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Systematic multiple diffraction in equi-inclination Weissenberg geometry.

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Multiple Bragg scattering has been recognized as a source of spurious X-ray diffraction effects from crystalline solids since its discovery by Renninger (1937). The necessary condition for the effect is the simultaneous intersection of two reciprocal lattice points hkl and HKL with the sphere of reflection. The diffracted beam from one reflection, say HKL, may then be rescattered into the direction of the diffracted beam from the other reflection, hkl, by a set of planes $h'k'l'$, where:

$$h'=h-H,\ k'=k-K,\ \text{and}\ l'=l-L\ . \qquad (1)$$

The effect has been observed for cases in which the reflection hkl normally has zero intensity due to a space group extinction (Hirshfeld, 1955) or to a special extinction (Renninger, 1937), but it should potentially affect the intensity of any reflections for which the multiple scattering condition is satisfied. Experimental data up to now have shown that intensities, not amplitudes, of reflections hkl and $h'k'l'$ are additive in the resultant beam (Fankuchen & Williamson, 1956; Hirshfeld, 1955).

Multiple diffraction is normally considered to be the result of accidental coincidence. The purpose of this note is to point out that the condition for multiple diffraction is systematically satisfied in upper layer equi-inclination Weissenberg geometry if the crystal rotation axis is parallel to a principal reciprocal lattice vector. If, for example, $\mathbf{c}^*$ is parallel to the rotation axis, the reciprocal lattice point $00l$ is always on the Ewald sphere. Moreover, if the points hkl and $00l$ are on the sphere, then so is the point $hk0$. The condition for multiple scattering is thus satisfied for all three reflections in pairs.

Since

$$\mathbf{s}_2-\mathbf{s}_0=\mathbf{h}_2(00l) \qquad (2a)$$

and

$$\mathbf{s}_3-\mathbf{s}_0=\mathbf{h}_3(hk0)\ , \qquad (2b)$$

the vector equations for all diffracted beams in the direction $\mathbf{s}_1$ are

$$\mathbf{s}_1-\mathbf{s}_0=\mathbf{h}_1(hkl)\ , \qquad (3a)$$

$$\mathbf{s}_1-\mathbf{s}_2=\mathbf{h}_1(hkl)-\mathbf{h}_2(00l)=\mathbf{h}_3(hk0)\ , \qquad (3b)$$

and

$$\mathbf{s}_1-\mathbf{s}_3=\mathbf{h}_1(hkl)-\mathbf{h}_3(hk0)=\mathbf{h}_2(00l)\ . \qquad (3c)$$

The measured intensity in the direction $\mathbf{s}_1$ will thus depend on three intensities, one of which depends on $F^2(hkl)$ while the other two depend on $F^2(00l).F^2(hk0)$.

The most serious changes in the intensity at $\mathbf{s}_1$ will obviously occur when $F^2(hk0)$ and $F^2(00l)$ are both large. They should be observed most easily if $F^2(hkl)$ is relatively small. Non-zero intensities for reflections excluded by a space group symmetry element may be observed if that element makes an angle other than 0° or 90° with the rotation axis.

The possibility of systematic multiple diffraction in equi-inclination Weissenberg geometry is implicit in the results of Fankuchen & Williamson (1956). While that experiment was primarily directed to a study of variations in the intensity of the reflection on the rotation axis ($00l$), the effect described in this note constitutes an obvious corollary.

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Zur Kristallstruktur des Natriumhypochlorits, $NaClO\,.\,5\,H_2O$.

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Das für die Untersuchungen verwendete Natriumhypochlorit wurde in Anlehnung an Gonzalez Barredo (1941) (dort auch weitere Literaturhinweise) durch Einleiten von etwas weniger als der theoretischen Menge Chlor in 50%ige Natronlauge zwischen +1 und +3 °C. erhalten. Das entstandene Natriumchlorid wurde bei etwa +10 °C. auf einer Glasfilternutsche abgesaugt, ohne dass auch nur die geringsten Anzeichen einer Zersetzung der Natriumhypochloritlösung auftraten. In der mit Eis-Kochsalzmischung gekühlten Vorlage kristallisierte das Natriumhypochlorit als grünlichgelbe Kristallmasse aus. Die Substanz wurde auf einem kleinen Glasfiltertiegel abgesaugt, zum Umkristallisieren in ein Reagenzglas gebracht und unter Zusatz von wenig Wasser auf etwa 25 °C. erwärmt. Beim langsamen Abkühlen der klaren gelben Lösung fielen lange, nach dem Trocknen fast farblose, glasige Nadeln aus. Diese wurden wiederum auf einem Glasfiltertiegel abgesaugt und zur Entfernung anhaftender Mutterlauge in diesem Tiegel zentrifugiert. Die Kristalle waren sehr trocken und zeigten keine Zersetzungserscheinungen, auch nicht nach monatelanger Aufbewahrung bei −18 °C.

Die Analyse ergab einwandfrei, dass es sich um ein Pentahydrat $NaClO\,.\,5\,H_2O$ (Fp = 25,5 °C.) handelt, was mit den Ergebnissen von Applebey (1919) (Fp = 24 °C.) bzw. Sanfourche & Gardent (1924) (Fp = 24,5 °C.) übereinstimmt.

Zu den röntgenographischen Untersuchungen wurden die Kristalle wegen ihrer grossen Zerfliesslichkeit und Luftempfindlichkeit in ein Markröhrchen gebracht und während der Aufnahme durch einen kalten Luftstrom gekühlt.

Das $NaClO.5H_2O$ kristallisiert rhombisch. Die Gitterkonstanten wurden zu $a = 8{,}08$, $b = 16{,}06$ und $c = 5{,}33$ Å bestimmt. (Fehér & Talpay (1944): $a = 7{,}91$, $b = 15{,}84$, $c = 5{,}28$ Å). Die Elementarzelle ist mit 4 Molekeln $NaClO.5H_2O$ besetzt. Aus diesen Werten errechnet sich die Dichte zu 1,574 g.cm.$^{-3}$. Aus den Auslöschungen folgen die Raumgruppen C_{2v}^1–$Pmm2$, D_{2h}^1–$Pmmm$ und D_2^1–$P222$.

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Crystal data(I) for some androstanes.*

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The natural androgenic hormones, a series of C_{19} steroids which are categorized by the lack of a C-17 side chain and by the presence of C-3 and C-17 oxygen substituents, are responsible for the development of primary and secondary male sex characteristics. Although the mechanism of action is not understood, the androgens (both natural and synthetic) are often used in the chemotherapeutic treatment of breast cancer. The response of the mammary tumor is proportional to the androgenic activity, some androgens being more potent than others. In addition to their use in substitution therapy in cases of androgen deficiency, such as hypogonadism, 'androgens' which have been modified to the point where their androgenic activity no longer exists are sometimes used exclusively for their anabolic function as illustrated by their ability to promote protein synthesis.

The crystal data (Table 1) for a series of androstanes, androgens, which are biosynthetic derivatives of testosterone, have been determined on a General Electric XRD-5 goniostat-equipped X-Ray diffraction unit using Cu $K\alpha$ radiation. Space groups have been established on the basis of systematic absences and optical activity. It is interesting to note that the isomers 2 and 3, 6 and 7, 6 and 8 are not isomorphous, whereas the isomers 7 and 8 have closely similar lattice constants and are possibly isomorphous. An additional point of interest is that the projection down the b-axis of compounds 1 and 5 has the same size and shape. As can be seen from the discrepancies between some of the calculated and measured densities, solvent of crystallization is present in some of the crystals.

Table 1. *Crystal data* (I) *for some androstanes*

	1	2	3	4	5	6	7	8	9
Formula	$C_{19}H_{30}O$	$C_{19}H_{30}O_2$	$C_{19}H_{30}O_2$	$C_{21}H_{32}O_3$	$C_{21}H_{32}O_3$	$C_{19}H_{32}O_2$	$C_{19}H_{32}O_2$	$C_{19}H_{32}O_2$	$C_{19}H_{28}O_3$
Molecular wt.	274·43	290·43	290·43	332·47	332·47	292·45	292·45	292·45	288·41
(g.cm.$^{-3}$, meas.)	1·125	1·123	1·150	1·181	1·193	1·141	1·189	1·159	1·150
(g.cm.$^{-3}$, calc.)	1·094	1·163	1·143	1·186	1·167	1·079	1·156	1·148	1·149
Z (calc.)	4·114	1·930	2·011	1·992	4·083	4·228	2·057	2·019	4·002
Space group	$P2_1$	$P2_1$	$P2_1$	$P2_1$	$P2_1$	$P2_12_12_1$	$P2_1$	$P2_1$	$P2_12_12_1$
a (Å)*	7·038	11·779	6·556	11·949	7·058	12·149	12·352	12·352	8·374
b (Å)*	41·807	7·912	21·573	7·299	48·170	23·015	7·189	7·148	25·106
c (Å)*	6·337	9·550	6·305	10·679	6·182	6·435	10·364	10·967	7·927
β	116·68°	111·34°	109·01°	91·26°	116·00°	—	114·10°	119·11°	—
V (Å^3)	1666	829	843	931	1889	1799	840	846	1667
Solvent	Ethanol	Heptane and acetone	Heptane	Ethanol	Ethanol	Dioxane	Unknown	Ethanol	Heptane and acetone

1. 5α–androstan–3–one.
2. 5α- androstan -3α–ol -17–one (androsterone).
3. 5α -androstan -3β–ol -17–one (epiandrosterone, isoandrosterone).
4. 5α–androstan -3α–ol -17–one acetate (androsterone acetate).
5. 5α–androstan–17β-ol- 3–one acetate (5α-dihydro testosterone acetate).
6. 5α–androstan- 3β, 17β–diol.
7. 5α–androstan–3α, 17β-diol.
8. 5β- androstan -3α, 17β–diol (etiocholane- 3α, 17β–diol).
9. 5β–androstan–3, 17 -dione (etiocholane- 3, 17–dione).

* ±0·004 Å.

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