Synthesis of 1-bromobutane

$$CH_3CH_2CH_2CH_2OH + NaBr + H_2SO_4 \rightarrow CH_3CH_2CH_2CH_2Br + NaHSO_4 + H_2O$$

Greenness optimization

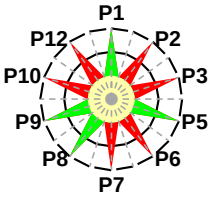
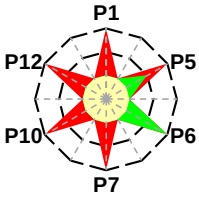
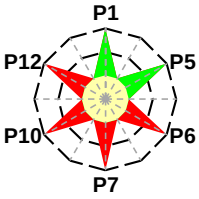
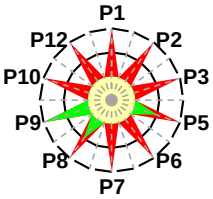
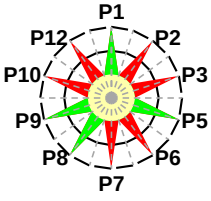
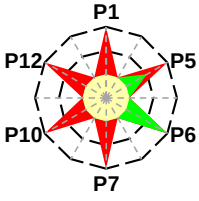
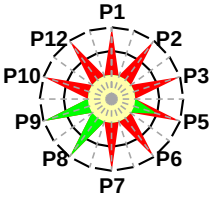
The greenest procedures for each step are: R_4[1] for the reaction, I_1[2] for the isolation and Pu_1[3-15] or Pu_5[6] (with same GS) for the purification. Combining these procedures with the one for the purification Pu_1[3-15] it is possible to obtain greener procedures than those of the original protocols.

In Table 1 are presented two combinations (1 and 2). Combination 2 presents the same greenness of the greenest of the analysed protocols (GSAI = 25.00).

When purification of the product is performed (combination 1), the greenness is reduced, but it is obtained a global protocol greener than those analysed where the three steps are performed.

Optimized protocols are described below.

Table 1. Green star obtained by combining the greenest procedures of each step

Combination	Reaction	Isolation	Purification	Global process
1	**R_4**  **IPE = 40,00**	**I_1**  **IPE = 25,00**	**Pu_1**  **IPE = 33,33**	 **IPE = 20,00**
2	**R_4**  **IPE = 40,00**	**I_1**  **IPE = 25,00**	Without purification	 **IPE = 25,00**

Optimized protocol 1

Reaction. In a 25 mL round bottomed flask, dissolve 2.66 g (25.9 mmol) of sodium bromide (about 20% excess) in 3 mL of distilled water and 1.60 g (21.6 mmol) of butan-1-ol. Cautiously, with steady swirling, add 2.2 mL (40.5 mmol) of concentrated sulphuric acid (about 88% excess) dropwise to the solution. Reflux the reaction using a sand bath for 45 minutes. Stick a wad of glass wool on the top of the condenser to prevent vapour from escaping.

Isolation. Transfer the mixture into a separatory funnel. Add 20 mL of ethyl ether and wash the organic layer with three 20 mL portions of distilled water. Wash the organic layer with two 20 mL portions of 5% sodium hydrogen carbonate solution and finally with more 20 mL of water. Dry the compound with a drying agent. *(No drying agent is indicated, so it's considered the use of magnesium sulphate, which is greener.)* Filter by gravity into a round bottomed flask and evaporate the solvent in a rotary evaporator, at room temperature.

Purification. Distill the 1-bromobutane, collecting the material that boils between 94 and 102 ºC.

Optimized protocol 2

Reaction. In a 25 mL round bottomed flask, dissolve 2.66 g (25.9 mmol) of sodium bromide (about 20% excess) in 3 mL of distilled water and 1.60 g (21.6 mmol) of butan-1-ol. Cautiously, with steady swirling, add 2.2 mL (40.5 mmol) of concentrated sulphuric acid (about 88% excess) dropwise to the solution. Reflux the reaction using a sand bath for 45 minutes. Stick a wad of glass wool on the top of the condenser to prevent vapour from escaping.

Isolation. Transfer the mixture into a separatory funnel. Add 20 mL of ethyl ether and wash the organic layer with three 20 mL portions of distilled water. Wash the organic layer with two 20 mL portions of 5% sodium hydrogen carbonate solution and finally with more 20 mL of water. Dry the compound with a drying agent. *(No drying agent is indicated, so it's considered the use of magnesium sulphate, which is greener.)* Filter by gravity into a round bottomed flask and evaporate the solvent in a rotary evaporator, at room temperature.

Purification. Not prescribed.

References

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