

CCXLVIII.—*Inorganic Complex Salts. Crystallographic and Optical Study. Part I.*

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THE crystals of the inorganic complex salts now to be described were prepared in the Cambridge University Chemical Laboratory by Mr. W. Thomas, who will shortly communicate an account of their preparation.

The crystals were extremely difficult to obtain and were for the most part very small and poorly developed. Their crystallographic and optical determination has for this reason proved a difficult task.

The following salts have been investigated : (1) Potassium ferri-oxalate + $3\text{H}_2\text{O}$; (2) potassium aluminium oxalate + $3\text{H}_2\text{O}$; (3) ammonium *cis*-diamminodinitro-oxalatocobaltate + H_2O ; (4) ammonium *trans*-diamminodinitro-oxalatocobaltate + H_2O ; (5) potassium *cis*-diamminodinitro-oxalatocobaltate + H_2O ; (6) potassium *trans*-diamminodinitro-oxalatocobaltate + H_2O ; (7) barium *cis*-diamminodinitro-oxalatocobaltate + $3\text{H}_2\text{O}$; (8) ammonium diamminotetranitrocobaltate.

So far only racemic compounds have been examined, all attempts to crystallise the optically active components having failed, owing to racemisation taking place during crystallisation.

The compounds (1) and (2) crystallise in the holohedral class of the monoclinic system and are isomorphous with each other. The *cis*-compounds, (3), (5), and (7), were found to show a close isomorphism and to belong to the ditrigonal-scalenohedral class of the rhombohedral system. The *trans*-compounds, (4) and (6), although they show close resemblances in the values of their crystal

angles and crystallise with the same symmetry, the full symmetry of the monoclinic system, are not truly isomorphous, as will be shown later. The compound (8) crystallises in the orthorhombic system with holohedral symmetry.

It is surprising to find that the *cis*-ammonium and *cis*-potassium compounds, (3) and (5), are isomorphous with the *cis*-barium compound (7), the former being salts of a univalent metal or radicle and the latter of a bivalent metal and therefore possessing a great difference in molecular weight and structure. Moreover, the compounds crystallise with a different number of molecules of water, the barium compound with three and the ammonium and potassium with one only.

Potassium Ferri-oxalate, $[\text{Fe}(\text{C}_2\text{O}_4)_3]\text{K}_3 \cdot 3\text{H}_2\text{O}$ (Figs. 1 and 2).

A complete crystallographic examination of this substance has been made by Jaeger (*Rec. trav. chim.*, 1919, **38**, 242) and others. The results given below agree sufficiently well with those of Jaeger. He gives the symmetry as monoclinic holohedral, the axial angle $\beta = 94^\circ 20'$, and the axial ratio $a : b : c = 0.9923 : 1 : 0.3925$. The forms observed by him are the same as those given below. The measurements are recorded now for the sake of comparison with the isomorphous aluminium salt. A detailed determination of the optical characters has not previously been made.

Crystal system: monoclinic. *Class*: holohedral. *Axial angle*: $\beta = 94^\circ 13\frac{1}{2}'$. *Axial ratio*: $a : b : c = 0.9916 : 1 : 0.3895$.

Forms observed: $B = \{010\}$, well developed, elongated parallel to the zone $[010, 11\bar{1}]$; $m = \{110\}$, small bright faces; $o = \{111\}$, small bright faces; $e = \{\bar{1}11\}$, large faces, striated and elongated parallel to the axis of the zone $[010, 11\bar{1}]$; $s = \{\bar{1}01\}$, when present, much striated and elongated parallel to the zone $[010, 11\bar{1}]$.

Angles measured:

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$Bm = (010) : (110)$	21	$45^\circ 8' - 45^\circ 28\frac{1}{2}'$	$45^\circ 19'$	*
$Bo = (010) : (111)$	19	$70 25 - 70 46$	$70 34$	*
$Be = (010) : (\bar{1}11)$	8	$69 29\frac{1}{2} - 69 46$	$69 39$	$69^\circ 38\frac{1}{2}'$
$mo = (110) : (111)$	19	$58 38\frac{1}{2} - 58 57$	$58 48\frac{1}{2}$	*
$oe = (111) : (\bar{1}\bar{1}1)$	19	$57 37\frac{1}{2} - 58 58$	$57 45\frac{1}{2}$	$57 48\frac{1}{2}$

Habit: the crystals are transparent, green in colour, and well developed. They can easily be obtained fairly large or small as desired. A common habit of the crystals is that of thin plates parallel to $B = \{010\}$. More usually, the habit is that shown in Fig. 1.

It might appear, at first sight, that a more natural mode of treatment would be to regard the faces of the forms $\{\bar{1}11\}$ and

$\{101\}$ as prisms of the form $\{210\}$ and pinacoids of the form $\{100\}$, respectively. The treatment adopted brings out, however, the strong pseudotetragonal symmetry which the crystals evidently possess, and also shows the marked similarity which exists between

FIG. 1.

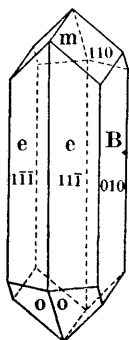


FIG. 2.

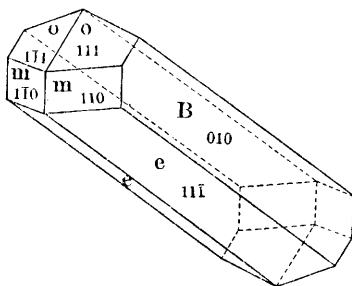


FIG. 3.

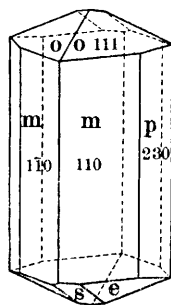


FIG. 4.

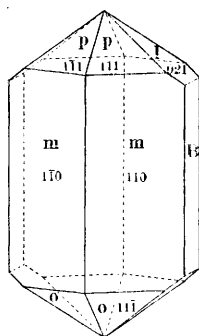


FIG. 5.

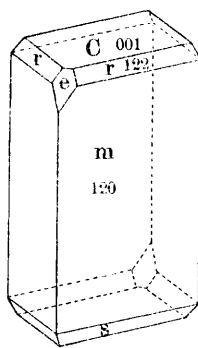


FIG. 7.

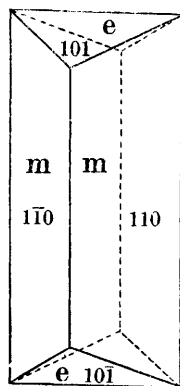
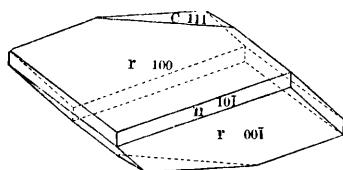


FIG. 6.



their angles and those of the crystals of the isomorphous aluminium compound. Fig. 2 shows a crystal drawn in the conventional position with the c -axis vertical.

Cleavage : none observed. A distinct parting parallel to $\{111\}$ was observed.

Density: determined by suspension in liquid, $d_4^{20} = 2.133$ (corrected).

Topic axes: $\chi : \psi : \omega = 8.354 : 8.424 : 3.199$.

Optical characters: refractive indices, measured by total reflection from $B = (010)$, $\alpha = 1.5019$, $\beta = 1.5558$, $\gamma = 1.5960$ for sodium light. The double refraction is therefore strong and the sign negative. The plane of the optic axes is B , and the acute bisectrix is inclined to the vertical axis, c , at an angle of about 2° in the obtuse axial angle. The optic axial angles in a liquid of refractive index about that of β were determined for lithium, sodium, and thallium lights and found to be as follows: Li, $79^\circ 36'$; Na, $78^\circ 49\frac{1}{2}'$; Tl, $77^\circ 53\frac{1}{2}'$. The dispersion shows $\rho > \nu$. Marked inclined dispersion of the bisectrices was also observed.

The pleochroism is fairly strong, the colour changing from dark green to very pale green. On looking through a crystal perpendicular to $B = (010)$, maximum absorption of light for vibrations perpendicular to the acute bisectrix and minimum absorption for vibrations parallel to that bisectrix were found.

Potassium Aluminium-oxalate, $[\text{Al}(\text{C}_2\text{O}_4)_3]\text{K}_3 + 3\text{H}_2\text{O}$ (Fig. 3).

Crystal system: monoclinic. Class: holohedral. Axial angle: $\beta = 93^\circ 23'$. Axial ratio: $a : b : c = 1.0061 : 1 : 0.3963$.

Forms observed: $B = \{010\}$, present occasionally and then as very narrow faces; $m = \{110\}$, and $p = \{230\}$, well developed but much striated; $o = \{111\}$, and $e = \{\bar{1}11\}$, small, fairly good faces; $s = \{\bar{1}01\}$, small, narrow faces.

Angles measured:

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$mm = (110) : (\bar{1}10)$	3	$89^\circ 40\frac{1}{2}' - 89^\circ 50'$	$89^\circ 46'$	*
$mp = (110) : (230)$	6	$11 \quad 1 \quad -11 \quad 14$	$11 \quad 7$	$11^\circ 18'$
$oo = (111) : (\bar{1}11)$	5	$39 \quad 29\frac{1}{2} - 39 \quad 51$	$39 \quad 37$	$39 \quad 42$
$es = (\bar{1}11) : (\bar{1}01)$	7	$20 \quad 17\frac{1}{2} - 20 \quad 55$	$20 \quad 35\frac{1}{2}$	*
$mo = (110) : (\bar{1}11)$	8	$58 \quad 44\frac{1}{2} - 59 \quad 17$	$59 \quad 0\frac{1}{2}$	$59 \quad 1$
$oe = (111) : (\bar{1}\bar{1}1)$	4	$58 \quad 6 - 58 \quad 31$	$58 \quad 19$	$58 \quad 20$
$em = (\bar{1}11) : (\bar{1}\bar{1}0)$	5	$62 \quad 40 - 63 \quad 0\frac{1}{2}$	$62 \quad 50$	*

Habit: the crystals were fairly large but poorly developed. They were colourless and more or less transparent. Their development was prismatic with either end terminated by four small pyramidal faces and one small dome.

Cleavage: none observed, but a well-marked although rough parting parallel to $\{\bar{1}01\}$ was observed.

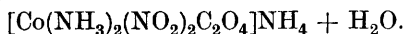
Density: determined by suspension in liquid, $d_4^{20} = 2.026$ (corrected).

Topic axes: $\chi : \psi : \omega = 8.300 : 8.442 : 3.273$.

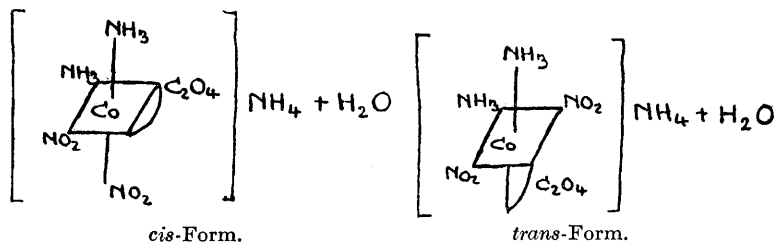
Optical characters : refractive indices as found by the immersion method, $\alpha = 1.49$, $\gamma = 1.50$. The double refraction is weak and the sign positive.

The plane of the optic axes is B , and the acute bisectrix is inclined to the a axis at an angle of about 11° in the obtuse axial angle. The optic axial angles in benzene ($\mu = 1.50$) were determined for lithium, sodium, and thallium lights and found to be as follows : Li, $70^\circ 44'$; Na, $71^\circ 12'$; Tl, $71^\circ 35\frac{1}{2}'$. The dispersion is apparently $v > p$. Marked inclined dispersion of the bisectrices was also observed.

Ammonium Diamminodinitro-oxalatocobaltiate,



Two isomeric forms of this composition occur, one crystallising in the rhombohedral system and one in the monoclinic. They correspond with the following structural formulæ :—



Ammonium cis-Diamminodinitro-oxalatocobaltiate + $1\text{H}_2\text{O}$.—
Crystal system : rhombohedral. *Class* : ditrigonal scalenohedral. *Millerian axial angle* : $\alpha = 107^\circ 57'$. Angle over the edges of the primary rhombohedron, $63^\circ 33'$.

Forms observed : $r = \{100\}$, well developed; $n = \{10\bar{1}\}$, small, narrow faces; $l = \{11\bar{1}\}$, small, square faces.

Angles measured :

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$rl = (100) : (1\bar{1}\bar{1})$	6	$85^\circ 35\frac{1}{2}' - 85^\circ 55'$	$85^\circ 43'$	$85^\circ 41'$
$nl = (10\bar{1}) : (1\bar{1}\bar{1})$	27	$43 \quad 3 \quad -43 \quad 54\frac{1}{2}$	$43 \quad 29$	$43 \quad 31$
$rl = (1\bar{1}\bar{1}) : (100)$	31	$46 \quad 9 \quad -46 \quad 54\frac{1}{2}$	$46 \quad 29$	*
$nr = (10\bar{1}) : (100)$	35	$57 \quad 48\frac{1}{2} - 58 \quad 47\frac{1}{2}$	$58 \quad 16$	$58 \quad 13\frac{1}{2}$

Habit : rhombohedral. The crystals were very small but well-developed. They were deep reddish-brown in colour and so opaque as to preclude any determination of optical characters. The crystals are similar in appearance to those of the isomorphous barium compound (Fig. 6) with the difference that in this case basal planes are absent.

Cleavage : none observed.

Density : measured by suspension in liquid, $d_4^{20} = 1.971$ (corr.).

Topic axes : $\chi = \psi = \omega = 5.784$.

Ammonium trans-Diamminodinitro-oxalatocobaltiate + $1\text{H}_2\text{O}$ (Fig. 4).—*Crystal system* : monoclinic. Class : holohedral. Axial angle : $\beta = 92^\circ 51\frac{1}{2}'$. Axial ratio : $a : b : c = 0.4089 : 1 : 0.3654$.

Forms observed : $B = \{010\}$, occasionally present as long, narrow faces; $m = \{110\}$, fairly good, long, narrow faces; $l = \{021\}$, $p = \{111\}$, $o = \{\bar{1}11\}$, all very small faces.

Angles measured :

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$mm = (110) : (\bar{1}\bar{1}0)$	5	$48^\circ 8' - 48^\circ 16'$	$48^\circ 12'$	*
$Bp = (010) : (111)$	3	$73 52\frac{1}{2} - 74 3$	$73 56\frac{1}{2}$	*
$Bo = (010) : (\bar{1}\bar{1}1)$	2	$72 48\frac{1}{2} - 73 3\frac{1}{2}$	$72 56$	$73^\circ 2\frac{1}{2}'$
$mp = (110) : (111)$	4	$44 46 - 44 52$	$44 48\frac{1}{2}$	*
$po = (111) : (\bar{1}\bar{1}1)$	5	$87 25\frac{1}{2} - 87 37\frac{1}{2}$	$87 32$	$87 45\frac{1}{2}$
$Bl = (010) : (021)$	6	$51 38 - 52 13\frac{1}{2}$	$51 54$	$51 50\frac{1}{2}$
$pl = (111) : (021)$	3	$41 27\frac{1}{2} - 41 36\frac{1}{2}$	$41 32$	$41 25\frac{1}{2}$
$lm = (021) : (\bar{1}\bar{1}0)$	4	$77 2\frac{1}{2} - 77 22$	$77 12$	$77 31$
$ol = (\bar{1}\bar{1}1) : (021)$	6	$42 22\frac{1}{2} - 42 46\frac{1}{2}$	$42 33$	$42 42$
$lm = (021) : (110)$	8	$73 2\frac{1}{2} - 73 54$	$73 36$	$73 15\frac{1}{2}$

Habit : prismatic, terminated by extremely small dome and pyramid faces. The crystals were very poorly developed. Hence it was not possible to obtain more than approximate measurements of the crystallographic angles. A strong pseudorhombic symmetry is shown, the axial angle differing from a right angle by less than 3° and the angles from $B = \{010\}$ on to $p\{111\}$ and $o = \{\bar{1}11\}$ differing respectively by less than 1° . The larger crystals were hollow right through the centre, rendering them useless for optical study, and it was not found possible to make a complete determination of optical properties with the extremely small crystals which were not hollow.

Cleavage : none observed.

Density : measured by suspension in liquid, $d_4^{20} = 1.879$ (corr.).

Topic axes : $\chi : \psi : \omega = 4.224 : 10.33 : 3.775$.

Optical characters : refractive indices as found by the immersion method, greater than 1.74 for vibrations parallel to the x and z axes and somewhat lower for those parallel to the y axis.

Pleochroism : colour changes from dark reddish-brown to light yellowish-brown, with maximum absorption of light for vibrations parallel to the x axis, minimum absorption for vibrations parallel to the z axis, and intermediate absorption for vibrations parallel to the y axis.

Nearly straight extinction parallel to the crystallographic axis c was observed.

Potassium Diamminodinitro-oxalatocobaltiate,
 $[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_2\text{C}_2\text{O}_4]\text{K} + \text{H}_2\text{O}$.

As in the case of the isomorphous ammonium salt, two isomeric forms of this composition occur, the *cis*-form, as before, crystallising in the rhombohedral, and the *trans*-form in the monoclinic, system.

Potassium cis-Diamminodinitro-oxalatocobaltiate + H_2O .—*Crystal system*: rhombohedral. *Class*: ditrigonal scalenohedral. *Millerian axial angle*: $\alpha = 107^\circ 54'$. *Angle over the edges of the primary rhombohedron*, $63^\circ 40'$.

Forms observed: $r = \{100\}$, well developed; $n = \{101\}$, small, narrow faces; $l = \{111\}$, small, square faces; $C = \{111\}$, small, triangular faces, which occur only rarely and are very poor when present.

Angles measured:

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$Cr = (111):(100)$	2	$37^\circ 24' - 37^\circ 44\frac{1}{2}'$	$37^\circ 34'$	$37^\circ 31'$
$rl = (100):(111)$	3	$85 32 - 85 44\frac{1}{2}$	$85 39$	$85 34\frac{1}{2}$
$nl = (101):(111)$	8	$43 28\frac{1}{2} - 43 41\frac{1}{2}$	$43 34$	$43 28\frac{1}{2}$
$lr = (111):(100)$	20	$46 11\frac{1}{2} - 46 55\frac{1}{2}$	$46 31\frac{1}{2}$	*
$nr = (101):(100)$	8	$58 1 - 58 21\frac{1}{2}$	$58 12\frac{1}{2}$	$58 10$

Habit: rhombohedral with basal terminations rare. The crystals were extremely small but fairly well-developed. They were dark reddish-brown and too opaque to allow of the determination of optical properties. They were similar in appearance to the crystals of the isomorphous barium compound (Fig. 6).

Cleavage: none observed.

Density: determined by suspension in liquid, $d_4^{20} = 2.007$ (corrected).

Topic axes: $\chi = \psi = \omega = 5.874$.

Potassium trans-Diamminodinitro-oxalatocobaltiate + H_2O (Fig. 5).—*Crystal system*: monoclinic. *Class*: holohedral. *Axial angle*: $\beta = 92^\circ 47\frac{1}{2}'$. *Axial ratio*: $a : b : c = 1.1558 : 1 : 0.9388$.

Forms observed: $A = \{100\}$, very narrow faces, not always present; $C = \{001\}$, fairly good, lozenge-shaped faces, always present and the largest of the faces terminating the prisms; $m = \{120\}$, fairly well-developed, long faces, often striated perpendicularly to the prism edge; $e = \{101\}$, extremely small, generally triangular faces; $r = \{122\}$, very narrow faces; $s = \{\bar{1}21\}$, similar in development to the form r ; $t = \{101\}$, similar in development to the form e , but only once found.

In addition to these forms, the crystals from one crystallisation also showed faces which evidently belonged to the forms $o = \{121\}$ and $p = \{\bar{1}22\}$, but as these crystals were extremely poor it was

not possible to obtain sufficiently accurate measurements of the angles from these faces on to O or m to establish their identity beyond doubt.

Angles measured :

	No. of measure- ments.	Limits.	Mean, Obs.	Calc.
$Am = (100) : (120)$	8	$66^\circ 14\frac{1}{2}' - 66^\circ 52'$	$66^\circ 35'$	*
$Ae = (100) : (101)$	2	$49 34\frac{1}{2} - 49 48\frac{1}{2}$	$49 41\frac{1}{2}$	$49^\circ 14\frac{1}{2}'$
$eC = (101) : (001)$	3	$37 56 - 38 0\frac{1}{2}$	$37 58$	*
$Ct = (001) : (\bar{1}01)$	1		$41 10\frac{1}{2}$	$40 11$
$Cm = (001) : (120)$	4	$88 40\frac{1}{2} - 89 6\frac{1}{2}$	$88 53\frac{1}{2}$	*
$Cr = (001) : (122)$	4	$44 28 - 45 16$	$44 52$	$45 3$
$ms = (120) : (12\bar{1})$	5	$25 27 - 26 32\frac{1}{2}$	$26 8\frac{1}{2}$	$26 17$
$sC = (12\bar{1}) : (00\bar{1})$	6	$63 6 - 65 48\frac{1}{2}$	$64 45\frac{1}{2}$	$64 49\frac{1}{2}$
$me = (120) : (101)$	5	$74 35\frac{1}{2} - 75 24$	$75 1\frac{1}{2}$	$74 58$
$mr = (\bar{1}20) : (122)$	5	$61 20\frac{1}{2} - 62 36$	$61 59\frac{1}{2}$	$61 55$

Habit : the crystals were extremely small and not well developed and only permitted approximate determinations of the crystallographic angles. They were prismatic with a prominent basal termination and with a certain number of very small pyramidal and domal faces.

Cleavage : none observed.

Density : measured by suspension in liquid, $d_4^{20^\circ} = 2.093$ (corrected).

Topic axes : $\chi : \psi : \omega = 6.080 : 5.260 : 5.043$.

Optical characters : refractive indices as found by the immersion method, $\alpha = 1.56$, $\beta = 1.65$, $\gamma > 1.74 < 1.77$. The double refraction is therefore strong and the sign positive.

The optic axes lie in the plane of symmetry and the obtuse bisectrix is very slightly inclined to the crystallographic axis c . Hence the extinction is nearly straight. The optic axial angle is large.

Pleochroism : the colour changes from deep reddish-brown to yellowish-brown. Maximum absorption of light takes place for vibrations parallel to the x axis, minimum absorption for vibrations parallel to the z axis, and intermediate absorption for vibrations parallel to the y axis.

A comparison between the values of the angles obtained for this compound and for the *trans*-ammonium compound indicates a close relationship between them. The two compounds are, however, not truly isomorphous, for there is a complete difference of orientation between them, the plane of symmetry and the diad axis in the ammonium compound being at right angles to the respective positions they should occupy if it were isomorphous with the potassium compound. In both compounds a strong pseudo-orthorhombic symmetry is evident. Had orthorhombic

symmetry been attained, the crystals of the two compounds would have shown true isomorphism.

Barium cis-Diamminodinitro-oxalatocobaltiate,
 $[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_2\text{C}_2\text{O}_4]_2\text{Ba} + 3\text{H}_2\text{O}$ (Fig. 6).

Crystal system : rhombohedral. *Class* : ditrigonal-scalenohedral. Millerian axial angle $\alpha = 108^\circ 16'$; angle over the edges of the primary rhombohedron, $63^\circ 6'$.

Forms observed : $C = \{111\}$, small, triangular faces, always present and fairly good; $r = \{100\}$, large, well-developed faces; $n = \{10\bar{1}\}$, very narrow and rather poor faces; $l = \{11\bar{1}\}$, extremely small faces, very rarely present.

Angles measured :

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$Cr = (111) : (100)$	14	$36^\circ 43\frac{1}{2}' - 37^\circ 24\frac{1}{2}'$	$37^\circ 2'$	*
$rl = (100) : (1\bar{1}\bar{1})$	1		$86^\circ 35\frac{1}{2}'$	$86^\circ 30'$
$nr = (10\bar{1}) : (100)$	21	$57^\circ 56\frac{1}{2}' - 59^\circ 8\frac{1}{2}'$	$58^\circ 32'$	$58^\circ 27'$

Habit : rhombohedral with basal terminations. The crystals were extremely small and rather opaque and of a deep reddish-brown colour.

Cleavage : none observed.

Density : determined by suspension in liquid, $d_4^{20} = 2.142$ (corrected).

Topic axes : $\chi = \psi = \omega = 7.544$.

Optical characters : refractive indices, measured by total reflection from $\{111\}$, $\epsilon = 1.5607$. ω is very high and could not be measured. The double refraction is strong and the sign is negative. On looking through some of the clearer crystals, perpendicular to the basal plane, in strong sodium light, a faint uniaxial interference figure was observed in convergent polarised light.

Table for the comparison of the *cis*-rhombohedral compounds.

Chemical composition.	Millerian axial angle.	Angle over edges of primary rhombo- hedron.	Molecular weight = M .	Density. d_4^{20} .	Topic axes, $\chi = \psi = \omega$.	
					(1) Calculated using M .	(2) Calculated using $2M$.
$[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_2\text{C}_2\text{O}_4]\text{NH}_4 + \text{H}_2\text{O}$	$107^\circ 57'$	$63^\circ 33'$	309.11	1.971	5.784	7.288
$[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_2\text{C}_2\text{O}_4]\text{K} + \text{H}_2\text{O}$	$107^\circ 54'$	$63^\circ 40'$	330.17	2.007	5.874	7.401
$[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_2\text{C}_2\text{O}_4]_2\text{Ba} + 3\text{H}_2\text{O}$	$108^\circ 16'$	$63^\circ 6'$	739.53	2.142	7.544	

A comparison of the crystallographic angles of the three rhombohedral *cis*-compounds makes evident their isomorphous relationship. This is a close one for all three compounds and in the case of the ammonium and potassium compounds is of an extreme closeness. The latter might be expected, since in such heavy

molecules the ammonium group and the potassium atom are a comparatively small part and therefore their substitution for one another would naturally have a very small effect on their crystallography. But, as pointed out early in this paper, the close isomorphism of the barium compound is not what would naturally be expected. No doubt, the reason given above for the extremely close isomorphism of the ammonium and potassium compounds applies in this case, but it does not seem sufficient to be the whole explanation. It may be that each barium atom takes the place of two ammonium groups or two potassium atoms in the crystal structure without greatly altering that structure. In this connexion, it is interesting to note that if the topic axes for the ammonium and potassium compounds are calculated, using double their molecular weights, the values obtained are fairly close to that of the barium compound in which the single molecular weight is used. It would seem, however, that only an exact knowledge of the arrangement of the atoms in the crystal structure could afford a complete explanation.

Ammonium Diamminotetranitrocobaltiate, $[\text{Co}(\text{NH}_3)_2(\text{NO}_2)_4]\text{NH}_4$
(Fig. 7).

Crystal system : orthorhombic. *Class* : holohedral. *Axial ratio*
 $a : b : c = 0.8677 : 1 : 0.5110$.

Forms observed : $m = \{110\}$, large faces; $e = \{101\}$, fairly large faces; $q = \{011\}$, very small faces, rarely present.

Angles measured :

	No. of measure- ments.	Limits.	Mean. Obs.	Calc.
$ee = (101) : (\bar{1}01)$	2	$60^\circ 58' - 61^\circ 0'$	$60^\circ 59'$	*
$me = (110) : (101)$	7	$67^\circ 5' - 67^\circ 47\frac{1}{2}'$	$67^\circ 28'$	*
$mm = (110) : (\bar{1}\bar{1}0)$	4	$81^\circ 16' - 82^\circ 58'$	$82^\circ 2\frac{1}{2}'$	$81^\circ 54'$

Habit : simple prismatic, terminated by a pair of macrodomes. The crystals were extremely small and poorly developed. The angular measurements obtained are therefore only approximate. Owing to the poor quality of the crystals, their optical study was not undertaken, but it was found that their refractive indices were high, lying between 1.78 and 1.74 for vibrations parallel to the c axis and about 1.73 for vibrations in a direction perpendicular to the c axis and parallel to either the a or the b axis. The crystals were of a deep reddish-brown colour.

Cleavage : none observed.

Density : determined by suspension in liquid, $d_4^{20} = 1.972$ (corrected).

Topic axes : $\chi : \psi : \omega = 6.042 : 6.963 : 3.558$.

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