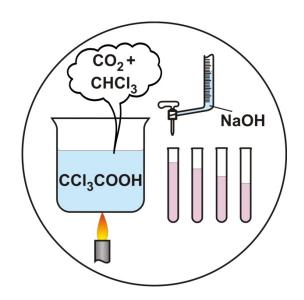
Measuring Rate Density by Titration

Equipment:

burner, laboratory tripod, wire gauze with ceramic center (alternatively: hot plate with magnetic stirrer)
beaker (400 mL)
6 beakers (100 mL)
burette (50 mL)
retort stand, clampholder, clamp funnel
5 test tubes
test tube stand
adhesive tape (as filling mark)
stopwatch
silicone rubber grip for hot beakers
dropping pipette



Chemicals:

trichloroacetic acid sodium hydroxide pellets sodium hydroxide standard solution (0.1 kmol m⁻³) phenolphthalein indicator solution deionized water

Safety:

trichloroacetic acid (C₂HCl₃O₂):







H314, H335, H410 P280, P301+330+331, P303+361+353, P305+351+338, P310

sodium hydroxide (NaOH):



H290, H314 P233, P280, P303+361+353, P305+351+338, P310

phenolphthalein solution (C₁₂H₁₄O₄) (in ethanol):







H225, H319, H341, H350 P210. P280, P305+351+338, P308+313

Since both trichloroacetic acid and sodium hydroxide can cause severe burns to the skin and serious eye damage, it is essential to wear suitable protective gloves and safety goggles when handling the substances. Since the decomposition of trichloroacetic acid produces chloroform, among others, the experiment has to be carried out under a fume hood.

Procedure:

<u>Preparation:</u> A test tube is marked at the level of the upper edge of the test tube stand (e.g. using an adhesive strip). 4 g of trichloroacetic acid are weighed out in a 100 mL beaker and then dissolved in about 10 mL of water. In a 400 mL beaker, 200 g of water are mixed with a sodium hydroxide pellet and heated to a gentle boil using a burner or a hotplate with a magnetic stirrer. The burette is attached to the retort stand by means of a clampholder and a clamp and filled with the sodium hydroxide standard solution via the funnel.

<u>Procedure:</u> The prepared trichloroacetic acid solution is poured into the boiling, slightly alkaline water. Subsequently, a small sample of the reacting mixture is taken every five minutes, i.e. the hot solution is poured into the marked test tube up to the filling mark. Then, each time, the liquid is transferred into a 100 mL beaker, a few drops of phenol-phthalein indicator solution are added and the solution is titrated with the sodium hydroxide standard solution until the color changes from colorless to pale pink. The titrated solution is then poured into an empty test tube and the test tubes are placed one after the other in the stand.

Observation:

The column of liquid in the test tubes protruding over the upper edge of the test tube stand continues to decrease during the course of the experiment until finally the height of the upper edge of the stand is reached, which is the case after about 20 to 30 minutes.

Explanation:

Trichloroacetic acid decomposes to chloroform (trichloromethane) and carbon dioxide when its aqueous solution is exposed to heat:

$$CCl_3 - COOH|w \rightarrow CCl_3H|g + CO_2|g$$
.

When investigating this slow reaction, a small sample of the reacting mixture is taken after certain periods of time to instantly determine the concentration of the acid by titration with sodium hydroxide solution. When the titrated solution is transferred into test tubes, the reaction process can be followed by observing the fill level. As the acid concentration decreases, so does the amount of sodium hydroxide that needs to be added. After about 20 to 30 minutes, the acid has completely decomposed.

Disposal:

The pH-neutral solutions are combined and disposed of in a container for aqueous neutral solvent waste.