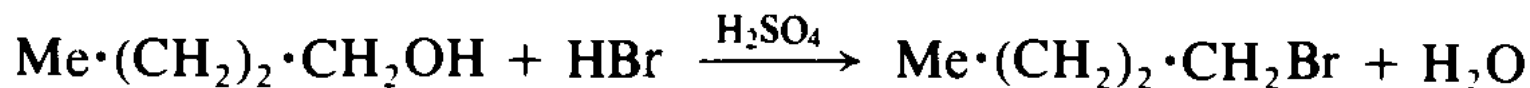


PROCEDIMENTO

Experiment 5.54 BUTYL BROMIDE (*1-Bromobutane*)

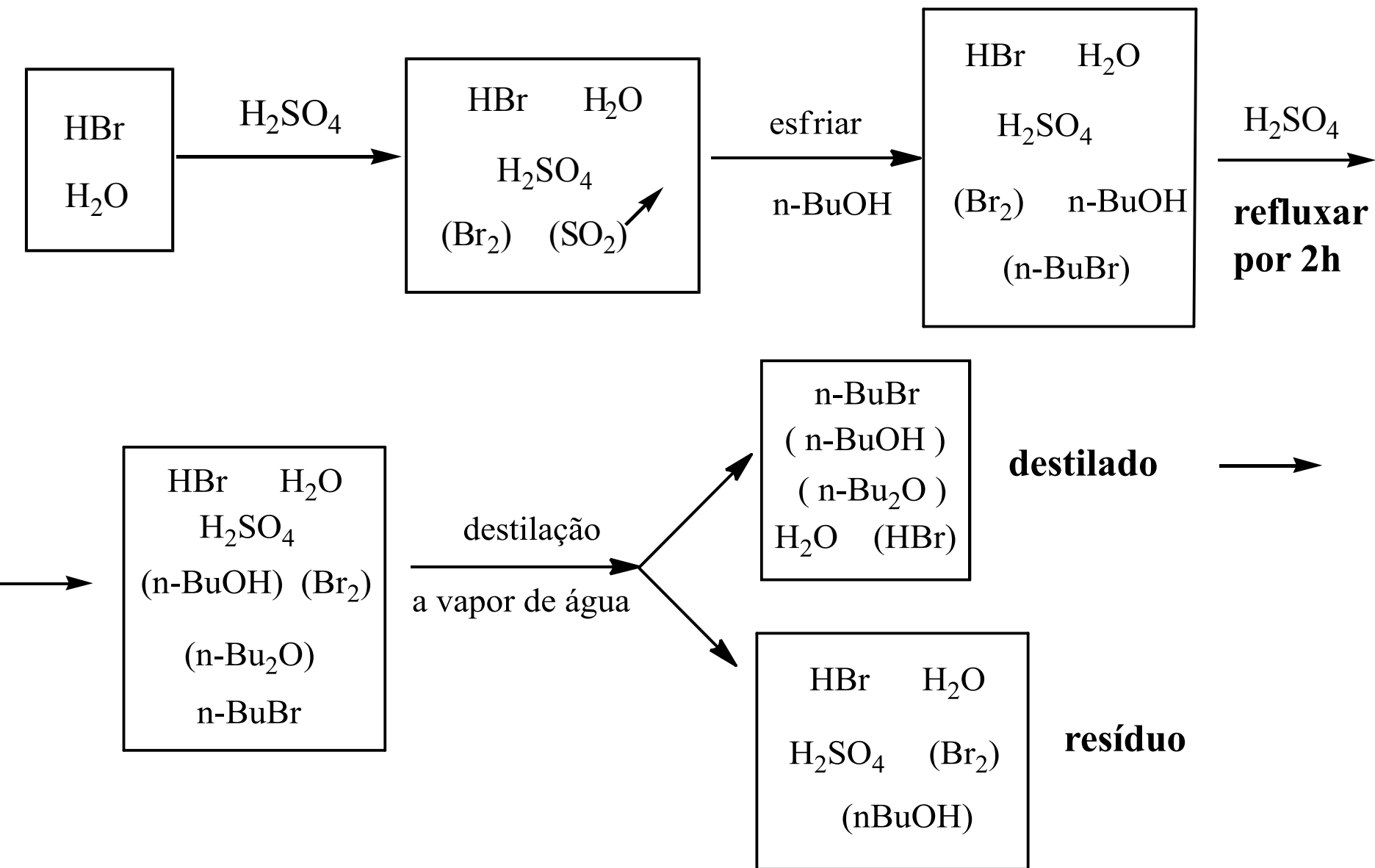


To 250 g of 48 per cent hydrobromic acid contained in a 500-ml round-bottomed flask add 75 g (41 ml) of concentrated sulphuric acid in portions with shaking; some hydrogen bromide may be evolved. Add 88 g (110 ml, 1.2 mol) of butan-1-ol, followed by 60 g (32.5 ml) of concentrated sulphuric acid in several 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%).

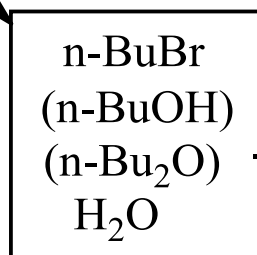
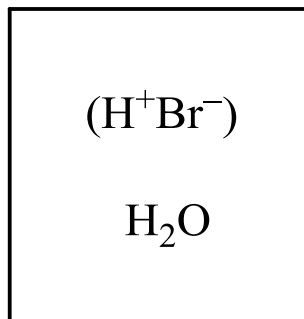
Usar ¼ da receita

FLUXOGRAMA: Brometo de n-Butila

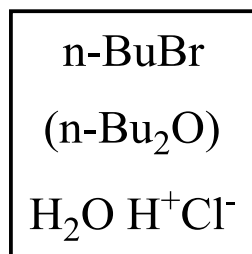
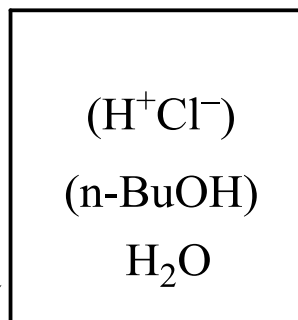


Brometo de n-Butila

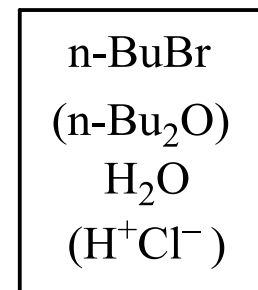
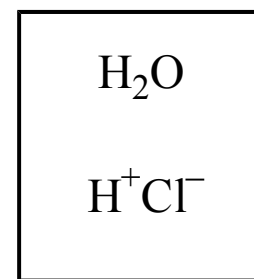
**Separação
de fases**



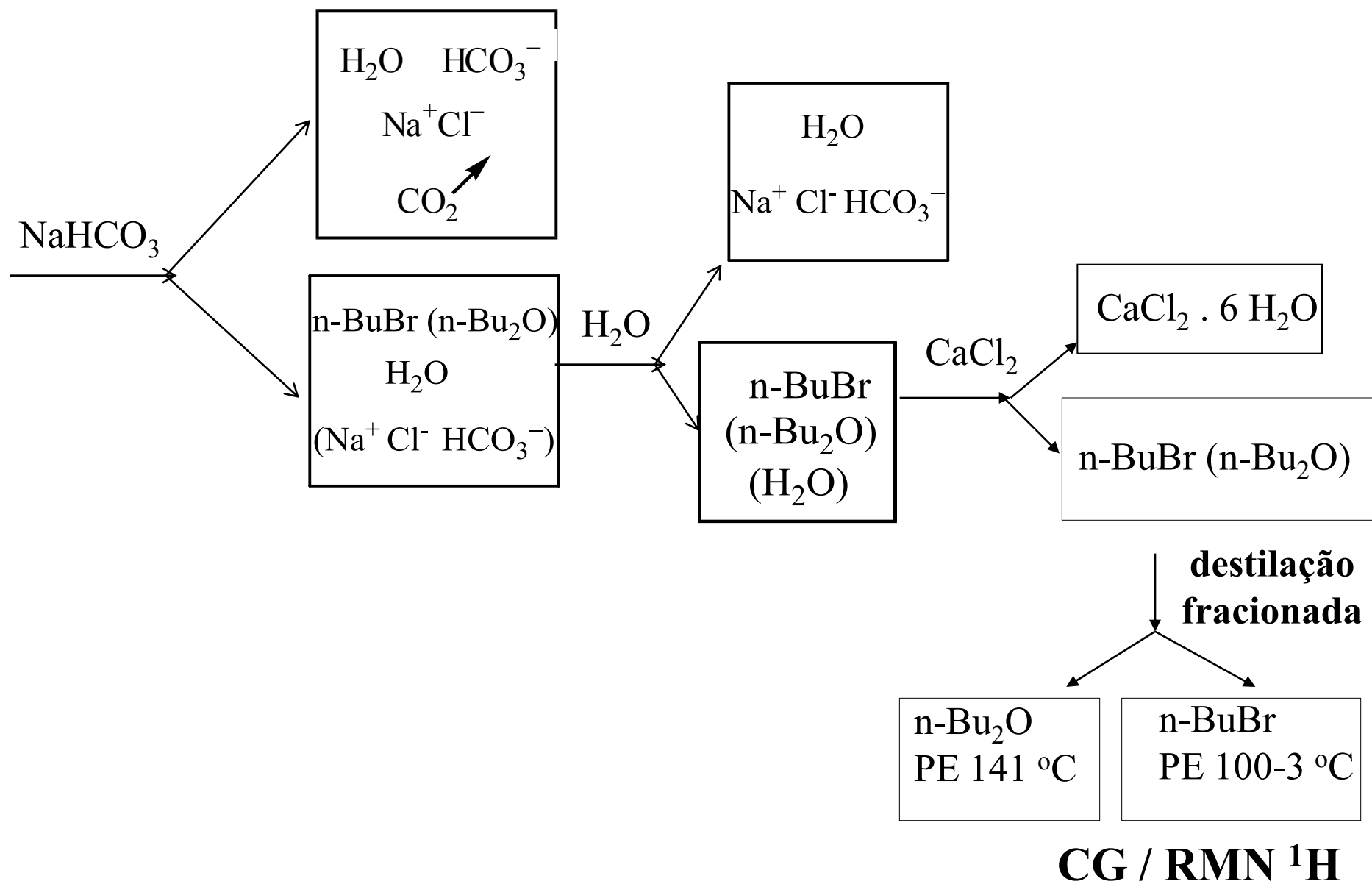
HCl conc.



H_2O



Brometo de n-Butila



Brometo de n-Butila

Quantidades: 1/4 da receita do Vogel (4ª Edição, p. 387)

HBr (48 %):	62,5 g (43 mL)	0,38 mol
H ₂ SO ₄ (conc.):	19 g (10 mL)	
+	15 g (8 mL)	
n-butanol:	22 g (28 mL)	0,30 mol
n-BuBr:	32,9 g (25,9 mL)	0,30 mol

Procedimento:

Cuidado ao misturar HBr e H_2SO_4 (capela! luvas!);
no balão de 250 mL.

Porque a adição de ácido sulfúrico é feita em duas porções?

Colocar condensador de refluxo e refluxar por 2 h, com manta, na capela;

Destilar a mistura até o destilado não passar mais turvo (observe a relação entre água e brometo de n-butila);

Separar as fases (+ água se necessário) e lavar com: 1. HCl conc.; 2. água; 3. NaHCO_3 (5 %); 4. água. *[Porque esta sequência?]*

Secar com “pouco” CaCl_2 ;

Destilação fracionada final para a purificação do produto, com coluna de Vigreux e fracionador (“vaquinha”).