

CHAPTER VI

CONCLUSION

The solid state characterization of pralidoxime chloride was studied in many techniques such as x-ray powder diffractometry, scanning electron microscopy and differential scanning calorimetry. These techniques were done to confirm the similarities and the differences between freeze dried product and the starting material powder.

DSC was used to detect a change in thermal behavior of the frozen solution. DSC thermogram reveals the exothermic peak at -20°C (data not shown). This exothermic event usually calls supercooling. DSC thermogram shows three endothermic peaks indicating that pralidoxime chloride formed eutectic mixtures with water. These endothermic peaks represent the two eutectic form combine in the frozen solution. The cooling and heating rate variation of thermal analysis has no influence to the low temperature thermal behavior of pralidoxime chloride frozen solution. Frozen thermal behavior with thermal treatment was studied in two conditions, annealing before and after reaching the predetermined freezing temperature. The annealing temperatures were set around eutectic temperature at -12 , -15 and -17°C . The thermal behavior from annealing before the predetermined freezing temperature condition is the same pattern of starting material because the molecules arrange in the same structure as the starting material. Annealing did not affect this condition. At annealing after the predetermined freezing temperature, the thermal behavior should change but the results show that the thermal behavior is not change.

Freeze drying experiment was set into two conditions follow DSC experiment, annealing before and after the predetermined freezing temperature (-40°C). On freeze drying process, the appearances of freeze dried pralidoxime chloride products are different between compact cake and the loosely aggregated cake. Annealing before reaching final freezing temperature caused more crystalline product show by XRPD pattern. DSC thermograms of freeze dried products of the annealing before condition show the broad endothermic melting peak with the onset of approximately 215°C . DSC thermograms of freeze dried products of the annealing after condition show the onset of the very sharp melting endotherm at approximately 225°C that is higher than the melting point of starting material.

Solid state stability of freeze dried pralidoxime chloride was studied between dry and humid conditions. When humid condition was introduced, the product looked moist and collapsed within the first day. X-ray powder diffractogram showed increase in crystallinity as time of the humidity exposure increase. No change was observed when kept in dry atmosphere at elevated temperature.

Low temperature thermal analysis was used to better understand the thermal behavior of freeze dried system. The thermal behavior of frozen solution is useful for manipulation and control of polymorphism or selects the different form of products after the freeze drying process. The pharmaceutical scientist could control the freeze drying condition to gain the desirable products of higher stability and at the same time must fulfill the optimal reconstitution time of drug especially the drug that will be used in emergency situation like pralidoxime chloride.