

Contributed by Henri Kessler and Abdallah Merrouche

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Type Material [Ga₁₂P₁₂O₄₈][(n-C₃H₇)₂NH₂F]₃: (H₂O)_w (w ~ 6 [1])

Method A. Merrouche, J. Patarin, M. Soulaire, H. Kessler, P. Anglerot [2]

Batch Composition 1 Ga₂O₃ : 1 P₂O₅ : 1 HF : 70 H₂O : 6.5 DPA (DPA = di-n-propylamine)

Source Materials

phosphoric acid (Fluka, 85% H₃PO₄)

distilled water

gallium sulfate hydrate (Strem Chemicals, Ga₂(SO₄)₃ · xH₂O (wt% Ga ~ 18))

hydrofluoric acid (Fluka, 40% HF)

di-n-propylamine (Fluka, 95%)

Batch Preparation (for 1 g dry product)

- (1) [1.16 g phosphoric acid + 2.2 g water + 3.8 g gallium sulfate hydrate + 2.5 g water], stir until dissolved
- (2) [(1) + 0.25 g hydrofluoric acid], mix thoroughly
- (3) [(2) + 3.25 g di-n-propylamine], mix thoroughly. Initial pH = 4 to 4.5

Crystallization

Vessel: PTFE-lined autoclave

Temperature: 140 °C

Time: 24 hours

Agitation: none

Product Recovery

- (4) Filter; wash with distilled water
- (5) Dry at 60 °C
- (6) Yield: approximately 60% with respect to starting oxides

Product Characterization

XRD characteristic strong reflections at d = 12.02, 8.50, 6.94, 6.01 Å

Elemental Analysis (anhydrous form): Ga_{0.49}P_{0.51}O₂(DPA)_{0.13}F_{0.12}

Crystal Size and Habit: cubes (5 - 40 μm)

References

- [1] A. Simmen, J. Patarin, C. Baerlocher, in Proceedings of the 9th International Zeolite Conference, Vol. 1, R. Von Ballmoos, J. B. Higgins, M. M. J. Treacy (eds.), Montreal, 1992, Butterworth-Heinemann, Stoneham, 1993, p. 433
- [2] A. Merrouche, J. Patarin, M. Soulaire, H. Kessler, D. Anglerot, in Molecular Sieves, Vol. 1, Synthesis of Microporous Materials, M. L. Occelli, H. F. Robson (eds.), Van Nostrand Reinhold, New York, 1992, p. 384