Nitromethane

Dissolve 50g of mono-chloroacetic acid in 100 ml water contained in a 500 ml round-bottomed bolt-head flask, and then neutralise the solution by the cautious addition of 30g of finely powdered anhydrous sodium carbonate. For this purpose, add the sodium carbonate in small quantities (about 1g) at a time, preferably with the aid of a spatula, and shake the solution gently around after each addition to facilitate the evolution of carbon dioxide: a clear solution is thus maintained throughout, whereas the rapid addition of large quantities of the carbonate produces lumps of material which are subsequently difficult to dissolve. Now dissolve 36.5g of sodium nitrite in 50 ml of water with gentle heating, cool the solution thoroughly in icewater, and then add it with shaking to that of the sodium monochloroacetate. Add some fragments of unglazed porcelain, and then fit the flask with a delivery-tube of moderately wide bore connected in turn to a water-condenser. Support the flask over a gauze, and then heat it gently with a small Bunsen flame. The solution slowly becomes yellow in colour, then greenish and finally a yellowish-brown, when a vigorous effervescence starts: at once remove the Bunsen flame, and allow the reaction to proceed spontaneously, carbon dioxide being evolved and the solution boiling vigorously. When the reaction subsides, replace the Bunsen flame and maintain a steady boiling. Nitromethane distills over in the steam and separates as a colourless oil at the bottom of the distillate: since nitromethane is slightly soluble in water, stop the distillation as soon as drops of nitromethane can no longer be detected in the distillate leaving the condenser. Transfer the distillate to a separating funnel, and carefully run off the lower layer of nitromethane and then dry it over anhydrous sodium sulfate for 30 minutes. Filter the dry nitromethane (preferably through a small dry Buchner funnel), transfer it to a 30 mL distilling-flask fitted with a water-condenser, and then slowly distill, collecting the fraction of bp 100-102°C. In view of the small volume of nitromethane to be manipulated, if a small Buchner funnel is not available, it is advisable to extract the crude nitromethane from the aqueous distillate with ether (30-40ml). Dry the ethereal extract over sodium sulfate, filter through a fluted filter-paper, and then distill off the ether on a water-bath, and finally distill the residual nitromethane. Nitromethane is obtained as a colourless liquid, of bp 101°C and d 1.10, yield, 10 g.