LETTERS TO THE EDITOR

PREPARATION AND PRELIMINARY X-RAY STUDIES OF ARGININE ASCORBATE, A CRYSTALLINE COMPLEX BETWEEN AN AMINO ACID AND A VITAMIN

ENZYME-COENZYME interactions, which are mostly noncovalent in character, are of considerable importance in biology. One approach to the study of the atomic details of such interactions is through the x-ray analysis of crystalline complexes between amino acids and short peptides on the one hand, and coenzymes and vitamins on the other. Here we report the preparation and preliminary x-ray analysis of such a complex between L-agrinine and L-ascorbic acid (Vitamin C). Ascorbic acid is also known to function as a coenzyme in some enzymatic reactions. It may be mentioned that the work reported in this note constitutes the first attempt at the x-ray analysis of a crystalline complex between an amino acid and a vitamin or a coenzyme.

The crystals of L-arginine L-ascorbate were prepared by the slow evaporation of an aqueous solution of the components in molar proportions. In order to prevent the oxidation of ascorbic acid, the crystallization experiments were conducted inside a desiccator in nitrogen atmosphere. An alkaline solution of pyrogallic acid was placed in the desiccator to remove the residual oxygen. The composition of the crystals was confirmed by comparing the ultraviolet absorption spectrum of the crystals with those of the components.

The space group and the unit cell dimensions of the crystals were determined from oscillation and Weissenberg photographs taken about crystallographic axes using CuKα radiation. The density of the sample was measured by flotation in a mixture of benzene and carbon tetrachloride. These data are given below.

Space group $P2_1$

$a = 5{\cdot}060 \pm 0{\cdot}008$, $b = 9{\cdot}977 \pm 0{\cdot}009$,
$c = 15{\cdot}330 \pm 0{\cdot}013$ Å,
$\beta = 97{\cdot}5 \pm 0{\cdot}2°$, $D_m = 1{\cdot}509 \pm 0{\cdot}008$,
$D_c = 1{\cdot}516$ gm/cc, $Z = 2$.

The complete structure determination of the complex is in progress.

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DETERMINATION OF PHENANTHRIDINE IN NON-AQUEOUS MEDIA

PHENANTHRIDINE assumes importance as its salts are found to be remarkably active against trypanosome infections[1]. Very few methods are available for the determination of the base. In the present work an attempt has been made to determine phenanthridine in ethyl methyl ketone-acetic acid medium using chlorosulphonic acid by employing visual, photometric and potentiometric techniques.

Chlorosulphonic acid was purified by fractional distillation. Ethyl methyl ketone and acetic acid were purified by standard methods[2,3], Phenanthridine was directly used after checking its melting point (108° C).

Approximately 1 M chlorosulphonic acid in acetic acid was diluted with ethyl methyl ketone to get the required concentrations. The solution was standardised by titration with anhydrous sodium acetate dissolved in the mixed solvent both potentiometrically and by visual titration.

In visual titrations 25 ml. of 0·02 M phenanthridine solution was tritrated with 0·05 M chlorosulphonic acid using methyl orange indicator. The indicator produced light yellow colour in base solutions and bright pink in acid solutions.

In photometric titrations, 25 ml. of 0·0001 M phenanthridine solution was tritrated against 0·01 M chlorosulphonic acid in presence of methyl red at 490 mμ.

In potentiometric titrations 25 ml. of 0·01 M base solution was titrated with 0·05 M chlorosulphonic acid. Glass electrode functioned as indicator electrode and the calomel electrode acted as reference electrode.

The experimental results obtained with the aid of the three different techniques are given in Table I.