_o^2]$, 274 refined parameters. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The refinement was terminated when the parameter shifts were less than $0{\cdot}04\sigma$. The final values for R and wR were 0·030 and 0·035, respectively. The maximum peak in the final ΔF map was 0·44 e Å^{-3}. Scattering factors were from *Interna-*

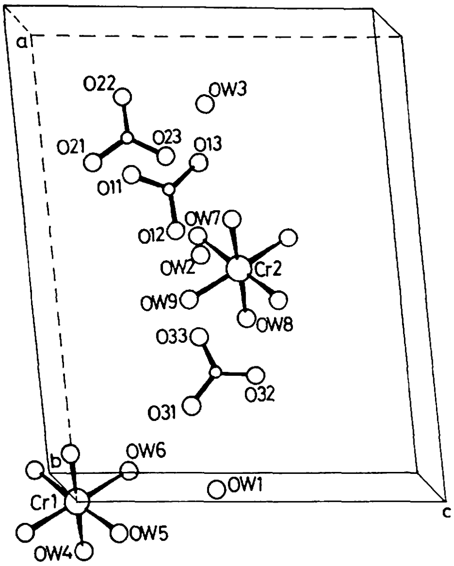

Fig. 1. Perspective view of the compound with atomic numbering scheme. The H atoms have been omitted for clarity.

tional Tables for X-ray Crystallography (1974, Vol. IV). The fractional coordinates and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows a perspective view of the asymmetric unit and the atomic numbering scheme. In fact, the asymmetric unit comprises two crystallographically independent half octahedra. The complete octahedra are generated by the $\bar{1}$ symmetry located at the central Cr ions.

The geometrical parameters are computed with *CSU* (Vicković, 1988). All calculations, including the crystal structure analysis, were performed on a PC-AT computer.

Related literature. Similar cell parameters and the existence of mixed crystals indicate the isomorphism of the title compound with $Al(NO_3)_3.9H_2O$ (Kannan & Viswamitra, 1965), contrary to the results given by Herpin & Sudarsanan (1965). On the other hand, the compound is undoubtedly found to be isostructural with $Fe(NO_3)_3.9H_2O$ (Hair & Beattie, 1977).

We thank D. B. Prelesnik for data collection.

References

Hair, N. J. & Beattie, J. K. (1977). *Inorg. Chem.* **16**, 245–250.
Herpin, P. & Sudarsanan, K. (1965). *Bull. Soc. Fr. Minéral. Cristallogr.* **88**, 595–601.
Kannan, K. K. & Viswamitra, M. A. (1965). *Acta Cryst.* **19**, 151–152.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
Vicković, I. (1988). *CSU*. Faculty of Sciences, Univ. of Zagreb, Yugoslavia.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles pertinent to the hydrogen bonded interactions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53737 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Lanthanum Orthosilicate Selenide, La_2SeSiO_4

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Abstract. The structure of La_2SeSiO_4 has been determined from single-crystal X-ray data, $M_r = 448{\cdot}9$, orthorhombic, space group D_{2h}^{11}–$Pbcm$, $a = 6{\cdot}279\,(4)$, $b = 7{\cdot}306\,(5)$, $c = 11{\cdot}177\,(7)$ Å, $V = 512{\cdot}7$ Å^3, $Z = 4$, $D_x = 5{\cdot}813$ g cm^{-3}, $\lambda(\text{Mo } K\alpha_1) = 0{\cdot}7093$ Å, $\mu = 236{\cdot}8$ cm^{-1}, $F(000) = 771$, (including anomalous dispersion), $T = 111$ K, $R(F^2) = 0{\cdot}100$ and $wR(F^2) = 0{\cdot}121$ for 3599 unique data, and $R = 0{\cdot}047$ and $wR = 0{\cdot}054$ for 2340 unique data with $F_o^2 > 3\sigma(F_o^2)$. The structure, which is similar to that of calcium chloride phosphate Ca_2ClPO_4 [Greenblatt, Banks & Post (1967). *Acta Cryst.* **23**, 166–171] contains discrete SiO_4^{4-} tetrahedra. The La atoms are eight-coordinate with six La—O bond lengths in the range 2·501 (3)–2·602 (4) Å and two La—Se bond lengths 3·048 (2), 3·233 (2) Å.

Experimental. Single crystals of La_2SeSiO_4 were isolated from reactions that produced single crystals of $La_2Ta_3Se_2O_8$ (Brennan, Aleandri & Ibers, 1990). La_2Se_3 was prepared from the elemental powders of lanthanum (REACTON, 99·9%) and selenium (Aldrich, 99·999+%) in evacuated quartz tubes heated at 1275 K. A combination of La_2Se_3, Ta_2O_5 (Aldrich, 99·99%) and tantalum (Aldrich, 99·9%) powders in a 1:1:1 ratio was reacted for three days at 1475 K in evacuated sealed quartz tubes to produce crystals of $La_2Ta_3Se_2O_8$ and La_2SeSiO_4, the source of silicon being the quartz tube. La_2SeSiO_4 grew as clear colorless prisms. EDAX microprobe analysis with a Hitachi S-570 SEM indicated the presence of lanthanum, selenium and silicon in approximately a 2:1:1 ratio. La_2SeSiO_4 powder was prepared by the reaction of La_2O_2Se with H_2SiO_3 (Aldrich thin layer chromatography high-purity-grade silica gel) in a 1:1 ratio at 1475 K for three days. La_2O_2Se was prepared by reaction of La_2O_3 with La_2Se_3 in a 2:1 ratio at 1275 K.

A clear irregular prism of La_2SeSiO_4 was ground into a 0·15 mm diameter sphere prior to X-ray diffraction measurements. The unit-cell parameters for La_2SeSiO_4 were determined from least-squares refinement of 15 reflections in the range $20 \leq 2\theta \leq 30°$ that had been hand centered on a Picker FACS-1

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