03.3–12 THE CRYSTAL STRUCTURE OF PHTHALYL SULFACETAMIDE. By T. P. Singh, Urmila Patel and M. Haridas, Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388120, Gujarat, India.

Phthalyl sulfacetamide, $C_{16}H_{14}N_2SO_6$, crystallizes in the monoclinic space group $P2_1/c$ with a = 8.01 (2), b = 13.00 (2), c = 18.04(3) Å, β = 111.6(5)°, V = 1746.59 Å^3, d_m = 1.412 (5), d_c = 1.378 Mg m^{-3}, Z = 4, λ(CuKα) = 1.5418 Å. Three-dimensional X-ray intensity data were collected photographically. The structure was determined by direct methods and difference Fourier techniques. It was refined by block-diagonal least squares procedure to an R value of 0.092 for 1805 observed reflections. The planar benzene rings in the structure are oriented with respect to each other at 62.0°. The coordination around sulfur atom is distorted from the ideal tetrahedral geometry. The dihedral angle of 114.6° around S-N bond is very different from those (60-80°) observed in other sulfonamide structures. The molecule is bent dramatically like a dog tail, thus bringing the two end moieties in the close proximity. The molecules are arranged in the form of layers. The intermolecular hydrogen bonding and the van der Waals forces stabilize the structure.

03.3–13 STRUCTURE OF SULFAMETHOXYPYRIDAZINE. By M. Haridas, Meera Singh and T.P. Singh, Department of Physics, Sardar Patel University Vallabh Vidyanagar 388120, Gujarat, India.

Sulfamethoxypyridazine (Laderkyn) 4 $C_{11}H_{12}N_4O_3S$, is a widely used anti-bacterial drug. It is a long acting drug with the general properties of sulfonamides. Yellow coloured and hexagonal shaped crystals of laderkyn, obtained from its solution in methanol at 4-5° C, belong to monoclinic, space group P_c with a = 8.86(3), b= 11.43(3), c = 27.44(4) Å, β = 100.6(4)°, V=2731.42Å^3, d_m = 1.483(8), d_c= 1.472 Mg m^{-3}, Z=8 and λ(CuKα) = 1.5418 Å. There are four crystallographically independent molecules consisting of 76 non-hydrogen atoms in the asymmetric unit. The structure was determined by a combination of direct methods and difference Fourier syntheses, and refined by block-diagonal structure factor least squares procedure. The final R value for 3224 observed reflections is 0.090.

The molecular dimensions of the four crystallographically independent molecules are not identical. The differences in them are substantially large in certain regions of these molecules. The electron density of sulfur atom belonging to one of these four molecules is disordered and distributed over three distinct sites with the occupancy values of 0.92, 0.05 and 0.03. The above are, presumably, due to steric hindrance caused by packing, the occurrence of dissimilar intermolecular forces among the crystallographically independent molecules and their liquid like behaviour. The angular disposition of bonds around S atoms is distorted from ideal tetrahedral geometry. The benzene and pyrimidine rings are essentially planar and inclined with respect to each other at 88.9, 78.5, 80.2 and 88.5°

03.3–14 THE CRYSTAL STRUCTURE OF VINZOLIDINE By Noel D. Jones, Stanislaw Rolski and John K. Swartzendruber, Lilly Research Laboratories, Eli Lilly and Co., Indianapolis, IN 46285, U.S.A.

Vinzolidine is a semi-synthetic derivative of the antineoplastic agent, vinblastine, an alkaloid from Vinca rosea Linn. The 1,5-napthalenedisulfonic acid salt of vinzolidine crystallizes from absolute methanol as colorless prisms containing three molecules of solvent per molecule of alkaloid. Asymmetric unit weight, 1269 for $(C_{48}H_{58}ClN_5O_9)(C_{10}H_8O_6S_2)\cdot 3CH_3OH$; a = 11.414(6), b = 20.617(7), c = 13.926(5) Å, β = 109.76(4)°; space group, $P2_1$; Z = 2; D_c = 1.366 g cm^{-3}; 3036 observed reflections, CuKα. The structure was solved by both the heavy atom method and by direct methods (SHELXTL) and refined to R = 0.065. The absolute configuration of vinzolidine was determined by anomalous dispersion and shown to be the same as that of vinblastine (Moncrief and Lipscomb, Acta Cryst. 21, 322, 1966). The close non-hydrogen-bonded approach of O5" and N9 (2.90 Å) may explain the unreactivity of N9.