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The crystal structures of some polymethylenediammonium adipates. II. Tetramethylenediammonium adipate.

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This investigation was undertaken to determine the molecular configuration of tetramethylenediamine, of which stereochemical data published seem to be rather few. Furthermore, this investigation may be interesting because the substance belongs to a series of polymethylenediammonium adipates studied by Hirokawa *et al.* (1954).

Crystallographic data of tetramethylenediammonium adipate $C_4H_8(NH_3)_2C_4H_8(CO_2)_2$ (4–6 nylon salt) are:

$$a = 15{\cdot}36,\ b = 5{\cdot}97,\ c = 14{\cdot}93\ \text{Å},\ \beta = 110{\cdot}5°,\ Z = 4$$

and the space group is $A2/a$, in some revisions as compared with those already given (Hirokawa *et al.*, 1954).

Using the three-dimensional technique, the crystal structure was determined, and at this stage, the error index is 16·8% by including the contributions of hydrogen atoms; further refinements are now being carried on. A composite drawing of the electron-density function with sections perpendicular to the *b*-axis is shown in Fig. 1, where some of the hydrogens are clearly shown.

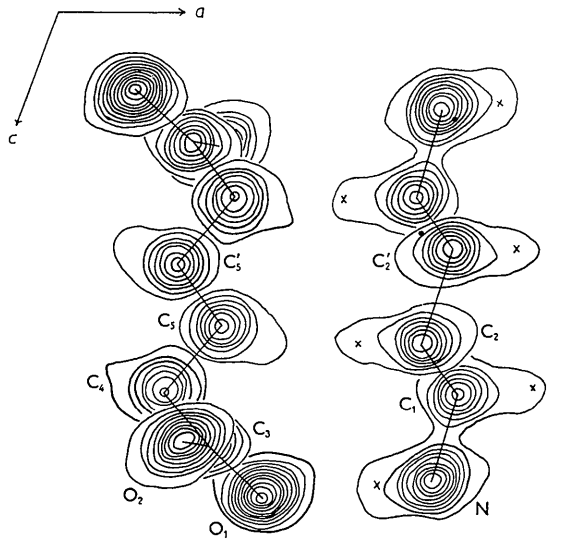

Fig. 1. A composite drawing of the electron-density function with sections perpendicular to the *b*-axis. The hydrogen atoms lying on the sections used in the calculation are also shown by crosses. Contours are drawn at arbitrary equal intervals.

The adipate ion possesses a center of symmetry like adipate ions in several other crystals; however, an imminent difference is found between the present result and those of other compounds. In the present case, the angle between the plane of the carboxyl group and that of the carbon zigzag chain is about 66°, which is larger than the values found in other compounds; for example, 6° in adipic acid (Morrison & Robertson, 1949), and 19° in hexamethylenediammonium adipate or 6–6 nylon salt (Hirokawa *et al.*, 1954). The situation may be due to different ways of the arrangements of the ions in the structures. The values of the bond lengths and angles in the adipate ion are quite reasonable.

The tetramethylenediammonium ion has a 2-fold axis at the middle point of the ion. The bond lengths and angles are also reasonable in this ion. The ion consists of two planar skeletal groups, each containing one half of the ion and the adjacent carbon atom (C_2'–C_2–C_1–N). The two planes thus formed make an angle of 72°; the tetramethylenediammonium ion is therefore found to possess a *gauche* form. This is characteristic if it is compared to the molecular configuration of the hexamethylenediammonium ion in the case of 6–6 nylon salt, where the ion possesses a *trans* form.

Each nitrogen makes three N–H · · · O hydrogen bonds to three different adipate ions. The situation is rather similar to the case of 6–6 nylon salt.

A detailed discussion will be published elsewhere.

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The thermal expansion of silicon.

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The results of the I.U.Cr. precision lattice parameter project, in which the lattice parameters of a series of substances were determined at ambient temperatures, have been published (Parrish, 1960). It may be of interest to note that using two 19 cm. high temperature powder cameras we have determined the lattice parameter of the silicon standard supplied for the project between 20 and 720 °C.

The camera used for the bulk of the work was a Unicam S150 (Goldschmidt type) slightly modified in so far as the 0·020″ platinum/platinum 13% rhodium thermocouple supplied was replaced by a 0·010″ ring type