UOV IM-17 Si(71.4), Ge(28.6)

Contributed by Jean-Louis Paillaud

Verified by Y. Kubota, W. Schmidt

Type Material: $|(C_{16}H_{38}N_2)_{5.5}\ (OH^-)_{11}\ (H_2O)_2|[Si_{125.7}Ge_{50.3}O_{352}]$
(SDA = decamethonium)

Method: Y. Lorgouilloux, M. Dodin, E. Mugnaioli, C. Marichal, P. Caullet, N. Bats, U. Kolb, J.-L. Paillaud [1]

Batch Composition: 0.6 SiO_2 : 0.4 GeO_2 : 0.25 $SDA(OH/Br)_2$: 10 H_2O

Source Materials
deionized water
decamethonium bromide (98%, Fluka)
resin Dowex® SBR LC NG, OH Form (Supelco)
amorphous germanium oxide GeO_2 (>99.99%, Aldrich)
silica (SiO_2, Degussa Aerosil 200)

Batch Preparation (for 1.08 g dry product)
(1) [10 g decamethonium bromide + water + 48 g Dowex®] in a polypropylene flask,[a] stir overnight, remove Dowex® by filtration, gently rotoevaporate the water to concentrate the solution to about 0.5 mol/L[b]
(2) [0.753 g Aerosil + 11.03 g solution (1)[c] + 0.836 g germanium oxide] in a polypropylene beaker, evaporate under stirring until it reaches a total weight of 3.65 g
(3) [(2) + slowly 288 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: 14 days
Agitation: no

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: 1.08 g

Product Characterization
XRD: UOV; competing phase: MFI when Si/Ge < 1
Elemental analysis: 2.5 SiO_2 : GeO_2[f]
Crystal size and habit: rhombus shaped plate-like crystals with dimension 1 µm.

Reference

[1] Y. Lorgouilloux, M. Dodin, E. Mugnaioli, C. Marichal, P. Caullet, N. Bats, U. Kolb, J.-L. Paillaud, RSC Advances 4 (2014) 19440

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 ($Br^- \rightarrow OH^{-)}$ is about 95 %, 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 0.453 mol/L.

d. Thick dough.

e. pH of the final mixture is 14.

f. A higher Si/Ge molar ratio is also possible with TEOS in the same procedure but the yield is low.