

REPORT ON THE EXPERIMENTAL WORK FOR THE MANUFACTURE OF AROMATIC AMINES
BY MEANS OF HYDROGEN UNDER PRESSURE

On the basis of experiments which showed that polysulfide and thiosulfate solutions could be reduced to the sulfide by means of hydrogen under pressure in the presence of a catalyst, experiments were undertaken to utilize this reaction for the reduction of nitro compounds to amines. It is well known that aromatic nitro compounds can be easily converted into the corresponding amines by means of alkali or ammonium sulfide solutions; the sulfide is oxidized to thiosulfate in the course of the reaction. By making use of the reduction process for thiosulfate, it should be possible to reduce any amount of nitro compounds to amines in liquid phase theoretically using a very small amount of sulfide. This process has the advantage over other similar processes which use hydrogen at high pressure that sulfur compounds, present either in the hydrogen or in the nitro compounds, do not affect the reaction and that the reaction can be carried out at comparatively low temperatures. The presence of sulfur-containing impurities might even be an advantage because it would counteract the hydrogenating or ammonia-splitting effect of strongly active catalysts and also make it unnecessary, under favorable circumstances, to add a large amount of the sulfur compound. In batch experiments it was found that catalysts containing iron are most suitable for this type of reaction. The hydrogen used may contain carbon monoxide, besides inert gases, which then takes part in the reaction. The ammonium sulfide solution which is either added at the beginning or formed in the course of the process is not completely reduced to sulfide after its oxidation to thiosulfate but is converted by well-known reactions to a small extent to sulfide and sulfate. By suitable choice of reaction conditions the conversion can be controlled so that only a pure sulfate solution is obtained besides the organic amine. Using nitrobenzene, the process was carried out continuously in the laboratory and in a semi-commercial plant. The work was then discontinued because it was found that no great advantage resulted from the use of this process as compared to the process operated at atmospheric pressure at Ludwigshafen.