

EUO

[Ga] EU-1

Si(96.5), Ga(3.5)

Contributed by A. N. Kotasthane

Verified by S. Lambert, H. Kessler, and T. Loiseau

Type Material Na₅[Ga₄Si₁₀₈O₂₂₄] · wH₂O (w ~ 26)

Method G. N. Rao, V. P. Shiralkar, A. N. Kotasthane, P. Ratnasamy [1]

Batch Composition 7.0 Na₂O : Ga₂O₃ : 36.6 SiO₂ : 4.0 R : 5.55 H₂SO₄ : 926 H₂O^a
(R = hexamethonium dibromide (C₁₂H₃₀N₂Br₂))

Source Materials

demineralized water
sodium hydroxide, reagent grade (97%)
silica sol (27.4% SiO₂, 0.5% Na₂O)
sulfuric acid (AR BDH, 98%)
gallium(III)sulfate (Aldrich 99.99%)
hexamethonium bromide monohydrate (HM-Br₂, Aldrich)

Batch Preparation (for 24 g product)

- (1) [30.0 g water + 5.41 g sodium hydroxide], mix until dissolved, pH = 13.8 ± 0.2
- (2) [81.0 g silica sol + 20 g water], mix until uniform, pH = 9.8 ± 0.2
- (3) [(1) + (2)], mix until uniform, pH = 13.6 ± 0.2
- (4) [30 g water + 2.6 g sulfuric acid + 4.3 g gallium sulfate], mix until dissolved; heating on hot plate for 15 minutes essential, pH = 0.08 ± 0.02
- (5) [(3) + (4)], add (4) drop-wise to (3) with good mixing
- (6) [30 g water + 15.2 g HM-Br₂], mix until dissolved. pH = 7.2 ± 0.2
- (7) [(5) + (6)], add (6) to (5) with good mixing. Adjust final batch to pH = 12.6 with 1 M NaOH^b

Crystallization

Vessel: stainless-steel autoclave (Parr, 300 mL capacity)
Temperature: 170 °C
Time: 6 days
Agitation: propeller mixer (250 RPM)

Product Recovery

- (1) Cool and filter to recover solids. Slurry pH after crystallization = 10.5 ± 0.2
- (2) Wash extensively with demineralized water until filtrate pH < 9
- (3) Dry at 110 °C for 12 to 15 hours
- (4) Yield: approximately 24 g (56% on Ga₂O₃, 79% on SiO₂)

Product Characterization

XRD: Pure EU-1 phase (EUO framework topology) having characteristic strong reflections at d = 4.30, 3.99 and 3.28 Å without any impurity phases. On longer crystallization (above 6 days), alpha quartz appears

Elemental Analysis: 92.4% SiO₂, 5.24% Ga₂O₃ 2.36% Na₂O (SiO₂/Ga₂O₃= 55.0, Na₂O/Ga₂O₃ = 1.3 6) ^{c,d}

Crystal Size and Habit: Homogeneously distributed small spheres (2-3 μm)

Reference

- [1] G. N. Rao, V. P. Shiralkar, A. N. Kotasthane, P. Ratnasamy, in Synthesis of Microporous Materials, Vol. I, M. L. Occelli, H. E. Robson (eds.), Van Nostrand Reinhold, New York (1992) p 153

Notes

- a. Increasing SiO₂/Ga₂O₃ in gel from 35 to 70 and to 110 gives SiO₂/Ga₂O₃ in product of 79 and 100 respectively. For products with SiO₂/Ga₂O₃ greater than 100, the preferred template is equimolar benzyldimethylamine + benzyl chloride. [1]
- b. The Na₂O content of the silica source is significant. If gel pH is less than 12.0, it should be adjusted with NaOH solution.
- c. By thermal analysis: template burnout 300-700°C (approximately 10% weight loss).
- d. By ⁷¹Ga MAS NMR: chemical shift is 170 ppm (Ga³⁺ in tetrahedral environment).