ITT ITQ-33 Si(19.98), Ge(26.02)

Contributed by Manuel Moliner

Verified by L. Tosheva, V. Georgieva, L. Lakiss

Type Material: [$Si_{19.98}Ge_{26.02}O_{92}$]

Method: A. Corma, M. J. Diaz-Cabanas, J. L. Jorda, C. Martinez, M. Moliner [1]

Batch Composition: 0.67 SiO_2 : 0.33 GeO_2 : 0.05 Al_2O_3 : 0.15 $Hex(OH)_2$: 0.1 $HexBr_2$: 0.3 HF : 1.5 H_2O

Source Materials

Ludox AS-40 (SiO_2) (40 %, Aldrich)
Germanium dioxide (GeO_2) (>99.9%, Aldrich)
Alumina (Al_2O_3) (78%, Sasol)
Hexamethonium hydroxide ($Hex(OH)_2$) (0.1M, Aldrich)
Hexamethonium bromide ($HexBr_2$) (>99.9%, Sigma)
Ammonium fluoride (NH_4F) (98 %, Sigma-Aldrich)
Double distilled water (H_2O)

Batch Preparation[a]

(1) [0.22 g $HexBr_2$ + 9.22 g $Hex(OH)_2$ + 0.04g Al_2O_3], dissolve under continuous stirring
(2) [(1) + 0.2 g GeO_2], dissolve under continuous stirring
(3) [(2) + 0.62 g Ludox AS-40], dissolve under continuous stirring
(4) [(3) + 0.07 g NH_4F], dissolve under continuous stirring
(5) Freeze-drying for 48h
(6) Absorption of water (77% humidity) [b]

Crystallization

Vessel: Teflon-lined stainless steel autoclave (5 mL)
Temperature: 180 °C
Time: 24 h

Product Recovery

(1) Filtration, rinsed with distilled water
(2) Dried at 70 ºC overnight
(3) Calcination of the material at 550 ˚C for 3 hours in an air stream

Product Characterization

XRD: ITQ-33
SEM: prismatic crystals, 5 μm

Reference

[1] A. Corma, M. J. Diaz-Cabanas, J. L. Jorda, C. Martinez, M. Moliner, Nature 443 (2006) 842

Notes

b. The starting mixture is prepared in a polypropylene bottle
c. Absorption of water in desiccator until the initial composition is reached