MISCELLANEOUS
Preparation of Allyl bromide.

The preparation involved two operations, (1) preparation of hydrobromic acid and (2) Allyl bromide.

The weak solution of hydrobromic acid which was collected (in a beaker) in the course of the preparation of bromobenzene by the method of Cohen and Gross (Cohen, Practical Organic Chem. 3rd Ed. p.155) was fractionally distilled and the fraction distilling at 123-126 ° at normal pressure was collected.

To 320 gms. of hydrobromic acid, 103 gms. of allyl alcohol were added in a 2 litre flask. The flask was fitted with a mechanical stirrer, separating funnel and an efficient condenser for downward distillation. Stirring was started and 100 gms. of concentrated $\text{H}_2\text{SO}_4$ were added gradually through the separating funnel to the warm solution. The allyl bromide distilled over in about an hour. The crude allyl bromide thus obtained was washed with sodium carbonate solution, dried over calcium chloride and distilled. The bromide distilled at 69-72 °. A portion of high boiling fraction was also obtained.

**Yield** 130 gms.
Preparation of acetaldehyde:

The ordinary method of preparation of acetaldehyde from acetylene could not be worked upon successfully in this laboratory and after some corrections, it was found possible to get an yield.

The method adopted was as follows:

Acetylene from a cylinder purified by passing through (1) sulphuric acid, (2) copper sulphate solution, (3) a tower packed with bleaching powder and lime and (4) ferric chloride solution with sulphuric acid, was led into a flask containing 300 c.c. of glacial acetic acid and 9.5 gms. of mercuric sulphate in solution, the temperature of which was kept at 30 c. The exit tube from the flask was connected to a ice cold (temp. kept too low) water condenser and to three wash bottles having condensing tube (spiral) in them and containing ether cooled in freezing mixture. The temperature of both the water condenser and that of the bath containing the wash bottles was always kept at 0-5 c. The gas was pressed for about fourteen hours at a moderate rate and water was added at intervals to replace that taken up in the reaction. The flask after the required time, was warmed to 60-70 c to drive all the aldehyde over into the ether. The ethereal layer was dried over sodium sulphate, then decanted and saturated with dry ammonia, when aldehyde ammonia was formed.

The yield obtained was not encouraging.
An amount of the crystals of aldehyde ammonia thus obtained was treated with a little amount of sulphuric acid and distilled carefully by passing ice cold water through the condenser and the receiver kept immersed in a freezing mixture. The fraction boiling at 20-21 °C was acetaldehyde.

Preparation of Butyl Chloride:

An amount of anhydrous zinc chloride (60 gms) was added to conc. HCl (40 gms). The mixture was kept cool to avoid loss of HCl. To this solution n-butyl chloride (19 gms.) was added. The mixture was then refluxed for about 5 hours on a water bath. The upper layer was separated after cooling. It was then placed in a distilling flask with an equal volume of calculated sulphuric acid. The side arm of the flask was closed and it was refluxed gently for about an hour. The butyl chloride was then distilled. The distillate was washed with water, dried over CaCI₂, filtered and redistilled. The fraction distilled at 76-78 °C was collected.

Yield 17 gms.

Calibration of a thermometer.

A furnace for the preparation of butadiene was made in the Engineering College. In order to measure the high temp. required (730-760 °C) in the experiment, a resistance thermometer was calibrated. The calibration
was made by the method of Carey Foster's bridge method. The resistance of the wire was measured at different temperatures. The attached graph was plotted with temp. and resistance and from the graph it was found that the resistance of the wire at 730 c is 1.01 ohm.

Preparation of p-nitroaniline.

An amount of finely ground acetanilide (one lb) was dissolved in conc. sulphuric acid (four lbs) by slowly stirring the contents with a mechanical stirrer, with the possible care that the temp. did not rise above 30 c. Then all was dissolved, the solution was cooled in a freezing mixture to 0 c, and a mixed acid previously cooled to 0, containing strong nitric acid (180 c.c.) and strong sulphuric acid (360 c.c.), gradually added, the temp. not exceeding 3 c. Then all was added, the solution was allowed to stand for three hours, when a sample on pouring into water and boiling with caustic soda gave no colour of aniline. The reaction mixture was poured on to a mixture of ice cold water (4000 c.c.), when the nitroacetanilide was precipitated. The precipitate was filtered off and washed with water both cold and hot. The melting point of the nitroacetanilide, when found to be 206 c or above, it was heated with twice its weight of sulphuric acid (0.5N).
on a water bath, until the liquid remained clear on diluting with water. The p-nitroaniline which was now present in the liquid as the sulphate was diluted with water and precipitated by the addition of an excess of caustic soda. When cold, the yellow crystalline precipitate was filtered, washed and recrystallised from boiling water and alcohol. Yellow needles were obtained m.p. 146-147° C. Yield 50%

Preparation of Dinaphthylamine.

An amount of -naphthol (25 gms.) was crushed to a powder and taken in a long necked round bottom flask. The flask was heated to 230-250° C and ammonia was passed in the flask for six hours and the temp. was kept in the range above mentioned throughout the period.

The contents of the flask were then treated with strong caustic soda solution and extracted with ether. After the removal of the solvent, a product was obtained which on recrystallisation from alcohol, gave a m.p. 170-171° C. All the positive tests for dinaphthyl amine were obtained. Yield 70%.