PTO PST-31 Si(94), Al(6)

Contributed by Donghui Jo and Suk Bong Hong

Verified by Independent verification of this synthesis procedure was not performed due to the use of homemade template. The recipe is published as received accompanied by the X-ray diffraction pattern provided from the contributing authors.

Type Material $|(SDA)_{1.9}Na_{0.1}(OH)_{1.1}|$ $[Al_{2.8}Si_{45.2}O_{96}]$
(SDA = 1,1'-(1,4-butanediyl)bis(2,4-dimethyl-1*H*-pyrazol-2-ium))

Method D. Jo, Y. Zhang, J. H. Lee, A. Mayoral, J. Shin, N. Y. Kang, Y.-K. Park, S. B. Hong [1]

Batch Composition 1.0 SiO_2 : 0.033 Al_2O_3 : 0.125 $SDABr_2$: 0.15 Na_2O : 30 H_2O
($SDABr_2$ = 1,1'-(1,4-butanediyl)bis(2,5-dimethyl-1*H*-pyrazol-2-ium) dibromide)

Source Materials
deionized water
aluminum hydroxide ($Al(OH)_3 \cdot H_2O$, Aldrich)
1,1'-(1,4-butanediyl)bis(2,4-dimethyl-1*H*-pyrazol-2-ium) dibromide ($SDA(Br)_2$, made in-house, 25% in water)[a]
sodium hydroxide (NaOH, Sigma-Aldrich, 1.515 g/mL, 50% in water)
colloidal silica (SiO_2 Ludox ® AS-40, Sigma-Aldrich, 40%)

Batch Preparation
(1) [0.1600 g aluminum hydroxide + 1.2756 g $SDABr_2$ + 10.93 g water + 396 µL sodium hydroxide] in a polypropylene bottle, stir for 1 hour
(2) [(1) + 3.76 g LUDOX AS-40], stir for 3 hours

Crystallization
Vessel: Teflon-lined stainless-steel autoclave
Time: 7 days
Temperature: 175 °C
Agitation: 60 rpm

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: < ~2 g

Product Characterization
XRD: PTO
competing phases: PWW (when $SiO_2/Al_2O_3 \leq 20$)
Elemental analysis: SiO_2/Al_2O_3 = 32 [1]
Crystal size and habit: very small (< 30 nm) nanospheres

References

[1] Jo, D.; Zhang, Y.; Lee, J. H.; Mayoral, A.; Shin, J.; Kang, N. Y.; Park, Y.-K.; Hong, S. B. An Aluminosilicate Zeolite Containing Rings of Tetrahedral Atoms with All Odd Numbers from Five to Eleven. *Angewandte Chemie International Edition* **2021**, *60* (11), 5936–5940.

Notes

a. First, 25.30 g of 1,4-dimethypyrazole was dissolved in 100 mL of acetonitrile. Then, 27.54 g of 1,4-dibromobuthane was added and the mixture was refluxed for 7 days. (*Caution*: this reaction can be highly exothermic and result in acidic mixtures, use appropriate precautions.) The solid product was recovered using filtration with 250 mL of *N*,*N*-dimethylformamide and subsequently with 500 mL of acetone.

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