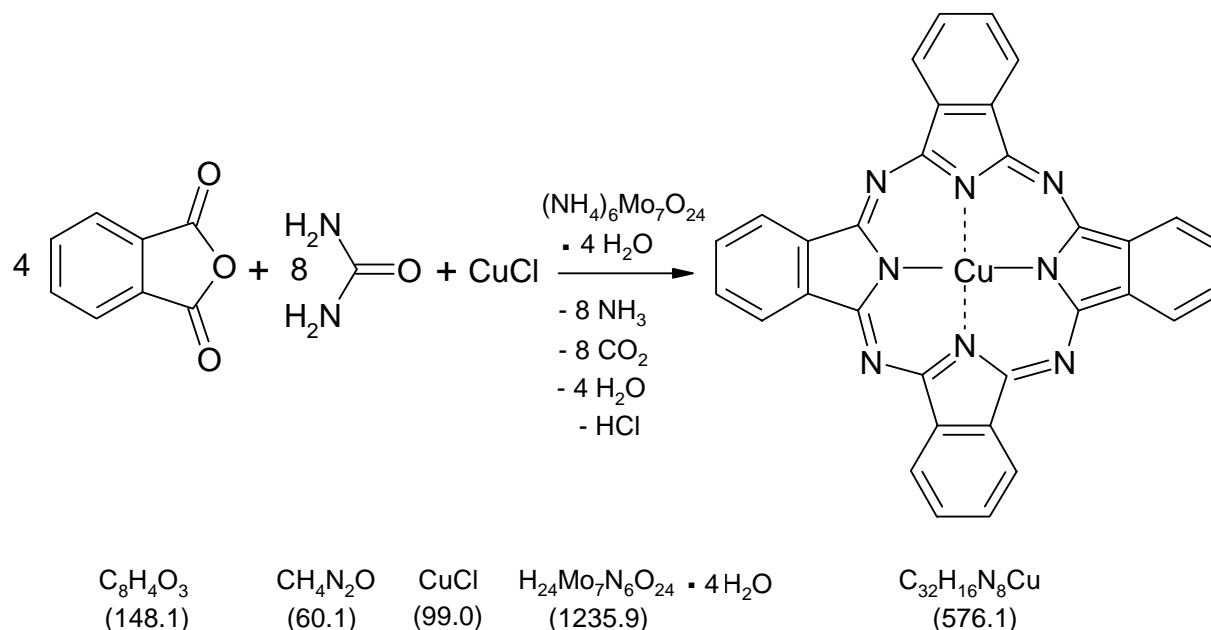


5009 Synthesis of copper phthalocyanine



Literature

Classic synthesis: F. H. Moser, A. L. Thomas, *Phthalocyanine Compounds*, Reinhold, New York **1963**; microwave application: A. Shaabani, *J. Chem. Research (S)* **1998**, 672-673

Classification

Reaction types and substance classes

reaction of the carbonyl group in carboxylic acid derivatives, ring closure reaction
 carboxylic acid anhydride, carbonic acid derivate, heterocycle, dye

Work methods

microwave-assisted reaction, stirring with magnetic stir bar, heating under reflux, extracting, filtering, draining of gases

Instruction (batch scale 4.5 mmol)

Equipment

microwave system ETHOS 1600, glass tube (40 cm, NS 29), 100 mL two-neck flask, magnetic stirrer, magnetic stir bar, reflux condenser, 2 wash bottles, adapter with ground-glass joint and hose coupling

Substances

urea (mp 132.5-134.5 °C)	5.53 g (92.0 mmol)
phthalic anhydride (mp 129-132 °C)	2.67 g (18.0 mmol)
copper(I) chloride	500 mg (5.00 mmol)
ammonium heptamolybdate	75 mg (0.061 mmol)

conc. hydrochloric acid (32%)	5 mL
ethanol (bp 78 °C)	80 mL

Reaction

The reaction apparatus consists of a 100 mL two-neck flask with temperature sensor and reflux condenser. The reflux condenser is equipped with an adapter with ground-glass joint and hose coupling, which is connected with a draining pipe for gases formed during the reaction. The pipe is connected with an empty safety wash bottle and this with a further wash bottle, filled with about 300 mL water.

A mixture of 5.53 g (92.0 mmol) urea, 2.67 g (18.0 mmol) phthalic anhydride, 500 mg (5.00 mmol) copper(I) chloride and 75 mg (0.061 mmol) ammonium heptamolybdate is filled in the reaction flask. After addition of two drops of water the reaction apparatus is installed in the microwave system with the glass tube (see "Technical Instructions. Standard refluxing apparatus for microwave system"). It is not necessary to homogenize the educt mixture, since the complete melt is achieved already after 2 minutes. The reaction mixture is irradiated for 10 minutes at a temperature restriction of 250 °C with 1000 W. Already after half of the time the melt solidifies to a porous violet mass.

Work up

The solid, which is cooled down to room temperature is chopped in the reaction flask, then 50 mL water and 5 mL conc. hydrochloric acid are added and the flask is installed in the microwave system - as aforementioned - with magnetic stir bar, temperature sensor and reflux condenser, but without gas draining pipe. The mixture is heated under stirring with 800 W for 10 minutes at 102 °C, whilst excessive or not reacted educts are extracted from the solid. After cooling down, the solid is filtered off over a folded filter, washed on the filter with 50 mL water and then with little ethanol, and then dried. The yellow-brown aqueous filtrate is disposed. Crude yield: 2.40 g, violet solid

Together with 50 mL ethanol, the crude product is filled in the two-neck flask again and installed in the microwave system – as aforementioned - with magnetic stir bar, temperature sensor and reflux condenser. With a radiation of 500 W it is heated under stirring to 80 °C for 10 minutes, whilst the side product dihydrophthalocyanine should be extracted from the solid. After cooling down to 50 °C the product is filtered over a folded filter, washed with 20 mL ethanol on the filter and dried in a desiccator at reduced pressure.

Yield: 2.15 g (3.73 mmol, 83%); gleaming, violet, fine-crystalline substance, insoluble in organic solvents.

The melting performance of the substance can serve as a criterion for purity: A pure product is given, if until temperatures of above 200 °C no melting or decomposition can be noticed. The here isolated substance was stable until 360 °C. The side product dihydrophthalocyanine melts under decomposition at 195-197 °C.

Comments

The addition of two drops of water before the beginning of the reaction facilitates the launching of the microwave energy in the solid mixture.

Waste management**Waste disposal**

Waste	Disposal
aqueous filtrate	neutralize, then: solvent water mixtures, containing halogen, containing heavy metal,
ethanolic filtrate	solvent water mixtures, halogen free

Time

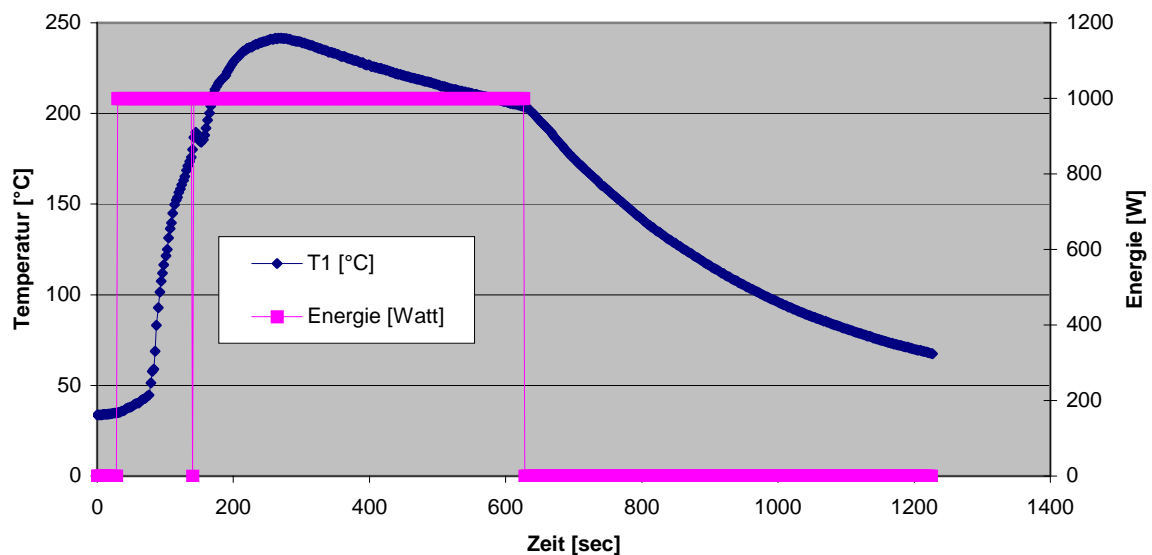
1 to 2 hours

Break

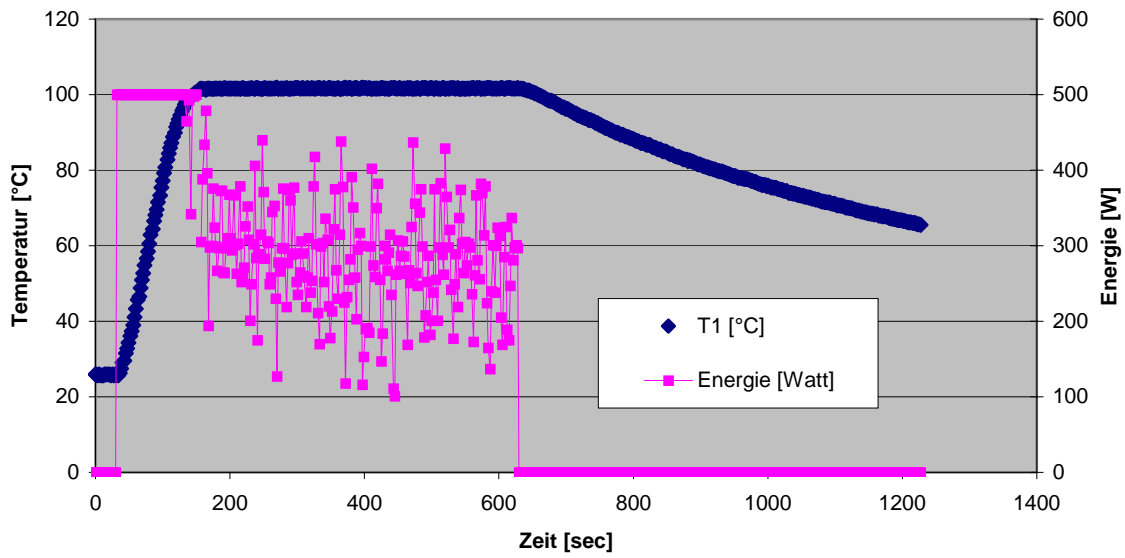
Between the microwave treatments

Degree of difficulty

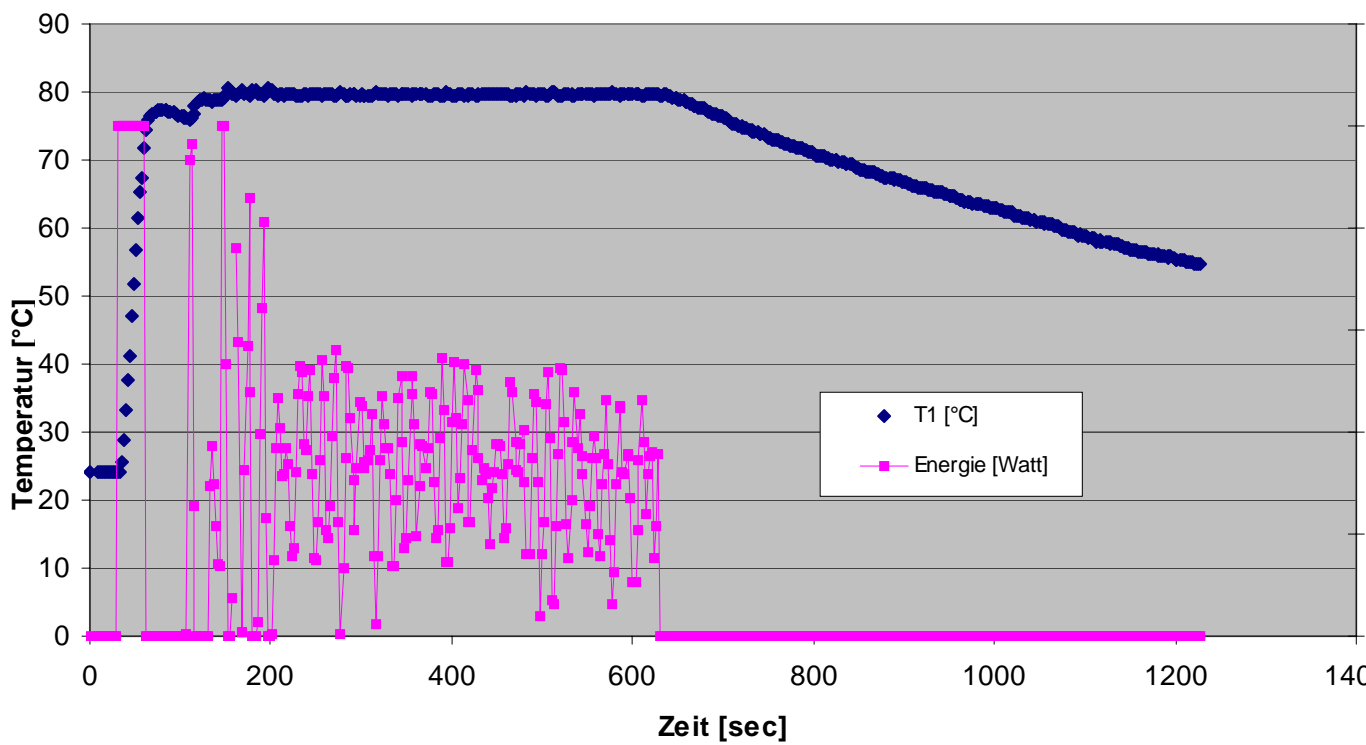
Easy

Analytics**Temperature-time-dependence of the working steps in the microwave field****Synthesis of copper(II)-phthalocyanine**

Extraction of crude copper(II)-phthalocyanine with diluted hydrochloric acid



Extraction of copper(II)-phthalocyanine with ethanol



IR-spectrum of the product copper(II)-phthalocyanine (KBr)