A solid-state transformation in 1,5-dichloroanthraquinone. By PO-WEN WANG, Physics Department, Polytechnic Institute of New York, New York, USA

(Received 4 January 1978; accepted 6 November 1978)

Abstract

1,5-Dichloroanthraquinone undergoes a solid-state transformation at 470 K. The two modifications are very similar, except that $a$ is greater than $b$ above the transition temperature and the inverse is true at room temperature.

The author prepared a powder pattern of 1,5-dichloroanthraquinone recently, but was unable to index several reflections satisfactorily on the basis of the unit-cell dimensions given by Bailey (1958).

Repeated recrystallizations of our material from saturated solutions in benzene, chloroform and methanol yielded identical powder patterns, but the indexing difficulty persisted.

Bailey's single-crystal specimen used for the complete structure analysis had been grown from the melt. The possibility that Bailey's specimen represented a high-temperature modification, while ours was the room-temperature form, was then investigated.

A powder pattern of material which had been heated to a few degrees above the melting point and then cooled, was found to differ significantly from our original patterns, but this could be indexed satisfactorily on the basis of Bailey's cell data (Table 1).

A differential scanning calorimeter trace of a specimen purified by recrystallization at room temperature was then prepared. A sharp dip at 525 K signaled the melting of the specimen. A solid-state transformation was indicated by a relatively minor endothermal maximum at 470 K.

Single crystals of the room-temperature modification, grown from solution in the form of long needles, are elongated along the $c$ axis of a monoclinic unit cell. Precession and Weissenberg photographs indicate that the space group is $P2_1/a$ or $P_{21}/a$ (i.e. $h0l$ occurs only with $h$ even) with $a = 13.762$, $b = 10.409$, $c = 3.866$ Å and $\beta = 93^\circ 25'$. There are two molecules per unit cell.

The space group of the high-temperature modification, as reported by Bailey, is $P2_1/a$. The cell dimensions are $a = 11.01$, $b = 13.06$, $c = 3.84$ Å and $\beta = 92^\circ 07'$. Clearly, the two modifications are very similar, the $c$ axis and $\beta$ angle are essentially unaffected by the transition. Details of structural rearrangements which accompany the transformation have not been investigated.

Helpful discussions with Professor B. Post are gratefully acknowledged.

Reference