PROCEDIMENTO

Experiment 5.54 BUTYL BROMIDE (*1-Bromobutane*)

$$Me\cdot(CH_2)_2\cdot CH_2OH + HBr \xrightarrow{H_2SO_4} Me\cdot(CH_2)_2\cdot CH_2Br + H_2O$$

To 250 g of 48 per cent hydrobromic acid contained in a 500-ml round-bottomed flask add 75 g (41 ml) of concentrated sulphuric acid in portions with shaking; some hydrogen bromide may be evolved. Add 88 g (110 ml, 1.2 mol) of butan-1-ol, followed by 60 g (32.5 ml) of concentrated sulphuric acid in several portions with shaking, and finally a few chips of porous porcelain. Attach a reflux condenser to the flask and reflux the mixture gently on a wire gauze for 2–3 hours; during this period the formation of butyl bromide is almost complete and a layer separates above the acid (1). If the preparation is carried out in the open laboratory, fit an absorption device (Fig. 2.61(*a*) or (*b*)) to the top of the condenser in order to absorb any hydrogen bromide and sulphur dioxide which may be evolved. Allow the contents of the flask to cool, remove the condenser and set it for downward distillation. Distil the mixture until no more oily drops of butyl bromide pass over (30–40 minutes). Transfer the distillate to a separatory funnel and remove the halide which

forms the lower layer. Wash it successively with water, an equal volume of concentrated hydrochloric acid (2), water, 5 per cent sodium hydrogen carbonate or sodium carbonate solution, and water. Separate the water as completely as possible and dry with 2–3 g of anhydrous calcium chloride or magnesium sulphate; the desiccant should be left in contact with the bromide for at least 30 minutes and shaken occasionally. Filter the dried product through a small funnel supporting a fluted filter paper into a 200-ml flask, add a few chips of porous porcelain and distil either from an air bath (Fig. 2.46) or on a ceramic-centred wire gauze. Collect the portion boiling at 100–103 °C. The yield is 155 g (95%).

Usar ¼ da receita