on and solute molecules move to the reactor walls, leading to severe encrustation and blockage when seeds of larger sizes are combined with smaller seed loading. Based on the above principles, when operating in the green zone as shown in Figure 3.23, formulated for a specific organic chemical compound, encrustation in DN15 has totally been avoided.

3.5.4.3 Case 3 – Due to Insufficient Nuclei

One of the largest chemical companies conducted continuous crystallisation trials for one of its fish oil products in a NiTech DN15 crystalliser; the starting and the end temperatures were 10 and −25 °C respectively, with a nucleation temperature of 3 °C. No seeds were used; spontaneous nucleation was the main means of generating nucleation. Two temperature zones were set up, 10 to 0 °C, and 0 to −25 °C. The cooling rates for the first and second zones were 2.5 and 0.75 °C min^{-1} respectively, for the purposes of generating supersaturation in zone 1 and growing crystals in zone 2. Cloudiness of the solution in DN15, the indication of nucleation, was noticed from 3 to 6 °C. Encrustation was observed close to the end of the zone 1 and the start of the zone 2. Following the same principles as the aforementioned seeding strategy, the combination of size and mass of nuclei is critically important for successful operation. When spontaneous nucleation takes place, nuclei are generated, and the size of nuclei is very small (not measured in this case) which is an advantage, meeting one of the two criteria; the amount of nuclei was however unknown and difficult to determine. The encrustation would suggest that the mass of nuclei was not sufficient according to the above principles. The two temperature zones were replaced by three as 10 to 5 °C, 5 to −10 °C and −10 to −25 °C, with cooling rates of 7.5, 7.5 and 0.75 °C min^{-1} respectively, on the hypothesis that more nuclei (*i.e.* more mass) would appear when sufficiently high supersaturation was generated quickly in zone 2. Previously the zone 1 was cooled from 10 to 0 °C at the rate of 2.5 °C min^{-1}, *i.e.* over 4 mins; the zone 2 was now from 5 to −10 °C at a rate of 7.5 °C min^{-1}, *i.e.* over 2 mins. Encrustation was still seen in zone 2 in further trials. One additional observation noted that the degree of blockage was decreased with the increase of oscillation. This hinted that there may be too many nuclei generated which has an adverse effect on the local mixing, causing blockage. By trial and error with a number of cooling rates in zone 2, encrustation was noticeably reduced and finally removed at one cooling rate that seemed to generate the correct mass of nuclei, enabling trouble-free runs thereafter! This is a practical dilemma; on one hand, some of the nuclei would stick on the lowest energy place, *e.g.* walls of the device, leading to encrustation, when the mass of nuclei from spontaneous nucleation is insufficient and cannot complete the nucleation process. On the other hand, when too many nuclei emerge from solution at once, local mixing is compromised with particle sedimentation and agglomeration that would also lead to surface encrustation. What would be the optimal loading of nuclei is compound dependent, for example, encrustation occurred in the DN15 a couple of days later when a new batch of starting