

UOS

IM-16

Si(57.8), Ge(42).2

Contributed by Jean-Louis Paillaud

Verified by Y. Kubota, C. Kirschhock, W. Schmidt

Type Material: $[(C_6N_2H_{11})_{6.88}] [Si_{55.5}Ge_{40.5}O_{192}F_{6.88}]$
(SDA = 1-ethyl-3-methyl-imidazol-3-ium)

Method: Y. Lorgouilloux, M. Dodin, J.-L. Paillaud, P. Caullet, L. Michelin, L. Josien, O. Ersen, N. Bats [1]

Batch Composition: 0.5 SiO₂ : 0.5 GeO₂ : 1 SDAOH/Br : 1 HF : 20 H₂O

Source Materials

deionized water

1-ethyl-3-methyl-imidazol-3-ium bromide (98%, Solvionic)

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

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

amorphous germanium oxide GeO₂ (>99.99%, Aldrich)

silica SiO₂ (Degussa Aerosil 200)

Batch Preparation (for 0.38 g dry product)

(1) [10 g 1-ethyl-3-methyl-imidazol-3-ium bromide + water + 52 g Dowex[®]] in a polypropylene flask,^a stir overnight, remove Dowex[®] by filtration, gently rotoevaporate the water to concentrate the solution to more than 2 mol/L^b

(2) [5.08 mL solution (1)^c + 0.58 g germanium oxide] in a polypropylene beaker, stir until dissolved^d

(3) [(2) + 0.333 g Aerosil], stir

(4) [(3) + slowly 487 μ L HF], stir^{e,f}

Crystallization

Vessel: 20 mL 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 90°C

(4) Yield: 0.78 g

Product Characterization

XRD: **UOS**; competing phase^e: MFI (when Si / Ge > 1.5)

Elemental analysis: 1.37 SiO₂ : GeO₂

Crystal size and habit: thin plate-like crystals displaying prismatic shape (reminding the one of MFI) with dimensions between 1 and 20 μ m

Reference

- [1] Y. Lorgouilloux, M. Dodin, J.-L. Paillaud, P. Caullet, L. Michelin, L. Josien, O. Ersen, N. Bats, *Journal of Solid State Chemistry* 182 (2009) 622

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 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 2.2 mol/L.
- d. Clear solution is obtained
- e. Translucent gel, low viscosity, adjust the water content (keep to evaporate at room temperature under stirring or add water) to obtain the right stoichiometry.
- f. pH of final mixture gel is 9.5.