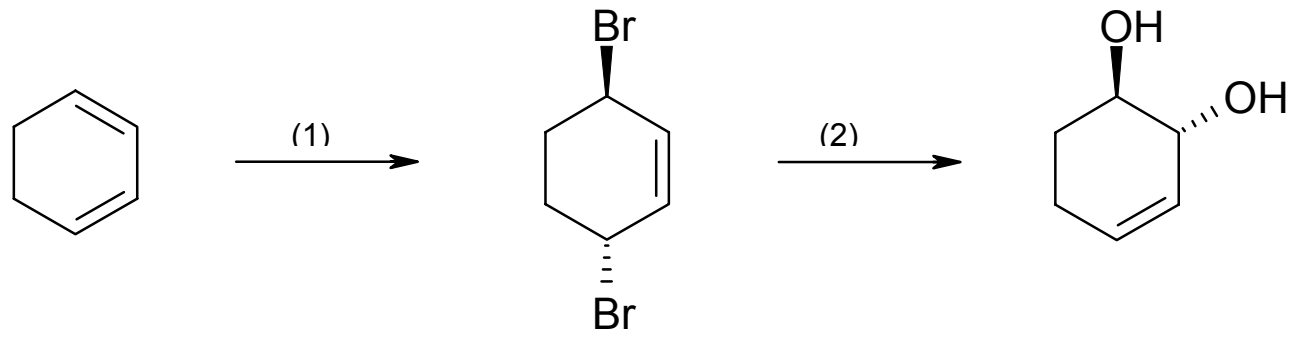
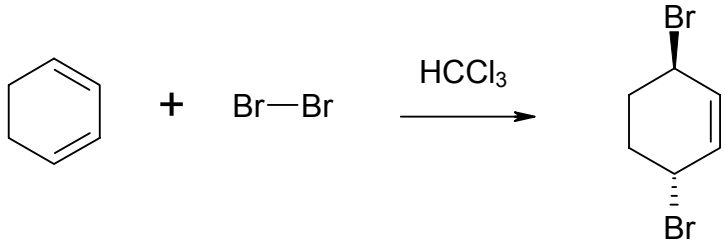
trans-cyclohex-3-ene-1,2-diol

Literatur: Helv. Chim. Acta, 36, 1953, 256



(1):



80 g/mol 160 g/mol 238 g/mol

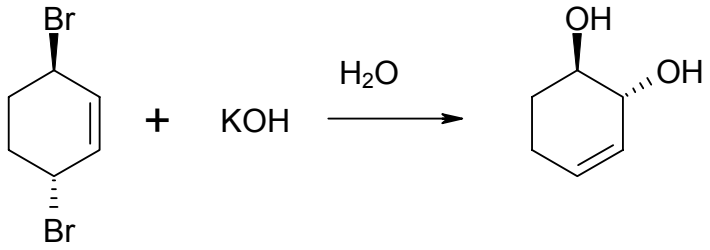
Substances:

1,3-cyclohexadiene	15,0 g = 188 mmol = 17,4 ml
Bromine	30.0 g = 188 mmol = 9,6 ml
Chloroforme	240 ml

The bromine and about 140 ml of the solvent is given in a dropping funnel and is slowly dropped in 1,3-cyclohexadiene in about 100 ml solvent. The flask is cooled in an ice-bath and the temperature may not increase 10 °C. The dark brown bromine is decolourated immediately in the cyclohexadiene. The reaction is done with argon protective gas. At the end you get a pale orange solution. The dropping funnel is washed with about 5 ml of chloroforme and the solution is stirred again for 15 minutes. The solvent will be evaporated and you get 42,8 g of an pale orange mixture of crystals and oil.

Yield: 42,8 g = 180 mmol; 180 mmol/ 188 mmol = 96 %

(2):



238 g/mol 56 g/mol 114 g/mol

Substances:

trans-1,4- dibromocyclo-2-hexene	42,8 g = 180 mmol
Potassium hydroxide	34,0 g = 600 mmol
Water	270 ml

The potassium hydroxide is soluted in water and cooled to room temperature. Then it is given to the dibromocyclohexene in a 1 l flask and it is stirred for four days. After that you add about 15 ml of conc. hydrochloric acid and $NaHCO_3$ to get pH 7. There is no more gas if this is reached. The water is evaporated. At the end you add 400 ml of toluene to get all of the water out. Then you add 500 ml of methylene chloride and you filtre. You add 50 g of silica gel an the solvent is evaporated. With column chromatography the impurities are separated. You use 100 g of silica gel and CH_2Cl_2/Et_2O 1:1 as solvent. Fractions 11-32 are evaporated and is recrystallized with about 50 ml of ethyl acetate. You get 8,9 g of a white solid.

Yield: 8,9 g = 78 mmol; 78 mmol/ 180 mmol = 43 %

^{1}H nmr: δ ($CDCl_3$) 1,61 (1H, m, CH_2-C6), 1,91 (1H, m, CH_2-C6), 2,14 (2H, m, CH_2-C5), 3,61 (1H, m, CH-C3 or –C4), 3,73 (2H, s, OH), 4,08 (1H, m, CH-C3 or –C4), 5,55 (1H, m, CH-C1 or –C2), 5,67 (1H, m, CH-C1 or –C2).