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THERMAL EXPANSION OF POTASSIUM ACETATE

From m.p. (304° C) to 23° C, potassium acetate is found to have undergone two solid-solid transformations. Initially these were detected by Hazlewood, Rhodes and Übbelohde¹ as discontinuities in its volume expansion. Hatibarua and Parry² investigated the structural basis of the transitions defined as:

Form I: Orthorhombic-Trans. temp. 155° C.—Form II: Monoclinic Trans. temp 75° C—Form III: Monoclinic with superlattice.

The transition I → II is characterised by the change in structural symmetry and termination of the rapid expansion ($\alpha_a \approx 500 \times 10^{-6}$) in the cell constant 'a'. The large thermal expansion may be seen in Fig. 1. The lattice parameters are given in Table I. The structural study revealed that the unit cell of form I consists of ionic double layers of potassium and oxylate ions parallel to (100) firmly bound by ionic forces. Between two such double layers are the acetate ions with their methyl ions in contact and bounded by much weaker van der Waal's forces. Another peculiarity is the presence of a void in the unit cell of form I extending through the whole crystal along [100]. The thermal

behaviour appears to stem from these two peculiarities. It has been observed that the directions of b and c axes in form I have remained unchanged through the transition to form II. This means that the orientation of the ionic double layers remains unchanged in the transition. From space group considerations it has been concluded that the acetate ion is no longer constrained to remain parallel to the a axis in form II. It moves in the plane (010) with respect to double layers parallel to (100). The progressive decrease of a axis from 9.744 Å at 155° C to 9.330 Å at 80° C and the increase in the angle β from 90° to 99.1° in the same range of temperature may be visualised as a shear deformation of the form I along [001] parallel to (100). Thus the structural behaviour is as follows: high temperature form I structure is the statistical average of two positions between which the acetate ions vibrate³

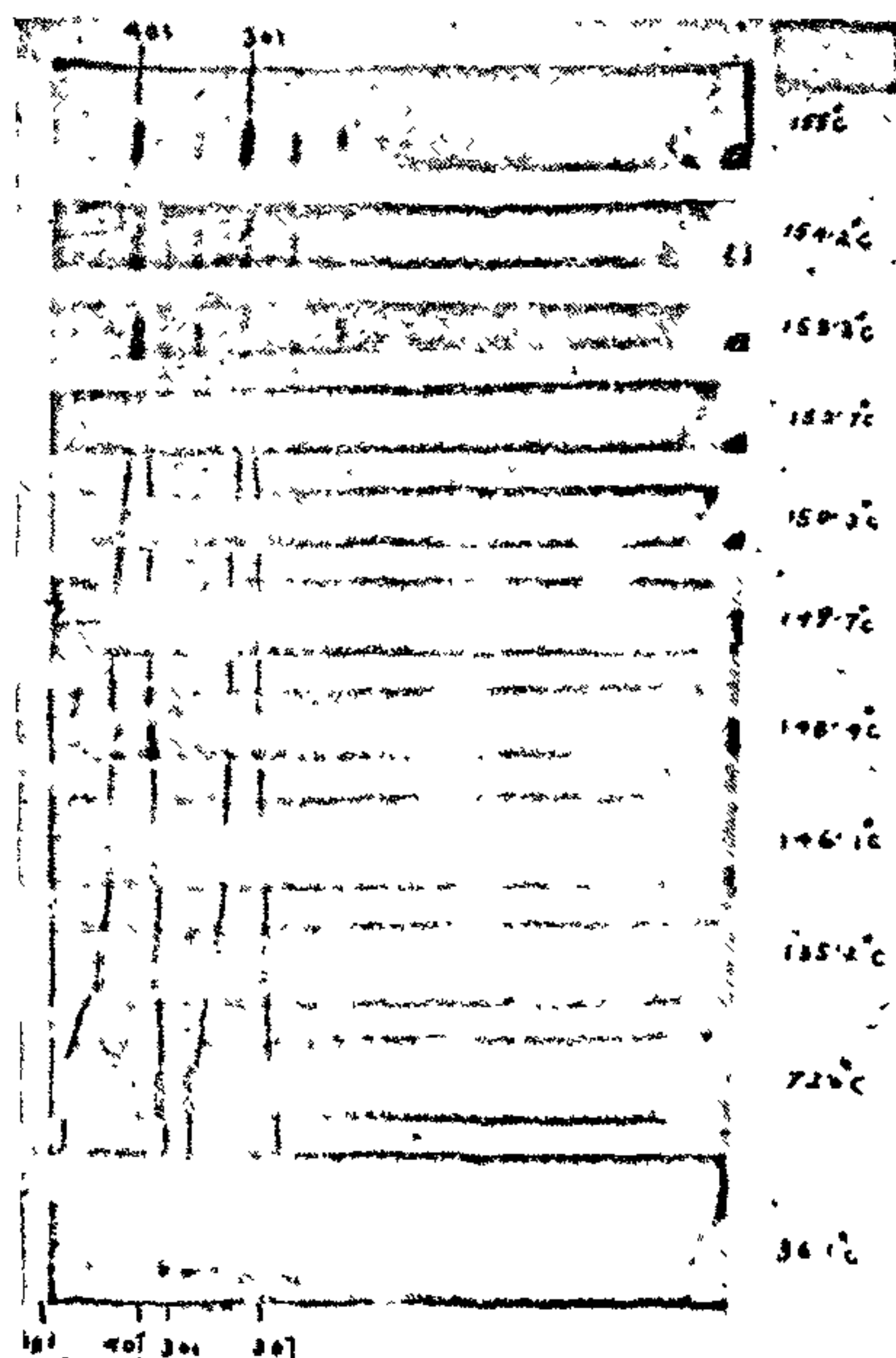


FIG. 1. The movement of pairs of reflexions of the type h0l and h0l towards each other with increasing temperature [in between room temperature and (II → I)].

The switching of the acetate ions between the two alternative sites begins at temperatures 3 to 4 degrees below the actual transition temperature. In form II, the a axis rapidly contracts and b and c expand ($\alpha_b \approx -100$, $\alpha_c \approx 40 \times 10^{-6}$) on cooling. As the crystal is cooled from 155° C, the methyl group of the acetate ion in contact with other two in the unit cell begins

TABLE I

Lattice parameters of potassium acetate (Relative accuracy : $\pm 0.005 \text{ \AA}$; absolute accuracy : 0–5%)

| Temperature °C | a Å | c Å | β° | Temperature °C | b Å |
|----------------|-----------|-------|---------------|----------------|-----------|
| 23.0 | 9.103 × 2 | 4.048 | 100.8 | 25.0 | 5.825 × 4 |
| 36.1 | 9.139 × 2 | 4.040 | 100.5 | 50.0 | 5.808 × 4 |
| 55.2 | 9.234 × 2 | 4.049 | 100.1 | 60.5 | 5.811 × 4 |
| 72.0 | 9.298 × 2 | 4.041 | 99.6 | 67.4 | 5.810 × 4 |
| 73.6 | 9.306 × 2 | 4.025 | 99.3 | 68.3 | 5.820 × 4 |
| 75.7 | 9.317 | 4.024 | 99.2 | 70.6 | 5.822 × 4 |
| 86.0 | 9.365 | 4.022 | 98.2 | 86.0 | 5.816 |
| 95.3 | 9.384 | 4.017 | 98.5 | 126.7 | 5.780 |
| 105.0 | 9.432 | 4.009 | 97.9 | 152.7 | 5.762 |
| 113.3 | 9.478 | 4.005 | 97.5 | 161.4 | 5.758 |
| 124.0 | 9.510 | 4.006 | 96.8 | 198.5 | 5.748 |
| 135.0 | 9.565 | 3.994 | 95.7 | | |
| 147.6 | 9.645 | 3.993 | 94.0 | | |
| 150.2 | 9.698 | 3.991 | 93.1 | | |
| 155.0 | 9.744 | 3.991 | 90.0 | | |
| 160.0 | 9.744 | 3.991 | 90.0 | | |
| 247.0 | 9.853 | 4.021 | 90.0 | | |

(Note : The values of the lattice constants 'a' and 'b' are to be multiplied by 3 and 4 respectively because of the appearance of the superlattice in form III. Thermal expansion has been calculated on the basis of the constants of the original lattice. The absolute accuracy of β is about one degree but the relative accuracy is $\pm 0.1^\circ$.)

to slide over them for closest packing. The penetration of the methyl group in between the other two ions separates them to some extent, so that this model accounts for both the contraction of *a* and expansion of *b*, on cooling in form II. The form III has not been investigated in detail; but from space group consideration and examination of diffraction patterns, it has been speculated to be a modulated structure obtained from form II structure.

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INFLUENCE OF PHENOBARBITONE ON SALICYLATE METABOLISM IN CARBON TETRACHLORIDE TREATED RATS

THE identification of the event that triggers the reaction of the liver to carbon tetrachloride has so far eluded investigators. Damage to mitochondria and endoplasmic reticulum¹⁻⁴, sympathoadrenal discharge and subsequent hepatic hyposia⁵ and peroxidative decomposition of cytoplasmic membrane lipids^{6,7} have been implicated. Racknagel⁸ has reviewed the relative importance of these and other factors. There is evidence to suggest that (carbon tetrachloride) induce inhibition of hepatic activity is due to the accumulation of catecholamines after its administration. Catecholamines exert profound lipolytic activity to the extent that the hepatic cells are unable to cope up with the increased need of lipid metabolism. Beta adrenergic agents are known to prevent liver damage caused by carbon tetrachloride⁹. It is well known that phenobarbitone can enhance hepatic microsomal activity¹⁰. One may therefore consider the possibility of antagonism between the influences of phenobarbitone and carbon tetrachloride on the drug metabolising capacity of liver. In the present report it was considered worthwhile examining whether phenobarbitone can be