MSE MCM-68 Si (101.6), Al (11.4)

Contributed by Yoshihiro Kubota

Verified by J.-L. Paillaud, C. Xia, F. Gao and X. Zou
Type Material: $Si_{101.6}Al_{11.4}O_{224}$
(SDA = $TEBOP^{2+}(I^-)_2$ = *N,N'*-diethylbicyclo[2.2.2]oct-7-ene-2,3:5,6-dipyrrolidine diiodide)

Method: D. L. Dorset, S. C. Weston, S. S. Dhingra [1]
Batch Composition: 1.0 SiO_2 : 0.1 $Al(OH)_3$[a] : 0.375 KOH : 0.1 $TEBOP^{2+}(I^-)_2$: 30 H_2O

Source Materials
colloidal silica (Ludox HS 40)
aluminum hydroxide (50 wt.%)
deionized water (DI)
8 mol/L potassium hydroxide aqueous solution (titrated as 5.93 mmol/g)
$TEBOP_2^+(I^-)_2$ synthesized from bicyclo[2.2.2]oct-7-ene-2,3:5,6- tetracarboxylic anhydride (Aldrich)

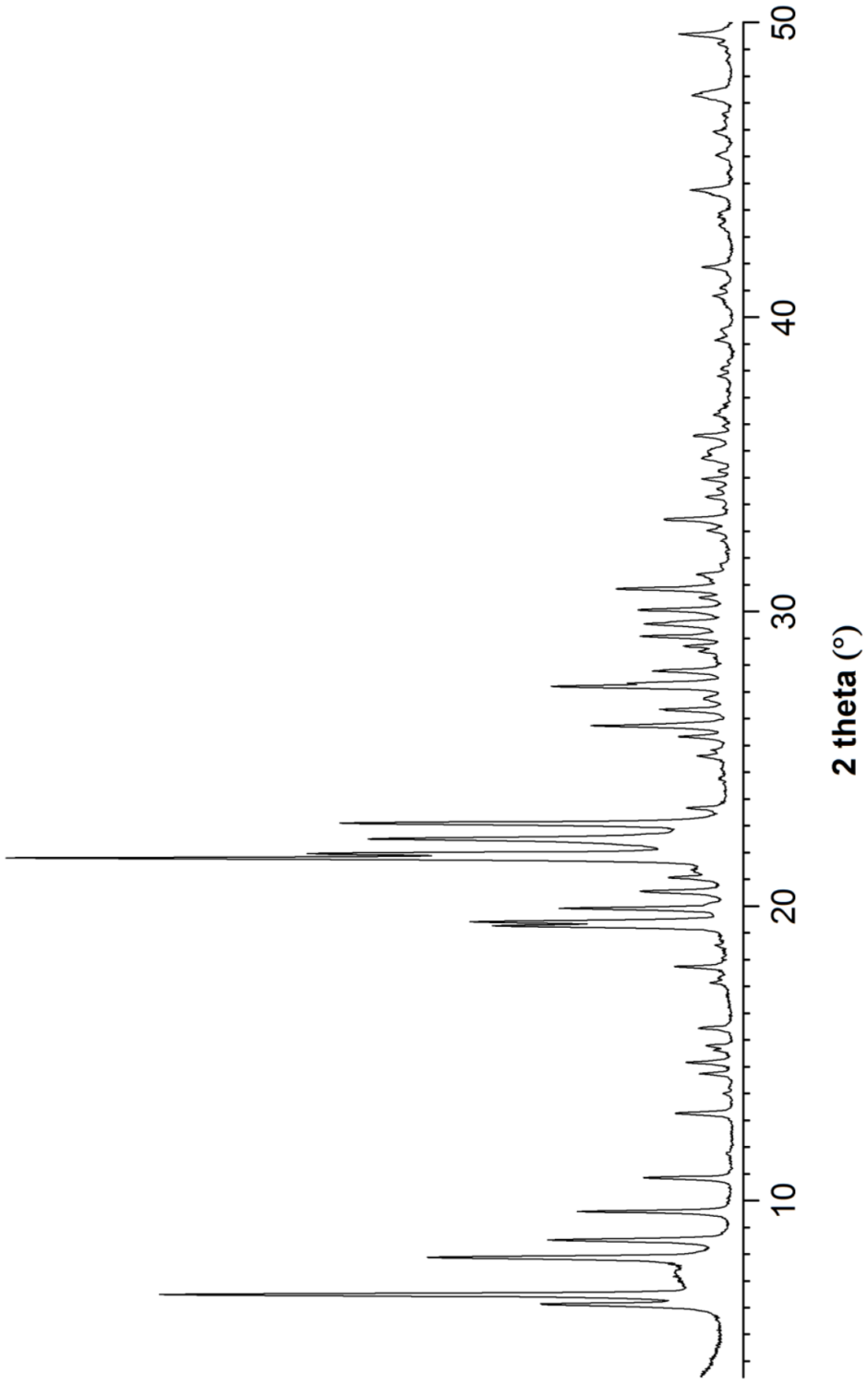
Batch Preparation (for ca. 6 g as-synthesized dry product)
(1) [15.0 g Ludox HS-40 + 40.0 g DI water + 0.78 g aluminum hydroxide], stir for 10 min in a Teflon beaker
(2) [(1) + 6.32 g potassium hydroxide aqueous solution 5.93 mmol/g]; stir for 30 min
(3) [(2) + 5.58 g $TEBOP_2^+(I^-)_2$]; stir for 4 h at room temperarure

Crystallization
Vessel: Teflon-lined stainless steel autoclave
Temperature: 160 °C
Time: 16 days
Agitation: 0 rpm

Product Recovery
(1) Dilute reaction mixture with water
(2) Filter and wash with water
(3) Dry at ambient temperature or at 80 °C
(4) Yield: 6.0 g
(5) Calcination conditions to remove SDA: 600°C for 10 h in air

Product Characterization
XRD: MSE
Crystal size: smaller than 100 nm

Reference
[1] D. L. Dorset, S. C. Weston, S. S. Dhingra, J. Phys. Chem. B 110 (2006) 2045
[2] S. Inagaki, Y. Tsuboi, Y. Nishita, T. Syahylah, T. Wakihara, Y. Kubota, Chem. Eur. J. 19 (2013) 7780

Notes
a. $Al(OH)_3$ source is important. The brand should be Pfalz and Bauer or Aldrich.

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