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Chemistry — "On some isomeric, complex cis- and trans-Diethylenediamine-Salts of Cobaltum, and on Tri-ethylenediamine-Zinc-Chloride." By Prof. Dr. F. M. JAEGER and Dr. Jul. KAHN. (Communicated in the meeting of June 30, 1917)

According to Werner's theory concerning the stereometrical configuration of inorganic salts derived from the complex radical: $\{MeX'_{\mathfrak{o}}\}$, there must exist two isomerides of derivatives containing ions of the special type: $\left(Me\frac{Y'_2}{X'_4}\right)$, which are distinguished as cisand trans-isomerides. If the six co-ordination-loci round the central atom be considered as situated in space like the six corners of a regular octahedron, the substitutes Y' are located in the cisderivatives as near as possible to each other, while, on the contrary, in the trans-derivatives they are elongated as far as possible from each other, being placed at the two ends of an axis of the octahedron. If in the complex salts of this kind, the four co-ordination-loci X'_4 be occupied by two bivalent radicals X''_2 , it is obvious that the configuration of the molecule in the cis-derivatives possesses the axial symmetry of C_2 ; the heteropolar binary symmetry-axis of these complex ions joins of course the middle of the octahedron-edge Y'Y'with that of the opposite and parallel edge. The symmetry of these ions is therefore exactly that of the monoclinic-sphenoidical class of crystallonomy and to every configuration of this kind corresponds therefore a non-superposable mirror-image, because the complex of atoms possesses only axial symmetry. The cis-compounds of the type $\left(Me\frac{Y'_{\bullet}}{X''_{\bullet}}\right)$ must, for that reason, be considered as racemic compounds · eventually resolvable into two optically active and oppositely rotating antipodes. The possibility of such a fission is demonstrated by WERNER in an experimental way for several salts of this kind.

The trans-derivatives of the same type $\left(Me \frac{Y'_2}{X''_2}\right)$, however, possess the symmetry of the group D_2^H . Their configuration is therefore identical with its mirror-image, so that they are not resolvable into such antipodes. 1)

¹⁾ See: F. M. JAEGER, Lectures on The Principle of Symmetry and Its Applications in all Natural Sciences, Elsevier-Company, Amsterdam, (1917), p. 228-256.

In the following paragraphs some of these resolvable and unresolvable salts will be described more in detail.

§ 2. RACEMIC CIS-DIAMINO-DIETHYLENEDIAMINE-COBALTI-CHLORIDE.

$$\left\{ Co \frac{(NH_s)_2}{(Eine)_2} \right\} Cl_s + 1H_2O.$$

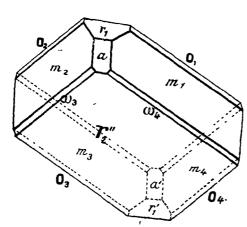


Fig. 1.

Red-brown, well-developed, and very lustrous small crystals, which obviously are isomorphous with the corresponding bromide and iodide.

Monoclinic-prismatic.

$$a:b:c=1,1172:1:0,8325;$$

 $\beta=87^{\circ}56^{1}/3'.$

Forms observed: $r'_2 = [\overline{101}]$, predominant; the external shape of the crystals appears ordinarily flattened parallel to this form.

Furthermore: m = [110], well-developed and very lustrous; $r_1 = [101]$, small, but giving sharp reflexes; o = [121] and $\omega = [\overline{121}]$, both very narrow, and almost equally developed; a = [100], small, but very lustrous; q = [011], mostly narrow, but yielding splendid reflexes.

Angles:	Observed:	Calculated.
a: m = (100): (110) =	* 480 9′	
$a: r'_2 = (\overline{1}00): (\overline{1}01) =$		
$a:r_1=(100):(101)=$	* 51 59	_
$m: r'_2 = \overline{(110)}: \overline{(101)} =$	67 14	67°17¹/₄
$m: \omega = (\overline{1}10): (\overline{1}21) =$	32 24	32 23
$m: m = (110): (\overline{1}10) =$	= 83 42	83 42
$r'_2: q = (\overline{101}): (011) =$	52 17	52 23
$r_1: r'_2 = (101): \overline{(101)} =$	= 73 21	73 2 3
$m: r_1 = (110): (101) =$	65 24	65 45
o:q = (121):(011) =	= 29 13	29 12
$r_1: q = (101): (011) =$	= 51 22	51 31
m: o = (110): (121) =	= 34 59	35 15

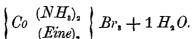
Perhaps cleavable parallel to m.

The crystals are only slightly dichroitic, in a way analogous to that of the *iodide*. They are evidently identical with crystals described

previously '), if only the following symbols be adopted there: $a = [\overline{101}], o = [\overline{110}], r = [\overline{100}], s = [101], and \omega = [\overline{121}].$

In contradiction to the data given in literature, all three halogenides must have the same content of crystallisation-water, and, according to the analytical investigation, $1 H_2O$.

§ 3. RACEMIC CIS-DIAMINO-DIETHYLENEDIAMINIC-COBALTI-BROMIDE.



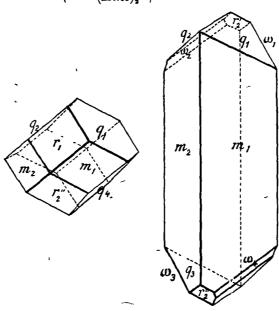


fig 2. cis-Diamino-diethylenediamine cobalti-bromide.

The substance crystallises in flat and long brownish-red needles -prismatic in the direction of the c-axis, or in short, thick and small crystals, having a slight elongation in the direction of the a-axis.

Monoclinic-prismatic.

$$a:b:c=1,1177:1:0,8322.$$

 $\beta=88^{\circ}5'.$

Forms observed: m = [110], and q = [011], large and lustrous. Occasionally m is predominant, and eventually q. Furthermore: $r_1 = [101]$ and $r'_2 = [\overline{101}]$, almost equally large and giving good reflexes; $\omega = [\overline{121}]$, commonly small, but also, if q = [011] be only slightly developed, occasionally almost equally large as r'_2 , a = [100], very narrow, and mostly absent. The substance is completely isomorphous with the corresponding iodide.

¹⁾ F. M. JAEGER, Zeits. f. Kryst. 39. 545. (1904).

Angles: Observed Calculated. $a: m = (100): (110) = 48^{\circ}10'$ $r'_2: m = (\overline{101}): (\overline{110}) = 67 15$ $r'_2: q = (101): (011) = 52 14$ $m: m = (110): (\overline{110}) = 83 \ 40$ 83°40′ $q: q = (011): (0\overline{11}) = 79 13$ 79 13 $r'_2: q = (\overline{101}): (0\overline{11}) = 52 14$ 52 14 $r_1: q_1 = (101): (011) = 51 \ 23^1/2$ 51 23 1/2 $r_1: m = (101) \cdot (110) = 6548$ 65 51 32 23 $m: \omega = (\overline{1}10): (\overline{1}21) = 32 19$ $\omega : q = (121) : (011) = 2752$ 28 1

The crystals are distinctly cleavable parallel to [110]. They are slightly dichroitic, analogously to the *iodide*. The angle of extinction also, about 20° with respect to the c-axis on m, has a size also comparable with that found in the case of the *iodide*.

§ 4. RACEMIC CIS-DIAMINO-DIETHYLLNEDIAMINE-COBALTI-IODIDE.

$$\left\{ Co \stackrel{(NH_3)_2}{(Eine)_2} \right\} I_3 + 1 H_2 O.$$

Big, splendidly developed, brown-red and highly lustrous crystals with very constant angular values.

Monoclinic-prismatic.

$$a:b:c=1,0975:1:0,8178;$$

 $\beta=87^{\circ}28^{2}/_{s}'$

Forms observed: m = [110], predominant and very lustrous; a = [100], smaller than m; q = [011], large and sharply reflecting, $r_1 = [101]$ and $r'_2 = [\overline{101}]$, almost equally well-developed and yielding excellent reflexes; o = [121], and $\omega = [121]$, almost equally large and well reflecting. The habit is prismatic parallel to the c-axis.

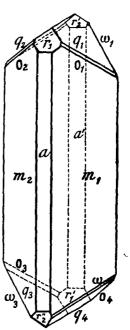


fig 3 Racemic cis-Diamino-diethylenediamine cobaltiiodide.

Angles:	J	Observed:	Calculated
a n	n = (100) : (110) = *		
$q \cdot q$	$=(011):(0\overline{1}1)=*$	78 301/2	
$r_1 : r_1$	$_{2}'=(101):(\overline{1}01)=*$	73 22	
m:n	n = (110) : (110) =	84 44	84°44′
a'.r	$g' = \overline{(100)} : \overline{(101)} =$	54 59 -	54 561/3
$r_1:a$	a = (101) : (100) =	51 44	51 40
$m \cdot c$	o = (110) : (121) =	35 20	35 31

Observed: Calculated: o: q = (121): (011) =29 13 29 12 32 14 $m:\omega = (\overline{1}10):(\overline{1}21) =$ 32 20 $m: r_1 = (110) \cdot (101) =$ 65 20 65 17 $m: r_2' = (\overline{1}10) \cdot (\overline{1}01) =$ 67 12 67 131/2 $r_1:q=(101):(011)=$ 51 16 51 6 $r_2: q = (101): (011) =$ 52 3 52 9 $m: q = \overline{(110)}: (011) =$ 64 38 64 43 m': q = (110): (011) =60 31 60 38 $\omega : q = (121) : (011) =$ 28 18

Perfectly cleavable parallel to m.

The crystals are feebly dichroitic: on a yellow-orange for vibrations in the direction of the c-axis, and for those perpendicular to them: red-orange. On m is the angle of extinction about 28° with respect to the vertical axis.

§ 5. TRANS-DIAMINO-DIETHYLENEDIAMINE-COBALTI-IODIDE.

$$Co_{(Eine)_2}^{(NH_3)_2}$$
 I_s .

Small, often badly developed crystals me with a dark red-brown colour; their aspect is that of hexagonal plates (fig. 4).

Rhombic-bipyramidal.

$$a:b:c=1,2449:1:1,2842.$$

Forms observed: a = [100], distinctly predominant and very lustrous; o = [111], and m = [120], well-developed and giving sharp mirror-images.

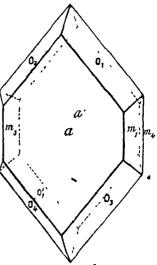


Fig. 4. trans-Diamino diethylenediamino-cobalti iodide.

Angles: Observed: Calculated: $a:o = (100):(111) = 57^{\circ}58' - a:m = (100):(120) = 68 7 - 60 = (111):(11\overline{11}) = 64 12 - 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}12 = 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}12 = 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}12 = 64^{\circ}12 = 64^{\circ}14' - 64^{\circ}12 = 64^{\circ}12 =$

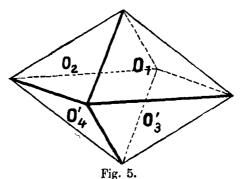
The crystals are slightly dichroitic: on a orange-red for vibrations parallel to the c-axis, dark orange-red for such as are perpendicular to them. The plane of the optical axes is [100]; the a-axis is first bisectrix. The apparent axial angle is large, the dispersion is rather strong, with e v round the e-axis.

§ 6. RACEMIC CIS-DIAMINO-DIETHYLENEDIAMINE-COBALTI-NITRATE.

$$\left\{ Co_{(Eine)_2}^{(NH_s)_2} \right\} (NO_s)_s$$
.

Beautiful, well-developed, yellow-brown or red-brown pyramids, with lustrous faces. They are nicely built, and have very constant angular values.

Rhombic-bipyramidal. a:b:c=0.9473:1:0.6758.



cis-Diamino-diethylenediamine-Cobalti-Nitrate.

Forms observed: o = [111], Nitrate. with highly lustrous faces. The external form of the crystals is that of flattened octahedra.

Angles. Observed Calculated: $o.o = (111) (11\overline{1}) = 910 0'$

$$o \cdot o = (111) \cdot (\overline{111}) = 61 \cdot 11$$

 $o \cdot o = (111) \cdot (\overline{111}) = 57 \cdot 48$
 $o \cdot o = (111) \cdot (\overline{111}) = 89 \cdot 2$
 $0 \cdot o = (111) \cdot (\overline{111}) = 89 \cdot 2$
 $0 \cdot o = (111) \cdot (\overline{111}) = 89 \cdot 2$

No distinct cleavage was observed.

§ 7. RACEMIC CIS-DINITRO-DIETHYLENEDIAMINE-COBALTI-NITRATE. (Flavo-salts).

$$\left\{ \begin{array}{ll} Co \stackrel{(NO_{\mathfrak{s}})_2}{(Eine)_{\mathfrak{s}}} \end{array} \right\} (NO_{\mathfrak{s}}).$$

Small, flattened, yellow-red prismatic crystals. They are identical with those previously measured 1) by us, which we had received from Werner, but they have other combination-forms, and somewhat deviating angular values. The aspect of the crystals is that of fig. 20 in the paper mentioned.

Monoclinic-prismatic.

$$a:b:c=1,5589:1:0,4073.$$

 $\beta=68^{\circ}30^{\circ}/4'.$

All forms previously observed were met with again; their relative development was also almost the same, only q = [01.1] was appreciably larger here. New forms are: r = [401], well developed and yielding good reflexes; and s = [410], very narrow, but lustrous.

Angles: Observed: Calculated:
$$m: m = (110): \overline{(110)} = 110^{\circ}50' - c: r = (001): \overline{(401)} = 57.36$$

20

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¹⁾ F. M. JAEGER, Zeits. f. Kryst. u. Miner. 39. 564. (1904).

Observed: Calculated:

m:c = (110):(001) = *	78 0	
m:b = (110):(010) =	34 35	34°35′
a:s = (100):(410) =	19 44	19 56
s: m = (410): (110) =	35 31	35 29
a: m = (100): (110) =	55 25	55 25
m: q = (110): (011) =	60 48	60 541/2
b:q = (010):(011) =	69 20	69 141/2
c:q=(001):(011)=	20 40	20 451/2

No distinct cleavability was observed.

On [010] feebly dichroitic: for vibrations parallel to the c-axis, yellow-orange, for such perpendicular to them red-orange. The angle of extinction on [010] is about 60° with respect to the c-axis, in the acute angle of the axes a and c. The plane of the optical axes is probably [010].

§ 8. Trans-Dinitro-diethylenediamine-Cobalti-Nitrate. (Croceo-salt).

,
$$\left\{ Co \frac{(NO)_2}{(Eine)_2} \right\} (NO_3)$$
.

This compound was obtained from the mother-liquid of the corresponding cis-derivative, in the form of splendidly developed, reddish brown, very lustrous and transparent, little crystals. They are evidently identical with those prepared by Werner, and previously described i); also in this case the angular values somewhat differ from those formerly measured. As a new form, b = [010] was found. The external aspect of the crystals is that of hexagonally or

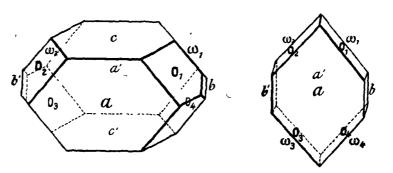


Fig. 6.

trans-Dinitro-diethylenediamine-Cobalti-Nitrate. octogonally bordered, rather thick crystal-plates.

Monoclinic-prismatic. a:b:c=1,3040:1:1,0085; $\beta=73^{\circ}31^{1}/4^{i}.$

¹⁾ F. M. JAEGER, Zeits. f. Kryst. v. Miner. 39. 565. (1904).

Forms observed: a = [100], predominant and splendidly reflecting; c = [001], smaller, often completely absent, but very lustrous; b = [010], small, well reflecting; o = [111], somewhat broader than $\omega = [111]$, perfectly reflecting. The external habit is flattened parallel to [100], with a slight elongation parallel to the b-, or the c-axis.

-		
Angles:	Observed	d: Calculated:
$a: \omega = (100) (1)$		-
$o: \omega = (\overline{1}1\overline{1}) (\overline{1})$	(11) = * 77 30	_
b:o=(010):(1	11)=* 55 54	
$\omega = (\overline{1}11) : (\overline{1}$	$\overline{1}1) = 83 53$	83°52′
$b: \omega = (010): (\overline{1})$	11) = 48 4	48 4
$o: \omega = (111) : (\overline{1})$	11) = 57 26	57 35
a: o = (100): (1	11) = 5240	52 12
a:c = (100):(0	(01) = 73.58	73 311/4
c:o = (001):(1	11) = 45 12	44 59
$c: \omega = (001): \overline{(1)}$	11) = 57, 22	57 22
o:o=(111):(1	$\bar{1}1) = 69 12$	69 12
	·	

No distinct cleavability was observed.

The crystals are distinctly dichroitic: on a for vibrations parallel to the c-axis: orange-yellow, for those perpendicular to them: lemon-yellow. The optical axial plane is [010]; one optical axis is almost perpendicular to a. Extremely strong dispersion, with o < v.

§ 9. RACEMIC CIS-DINITRO-DIETHYLENEDIAMINE-COBALTI-NITRITE.

(Flavo-salt).

$$\left\{ Co \frac{(No_2)_2}{(Eine)_2} \right\} (NO_2).$$

Dark coloured crystals, which in their external aspect, are highly analogous to those of the corresponding trans-derivative. They are very beautifully developed, and possess constant angular values.

Monoclinic-prismatic.

$$a:b:c = 0.7382:1:0.9094;$$

 $\beta = 82^{\circ} 57'.$

Forms observed: m = [110], and b = [010], large and very lustrous; o = [111], large, perfectly reflecting; $\omega = [\overline{2}23]$, small but well reflecting; c = [001], small, giving good reflexes. The external habit is prismatic parallel to the c-axis, and commonly somewhat flattened parallel to [010]. Occasionally [010] is absent, in other cases, however, the same is true for $[\overline{2}23]$.

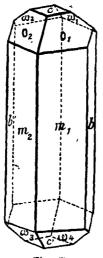


Fig. 7.
Racemic cis-Dinitrodiethylenediamine-CobaltiNitrite.

Angles: Observed: Calculated: $m: m = (110): (1\overline{10}) = 72^{\circ}27'$ c: m = (001): (110) = 84 19 $o:o = (111):(\overline{111}) = 59 38$ 530461/2 b: m = (010): (110) = 5349 $c: \overline{\omega} = (001): \overline{(223)} = 4834$ 48 241/2 $c \cdot o = (001) : (111) = 56 \ 46$ 56 51 o: m = (111): (110) = 384138 50 $\omega: m = (\overline{223}) : (\overline{110}) = 35 35$ 35 541/2 b:o = (010):(111) = 60 1060 11 $b: \omega = (010) : (223) = 63 \ 37$ 63 38

No distinct cleavability was found.

In contrast with the water-containing crystals of the *trans*-derivative, these crystals do *not* manifest any trace of change. They are distinctly dichrottic: on [010] orange-red for vibrations parallel to the c-axis, for such as are perpendicular to them: blood-red. The angle of extinction on b includes 33° with the direction of the c-axis, in the quadrant of the acute angle a:c.

 $\omega:\omega = \overline{(223)}:\overline{(223)} = 5249$

§ 10. Trans-Dinitro-diethylenediamine-Cobalti-Nitrite (Croceo-salt).

$$\left\{ Co \frac{(No_2)_2}{(Eine)_2} \right\} (NO_2) + 2H_2O.$$

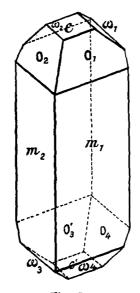


Fig. 8
Trans-Dinitro diethylenediamineCobalti-Nitrite.

Big, splendidly developed, dark brown crystals which, however, soon lose their water of crystallisation, becoming gradually orange-yellow, most rapidly on the faces of the prism.

52 44

Monoclinic-prismatic. . a:b:c=1,0458:1:1,7995; $\beta=68^{\circ}33^{2}/_{b}$.

Forms observed: m = [110], predominant and very lustrous; o = [111], large and giving good reflexes, like c = [001], which is much smaller than m; $\omega = [225]$, very small, and often absent. Notwithstanding the different angular values there exists a striking analogy in aspect and development of these crystals, and those of the anhydrous cis-derivative.

Angles: Observed: Calculated. $c:o\ (001):(111)=^*54^\circ57'$ $o:o\ (111):(1\overline{11})=^*72\ 31$ $o.m\ (111):(110)=^*50\ 17$ $c:m\ (001):(110)=74\ 41$ $m:m\ (110):(1\overline{10})=91\ 30$ $g1\ 32$ $g1\ 33$ $g1\ 34$ $g2\ 34$

A distinct cleavability was not found.

The crystals are dichroitic in a way analogous to that found in the case of the cis-derivative. On m is the angle of extinction oblique.

\$11. From a solution containing both the cis- and trans-derivatives, crystals of the form shown in fig. 9 were deposited, which imitate in a striking way the aspect of the cis-derivative. In contradiction with those of this substance, however, the crystals of this kind do not decay, nor do they lose any water of crystallisation. Moreover, they have often rather oscillating angular values in the zône of the prism, and uneven faces. Their dichroism is analogous to that observed with the pure cis-, or trans-compounds.

Monoclinic-prismatic.

$$a \ b \cdot c = 1,0169 : 1 : 0,9030, \beta = 66^{\circ} 16'.$$

Forms observed: m = [110] predominant and lustrous; o = [111], large, giving good reflexes; b = [010], much smaller than m; c = [001], small, but well measurable. The external form is prismatic cistowards the c-axis.

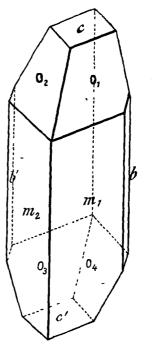


Fig 9.

cis- + trans-Dinitro-diethylenediamine Cobalti-Nitrite.

Ang	gles	Observed		Calculated
c	m = (001)	(110)=*	72°52′	-
b	m = (010)	(110)=*	47 3	
0	o = (111)	$(1\overline{1}1) = *$	64 291/2	
m	m = (110)	$(1\overline{1}0) =$	85 58	85°54′
C	o = (001)	(111) =	48 50	48 261/2
0	m = (111)	(110) =	57 55	58 18
b	o = (010)	(111) =	57 47	57 451/4

No distinct cleavability was observed.

Notwithstanding the striking analogy in form, a distinct and real difference in the angular values and parameters is found to exist; the ratio a:b is analogous to that of the trans-compound, while for b:c exactly the same is true with respect to the cis-derivative. It is difficult to make sure whether here a double-compound of the isomeric salts is present, or a mixed-crystal between them. The fact that the angular values are somewhat fluctuating, and that the faces often reflect irregularly, might be looked upon as an argument of the greater probability of the last mentioned supposition. In any case, the peculiar crystallonomical analogy, and the intermediate shape of these crystals between those of the components, are highly remarkable phenomena, perhaps explicable by the only feeble morphotropic influence of NO_2 -groups in general.

It is worth drawing attention, moreover, to the fact that all the cis-Diethylenediamine-derivatives here studied, have monoclinic symmetry, however not that of the group C_2 . Probably there will be salts of this particular symmetry amongst the optically-active components of these racemic salts.

§ 12. Finally we give here a description of RACEMIC TRI-ETHYLENEDIAMINE-ZINC-CHLORIDE, the fission of which is also attempted in this laboratory.

 $\{Z_n \ (Eine)_{s}\} \ Cl_{s} + 2 \ H_{s}O.$

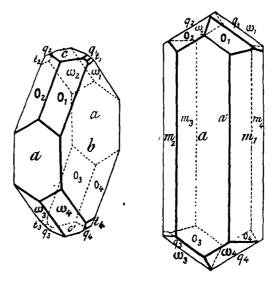


Fig. 10.
Tri-ethylenediamine-Zinc-Chloride.

Beautiful, colourless, very lustrous and transparent crystals, which allow very exact measurements. The substance was prepared from ZnCl, and ethylenediamine in slight excess; it was recrystallised from water. Analysis gave: 20 °/°, Cl, corresponding with a content of 2 molecules of water.

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Monoclinic-prismatic.

a:b:c = 0.9238:1:0.6299;

\beta = 86°33'.
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Forms observed: b = [010], commonly predominant and highly lustrous, occasionally totally absent; a = [100], well-developed and always present; o = [111], yielding splendid reflexes; m = [350], lustrous, and when present, rather large; c = [001], small, often absent, giving somewhat dull reflexes; q = [011] and t = [031], very dimly reflecting; $\omega = [111]$, highly lustrous and well-developed.

The external form is that of hexagonally bordered, thick plates parallel to [010], or that of short prisms parallel to the c-axis.

gles:	Ob:	served:	Calculated
a:c	$=(100)\cdot(001)=*$	86 33	_
$b \cdot b$	=(010):(111)=*	63 11	_
a : 0	$=(\overline{1}00):(\overline{1}11)=*$	$62 \ 12^{1}/_{2}$	
0:0	$=(111)\cdot(1\overline{11})=$	53 44	53°44′
ω:ω	$=(\overline{111})\cdot(\overline{111})=$	56 16	56 1 6
b : ω	$=(010)(\overline{1}11)=$	61 52	61 52
ο:ω	$=(111)(\overline{1}11)=$	59 50	_
a:o	=(100).(111)=	57 58	57 49
c . b	=(001) · (011) =	32 1	$32 9^{2}/_{3}$
q:t	=(011):(031)=	29 51	29 55
t:b	=(031):(010)=	27 53	27 551/2
a:m	=(100):(350)=	55 0	55 47
m m	$=(350):(\overline{350})=$	67 58	68 16
, ,	.1	1	

No distinct cleavability was found.

On [010] is the angle of extinction 11° with respect to the c-axis; the plane of the optical axes is probably [010].

Laboratory for Inorganic and Physical Chemistry of the University, Groningen, Holland.