

UOZ

IM-10

Ge(100)

Contributed by Jean-Louis Paillaud

Verified by Y. Kubota, W. Schmidt

Type Material: $[\text{C}_{12}\text{N}_2\text{H}_{30}]_2[\text{F}_4\text{Ge}_{40}\text{O}_{80}]$
(SDA = hexamethonium)

Method: Y. Mathieu, J.-L. Paillaud, P. Caullet, N. Bats [1]

Batch Composition: 1 GeO_2 : 0.25 $\text{SDA}(\text{OH}/\text{Br})_2$: 0.5 HF : 5 H_2O

Source Materials

deionized water

hexamethonium bromide (98%, Aldrich)

resin Dowex[®] SBR LC NG, OH Form (Supelco)

hydrofluoric acid (HF, 40% in water, Carlo Erba),

amorphous germanium oxide GeO_2 (>99.99%, Aldrich)

Batch Preparation (for 0.375 g dry product)

(1) [10 g hexamethonium bromide + water + 55 g Dowex[®]] in a polypropylene flask,^a stir overnight, remove Dowex[®] by filtration, gently rotoevaporate the water to concentrate the solution to about 1 mol/L^b

(2) [6.34 g solution (1)^c + 1.74 g germanium oxide] in a polypropylene beaker, evaporate under stirring until it reaches a total weight of 2.214 g

(3) [(2) + slowly 365.7 μL HF], stir manually with a non metallic spatula (ideally with a Teflon stirrer)^{d,e}

Crystallization

Vessel: Teflon-lined stainless steel autoclave

Temperature: 170° C

Time: 7 days

Agitation: 60 rpm

Product Recovery

(1) Dilute reaction mixture with water

(2) Filter and wash with water

(3) Dry at ambient temperature or at 70°C

(4) Yield: 0.375 g

Product Characterization

XRD: UOZ; competing phase: no

Elemental analysis: GeO_2

Crystal size and habit: truncated square bipyramidal morphology, with dimension 1 μm .

Reference

- [1] Y. Mathieu, J.-L. Paillaud, P. Caullet, N. Bats, *Micropor. Mesopor. Mater.* 75 (2004) 13

Notes

- a. The water volume is such that the height of the solution is twice the resin layer in the flask.
- b. The exchange rate ($\text{Br}^- \rightarrow \text{OH}^-$) is about 98 %, which is determined by acid-base titration and liquid proton NMR. If necessary, a second exchange may be achieved if the first exchange rate is too low.
- c. Here the concentration of the SDA solution is 0.65 mol/L.
- d. Thick dough.
- e. pH of final mixture is 6.5-7.