Contributed by Yoshihiro Kubota


Type Material: CDS-1, Si$_{38}$O$_{72}$
PLS-1, Si$_{18}$O$_{34}$(OH)$_4$K$_{1.3}$.1.7(CH$_3$)$_4$NOH (precursor material of CDS-1)

Method: T. Ikeda, Y. Akiyama, Y. Oumi, A. Kawai, and F. Mizukami [1]

Batch Composition: 1.0 SiO$_2$ : 0.015 KOH : 0.22 (CH$_3$)$_4$NOH : 16.2 H$_2$O : 3.41 1,4-dioxane

Source Materials
- deionized water (DI)
- potassium hydroxide (0.5 mol/L aqueous solution, KOH)
- tetramethylammonium hydroxide (15 wt.% aqueous solution, (CH$_3$)$_4$NOH)
- 1,4-dioxane (Sigma Aldrich)
- silica (Cab-O-Sil M5)

Batch Preparation (for 4 g dry product as PLS-1)
1. [25.0 g water + 10.0 g silica + 22.0 g (CH$_3$)$_4$NOH aq.$^a$ + 0.5 g KOH aq.$^b$], stir in a vessel
2. [(1) + 50.0 g 1,4-dioxane], vigorously stir for 1 h at room temperature

Crystallization
- Vessel: Teflon-lined stainless steel autoclave (300 mL)$^c$
- Temperature: 150 °C
- Time: 10 days
- Agitation: no

Product Recovery
- (3) Filter and wash with acetone and water
- (4) Dry at 70 °C in a convection oven for 12 h
- (5) Yield: 3.5 g

Calcination$^d$
- From 400 to 900 °C; under vacuum at 10$^{-3}$ to 10$^{-8}$ Torr$^e$

Product Characterization
- XRD: CDO
- Crystal size and habit: thin platelet-like crystals with a length < 2 $\mu$m and a thickness of > 100 nm (approximately).

Reference

Notes
a. 15 wt.% aqueous solution
b. 0.5 mol/L aqueous solution
c. 150 mL autoclave is also utilized
d. The topotactic conversion from PLS-1 to CDS-1 requires the calcination treatment.

e. Typically, 500 °C, 12 h, ambient pressure (evacuation not required), according to Ref. [2]