Acta Cryst. (1962). **15**, 1050

Crystallographic data on cortisone alcohol ($\Delta_4$pregnene-17*a*, 21-diol-3, 11, 20-trione).

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(*Received* 24 *April* 1962)

During an investigation of the polymorphism of cortisone acetate we had an opportunity of comparing the powder patterns of both free cortisone (cortisone alcohol) and of cortisone acetate with the interplanar spacings published for these compounds. (Beher, Parsons & Baker, 1955; Parsons, Beher, Baker, 1958). As reported elsewhere (Callow & Kennard, 1961) cortisone acetate occurs in five different polymorphic modifications, four of which are unstable in the presence of water and transform to a stable modification designated as Form I. Powder photographs of all five forms were recorded. The interplanal spacings and relative intensities of Form I were found to agree with the powder data quoted for *free* cortisone, Δ^4pregnene-17*a*, 21-diol-3, 11, 20 trione, (Pattern No. 97) quoted in the X-ray Diffraction Powder Data and Index for the Steroid (Parsons, Beher & Baker, 1958). Our measurements on free cortisone on the other hand agreed with Pattorn No. 98 of the Storoid Index quoted as for cortisone acetate, Δ^4pregnene-17*a*, 21-diol-3, 11, 20-trione-21-acetate. It seemed therefore that in these publications figures for the two compounds were inadvertently transposed and some corrections of the published data were necessary. Dr. Parsons (private communication) has rechecked the powder pattern of both samples and agrees with the findings reported in this note.

Single crystal and indexed powder data are reported below for free cortisone crystallized from methanol. Single-crystal measurements were taken from full rotation and Weissenberg photographs recorded with Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å). The interplanar spacings were derived from photographs taken on a 114·6 mm. Guinier camera and the relative intensities of the lines were estimated photometrically. I should like to thank Prof. P. M. de Wolff for the powder data quoted in this paper.

Cortisone alcohol $C_{21}H_{28}O_5$ crystallized in flat, diamond-shaped plates with diagonal extension. The crystals were orthorhombic with

$$a = 10{\cdot}06,\ b = 23{\cdot}70,\ c = 7{\cdot}79\ \text{Å}.$$

Density, measured by centrifuging in a density gradient column was 1·23 g.ml.$^{-1}$. Density calculated for four molecules in the unit cell was 1·28 g.ml.$^{-1}$. The space group was determined from absent reflexions as $P2_12_12_1$.

References

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Table 1. *Interplanar spacings and relative intensities of cortisone alcohol*

d (Å)	I/I_1	*hkl*	*d* (Å)	I/I_1	*hkl*	*d* (Å)	I/I_1	*hkl*
11·84	< 1	020	3·25	2	042	2·315	1	440, 143
7·65	2	120	3·23	< 1	320	2·307	2	1,10,0, 203
7·39	7	011	3·15	3	251	2·296	3	182, 213
6·51	2	021	3·11	3	071, 260	2·277	1	371, 272, 053
6·15	66	101	3·09	3	142	2·266	2	223
5·96	64	111	3·05	10	311, 212	2·237	< 1	291, 352
5·54	3	031	2·981	9	321, 222	2·215*B*	7	450, 380, 153, 441,
5·46	13	121	2·963	1	080			1,10,1, 233
5·10	3	140	2·885	10	261, 152	2·170	2	061
4·92	100	210	2·871	6	331, 232	2·147	2	451, 362, 243, 381, 282
4·85	31	131	2·841	3	180	2·137	2	192, 2,10,0
4·63	8	220	2·773	1	081, 062	2·123	3	460, 163
4·27	5	141	2·733	2	341, 242	2·107	2	1,11,0
4·22	1	201	2·673	3	181, 162	2·076	3	253, 0,11,1
4·16	4	211	2·643	2	271	2·061	1	073
4·05	3	051	2·583	3	351, 013, 252	2·054	1	303
3·98	9	221	2·553	3	072, 280, 360	2·044	2	432, 313
3·84	6	012, 240	2·527	1	312	2·019	1	470, 173
3·76	10	151	2·516	10	400, 103	1·991	1	442, 263
3·73	7	231	2·494	2	091	1·985	2	1,10,2, 333, 520
3·70	6	022	2·478	1	172	1·945	2	501, 004, 530
3·68	4	160	2·461	2	420, 123	1·940	3	511, 343, 014, 1,12,0
3·63	1	102	2·428	4	191, 332, 361, 281, 262	1·914	1	183, 0,12,1, 104, 480
3·59	3	112	2·397	1	430, 133	1·908	1	273, 114
3·47	1	122	2·380	4	411, 043, 370	1·883	3	353, 1,12,1
3·44	9	241, 250	2·371	< 1	0,10,0	1·858	1	134
3·32	6	161, 310	2·359	< 1	082	1·820	3	144, 363, 193, 490, 283
3·30	5	132	2·334	< 1	342, 290			