

Chemistry. — “*The Crystalforms of Some Organic Nitrogen-compounds.*
By Prof. F. M. JAEGER.

(Communicated at the meeting of January 30, 1926).

§ 1. In this paper the crystalforms are described of the following fifteen nitrogen-derivatives, which have been investigated during the last few years: *p*-Amino-acetophenone; Acetyl-phenyl-urethane; Acetyl- α -amino-pyridine; α -Amino-pyridine-urethane; Acetyl-chinine; Tartronic-acid; *d*-Tartramidic-acid; Trihydrazide-hydrochloride of Tricarballic acid; Amido-sulphonic acid; Phtalimidine; Ammonium-phtalimidine-acetate; Ethylenediamine - hydrochloride; *p*-Nitro-mono-propyl-aniline; Benzoylcyno-acetic methyl-ether and *m*-Nitro-benzoylcyno-acetic methyl-ether. Also the description is added here of Triethylphosphine-carbodi-sulphide, the remarkable addition-product of carbondisulphide: CS_2 and $P(C_2H_5)_3$.

§ 2. *p*-Amino-Acetophenone ¹⁾: $CH_3 \cdot CO \cdot C_6H_4(NH_2)$; Mpt: $106^\circ C$.

From benzene in large, flat, pale yellowish, transparent crystals; from a mixture of alcohol + chloroform in rectangular plates, elongated either parallel to the *b*-, or to the *a*-axis (fig. 1).

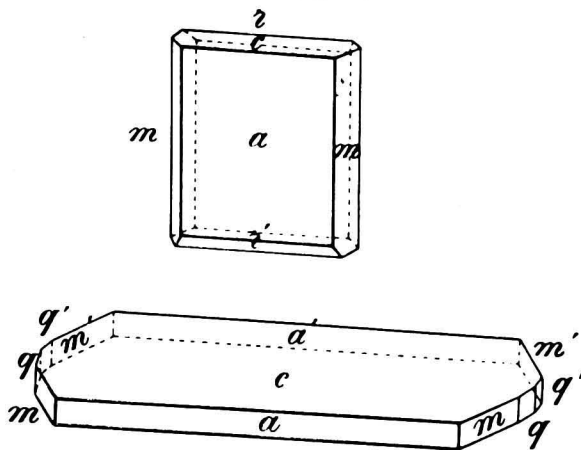


Fig. 1.

Monoclinic-prismatic.

$$a:b:c = 1,7447:1:1,6662;$$

$$\beta = 73^\circ 16'.$$

Forms observed: $c = \{001\}$, mostly predominant and lustrous; $a = \{100\}$, well developed; $m = \{110\}$, broader than $p = \{120\}$; $r = \{\bar{1}01\}$, well developed and yielding good reflections. The habit is often elongated parallel to the *b*-axis, with $\{001\}$ and $\{100\}$ predominant, and in the last case laso prismatic along the *c*-axis.

¹⁾ V. B. DREWSSEN, Lieb. Ann. der Chemie, 212, 162, (1882).

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$a : r = (100) : (\bar{1}01) = * 55^{\circ} 9'$		—
$a : m = (100) : (110) = * 59 \quad 6$		—
$c : a = (001) : (100) = * 73 \quad 16$		—
$c : r = (001) : (\bar{1}01) = 51 \quad 33$		$51^{\circ} 35'$
$p : p = (120) : (\bar{1}20) = 33 \quad 55$		$33 \quad 19$
$p : m = (120) : (110) = 14 \quad 3$		$14 \quad 14\frac{1}{2}$
$c : p = (001) : (120) = 85 \quad 20\frac{1}{2}$		$85 \quad 16$
$c : m = (001) : (110) = 81 \quad 35$		$81 \quad 29$

In the prism-zone the angular values vary not inappreciably. No distinct cleavability. The optical axial plane is $\{010\}$; the first bisectrix is almost perpendicular to $\{001\}$. The angle of the optical axes is evidently very small; weak and inclined dispersion.

§ 3. *Acetyl-phenyl-Urethane*¹⁾: $C_6H_5 \cdot N \begin{matrix} \text{CO} \cdot \text{CH}_3 \\ \text{CO} \cdot \text{OC}_2\text{H}_5 \end{matrix}$; Mpt.: 59°C .

From ligroine in colourless, well-developed crystals. (Fig. 2).

Rhombic-bipyramidal.

$a : b : c = 1,2323 : 1 : 15141$.

Forms observed: $c = \{001\}$, large and lustrous; $m = \{110\}$, also well developed and giving sharp images; $a = \{100\}$ and $b = \{010\}$, generally almost equally large; $o = \{211\}$, well reflecting, mostly smaller, but occasionally much larger than m . The habit is short-prismatic with tabular form parallel to $\{001\}$, or that of thick plates parallel to $\{001\}$.

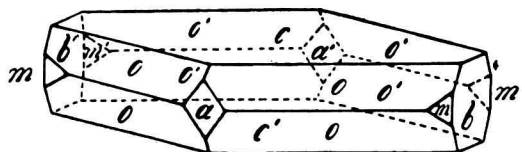
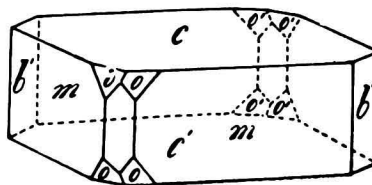


Fig. 2.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$b : m = (010) : (110) = * 39^{\circ} 3\frac{1}{2}'$		—
$c : o = (001) : (211) = * 55 \quad 17$		—
$a : o = (100) : (211) = 45 \quad 32$		$45^{\circ} 30'$
$b : o = (010) : (211) = 64 \quad 25$		$64 \quad 28$
$o : o = (211) : (\bar{2}\bar{1}\bar{1}) = 51 \quad 10$		$51 \quad 4$
$a : m = (100) : (110) = 50 \quad 56\frac{1}{2}$		$50 \quad 56\frac{1}{2}$
$m : m = (110) : (\bar{1}\bar{1}0) = 101 \quad 53$		$101 \quad 53$
$m : o = (110) : (211) = 39 \quad 12$		$39 \quad 7$
$o : o = (211) : (\bar{2}\bar{1}\bar{1}) = 69 \quad 28$		$69 \quad 26$
$m : o = (\bar{1}\bar{1}0) : (211) = 83 \quad 55$		$83 \quad 54\frac{1}{2}$

No distinct cleavage could be found.

¹⁾ D. R. NIJK, Rec. d. Trav. chim. d. Pays-Bas, **39**, 701, (1920).

The plane of the optical axes is probably $\{100\}$; the c -axis is first bisectrix. The apparent axial angle is only small; the dispersion is weak.

§ 4. *Acetyl- α -Amino-pyridine*¹⁾: $C_5H_4N.NH.CO.CH_3$; *Mpt*: $71^\circ C$.
From benzene + ligroine in colourless, very lustrous crystals. (Fig. 3).

Monoclinic-prismatic.

$$a : b : c = 1,4939 : 1 : 2,0719;$$

$$\beta = 89^\circ 14' \frac{1}{2}.$$

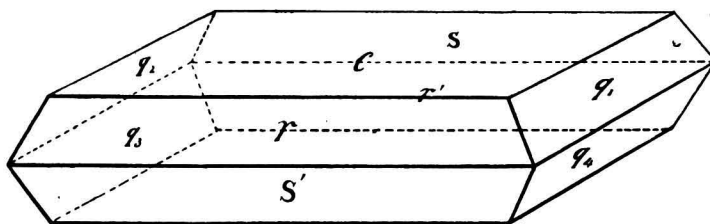


Fig. 3.

Forms observed: $c = \{001\}$, predominant, yielding good reflections; $r = \{100\}$ and $s = \{\bar{1}01\}$, equally broad, well developed and giving sharp images; $q = \{011\}$, broad and lustrous. The habit is that of thick tables parallel to $\{001\}$, with elongation in the direction of the b -axis.

Angular values: *Observed*: *Calculated*:

$c : r = (001) : (101) =$	$^* 53^\circ 42\frac{1}{2}'$	—
$c : s = (001) : (\bar{1}01) =$	$^* 54 42\frac{1}{2}$	—
$c : q = (001) : (011) =$	$^* 64 14$	—
$r : s = (101) : (10\bar{1}) =$	$71 35$	$71^\circ 35'$
$q : q = (011) : (01\bar{1}) =$	$51 32$	$51 32$
$r : q = (101) : (011) =$	$74 59$	$75 5\frac{1}{2}$

Perfectly cleavable parallel to $\{010\}$.

§ 5. *α -Amino-pyridine-Urethane*²⁾: $C_5H_4N.NH.CO.OC_2H_5$; *Mpt*: $105^\circ,5 C$.

From ether this substance is obtained in well developed, but very soft and plastic crystals; they are colourless and commonly possess a prismatic or parallelepipedal shape. By the translation-planes present the crystals can only be manipulated, if the necessary precautions be taken; the angular values vary not inappreciably in the different zones.

Monoclinic-prismatic.

$$a : b : c = 0,7946 : 1 : 1,3552;$$

$$\beta = 73^\circ 21'.$$

¹⁾ E. DINGEMANSE and J. P. WIBAUT, Rec. d. Trav. Chim. d. Pays-Bas, **42**, 240, (1923),

²⁾ E. DINGEMANSE and J. P. WIBAUT, Rec. d. Trav. Chim. d. Pays-Bas, loco cit.

Forms observed: $m = \{110\}$, large and lustrous; $a = \{100\}$, narrower than m , often absent, but giving good reflections; $c = \{100\}$, well devel-

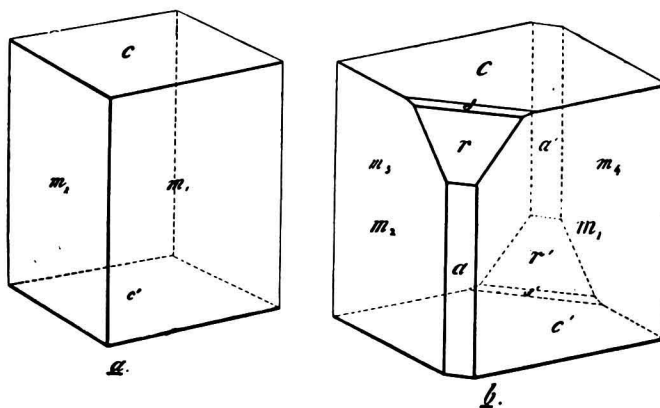


Fig. 4.

oped and yielding very good reflections; $r = \{101\}$, well reflecting, much broader than $s = \{108\}$, which form is often absent.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$m : m = (110) : (\bar{1}\bar{1}0) =$	$74^\circ 34'$	—
$c : m = (001) : (110) =$	$76 49\frac{1}{2}$	—
$c : r = (001) : (101) =$	47 40	—
$c : s = (001) : (108) =$	10 40	$10^\circ 54'$
$a : m = (100) : (110) =$	37 23	37 17
$a : s = (100) : (108) =$	63 0	62 27
$r : s = (101) : (108) =$	36 45	36 46

Cleavage perfect parallel to $\{101\}$.

On $\{001\}$ diagonal, on $\{110\}$ oblique extinction.

§ 6. *Acetyl-chinine:* ¹⁾ $C_{20}H_{23}(CO \cdot CH_3) \cdot N_2O_2$; *Mpt:* $116^\circ C$.

This compound crystallizes at room-temperature from ether in splendid, colourless and transparent crystals, having a high lustre and measuring often several cm. in length. (Fig. 5).

Rhombic-bisphenoidal.

$$a : b : c = 1,1142 : 1 : 0,6119.$$

Forms observed: $a = \{100\}$ and $m = \{110\}$, about equally large and very lustrous; they yield excellent reflections, although on a often multiple images. Further: $b = \{010\}$, much narrower, often only with a single plane, well reflecting; $s = \{201\}$ and $r = \{101\}$, both giving excellent

¹⁾ L. SEEKLES, Dissertation, Leiden, (1922), p. 33.

images, s mostly much smaller than r ; $o = \{\bar{1}11\}$, large and lustrous, almost always showing a single plane; $\omega = \{111\}$, small, dull, but well measurable. The crystals are well built and have constant angular values. Their habit is thick-prismatic and elongated parallel to the c -axis or flattened parallel to two planes of m and a .

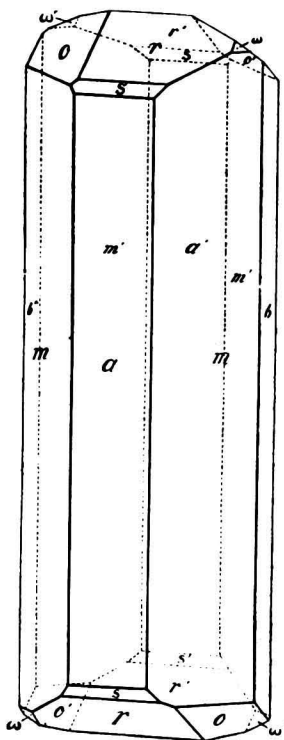


Fig. 5.

Angular values: Observed: Calculated:

$a : m = (100) : (110) =$	$48^\circ 5\frac{1}{2}'$	—
$a : s = (100) : (201) =$	$42 19$	—
$b : m = (010) : (110) =$	$41 58\frac{1}{2}$	$41^\circ 54\frac{1}{2}$
$m : m = (110) : (\bar{1}10) =$	$83 57$	$83 49$
$s : r = (201) : (101) =$	$18 49$	$18 54\frac{1}{2}$
$r : r = (101) : (\bar{1}01) =$	$57 59$	$57 33$
$a : o = (\bar{1}00) : (\bar{1}11) =$	$64 48$	$64 53\frac{1}{2}$
$o : \omega = (\bar{1}11) : (111) =$	$50 20$	$50 13$
$\omega : a = (111) : (100) =$	$64 48$	$64 53\frac{1}{2}$
$m : \omega = (110) : (111) =$	$50 28$	$50 33\frac{3}{4}$
$m : s = (110) : (201) =$	$60 19$	$60 24$
$s : o = (201) : (\bar{1}11) =$	$33 38$	$33 31\frac{1}{4}$
$o : m = (\bar{1}11) : (110) =$	$86 18$	$86 4\frac{3}{4}$
$b : \omega = (010) : (111) =$	$61 36$	$61 47\frac{1}{2}$
$\omega : r = (111) : (101) =$	$28 23$	$28 12\frac{1}{2}$
$m : r = (110) : (101) =$	$71 21$	$71 15$
$b : o = (010) : (\bar{1}11) =$	$61 32$	$61 47\frac{1}{2}$
$o : r = (\bar{1}11) : (\bar{1}01) =$	$28 28$	$28 12\frac{1}{2}$

No distinct cleavage was found.

In solution the substance is levogyrate:
 $[a]_D = -120,08$.

§ 7. *Tartronamic Acid*¹⁾: $NH_2 \cdot CO \cdot CH(OH) \cdot COOH$; M_{pt} : $160^\circ C$. with decomposition.

From water this compound crystallizes commonly in colourless flat needles, which are arranged in spherulithes, but rarely also in big, colourless, prismatic crystals. They are well built and allow accurate measurements.

Monoclinic-prismatic.

$a : b : c = 3,0003 : 1 : 2,4063$;

$\beta = 64^\circ 19'$.

Forms observed: $c = \{001\}$ and $a = \{100\}$, both giving very sharp images and almost equally broad; $o = \{111\}$,

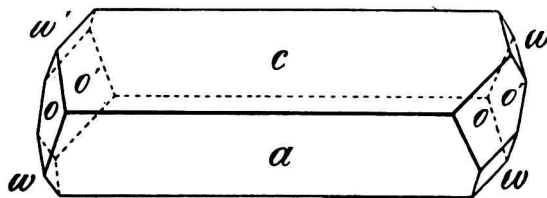


Fig. 6.

¹⁾ N. MENSCHUTKIN, Lieb. Ann. der Chemie, 182, 82, (1876).

splendidly reflecting and much better than $\omega = \{\bar{1}11\}$, which form shows only narrow and dull faces. The habit is thick-prismatic parallel to the b -axis.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$a : c = (100) : (001) =^* 64^\circ 19'$		—
$c : o = (001) : (111) =^* 59 31$		—
$a : o = (100) : (111) =^* 62 16$		—
$o : o = (111) : (\bar{1}\bar{1}\bar{1}) = 70 34$		$70^\circ 23'$
$c : \omega = (001) : (\bar{1}11) = 74 8$		$74 4\frac{1}{2}$
$\omega : o = (\bar{1}11) : (\bar{1}\bar{1}\bar{1}) = 46 21$		$46 24\frac{1}{2}$
$a : \omega = (\bar{1}00) : (\bar{1}11) = 80 59$		$81 9$
$\omega : o = (\bar{1}\bar{1}\bar{1}) : (111) = 36 40$		$36 35$

No distinct cleavage could be observed.

On a and c normal extinction. The plane of the optical axes is $\{010\}$; inclined dispersion. One branch of the hyperbola is on $\{001\}$ visible at the border of the field of the microscope.

§ 8. *Dextrogyrate Tartramidic Acid*¹⁾: $\text{COOH} \cdot \text{CHOH} \cdot \text{CHOH} \cdot \text{CO} \cdot \text{NH}_2$; *Mpt*: 172°C .

Crystals of the shape reproduced in Fig. 7 were obtained from a

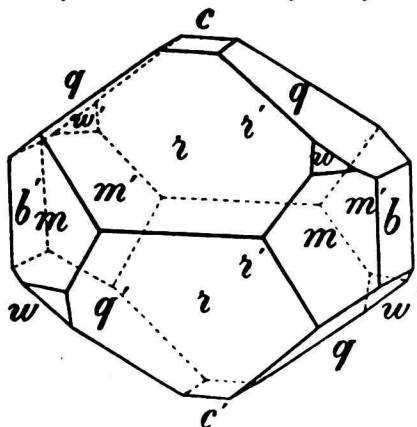


Fig. 7.

solution in water. They are colourless, highly lustrous and yield excellent images.

Rhombic-bisphenoidal; (*pseudotetragonal*).

$a : b : c = 0,7352 : 1 : 0,7393$.

Forms observed: $m = \{110\}$, $r = \{201\}$ and $q = \{011\}$, all about equally well developed, very lustrous and giving good reflections; $\omega = \{221\}$, somewhat smaller, but yielding sharp images, as also $b = \{010\}$; $c = \{001\}$, distinct, but a little duller. The cry-

ystals are nearly isometrical, with a slight flattening in the direction of the a -axis.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$b : m = (010) : (110) =^* 53^\circ 40\frac{3}{4}'$		—
$q : q = (011) : (0\bar{1}\bar{1}) =^* 72 57$		—
$r : r = (201) : (20\bar{1}) = 52 58$		$52^\circ 52\frac{1}{2}$
$c : r = (001) : (201) = 63 31$		$63 33\frac{1}{2}$
$b : \omega = (010) : (221) = 56 43$		$56 38\frac{1}{2}$

¹⁾ Comp, e. g.: L. PASTEUR, *Ann. de Chim. et Phys.*, (3), 38, 454, (1853); P. GROTH, *Chem. Kryst.*, III, 302, 308. The preparation here investigated was made by Dr. R. A. WEERMAN *Diss. Delft*, (1916), p. 109.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$\omega : r = (221) : (201) =$	33 18	33 21½
$m : m = (110) : (\bar{1}\bar{1}0) =$	72 39½	72 38⅔
$b : q = (010) : (011) =$	53 31½	53 31½
$c : q = (001) : (011) =$	36 28½	36 28½
$m : q = (110) : (011) =$	49 37½	49 37½
$r : q = (201) : (011) =$	69 8	69 1
$m : r = (110) : (201) =$	43 41	43 49⅔

No distinct cleavage was found.

On {010} and {001} normal extinction. The plane of the optical axes is {100}, with the *c*-axis probably as first bisectrix. PASTEUR observed in the crystals of the corresponding levogyrate isomeride, instead of ω , a form $o = \{221\}$.

§ 9. *Triethylphosphine-Carbodisulphide*¹⁾: $P(C_2H_5)_3 \cdot CS_2$; Mpt: 122° C.

This compound crystallizes from alcohol in flat, blood-red, very brittle needles.

Monoclinic-prismatic (pseudo-rhombic).

$$a : b : c = 0,6306 : 1 : 0,9716;$$

$$\beta = 89^\circ 51\frac{1}{2}.$$

Forms observed: $b = \{010\}$, strongly predominant and, just as $r = \{101\}$ and $r'' = \{\bar{1}01\}$, yielding very sharp images; $\omega = \{111\}$, narrow, but well measurable; $c = \{001\}$, lustrous and well reflecting.

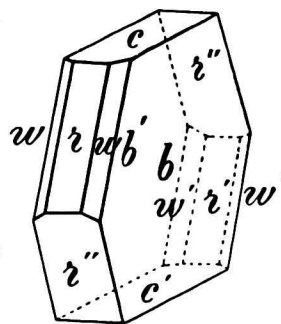


Fig. 8.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$r : r'' = (101) : (\bar{1}01) =^*$	114° 2'	—
$b : \omega = (010) : (111) =^*$	62 9	—
$c : r = (001) : (101) =^*$	56 55	—
$r : \omega = (101) : (111) =$	27 51	27° 51'
$r : b = (101) : (010) =$	90 0	90°

Perfectly cleavable parallel to {010} and {101}. The plane of the optical axes is practically parallel to {001}; the first bisectrix is perpendicular to {010} and has a positive character. Very strongly dichroitic: on *b* for vibrations parallel to {001} dark-red, for those perpendicular to this, orange-yellow.

¹⁾ According to J. P. WIBAUT (Rec. d. Trav. Chim. d. Pays-Bas, 44, (1925), 239) the meltingpoint of 95° C., as given in literature, must be considered as wrong. With some other choice of the parameters, the crystals investigated would be identical with those investigated by SELLA (Rend. Acad. Lincei, (2), 20, 361, (1863).

§ 10. *Trihydrazide-hydrochloride of Tricarallylic Acid*¹⁾: $C_3H_5(CO \cdot NH \cdot NH_2)_3, 3HCl$.

From alcohol by slow evaporation in vacuo beautiful, colourless crystals were obtained.

Cubic-hexakisoctahedral.

Well built octahedra, with $(111) : (\bar{1}\bar{1}\bar{1}) = 70^\circ 33'$. The small corrosion-figures on the faces of $\{111\}$ were hexagons with their edges parallel to those of the octahedron. The crystals are isotropous, without circular polarisation.

§ 11. *Amidosulphonic Acid*: $NH_2 \cdot SO_3H$.

From a solution in water big, colourless, splendidly built and perfectly transparent crystals are obtained, as reproduced in Fig. 9.

Rhombic-bipyramidal;
(pseudo-tetragonal).

$$a : b : c = 0,8720 : 1 : 0,8774.$$

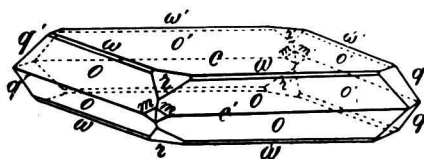


Fig. 9

Forms observed: $c = \{001\}$, large and very lustrous; $o = \{111\}$, also well developed and yielding sharp reflections; $r = \{102\}$ and $q = \{021\}$, almost equally large and well reflecting; $m = \{210\}$, appreciably smaller, but distinct and exactly measurable; $\omega = \{114\}$, very narrow and giving somewhat duller images. The habit is almost isometrical, with flattening parallel to $\{001\}$; often r and ω show only half the number of faces, the crystals therefore showing an apparent monoclinic symmetry. However, the crystals are really pseudo-tetragonal: the angle $(001) : (101) = 45^\circ 10' \frac{1}{2}$, so that the b -axis would be the vertical axis of the apparently tetragonal individual; $a : c$ becomes then about: 1 : 1,142. The extinction on $\{210\}$ and the other optical properties prove, however, the true rhombic symmetry of the crystals.

Angular values: *Observed:* *Calculated:*

$c : o = (001) : (111) =^* 53^\circ$	$9\frac{3}{4}'$	—
$m : m = (210) : (\bar{2}\bar{1}0) =^* 47$	7	—
$c : q = (001) : (021) = 60$	$20\frac{1}{4}$	$60^\circ 19\frac{1}{2}'$
$q : q = (021) : (0\bar{2}\bar{1}) = 59$	19	59 21
$c : r = (001) : (102) = 26$	$57\frac{1}{2}$	26 $42\frac{1}{4}$
$o : o = (111) : (\bar{1}\bar{1}\bar{1}) = 63$	30	63 $28\frac{2}{3}$
$o : o = (111) : (\bar{1}\bar{1}\bar{1}) = 73$	$40\frac{1}{2}$	73 $40\frac{1}{2}$
$c : \omega = (001) : (112) = 18$	45	18 27

No distinct cleavage could be found.

The plane of the optical axes is $\{001\}$, with the b -axis as first bisectrix. The apparent axial angle is large, the dispersion weak.

¹⁾ TH. CURTIUS and A. HESSE, Journ. für prakt. Chem., 62, 236, (1900).

Evidently the crystals are identical with those described by A. FOCK (Zeits. f. Kryst., **14**, 531, (1888)), where $\{010\} = \{001\}$ in our crystals.

§ 12. *Phtalimidine*: $C_6H_4 \left\langle \begin{array}{c} CO \\ CH_2 \end{array} \right\rangle NH$; Mpt: $150^\circ C$.

From a mixture of chloroform and carbondisulphide flat parallelogram-shaped crystals were obtained, as reproduced in fig. 10.

Monoclinic-prismatic.

$$b : c = 1 : 0,4913;$$

$$\beta = 79^\circ 20\frac{2}{3}'.$$

Forms observed: $b = \{010\}$, strongly predominant and yielding sharp reflections, as also $a = \{100\}$; $q = \{100\}$, well developed.

Angular values:

	Observed:	Calculated:
$a : q = (100) : (011) =$	$80^\circ 25'$	—
$b : q = (010) : (011) =$	$64 \quad 13\frac{1}{2}$	—
$q : q = (011) : (0\bar{1}1) =$	$51 \quad 33\frac{1}{4}$	$51^\circ 33'$
$a : b = (100) : (010) =$	$90 \quad 0$	90°

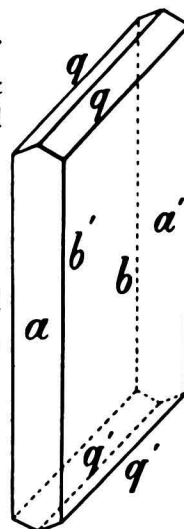


Fig. 10.

Perfectly cleavable parallel to $\{010\}$; distinctly parallel to $\{100\}$.

The plane of the optical axis is probably parallel to $\{010\}$. On b one of the optical principal sections makes an angle of $19\frac{1}{2}^\circ$ with the c -axis in the posterior quadrant.

§ 13. *Ammonium - Phtalimidine - acetate*: $C_6 H_4 \left\langle \begin{array}{c} CO \\ CH_2 \end{array} \right\rangle N-CH_2-COO(NH_4) + 2 H_2O$.

This salt was deposited from an aqueous solution in the form of big, colourless crystals of the shape reproduced in Fig. 11.

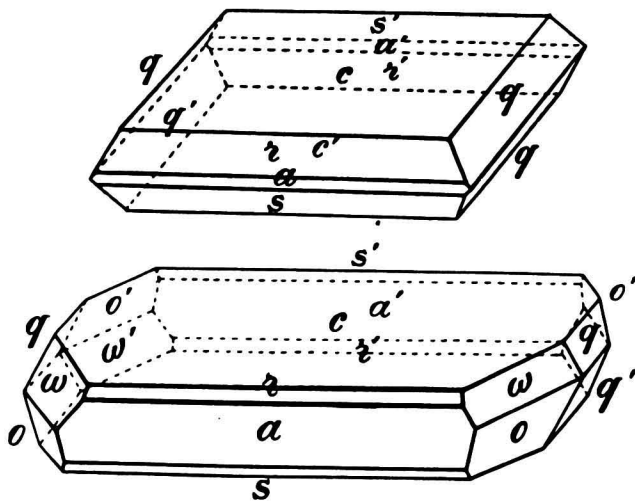


Fig. 11.

Monoclinic-prismatic.

$$a : b : c = 0,8837 : 1 : 1, 4301 :$$

$$\beta = 77^{\circ}46'.$$

Forms observed: $c = \{001\}$, strongly predominant and giving excellent images; $q = \{011\}$, broad, also very lustrous; $r = \{101\}$, broad reflecting; $a = \{100\}$, narrow and dull, but occasionally broader than r and yielding good reflexes; $s = \{\bar{1}01\}$, narrower than r , well measurable; $\omega = \{122\}$, small, well reflecting; $o = \{\bar{3}62\}$, larger than ω and yielding rather good images. The habit is tabular parallel to $\{001\}$ with elongation in the direction of the b -axis.

Angular values: *Observed:* *Calculated:*

$c : r = (001) : (101) =$	$\ast 49^{\circ} 40'$	—
$r : a = (101) : (100) =$	$\ast 28 \quad 6$	—
$c : q = (001) : (011) =$	$\ast 54 \quad 25$	—
$a : s = (100) : (\bar{1}0\bar{1}) =$	$35 \quad 2$	$34^{\circ} 48'$
$s : c = (\bar{1}0\bar{1}) : (00\bar{1}) =$	$67 \quad 27$	$67 \quad 26\frac{1}{2}$
$q : q = (011) : (0\bar{1}\bar{1}) =$	$71 \quad 12$	$71 \quad 10$
$r : q = (101) : (011) =$	$67 \quad 54$	$67 \quad 52\frac{1}{2}$
$s : q = (\bar{1}0\bar{1}) : (011) =$	$76 \quad 55$	$77 \quad 6$
$a : q = (100) : (011) =$	$82 \quad 48$	$82 \quad 55$
$c : \omega = (001) : (122) =$	$54 \quad 4$	$53 \quad 53\frac{1}{2}$
$\omega : o = (122) : (\bar{3}6\bar{2}) =$	$30 \quad 30$	$30 \quad 21$
$o : c = (\bar{3}6\bar{2}) : (00\bar{1}) =$	$95 \quad 46$	$95 \quad 45\frac{1}{2}$
$a : \omega = (100) : (122) =$	$58 \quad 41$	$59 \quad 5$
$\omega : q = (122) : (011) =$	$23 \quad 53$	$23 \quad 50$

No distinct cleavability.

§ 14. *Ethylenediamine-hydrochloride:* $C_2H_4(NH_2)_2, 2HCl$.

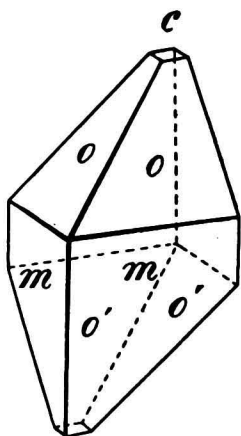


Fig. 12.

From water at room-temperature, this compound is obtained in splendid, colourless, perfectly transparent and strongly refracting crystals. They allow exact measurements to be made. (Fig. 12).

Monoclinic-prismatic.

$$a : b : c = 1,4496 : 1 : 0,6597 ;$$

$$\beta = 88^{\circ} 26\frac{3}{4}'.$$

Forms observed: $m = \{110\}$, very lustrous and yielding excellent images, as also $o = \{111\}$; $c = \{001\}$, small, well measurable; $\omega = \{122\}$, in most cases only rudimentary, with strongly curved faces; often even totally absent. The habitus is that of flat, kite-shaped crystals or of steep needles or long prisms.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$m : m = (110) : (\bar{1}\bar{1}0) =^* 110^\circ 47'$		—
$c : m = (001) : (110) =^* 89 \quad 7$		—
$o : o = (\bar{1}\bar{1}1) : (\bar{1}\bar{1}\bar{1}) =^* 61 \quad 26$		—
$m : o = (\bar{1}\bar{1}0) : (111) = 77 \quad 56$		$77^\circ 58\frac{1}{2}'$
$m : o = (110) : (111) = 50 \quad 40$		$50 \quad 45\frac{1}{2}$
$c : o = (001) : (111) = 38 \quad 12$		$38 \quad 21\frac{1}{2}$
$m : \omega = (\bar{1}\bar{1}0) : (122) = 71 \quad 19$		$71 \quad 1$

It seems that no distinct cleavage is present here.

§ 15. *p*-Nitro-mono-Propyl-Aniline: $C_6H_4(NO_2) \cdot NH(C_3H_7)$.

From alcohol this compound is deposited in the form of elongated, deep yellow, flat prisms with oblique terminal faces. They are highly lustrous, have a lilac surface-hue and yield multiple images. (Fig. 13).

Triclinic-pinacoidal.

$$a : b : c = 0,6135 : 1 : 0,2992;$$

$$A = 59^\circ 38'; \alpha = 64^\circ 42'.$$

$$B = 125 \quad 21; \beta = 121^\circ 17\frac{1}{2}'.$$

$$C = 72 \quad 38; \gamma = 89^\circ 30'.$$

Forms observed: $b = \{010\}$, predominant and, like $a = \{100\}$ and $c = \{001\}$, all yielding very sharp reflexes; $m = \{230\}$, $p = \{130\}$ and $t = \{2\bar{3}0\}$, all about equally broad and well reflecting; $q = \{011\}$, distinctly developed, lustrous; $s = \{0\bar{1}1\}$, small and faintly reflecting. The habit is elongated in the direction of the c -axis, with flattening parallel $\{010\}$.

Angular values : *Observed :* *Calculated :*

$a : c = (100) : (001) =^* 54^\circ 39'$		—
$a : b = (100) : (010) =^* 107 \quad 22$		—
$b : c = (010) : (001) =^* 120 \quad 22$		—
$c : q = (001) : (011) =^* 14 \quad 13$		—
$p : b = (130) : (010) =^* 33 \quad 31$		—
$b : t = (010) : (2\bar{3}0) = 39 \quad 22$		$39^\circ 15'$
$a : t = (100) : (2\bar{3}0) = 33 \quad 16$		$33 \quad 23$
$a : m = (100) : (230) = 48 \quad 1$		$48 \quad 17$
$m : p = (230) : (130) = 25 \quad 40$		$25 \quad 34$
$b : q = (010) : (011) = 106 \quad 9$		$106 \quad 9$
$c : s = (001) : (0\bar{1}1) = 11 \quad 3$		$11 \quad 3\frac{1}{2}$
$s : b = (0\bar{1}1) : (010) = 48 \quad 35$		$48 \quad 34\frac{1}{2}$
$c : m = (001) : (230) = 82 \quad 42$		$82 \quad 54$

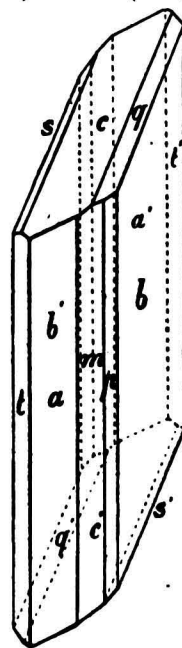


Fig. 13.

On $\{010\}$ oblique extinction; the optical principal section makes, on

this face, an angle of about 12° with the direction of the c -axis. Strongly dichroitic: orange-yellow-pale yellow.

§ 16. *Methylether of Benzoyl-cyano-acetic Acid*: $(CN) \cdot CH(CO \cdot C_6H_5) \cdot CO \cdot OCH_3$; Mpt: $75^\circ, 2$ C.

The compound was prepared from *benzoylchloride* and the *sodium-salt of methyl-cyano-acetate* at a low temperature.¹⁾

From absolute alcohol the substance crystallizes at 60° C in beautiful, pale yellow, very lustrous prisms, which are often flattened parallel to two opposite planes of m .

Rhombic-bipyramidal.

$$a : b : c = 1,1433 : 1 : 0,7052.$$

Forms observed: $m = \{110\}$, predominant and yielding excellent reflections; $q = \{011\}$, large and very lustrous; $o = \{111\}$, smaller than

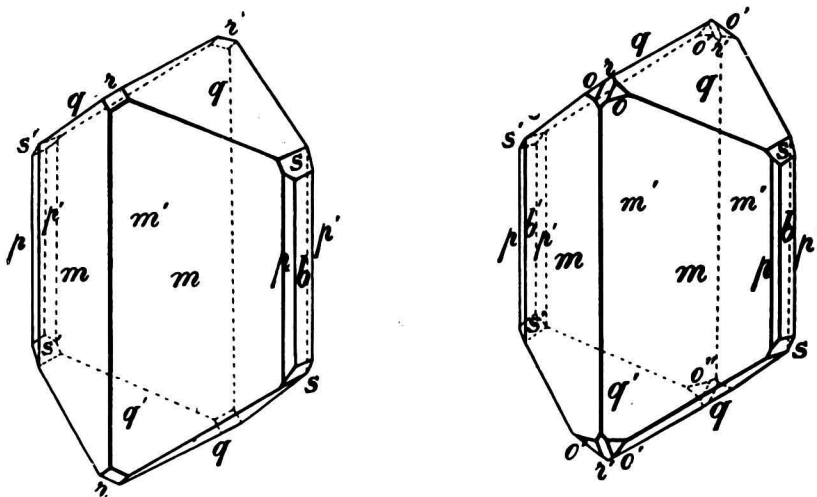


Fig. 14.

q , often apparently in two bisphenoids: the corrosion-figures on m and o prove, however, beyond doubt that the symmetry is really bipyramidal. Further: $b = \{010\}$, very narrow, often hardly visible and yielding only weak images; $p = \{120\}$, well developed and lustrous, often absent; $r = \{101\}$, small and dull; $s = \{021\}$, also subordinate, but giving good reflections. The habit is short-prismatic parallel to the c -axis. A number of combinations were observed, dependent on circumstances during the crystallisation: e.g.: $\{110\}$, $\{011\}$ and $\{111\}$; $\{110\}$, $\{011\}$, $\{111\}$, $\{021\}$ and $\{010\}$; $\{110\}$ and $\{111\}$; $\{110\}$ $\{111\}$ and $\{011\}$; $\{110\}$, $\{111\}$, $\{011\}$, $\{021\}$, $\{010\}$; $\{101\}$ and $\{120\}$; etc. Compare Fig. 14.

¹⁾ See also: L. BARTHE, *Compt. rend. Paris*, **106**, 1416, (1888); A. HALLER, *ibid.*, **101**, 1270, (1883).

Angular values : Observed : Calculated :

$o : o = (111) : (\bar{1}\bar{1}1) =$	$86^\circ 16'$	—
$m : q = (110) : (011) =$	$64 17\frac{1}{2}$	—
$m : m = (110) : (1\bar{1}0) =$	$97 35\frac{1}{2}$	$97^\circ 39'$
$q : q = (011) : (0\bar{1}1) =$	$70 18$	$70 23$
$m : o = (1\bar{1}0) : (111) =$	$46 52$	$46 52$
$o : o = (111) : (1\bar{1}1) =$	$61 54$	$61 57$
$o : r = (111) : (101) =$	$30 57$	$30 58\frac{1}{2}$
$b : p = (010) : (120) =$	$23 44$	$23 37\frac{1}{2}$
$p : m = (120) : (110) =$	$17 32$	$17 33$
$m : r = (110) : (101) =$	$69 39\frac{1}{2}$	$69 47$
$r : q = (101) : (011) =$	$45 57\frac{1}{2}$	$45 55\frac{1}{2}$
$b : s = (010) : (021) =$	$35 29$	$35 20$
$s : q = (021) : (011) =$	$19 22$	$19 28\frac{1}{2}$

No distinct cleavability was stated.

§ 17. *Methyl-ether of m-Nitro-benzoyl-cyano-acetic Acid*: $C_6H_4(NO_2)CO.CH(CN).CO.OCH_3$.

The substance was prepared in an analogous way as the former, *m-Nitrobenzoylchloride* being used in this case.

From a *hot* solution in ether or from a mixture of ether and acetone, small, almost colourless, very lustrous crystals were obtained.

Monoclinic-prismatic.

$$a : b : c = 0,7958 : 1 : 0,8067 ;$$

$$\beta = 82^\circ 9'.$$

Forms observed: $a = \{100\}$ and $m = \{110\}$, well developed and highly lustrous; $c = \{001\}$, also large, but much duller; $r = \{905\}$ and $o = \{111\}$, distinct, but yielding mostly only faint reflections. The angles in the zone of the b -axis oscillate within rather wide limits with separate

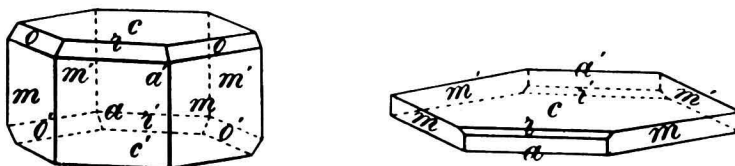


Fig. 15

individuals. The habit is short-prismatic parallel the c -axis or tabular parallel $\{001\}$. See Fig. 15.

<i>Angular values :</i>	<i>Observed :</i>	<i>Calculated :</i>
$a : m = (100) : (110) =^* 51^\circ 45'$		—
$m : o = (110) : (111) =^* 32 49$		—
$m : c = (110) : (001) =^* 85 9$		—
$m : m = (110) : (\bar{1}\bar{1}0) = 76 30$		$76^\circ 30'$
$a : r = (100) : (905) = 26 53\frac{1}{2}$		26 48
$r : c = (905) : (001) = 55 10\frac{1}{2}$		55 21
$a : c = (100) : (101) = 82 4$		82 9
$c : o = (001) : (111) = 52 20$		52 20

Distinctly cleavable parallel to $\{001\}$. The corrosion-figures obtained on $\{100\}$ with Cassia-oil, were in full agreement with the symmetry mentioned.

On a and c normal extinction. The plane of the optical axes is $\{010\}$; the dispersion is inclined and extraordinarily strong, with: $\rho > \nu$.

The apparent axial angle (in Cassia-oil) is about $47\frac{1}{2}^\circ$; one of the axes is seen almost perpendicular to $\{001\}$, the first bisectrix almost halving the angle of the edges of (100) and (001) with (010) .

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