

Table 2. Results of three gas chromatographic analyses (GLC) of a mixture of four organic acids compared with gravimetric determination.

Acid	Gravimetric determination Weight per cent (as methyl esters)	GLC determination			
		I	II	III	Mean
Pyruvic	23.9	23.7	24.0	24.9	24.2
Lactic	28.9	28.3	29.6	28.5	28.8
Acetoacetic	19.8	20.9	19.7	19.6	20.1
β -Hydroxybutyric	27.4	27.1	26.7	27.0	26.9

organic acids from the urine of a diabetic patient with a slight ketoacidosis is shown in Fig. 3. In this chromatogram, however, several peaks still remain to be identified. The same technique has also been successfully applied for the determination of citrate in plasma from patients under various clinical conditions.

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The Crystal Structure of N-(α -glutarimido)-4-bromophthalimide

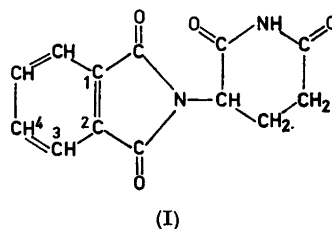
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The glutarimido derivative of phthalimide (I) has various striking pharmacological activities. It would appear to be of impor-

tance to know its molecular structure, and we have therefore started X-ray crystallographic investigations of this and related compounds.

A sample of (I) was kindly supplied by Norsk Astra. It consisted of small well-developed crystals elongated along the *b* axis. The X-ray diagrams showed them to be monoclinic, with $a = 8.28 \text{ \AA}$,



$b = 10.07 \text{ \AA}$, $c = 15.37 \text{ \AA}$, and $\beta = 109^\circ$ (all $\pm 1\%$). By flotation the density was found to be about 1.42 g cm^{-3} and there are four (calc. 4.01) molecules in the unit cell. The space group is $P2_1/c$.

Although the space group is favourable, the crystallographic axes are all rather long and it might be difficult to determine the crystal structure. The 4-bromoderivative of (I) was then prepared by Dr. Else Kloster-Jensen of this Institute, and we continued the investigation using this compound, in which the essential structural features of (I) must be expected to be retained. An account of the synthesis will be published separately.

Small single crystals were eventually obtained by sublimation in vacuum, and X-ray Weissenberg photographs taken around the *a* and *b* axes. The crystals were

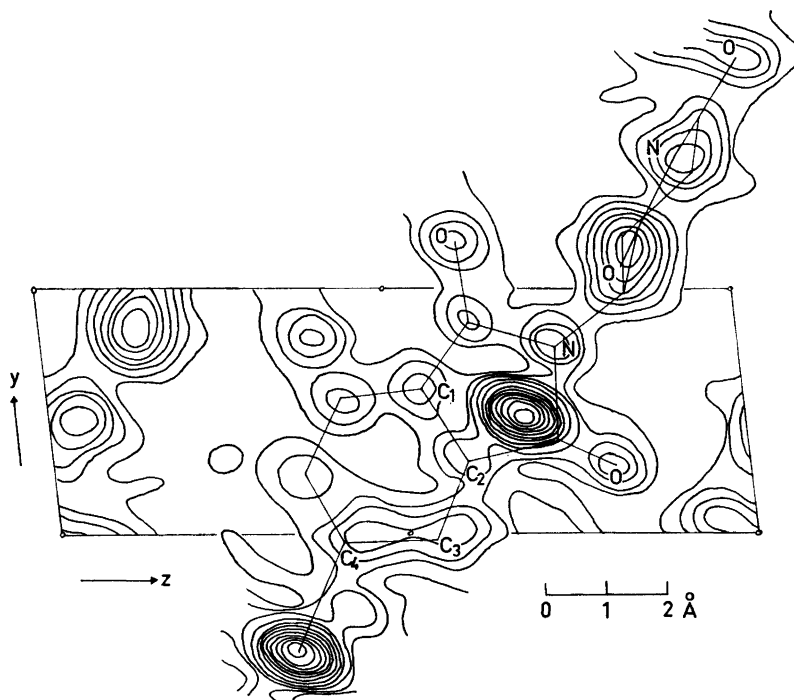


Fig. 1. Electron density projection in direction of a axis.

triclinic, with $a = 7.12$ Å, $b = 7.96$ Å, $c = 11.51$ Å, $\alpha = 96.5^\circ$, $\beta = 97.0^\circ$, and $\gamma = 90^\circ$. The density is 1.75 g cm $^{-3}$ and there are two (calc. 2.01) molecules in the unit cell. The space group was assumed to be $P\bar{1}$ and this was verified by the structure analysis.

The structure was solved by the standard heavy atom procedure in two projections, one of which is shown in Fig. 1. The corresponding values of the reliability index R are 0.16 and 0.18 at the present stage. There is considerable overlap of atoms, and further refinements will be carried out on three-dimensional data. However, the general features of the molecular structure are evident from the electron density projections.

The phthalimide part of the molecule is planar. In the glutarimide residue all non-hydrogen atoms except the β -carbon atom lie approximately in one plane,

which is nearly at right angles to the phthalimide plane and forms an angle of roughly 25° with its twofold axis. The central C–N bond is in "equatorial" position and coplanar with the phthalimide within the limits of error. All bond lengths and angles appear to be normal.

We would like to point out that this molecular structure has a general resemblance in size and shape with certain important biomolecules, especially the nucleosides, a fact which possibly may be related to its biological activities.

A full account of the work will be published later.

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