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CRYSTAL STRUCTURE OF DIAQUA NITRATOGLYCINECALCIUM(II) NITRATE

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GLYCINE forms complexes with many inorganic salts and acids[1,2]. Some of these complexes have therapeu tic values and all of these are of chemical and bilogical interest[3,4]. The crystal structures of these simple molecules may serve as model systems in understanding the complicated structures of macromolecules. Hence, a systematic study of the complexes of glycine with many inorganic salts and acids was taken up. The crystal structures of the complexes of glycine with $CaCl_2$,[5,6] $CaBr_2$,[7,8] CaI_2,[9,10] $CdCl_2$,[11] $CdBr_2$[11] and H_3PO_4[12] had earlier been elucidated. In the present study the crystal structure determination of diaquanitratoglycinecalcium(II) nitrate was taken up.

Single crystals of the above complex $(NH_2CH_2COOH)\ Ca(NO_3)_2.\ 2H_2O$ were grown from a saturated aqueous solution, containing stoichiometric amounts of glycine and calcium nitrate. The crystal data are as follows: $a = 6.865(5)$, $b = 13.250(10)$, $c = 11.275(6)$ A, $V = 1025.6$ A^3, F.W. $= 275.2$ $D_{mea} = 1.82$ $g.cm^{-3}$, $D_{cal} = 1.78$ $g.cm^{-3}$, $Z = 4$, $\mu(CuK\alpha) = 64$ cm^{-1} and the space group is $P2_12_12_1$. The density was measured by flotation method using a mixture of bromoform and carbon tetrachloride.

The three-dimensional intensity data were collected using an Enraf-Nonius CAD-4 diffractometer, with graphite monochromatised $CuK\alpha$ radiation at IIT, Madras. Absorption, Lorentz and polarisation corrections were applied on these 1165 unique reflections for which intensity data were collected. From a three-dimensional Patterson synthesis, the position of the calcium atom was determined.

Thereafter, successive Fourier and difference Fourier syntheses revealed the rest of the structure. Structure-factor least-squares refinement using the block-diagonal approximation was carried out on an IBM 1130 computer. With anisotropic thermal parameters for all the non-hydrogen atoms, the resi-

TABLE 1.
Fractional atomic coordinates for diaquanitratoglycinecalcium(II) nitrate.

Atom	x	y	z
Ca	0.0645	0.2288	0.0896
C(1)	0.582	0.186	0.147
C(2)	0.585	0.126	0.264
O(1)	0.722	0.218	0.103
O(2)	0.394	0.199	0.102
O(3)	0.470	0.067	0.019
O(4)	0.020	0.049	0.171
O(5)	0.016	0.082	0.054
O(6)	0.400	0.373	0.322
O(7)	0.579	0.460	0.458
O(8)	0.393	0.316	0.494
Ow(1)	0.050	0.405	0.134
Ow(2)	0.133	0.228	0.304
N(1)	0.428	0.065	0.283
N(2)	0.019	0.010	0.069
N(3)	0.466	0.385	0.427

dual index at present is about 0.12. Further refinement is under progress. The present fractional atomic coordinates are presented in table 1. A view of the structure looking down the *a*-axis is shown in figure 1.

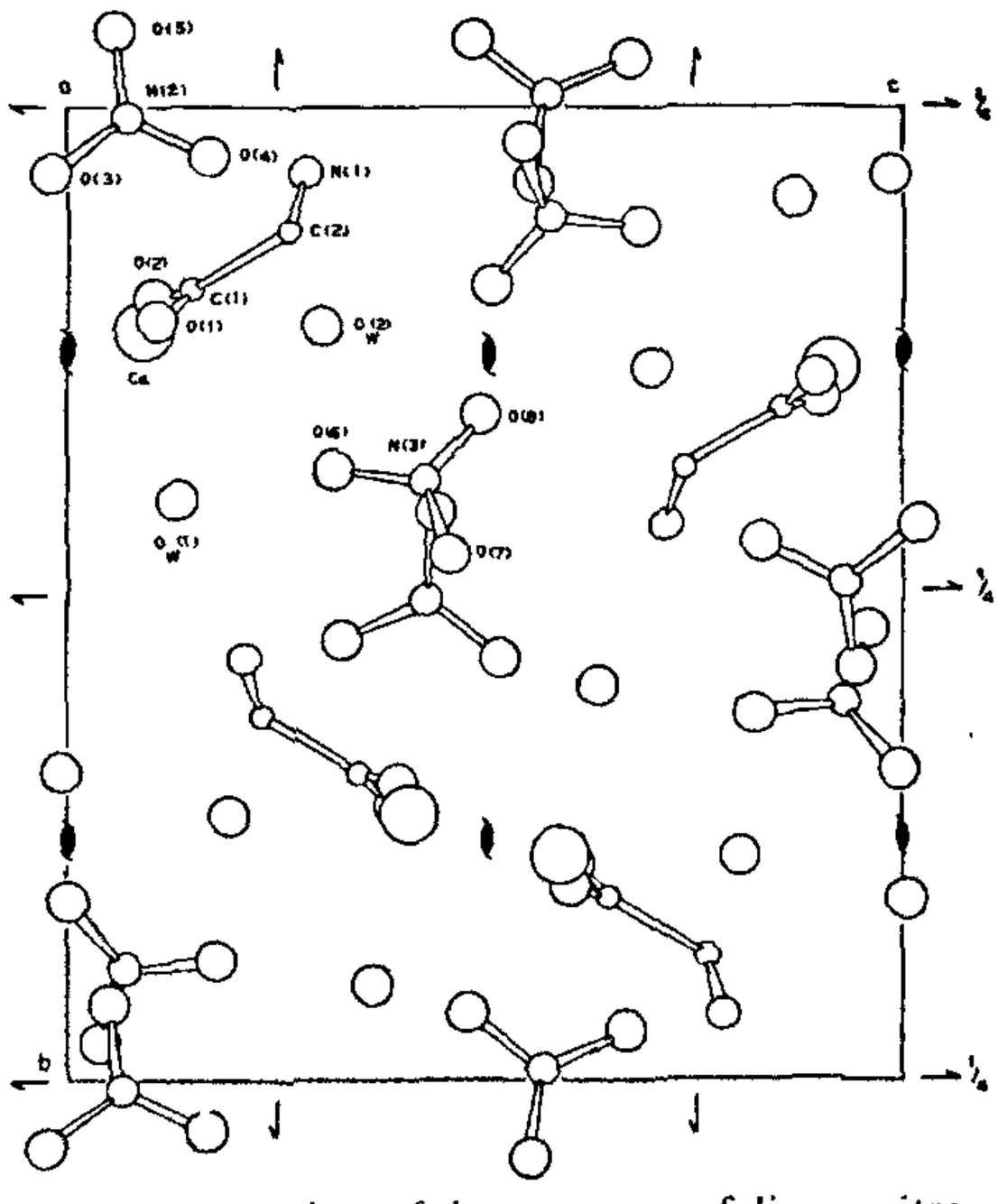

Figure 1. Projection of the structure of diaquanitratoglycinecalcium(II) nitrate down the *a*-axis.

Calcium is coordinated to eight oxygen atoms, two of them belonging to water molecules, another two belonging to a nitrate group and the rest to the carboxyl group of the glycine molecule. The Ca-O distance range from 2.3 to 2.64 Å. Glycine molecule and the nitrate groups have the normal bond distances and bond angles as found in other similar structures[2].

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A NEW ROUTE FOR SYNTHESIS OF 2,4,6-TRIARYLPYRIDINES *VIA* PHOSPHONIUM YLIDES

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A NEW route for the synthesis of 2,4, 6-triarylpyridines is reported. It involves the reaction of phenacylidenetriphenylphosphoranes with α,β -unsaturated ketones with ammonium acetate as cyclization agent.