

**Contributed by** Lovat V. C. Rees

**Verified by** Yingcai Long, P. Sidheswaran and G. Price

**Type Material**  $(\text{NaAlO}_2)_7(\text{SiO}_2)_9$

**Method** L. V. C. Rees and S. Chandrasekhar [1]

**Batch Composition**  $\text{Al}_2\text{O}_3$  : 2.2  $\text{SiO}_2$  : 5.28 NaF : 105.6  $\text{H}_2\text{O}$

#### Source Materials

distilled water  
sodium fluoride (BDH Analar)  
kaolinite ( $\text{Al}_2\text{O}_3$  : 2.2  $\text{SiO}_2$  : 2  $\text{H}_2\text{O}$ )<sup>a</sup>

**Batch Preparation** (for 10 g dry product)

- (1) [87.7 g water + 10.4 g sodium fluoride], stir and make a slurry
- (2) [(1) + 12.7 g kaolin], mix thoroughly

#### Crystallization

Vessel: sealed polypropylene  
Time: 60 days  
Temperature: 85°C  
Agitation: none  
pH: initial 7.5, final 9-10

#### Product Recovery

- (1) Filter and wash thoroughly with distilled water
- (2) Exchange twice with NaCl solution
- (3) Wash with distilled water (adjusted to pH = 10 with NaOH)
- (4) Dry at 85°C for 24 hours
- (5) Rehydrate over water vapor from saturated NaCl solution

#### Product Characterization

XRD: Strong zeolite P competing phases: CHA, no SOD  
Elemental Analysis:  $\text{NaAlO}_2 \cdot 1.18 \text{SiO}_2$  (by atomic absorption spectroscopy)<sup>b,c</sup>  
Crystal Size and Habit: spherulitic particles of approximately 10  $\mu\text{m}$  dia.

#### Reference

- [1] L. V. C. Rees, S. Chandrasekhar, Zeolites 13 (1993) 535

#### Notes

- a. Kaolinite from Trivandrum, Kerela, India
- b. Dissolution of the zeolite sample for atomic adsorption: 0.1 g sample was treated with 30 mL of a mixture of HCl (37%), HF (48%) and distilled water (ratio 1:1:1); the same was kept for two to three days for complete dissolution.
- c. <sup>29</sup>Si MAS NMR gave five lines with chemical shifts of -86.79, -91.55, -97.19, -102.86 and -107.10 ppm. <sup>27</sup>Al MAS NMR gave two lines, intense Al (tet) at 58.58 and weak (oct) at -0.42 ppm.