MFI [Ti] ZSM-5 Si(98.6), Ti(1.4)

Contributed by Jan H. C. van Hooff and Arjan van der Pol

Verified by D. Serrano and Z. Gabelica

Type Material $[Ti_{1.3}Si_{94.7}O_{192}] : wH_2O$

Method M. Taramasso, G. Perego and B. Notari [1-3]

Batch Composition TiO_2: 70 SiO_2:1980 H_2O : 30 TPA-OH (TPA tetra-n-propylammonium)

Source Materials

distilled water
tetraethylorthosilicate $[Si(OC_2H_5)_4]$ (Merck 800658)
tetraethylorthotitanate $[Ti(OC_2H_5)_4]$ (Merck 821083)
tetrapropylammonium hydroxide (Alfa 17456, 40% solution in water)

Batch Preparation (for 43 g product)

(1) [163.3 g tetraethylorthosilicate + 2.56 g tetraethylorthotitanate], mix at 35˚C
(2) [(1) + 170 g tetrapropylammonium hydroxide (40% solution)], add slowly at 0˚C to prevent hydrolysis
(3) Heat at 80˚C to evaporate ethanol
(4) Add water to restore initial volume; final pH 12.2

Crystallization

Vessel: 500 mL stirred autoclave
Temperature: 175˚C
Time: 2 days
Agitation: 120 rpm

Product Recovery

(1) Centrifuge to recover solids and wash with water (three times)
(2) Dry at 120˚C
(3) Heat in air to 550˚C (heating rate 5˚C/min.) and hold at 550˚C for 3 hours
(4) Yield 90% on SiO_2

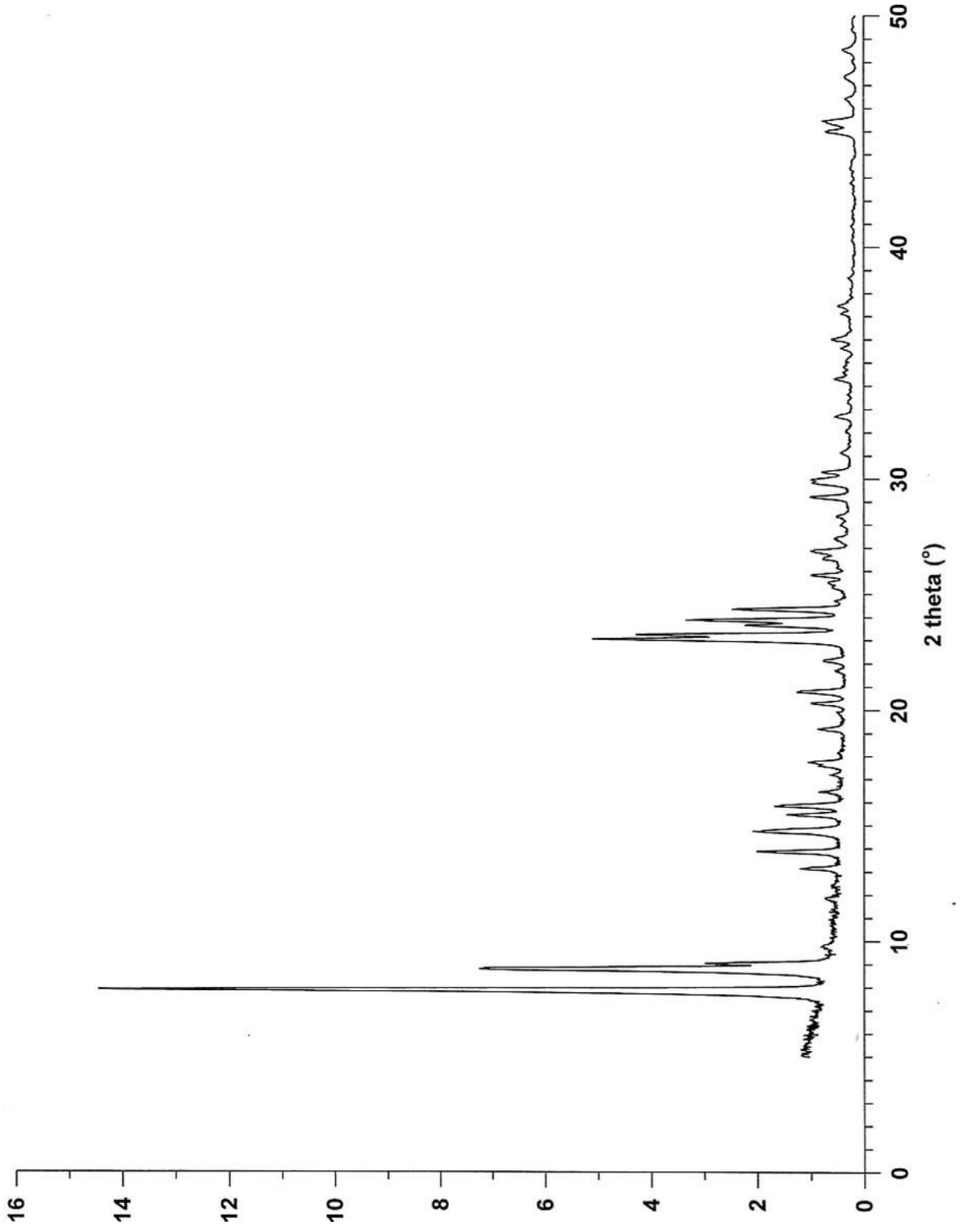
Product Characterization

XRD: orthorhombic MFI (only crystalline phase)
Elemental Analysis: 1.37 mol% Ti (72 SiO_2/TiO_2 by AAS) [a]
Crystal Size and Habit: 0.3 μm cubes (SEM)

References

[1] M. Tarainasso, G. Perego, B. Notari, US Patent 4 410 501 (1983)
[2] A. J. H. P. van der Pol, J. H. C. van Hooff, Appl. Catal. 92 (1992) 93
[3] J. A. Martens, P. Buskens, P. A. Jacobs, A. van der Pol, J. van Hooff, P. J.Kooymann, H. van Bekkum, Appl. Catal. 99 (1993) 71
[4] A. Tuel, Y. Ben Taarit, Zeolites 14 (1994) 272

Note

a. No extra-framework TiO_2 can be observed by IR or UV-VIS. No IR bands around 700 cm^{-1} and no UV-VIS bands above 250 nm were observed [4].

MFI **[Ti] ZSM-5** **Si(98.6), Ti(1.4)**