Performing a standard Japp-Klingemann synthesis that involved diazotation of 2,6-dinitroaniline and reaction with pentane-2,4-dione in aqueous hydrochloric acid medium surprisingly yielded a mixture of the expected 2,6-dinitro- and the unexpected 2-chloro-6-nitro-substituted hydrazones. This mixture was isolated as solid-solution crystals containing the components in an approximate ratio of 1 to 2. Based on both, selected compounds of comparison that show the aromatic ring substituents changed in position and intermediate diazonium salts, potential causal connections of the unusual behaviour during the route of the particular Japp-Klingemann reaction were studied. X-Ray crystal structures of the relevant compounds have been determined to support the discussion.

**Key words:** Pentane-2,4-dione, Anilines, Diazotation, Japp-Klingemann Reaction, X-Ray Diffraction Analysis