Dihydroxyphenylalanine (DOPA) is an amino acid found in seedlings, pods and beans. Atomeut, the anti-hypertensive drug is an alpha-methylated analog of DOPA. The crystal structure of the title compound, which is a 3,4-dimethoxy analog of Atomeut, was undertaken to study the conformation of methylated amino acids in the solid state.

The crystal structure of the title compound are triclinic, space group PT, with cell dimensions: a = 11.437(2), b = 20.434(1), c = 95.74(1), α = 96.91(1), γ = 92.8(1)°, V = 1355.5(3), Z = 4 (two molecules per asymmetric unit) and Dcalc. = 1.303 g/cc. The density was measured by flotation in bromoform and benzene at 1.29 g/c.c. The preliminary data was collected on a GEXRD-6 diffractometer and the complete three-dimensional data collected on a CAD-4 diffractometer. A total of 6313 reflections were collected, out of which 4006 were considered significant. The data was processed on a PDP 11/34 computer. The structure was obtained by a multi-solution technique, using the MULTAN program and refined by full-matrix least-squares method.

Both the molecules in the asymmetric unit exist as zwitterions in the solid state. Although both molecules have similar conformations, there are some essential differences. The C-01 is shorter than C-02 by 0.016 and 0.039 Å respectively in molecules A and B. C-02 of molecule B is considerably longer (0.033 Å) than C-02 of molecule A, because O2 of molecule B is involved in several hydrogen bonding interactions in molecule A. The torsion angles δ1 and δ2 are ±47.2°, ±73.9° in molecule A and ±67.1° and ±71.3° in molecule B respectively. Looking along C1-C8, the three substituents α-methyl group, NH1 and COO- are at dihedral angles of ±67.2, ±56.8° at ±71.3, ±75.6° and ±59.2° and ±59.3° in molecules A and B respectively. Atom A is coplanar with the piperidone ring and the ethylenic hydrogen atoms stabilize the conformation of the piperidone ring is evident from the observed planarity of the nitrogen atom lone pair and the piperidyl nitrogen, indicates interaction between the nitrogen atom lone pair and the carbonyl group. The half chair conformation of the piperidone ring is evident from the endocyclic torsion angles. A pair of intramolecular C=O...O hydrogen bonds involving the ethylenic hydrogen atoms stabilize the conformation of the molecule.

03. CRYSTALLOGRAPHY IN BIOCHEMISTRY AND PHARMACOLOGY