

Synthesis and Extraction of Anhydrous High Purity Hydrazine (HPH) from the Hydrazine Hydrate

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Hydrazine of space grade (HPH) and its alkylated derivatives are propellants used in aerospace and military industry (space shuttles, engines of midcourse adjustment of satellites, missiles). The know-how of our team made it possible to develop an integrated original process, including the synthesis, extraction and purification phases for producing a hydrazine in conformity with the particularly restricting specifications with a percentage of carbon (organic compounds and CO₂) much lower than 30 ppm.

The synthesis of N₂H₄ is carried out by the Raschig way, method of strictly mineral preparation utilizing no carbon atom. A predictive kinetic model permits to determine the optimal conditions according to the concentration of the reagents, the pH, the temperature and the pressure. The design of an innovating device of synthesis under pressure (403 K / 50 bars) makes possible to directly inject the chloramine stabilized in liquid ammonia under pressure so as to save energy. The optimized conditions (10 bars / 323 K) are less constraining than those denoted in the literature and industry (30 bars / 403 K) for higher outputs (70-80%).

The optimization of the various steps of the process of extraction and purification rests exclusively on equilibria between a liquid and another liquid, a vapour or a crystallized phase. The aqueous hydrazine solutions which are the real problem were the subject of a particular study and equilibria between solid/liquid/vapor phases of the binary system H₂O-N₂H₄ were modeled. A distillation permits to extract N₂H₄ as an azeotropic solution, solution which is then concentrated by liquid/liquid demixion and salting out with soda (gap of miscibility in the ternary system N₂H₄-NaOH-H₂O). The extraction and the purification of anhydrous hydrazine are done then by fractional crystallization at low temperature. These last steps call out no vapor phase what excludes any danger from explosion.