P R E F A C E

One of the most outstanding developments of chemical industry during the past three decades, has been the use of high pressure processes for chemical synthesis. In pressure, the chemist has discovered a valuable tool with which he is rapidly opening up new developments of chemical processes. Besides the well known methods of synthesising ammonia, methanol, urea, etc., which are now being manufactured economically in large quantities, a number of other vitally important high pressure processes have been reported. As examples, the production of synthetic petrol by the hydrogenation of coal, coal tar, and petroleum residues, gasolines by the cracking of heavy petroleum fractions, lubricating oils by polymerisation of ethylene, polythene plastic etc. may be cited.

Among the many workers in the field, the researches of Ipatieff, Haber, Bergius, Fischer, Bone, Newitt, Morgan, Pines, Reppe, Storch and Hardy are outstanding. Just prior to and during the last war, extensive and intensive researches were carried out throughout the world, particularly in Germany by I.G.Farbenindustrie and Ruhrchemie A.G. under the able guidance of eminent chemists like Dr.J.W.Reppe and Dr.O.Roelen, which culminated in the discoveries of a large number of new

syntheses of great industrial and theoretical importance.

As a consequence of Reppe's pioneering researches, the comparatively little known field of the chemistry of carbon monoxide, acetylenes and olefines opened up with immense possibilities. His researches in acetylene and carbon monoxide chemistry may be divided into four groups : vinylation, ethynylation, polymerisation and carboxylation. As examples of the last group may be mentioned the synthesis of acrylic acid and ~~their~~ its derivatives from acetylene, carbon monoxide and compounds containing active hydrogen atoms, saturated carboxylic acids and their derivatives from olefines, carbon monoxide and compounds containing active hydrogen atoms, saturated carboxylic acids from alcohols and carbon monoxide, and carboxylic acids from ethers, carbon monoxide and water, using in all cases a carbonyl forming metal like nickel, cobalt or iron as a catalyst.

It is rather surprising that there are not many published papers on the above reactions. Most of Reppe's and others' work were patented, the only available information being found in the various B.I.O.S. and F.I.A.T. reports and a few published papers[1-15].

Literature survey reveals that inspite of great

technical importance of adipic acid, comprehensive data on the synthesis of adipic acid from tetrahydrofuran, carbon monoxide and water are not available. Hardly any scientific paper has been published on the subject. Hence it was deemed necessary to take up systematic study on the synthesis of the acid.

Delta-valerolactone, which can easily be converted into epsilon - caprolactam, an important material for polyamide synthesis, can be obtained from tetrahydrofuran and carbon monoxide under high pressure. As no systematic study has been made on the synthesis, a number of experiments are also conducted to synthesis the lactone.

The present thesis consists of two parts :-

The first part of the thesis is concerned with the synthesis of adipic acid from tetrahydrofuran, carbon monoxide and water at high pressure in presence of nickel, cobalt and iron catalysts. To elucidate the mechanism of the formation of various syntheses products, the decomposition of tetrahydrofuran, adipic acid and carbon monoxide have been studied under identical condition of temperature and pressure, in presence of nickel iodide-silica gel catalyst.

The second part of the thesis is concerned with the synthesis of delta-valerolactone from tetrahydrofuran and

carbon monoxide at high pressure using the above mentioned catalysts.

The progress of the reactions has been studied in detail under various experimental conditions and the results have been embodied in the body of the thesis.