of the NH_4^+ ion and 1·81 Å for the radius of the Cl^- ion, an N–Cl distance of at least 3·29 Å might have been expected. The much shorter observed distance, therefore, strongly suggests that Cl′ is hydrogen-bonded to N(1), (N–H · · · Cl). This conclusion receives support from the values of the pertinent bond angles:

Cl′–N(1)–CH_3', 110·0°; Cl′–N(1)–C(2), 106·6°;
Cl′–N(1)–C(6), 107·6°; CH_3'–N(1)–C(2), 110·4°;
CH_3'–N(1)–C(6), 111·4°; C(2)–N(1)–C(6), 110·7°, (mean, 109·4°),

all of which are very close to the tetrahedral value of 109° 28′. As Dr Marsh also has pointed out, the direction of this hydrogen bond is almost parallel to the *z* axis and, thus, might be responsible for the large apparent temperature-factor anisotropy of the chlorine ion.

References

KARTHA, G., AHMED, F. R. & BARNES, W. H. (1960). *Acta Cryst.* **13**, 525.
MARSH, R. E. (1960). Private communication.

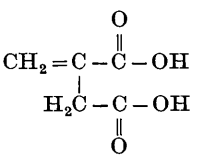
Acta Cryst. (1961). **14**, 94

Unit cell and space group of itaconic acid.

By AMNON GOLDSTEIN, GERALD MANDEL and DANIEL PINDZOLA,* *Polytechnic Institute of Brooklyn, Brooklyn* 1, *N.Y., U.S.A.*

(*Received* 8 *June* 1959 *and in revised form* 24 *May* 1960)

Itaconic acid (methylene succinic acid):



is of importance as an intermediate in the synthesis of polymers, dyestuffs, and pharmaceuticals.

Material prepared by Chas. Pfizer & Co. was recrystallized twice from water. Large single crystals in the form of octahedra up to 2 cm. in diameter were readily grown by slow evaporation of an aqueous solution of the acid. Analysis of these crystals by the method of neutralization equivalents indicated that they contained 99·5% of the expected acid equivalents. No hydration of the crystals was detected. The crystals showed pronounced cleavage perpendicular to *a*.

The crystals were investigated with precession and Weissenberg cameras. The unit cell is orthorhombic. Systematic absences are consistent with either of two possible space groups: C_{2v}^5–*Pca*2_1 or D_{2h}^{11}–*Pcam*.

Unit-cell dimensions were computed from powder data obtained using a recording diffractometer. The dimensions are:

$$a = 5{\cdot}45 \pm 0{\cdot}01,\ b = 18{\cdot}45 \pm 0{\cdot}03,\ c = 11{\cdot}45 \pm 0{\cdot}02\ \text{Å}.$$

The density calculated on the assumption that there are eight molecules per unit cell is 1·50 g.cm.$^{-3}$. The density measured by floatation in a mixture of CCl_4 and $CHCl_3$ is 1·49 ± 0·01 g.cm.$^{-3}$. It should be noted that the density generally quoted in the literature,† namely 1·63 g.cm.$^{-3}$, is incorrect.

Efforts were made to resolve the space group ambiguity by means of piezoelectric tests. A positive piezoelectric test would have indicated that the unit cell is non-centrosymmetric. This would have ruled out *Pcam* unambiguously. However, no piezoelectric effect was detected in a series of experiments using several single crystals of itaconic acid of varying sizes.‡ Therefore, we are left with a twofold ambiguity with regard to the space group.

* Submitted in partial fulfillment of the requirements for the course in X-ray crystallography at The Polytechnic Institute of Brooklyn.

† Beilstein's Handbuch der organischen Chemie. J. Springer, Berlin 1918.

‡ Piezoelectric tests were made by Dr F. Holtzberg of the IBM Watson Scientific Labs., New York City.