A study of extremely pure dipalmitoyl-phosphatidylcholine (DPDC) by thermal analysis and x-ray scattering has revealed several features not reported previously. The experimental observations were very sensitive to sample purity and experimental procedure. Nevertheless, reproducible results have been obtained. From both single domain and powder X-ray patterns we have deduced the existence of several novel structural features such as a three-dimensional "L6\" phase or phase transitions in the high temperature phase region P6 + Qq.

In an effort to determine the membrane-bound structure of gramicidin A we have grown crystals for X-ray diffraction which contain gramicidin and phosphatidylcholine. Since CD studies suggest that the conformation of gramicidin in crystals prepared without lipids is unlikely to be the same as in membranes, several crystal forms containing both lipids and gramicidin have been prepared. The first, rolled up stacks of sheets with a high lipid/peptide ratio (19:1), exhibits primarily lipid reflections altered by the presence of peptide, as well as a number of reflections due to peptide itself. This form is useful for examining the effects of peptide on lipid molecules. The second form, which has a much lower lipid/peptide ratio (2:1), is a well-ordered single crystal which diffracts strongly to 2.4\AA, and differs from any of the crystals prepared in the absence of lipid. The symmetry of these crystals is orthorhombic with lattice dimensions 26 A x 26 A x 32 A. A third crystal form which contains noncovalent thallium in addition to lipid and protein is currently under investigation. Preliminary spectroscopic evidence suggests the gramicidin conformation in the mother liquor from which these crystals were formed is likely to be of the membrane-bound type. These crystal forms will be useful in defining the membrane-bound ion channel conformation at a resolution heretofore unseen for any membrane proteins. This work is funded by NSF PCM 8020063 and NIH GM27292.

In the flexible activation domain of trypsinogen the disulfide Cys191-Cys220 can be selectively reduced and mercerated. The crystal structures of such modified trypsinogen have been analyzed in its native form and in its complex with P1 and the dipeptide LEV-VAL. The mercury atom, covalently bound between the two sulfur atoms, is crystallographically undetectable in the free form of trypsinogen, whereas well defined in the complexed form. After crystallographic refinement of the complex at 2.0\AA resolution, the R-value was 0.20.

A trypsinogen crystal was crystallographically analyzed and refined at 1.8\AA. The coordinates as well as the thermal parameters were compared with the room temperature data. The overall B-factor is reduced by 4.4\AA. A number of additional ordered water molecules are observed. The order of the activation domain is not increased detectably at 1.8\AA. Residues at the boundaries of the flexible segments, except at the N-terminus, indicate a considerable contribution of temperature independent static disorder. Within the significance level of our measurements it seems that most of the other residues behave like harmonic oscillators.