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The crystal structure of $(+)_{589}$-tris-[(−)-*trans*-1,2-diaminocyclohexane]cobalt(III) chloride monohydrate ('*ob*'isomer). Corrigenda.

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The atomic parameters of O(1) in the paper by Kobayashi, Marumo & Saito [*Acta Cryst.* (1972). B**28**, 2709–2715] are incorrect and the last line of Table 2(*a*) should read O(1) 2968 (6) 4991 (13) 7042 (10) 94 (6) 194 (11) 254 (16) 40 (12) 90 (8) 9 (20).

An incorrect population factor was used for O(1) in our recent paper on the crystal structure of

$$(+)_{589}\text{-}[\mathrm{Co(\text{-}chxn)_3]Cl_3.H_2O}$$

(Kobayashi, Marumo & Saito, 1972). The oxygen atoms are randomly distributed on the fourfold general position with population of 0·5. The last line in Table 2(*a*) should read as in the above abstract.

There is no significant change in the positional and thermal parameters of the other atoms. The final *R* value is 0·031.

Reference

Kobayashi, A., Marumo, F. & Saito, Y. (1972). *Acta Cryst.* B**28**, 2709–2715.

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The structures of fluorides. V. The *x*-parameter in NiF_2.

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A least-squares refinement with 22 integrated intensities from a neutron powder pattern of NiF_2 gave an unweighted *R* index on F^2 of 3·5 % and a fluorine *x* parameter 0·3052 (6). The octahedron was found to be undistorted, confirming that rutile-type fluorides as well as oxides cannot be completely described by a simple Born model.

The observed distortions of octahedra found in rutile-type AB_2 oxides and fluorides are of interest because of their correlation with theoretical predictions. These distortions can be expressed in terms of the difference between the lengths of the 4 coplanar bonds (l_1) located in the (110) plane and the 2 axial bonds (l_2) normal to (110). A Born model predicts $l_1 > l_2$. Baur & Khan (1971) found with X-ray diffraction that, while CrO_2, RuO_2, SnO_2, OsO_2, PbO_2, MgF_2, MnF_2, FeF_2, CoF_2, NiF_2 and ZnF_2 have $l_1 > l_2$, SiO_2, TiO_2 and GeO_2 have $l_1 < l_2$. In view of this, they pointed out that the Born model was probably inadequate. Jahn–Teller distortions are only likely in FeF_2 (Knox, 1961).

While studying fluorides present in fluorine generators, we measured the x_F parameter of NiF_2 at 21 °C by powder neutron diffraction. This technique does not have the problems of absorption and extinction encountered in the X-ray method. Pure NiF_2 was prepared by heating $NiCl_2$ in F_2 at 275 °C in a flow reactor (Brauer, 1963), and removing absorbed F_2 (Watanabe & Takashima, 1971) by pumping. The pattern was collected on HIFAR, the AAEC research reactor, by the elastic diffraction technique (Caglioti, 1970) with $\lambda = 1{\cdot}075$ Å. x_F, B_{Ni}, and B_F where *B* is the isotropic Debye–Waller factor, were determined by a least-squares refinement of 22 integrated intensities, the function minimized being $\sum_i [w_i(\sum_j JF_o^2 - \sum_j JF_c^2)^2]$, where *i* is a peak with *j* components each with multiplicity *J*.

The final discrepancy index, $R = \sum_i (\sum JF_o^2 - \sum JF_c^2)/$

Table 1. *Observed and calculated integrated intensities in the neutron powder pattern of* NiF_2 *at* 21 °C

hkl	$\sum JF_o^2$	$\sum JF_c^2$	σ
110	21·1	21·0	0·4
101	12·5	13·4	0·4
020	0·3	0·6	0·3
111	29·3	28·7	0·6
120	11·6	13·1	0·5
121	103·1	102·6	1·5
220	39·6	41·3	1·1
002	33·1	32·6	1·2
130	15·1	14·6	1·0
221	5·9	5·7	0·9
112 031	151·6	148·0	2·3
131	15·5	15·0	1·3
230 022	4·9	4·5	1·1
122	24·9	23·3	1·4
231	5·1	5·4	1·1
040	21·4	20·4	1·6
140 222	102·3	101·0	2·5
330	42·1	44·4	1·9
132 141	72·7	75·6	2·5
240 331	24·5	22·1	1·9
103	12·7	11·4	1·7
232 241 113	48·1	51·6	2·3

$\sum_i\sum_j JF_o^2$ was 0·035. The observed and calculated intensities are given in Table 1. The thermal parameters found were $B_{\mathrm{Ni}}=0{\cdot}45$ (8) Å^2 and $B_{\mathrm{F}}=0{\cdot}56$ (9) Å^2. These may be low if thermal diffuse scattering (TDS) is present, but TDS normally has little effect on positional parameters (Cooper, 1970). The correlation coefficients involving x_{F} and the scale factor, B_{Ni} and B_{F} were low (−0·007, 0·008 and 0·084 respectively).

Whereas the previous X-ray results indicated a slight distortion, the neutron refinement gives an undistorted octahedron in NiF_2. The present values of l_1 and l_2 are similar to the Ni–F distance of 2·006 Å in the perovskite $KNiF_3$, where the octahedron is regular by symmetry (Knox, 1961). The deviation from the Born model for NiF_2 is greater than in the previous work, lending further support for the conclusion of Baur & Khan (1971) that the simple Born model alone may not explain the observations.

The results of the present neutron and previous X-ray work are given below:

Study	x_F	l_1(Å)	l_2(Å)
Present work (neutron)	0·3052 (6)	2·005 (3)	2·007 (4)
Baur & Kahn (1971) (X-ray)	0·3012 (13)	2·022 (6)	1·981 (9)

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Cell constants of two hexachloropalladates.

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The unit-cell constants of potassium and ammonium hexachloropalladate have been redetermined as 9·7066 (3) and 9·8222 (4) Å respectively at 20°C.

Both potassium and ammonium hexachloropalladates have the K_2PtCl_6 structure (Wyckoff, 1965), with space group *Fm*3*m* (No. 225). Sharpe (1953) determined their lattice constants to be 9·74 (1) and 9·84 (1) Å. Bell, Hall & Wates (1966) redetermined the lattice constant of the ammonium salt to an accuracy of 0·01 Å. Greater accuracy has now been obtained by the use of relatively standard equipment and techniques.

Samples were obtained from FLUKA AG. Analysis results were:

K_2PdCl_6	26·8 % Pd calc.,	26·7 % Pd found;
$(NH_4)_2PdCl_6$	30·0 % Pd calc.,	30·6 % Pd found.

The positions of the peaks for angles (2θ) from 2° to 150° were obtained with unfiltered copper radiation on a Philips PW1050 diffractometer. Wavelengths of the different X-ray lines used were:

Cu $K\bar{\alpha}$	1·54178 Å
Cu $K\alpha_1$	1·54051
Cu $K\alpha_2$	1·54433
Cu $K\beta$	1·39217.

For all the reflexions arising from Cu $K\alpha_1$ a graph of d_{obs} against cot (θ_{obs}) was drawn adapting the method of Bracher & Small (1967) to eliminate zero errors in θ. The θ values for all of the reflexions were corrected and used in the program *CELFIT* of Bracher (1967) to refine the cell parameter. The parameters obtained for the two compounds at 20°C are:

K_2PdCl_6	9·7066 (3) Å
$(NH_4)_2PdCl_6$	9·8222 (4) Å.

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