

Supplementary Materials: Temperature-Induced Desorption of Methyl Tert-Butyl Ether Confined on ZSM-5: An In Situ Synchrotron XRD Powder Diffraction Study

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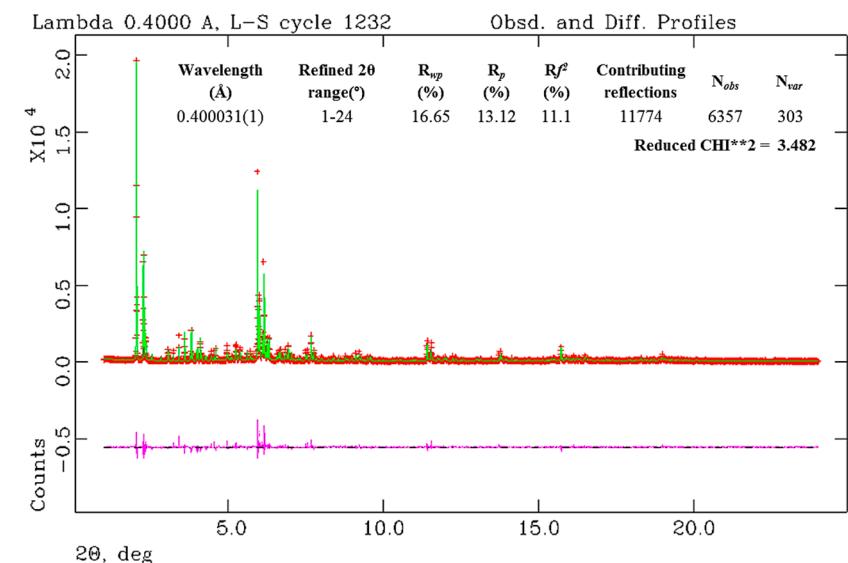


Figure S1. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 30 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

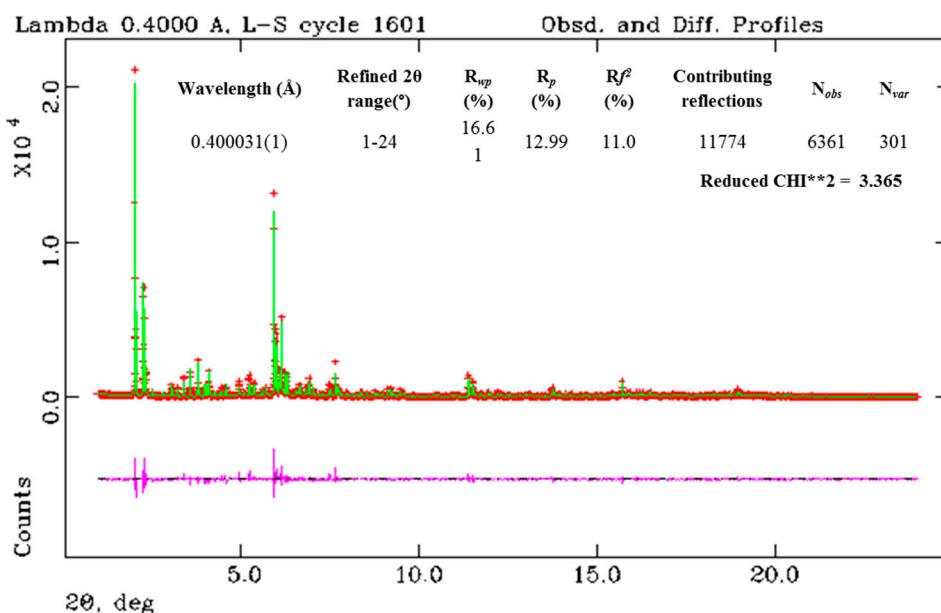


Figure S2. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 50 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

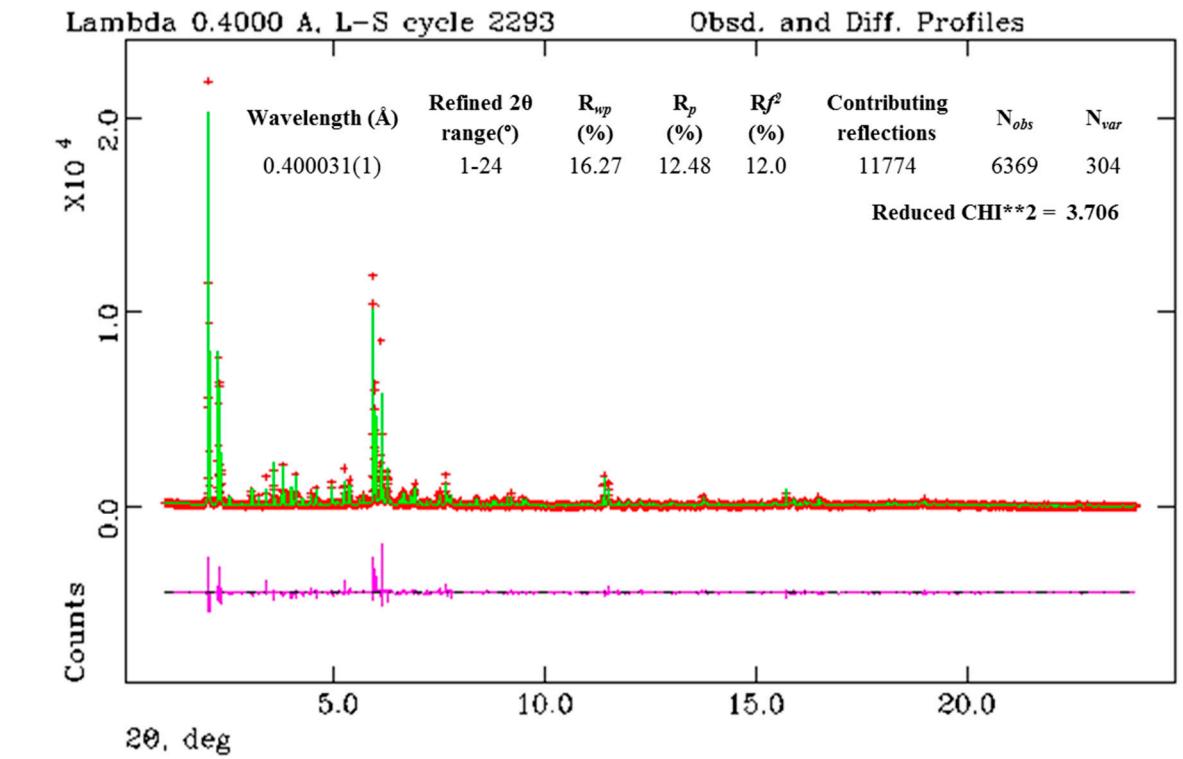


Figure S3. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 75 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

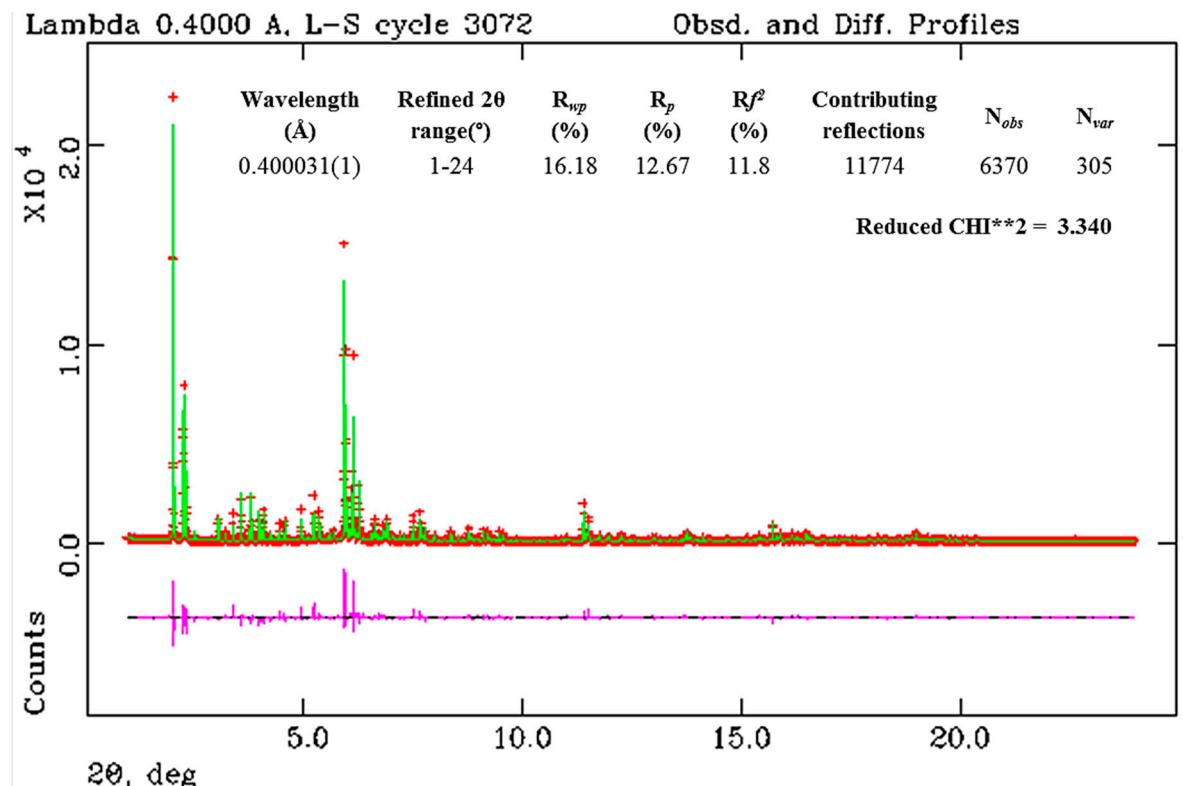


Figure S4. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 90 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe

the goodness of fit testing.

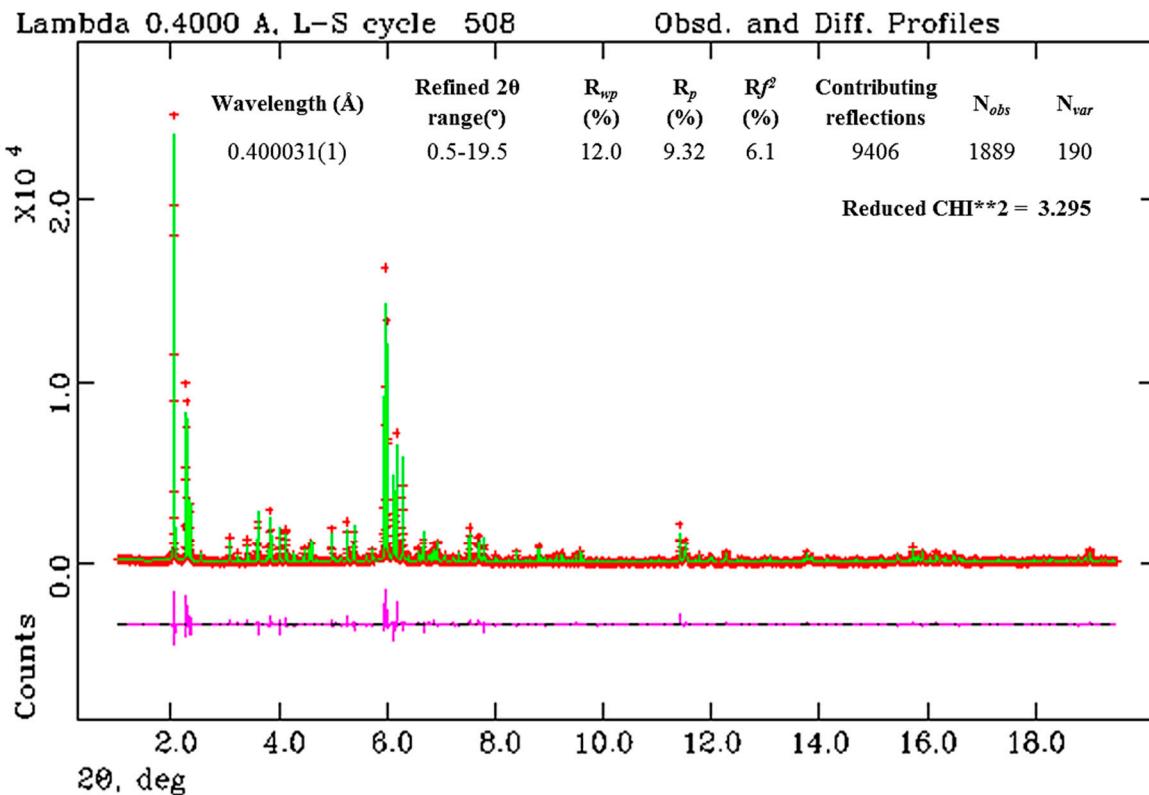


Figure S5. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 125°C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

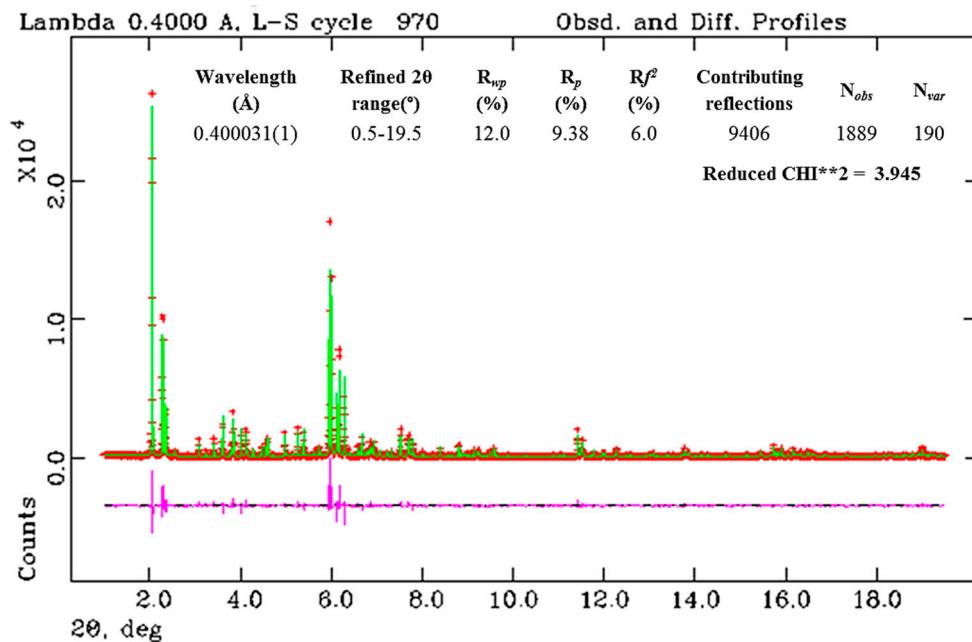


Figure S6. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 150 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

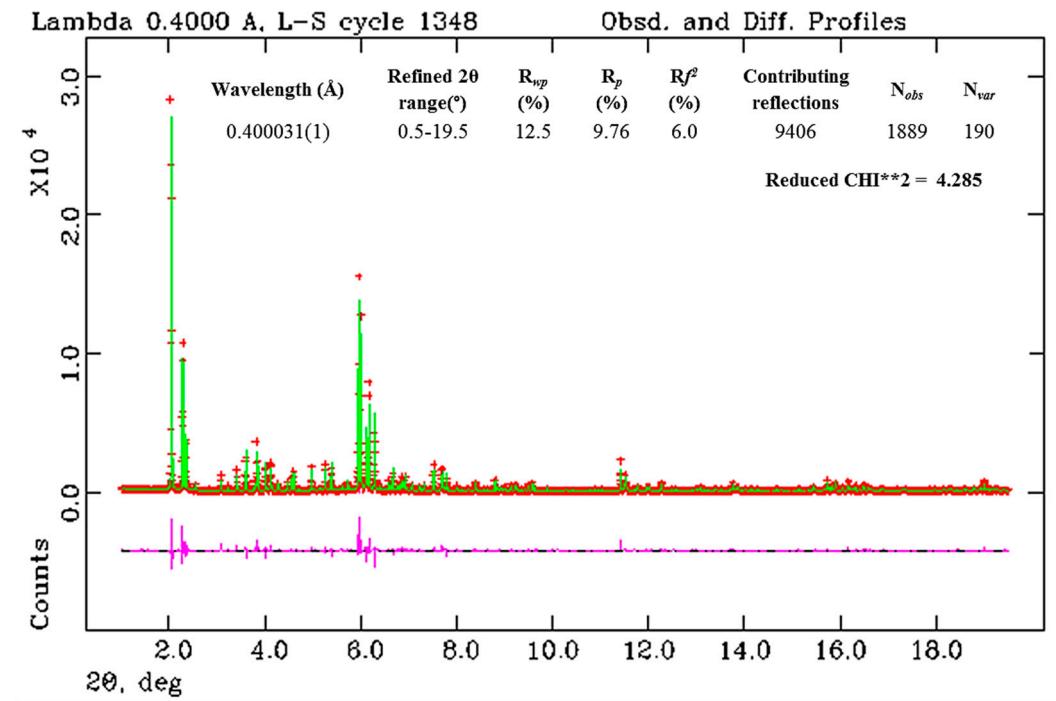


Figure S7. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 175 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

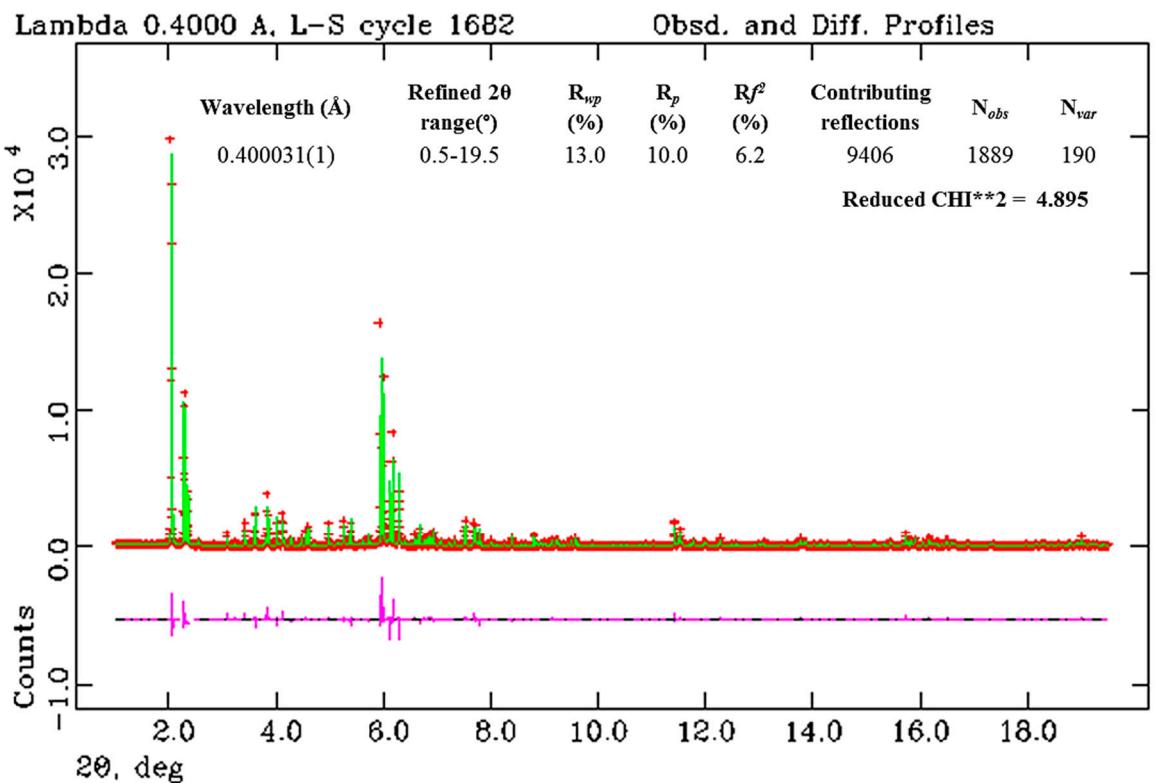


Figure S8. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 200 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

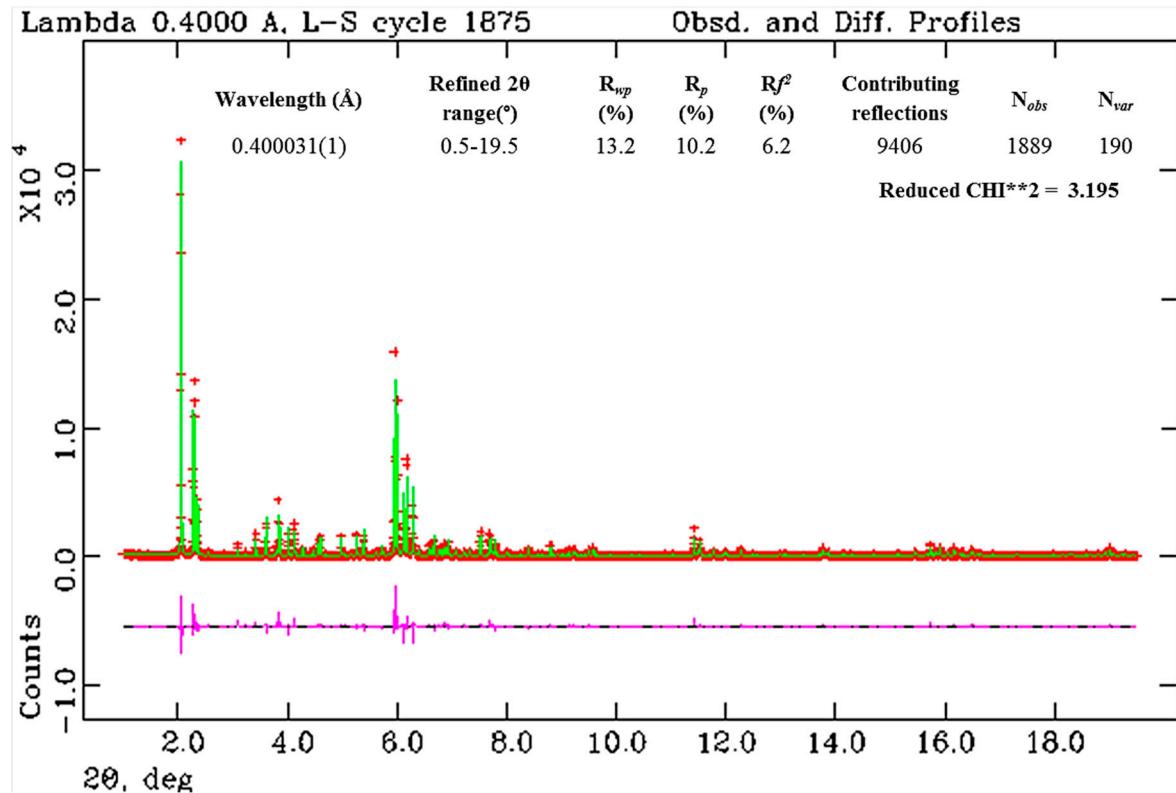


Figure S9. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 225 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

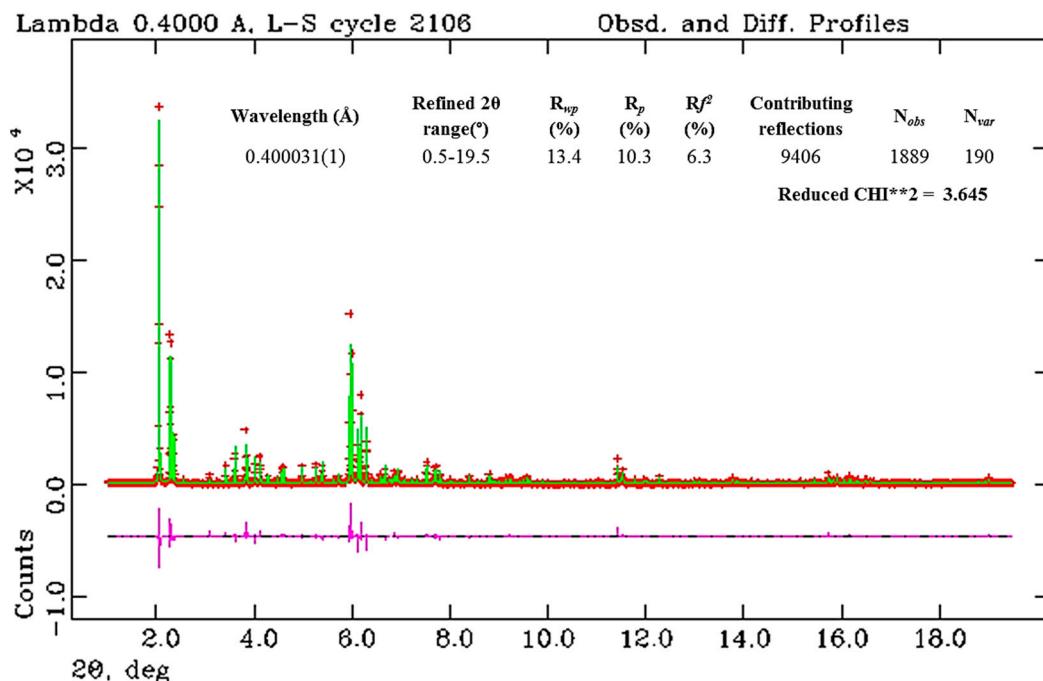


Figure S10. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 250 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

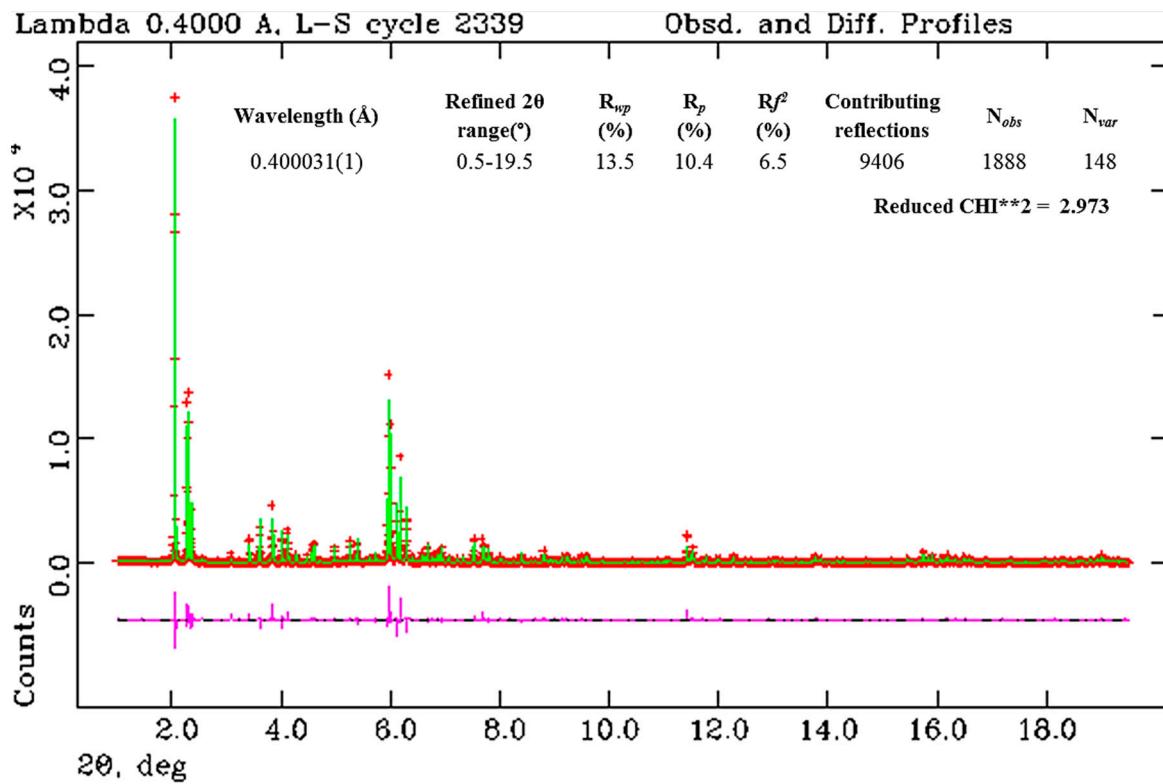


Figure S11. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 300 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

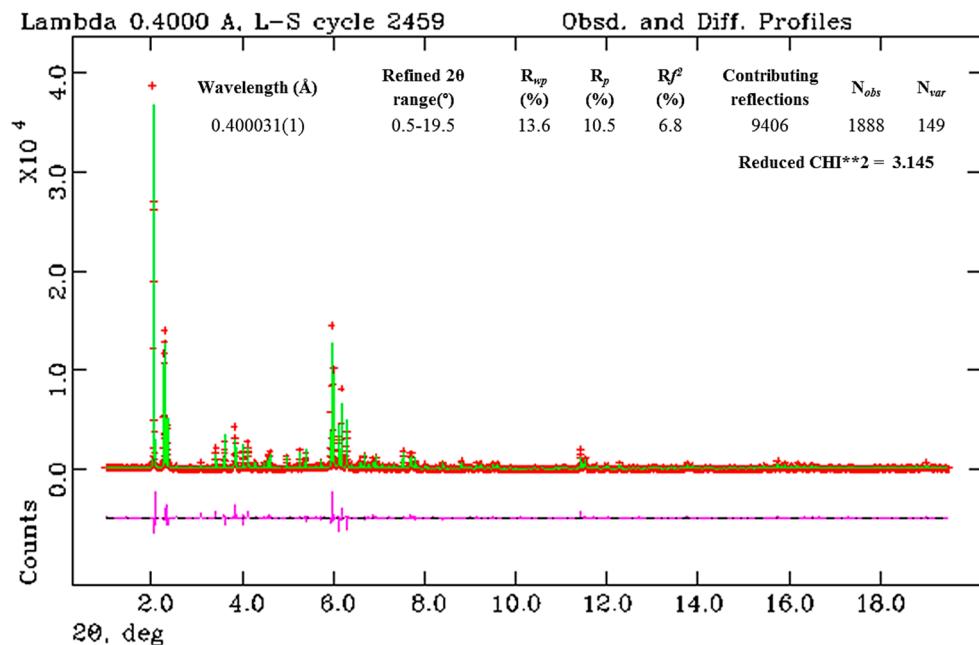


Figure S12. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 350 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI^{**2} : reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

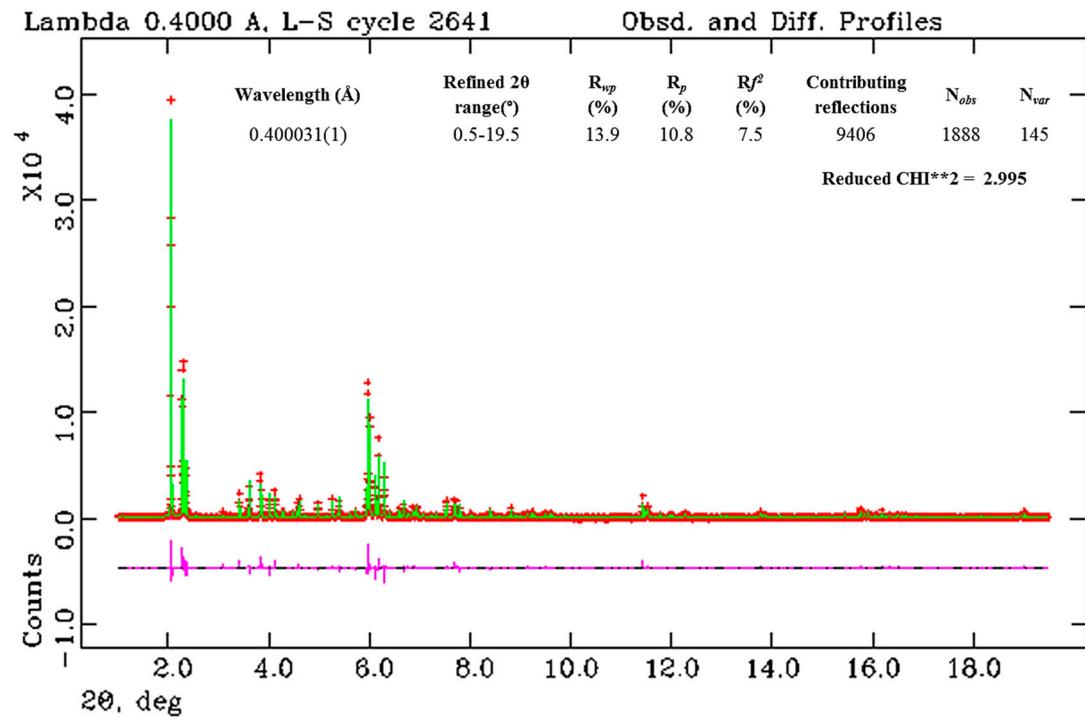


Figure S13. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 400 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

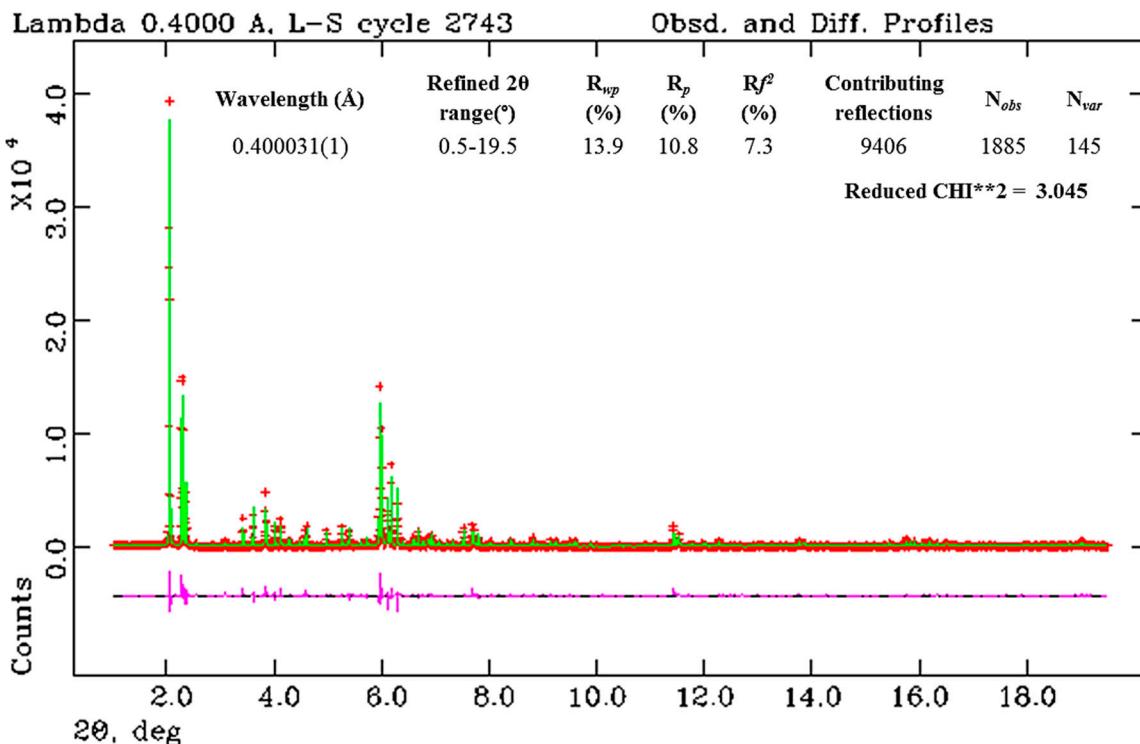


Figure S14. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 450 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

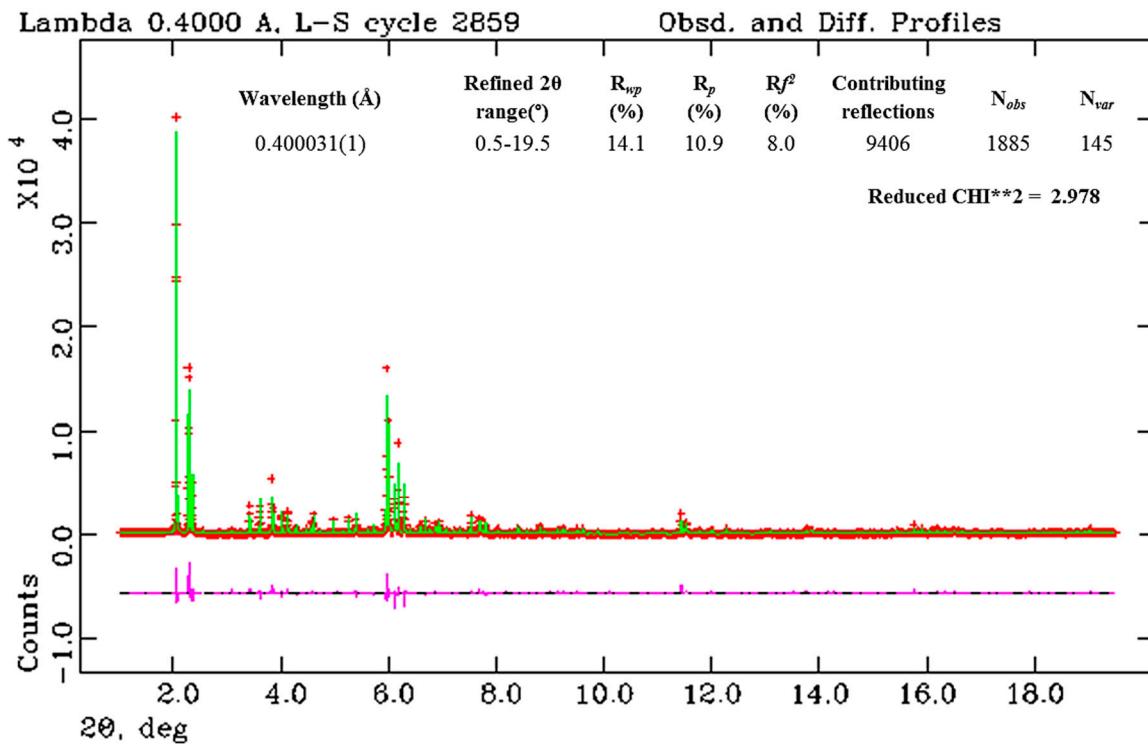


Figure S15. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 500 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

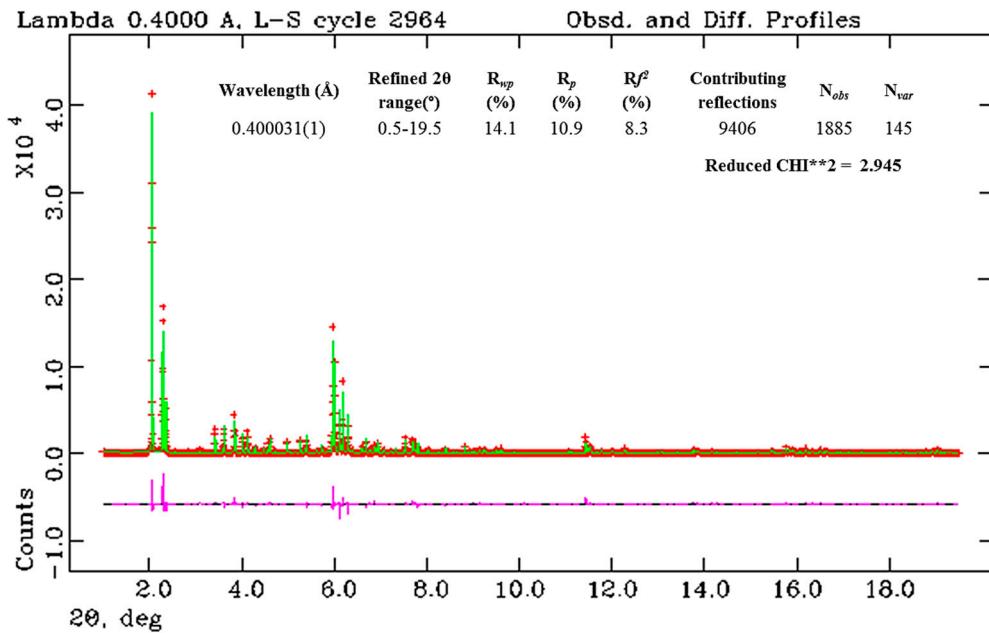


Figure S16. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 550 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

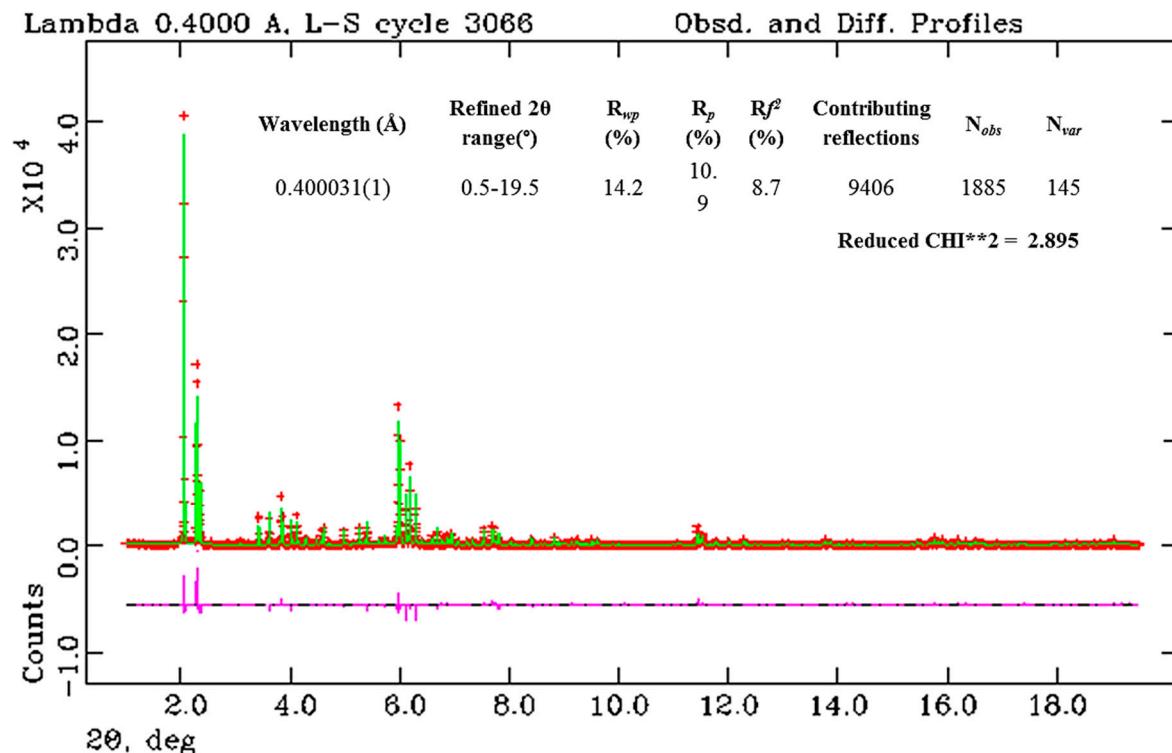


Figure S17. Observed (dotted upper line), calculated (solid upper line), and difference (solid lower line) powder diffraction pattern of ZSM-5 at 600 °C. Crystallographic data from the Rietveld refinement are also reported. Reduced CHI**2: reduced chi-squared statistic (χ^2_v) used to describe the goodness of fit testing.

Table S1. Fractional atomic coordinates of ZSM-5 loaded with MTBE at Room Temperature (30 °C).

Site	ZSM-5-MTBE 30 °C				
	x/a	y/b	z/c	Uiso	Fraction
T1	0.05422(24)	0.42299(20)	-0.32853(38)	0.0142(9)	1
T2	0.03289(24)	0.31648(24)	-0.16757(38)	0.0142(9)	1
T3	0.06535(21)	0.28079(22)	0.04463(42)	0.0142(9)	1
T4	0.06465(25)	0.12496(22)	0.03471(35)	0.0142(9)	1
T5	0.02811(23)	0.07270(24)	-0.17287(40)	0.0142(9)	1
T6	0.05738(21)	0.19424(23)	-0.30775(32)	0.0142(9)	1
T7	-0.17116(22)	0.42583(22)	-0.32245(39)	0.0142(9)	1
T8	-0.12479(24)	0.31179(24)	-0.17574(39)	0.0142(9)	1
T9	-0.17543(24)	0.26949(24)	0.03311(42)	0.0142(9)	1
T10	-0.17825(23)	0.11428(23)	0.03045(44)	0.0142(9)	1
T11	-0.12891(24)	0.06690(25)	-0.17525(45)	0.0142(9)	1
T12	-0.16457(24)	0.18877(23)	-0.30996(35)	0.0142(9)	1
T13	0.44273(23)	0.42431(22)	-0.33351(34)	0.0142(9)	1
T14	0.47104(18)	0.31072(23)	-0.18160(33)	0.0142(9)	1
T15	0.43780(15)	0.27990(21)	0.03490(24)	0.0142(9)	1
T16	0.43301(25)	0.12309(21)	0.03032(36)	0.0142(9)	1
T17	0.47292(24)	0.07354(23)	-0.18408(42)	0.0142(9)	1
T18	0.44110(22)	0.18896(21)	-0.31945(37)	0.0142(9)	1
T19	0.67263(21)	0.42165(23)	-0.31214(36)	0.0142(9)	1
T20	0.63190(22)	0.31124(25)	-0.16729(33)	0.0142(9)	1

T21	0.66960(23)	0.27186(26)	0.04872(40)	0.0142(9)	1
T22	0.67089(24)	0.11689(25)	0.03547(42)	0.0142(9)	1
T23	0.63030(25)	0.07300(27)	-0.18315(42)	0.0142(9)	1
T24	0.67949(26)	0.19301(25)	-0.30059(32)	0.0142(9)	1
O1	0.06146(38)	0.37608(32)	-0.23321(54)	0.0265(18)	1
O2	0.06726(31)	0.31902(29)	-0.05965(31)	0.0265(18)	1
O3	0.05498(38)	0.20338(18)	0.02473(74)	0.0265(18)	1
O4	0.06844(32)	0.09249(41)	-0.07377(35)	0.0265(18)	1
O5	0.04798(39)	0.12189(24)	-0.26137(47)	0.0265(18)	1
O6	0.05047(35)	0.24791(29)	-0.22068(44)	0.0265(18)	1
O7	-0.15563(43)	0.37305(34)	-0.23642(59)	0.0265(18)	1
O8	-0.16183(39)	0.30522(37)	-0.07123(52)	0.0265(18)	1
O9	-0.16226(52)	0.19167(19)	0.02132(75)	0.0265(18)	1
O10	-0.17055(29)	0.07977(38)	-0.07604(41)	0.0265(18)	1
O11	-0.15305(43)	0.11786(29)	-0.25968(57)	0.0265(18)	1
O12	-0.13460(40)	0.24524(34)	-0.23883(63)	0.0265(18)	1
O13	-0.04637(20)	0.32394(34)	-0.15701(68)	0.0265(18)	1
O14	-0.05068(19)	0.07744(42)	-0.15319(75)	0.0265(18)	1
O15	0.12231(27)	0.42139(32)	-0.39012(50)	0.0265(18)	1
O16	-0.00610(31)	0.39730(35)	-0.39772(64)	0.0265(18)	1
O17	-0.13576(28)	0.40270(40)	-0.42322(52)	0.0265(18)	1
O18	0.12970(22)	0.20008(44)	-0.35745(36)	0.0265(18)	1
O19	0.00057(26)	0.20709(33)	-0.39040(42)	0.0265(18)	1
O20	-0.12807(24)	0.19087(45)	-0.41556(42)	0.0265(18)	1
O21	0.04688(41)	-0.00138(21)	-0.20486(40)	0.0265(18)	1
O22	-0.14151(37)	-0.00732(23)	-0.21337(53)	0.0265(18)	1
O23	-0.25028(17)	0.43040(38)	-0.34101(49)	0.0265(18)	1
O24	-0.24293(21)	0.20141(35)	-0.32639(24)	0.0265(18)	1
O25	-0.25142(18)	0.28204(53)	0.06530(54)	0.0265(18)	1
O26	-0.25308(18)	0.10506(50)	0.06956(52)	0.0265(18)	1
O27	0.44229(37)	0.37445(29)	-0.24093(46)	0.0265(18)	1
O28	0.45256(29)	0.31606(26)	-0.06799(25)	0.0265(18)	1
O29	0.43035(40)	0.20195(17)	0.01880(42)	0.0265(18)	1
O30	0.44315(49)	0.09043(38)	-0.07694(45)	0.0265(18)	1
O31	0.43670(39)	0.11796(22)	-0.26704(59)	0.0265(18)	1
O32	0.44854(34)	0.24521(28)	-0.23598(42)	0.0265(18)	1
O33	0.66735(28)	0.37392(35)	-0.21734(50)	0.0265(18)	1
O34	0.64609(41)	0.31062(27)	-0.04980(28)	0.0265(18)	1
O35	0.65421(49)	0.19438(21)	0.03639(79)	0.0265(18)	1
O36	0.66041(43)	0.08847(49)	-0.07489(39)	0.0265(18)	1
O37	0.66624(43)	0.11901(27)	-0.26347(52)	0.0265(18)	1
O38	0.66036(41)	0.24441(33)	-0.21484(38)	0.0265(18)	1
O39	0.55290(16)	0.31543(44)	-0.18834(62)	0.0265(18)	1
O40	0.55165(21)	0.08823(35)	-0.18408(87)	0.0265(18)	1
O41	0.37281(25)	0.41915(38)	-0.39216(48)	0.0265(18)	1
O42	0.50237(30)	0.40541(40)	-0.40683(50)	0.0265(18)	1
O43	0.63233(29)	0.39012(36)	-0.40436(43)	0.0265(18)	1

O44	0.37444(27)	0.20140(39)	-0.38382(62)	0.0265(18)	1
O45	0.50498(26)	0.19123(36)	-0.39040(48)	0.0265(18)	1
O46	0.63497(18)	0.20763(40)	-0.39774(31)	0.0265(18)	1
O47	0.46054(32)	-0.00312(17)	-0.20787(47)	0.0265(18)	1
O48	0.64278(38)	-0.00319(23)	-0.20991(57)	0.0265(18)	1
MTBE1					
Site	x/a	y/b	z/c	Uiso	Frac
C1a	0.7424	0.7197	0.0722	0.121	0.419
C2a	0.7470	0.6937	0.1733	0.121	0.419
C3a	0.7691	0.6740	0.0002	0.121	0.419
C4a	0.7733	0.7849	0.0656	0.121	0.419
O1a	0.6695	0.7209	0.0519	0.121	0.419
C6a	0.6210	0.7564	0.1131	0.121	0.419
MTBE2					
Site	x/a	y/b	z/c	Uiso	Frac
C1b	0.7413	0.5648	0.1387	0.197	0.796
C2b	0.7566	0.4946	0.1307	0.197	0.796
C3b	0.1765	0.5163	-0.0915	0.197	0.796
O1b	0.7051	0.4679	0.0647	0.197	0.796
C4b	0.7473	0.4604	0.2249	0.197	0.796
C5b	0.6839	0.5074	-0.0157	0.197	0.796
W					
Site	x/a	y/b	z/c	Uiso	Frac
W	0.1468	0.5186	-0.0023	0.250	0.5

Table S2. Fractional atomic coordinates of ZSM-5 loaded with MTBE at 100 °C

Site	ZSM-5-MTBE 100 °C			
	x/a	y/b	z/c	Uiso
T1	0.22697(24)	-0.17189(16)	0.03435(32)	0.0181(7)
T2	-0.07688(20)	-0.05589(21)	0.33995(30)	0.0181(7)
T3	0.19088(24)	-0.12981(21)	-0.18254(33)	0.0181(7)
T4	0.07116(23)	0.12925(23)	0.31793(36)	0.0181(7)
T5	-0.07587(22)	0.17191(15)	0.32380(32)	0.0181(7)
T6	0.21965(20)	0.06216(21)	0.02932(29)	0.0181(7)
T7	0.12260(20)	-0.06602(21)	0.52457(32)	0.0181(7)
T8	-0.11859(24)	-0.17388(16)	0.47035(35)	0.0181(7)
T9	0.30902(24)	-0.03007(21)	0.31059(31)	0.0181(7)
T10	0.18619(21)	-0.05830(23)	0.17352(34)	0.0181(7)
T11	0.18755(24)	0.17202(16)	0.18192(33)	0.0181(7)
T12	0.07154(22)	-0.02829(22)	0.31571(32)	0.0181(7)
O1	-0.11000(48)	-0.250000	0.43509(48)	0.0162(1)
O2	0.11447(22)	-0.05243(32)	0.22289(36)	0.0162(1)
O3	0.19663(33)	0.12961(23)	0.08259(36)	0.0162(1)
O4	0.19159(50)	0.250000	0.15546(65)	0.0162(1)
O5	0.19252(33)	-0.12980(22)	0.12099(35)	0.0162(1)

O6	-0.00486(18)	-0.04360(31)	0.29420(35)	0.0162(1)
O7	0.21780(45)	-0.250000	0.05639(61)	0.0162(1)
O8	0.24141(26)	-0.05163(43)	0.25791(52)	0.0162(1)
O9	-0.00469(22)	0.14406(37)	0.29226(42)	0.0162(1)
O10	0.19538(36)	-0.00083(25)	0.09255(49)	0.0162(1)
O11	0.11676(26)	0.15599(42)	0.22954(39)	0.0162(1)
O12	-0.07808(27)	-0.12600(18)	0.39749(40)	0.0162(1)
O13	0.31188(29)	-0.05939(35)	0.42039(31)	0.0162(1)
O14	-0.12978(27)	-0.05781(34)	0.25214(39)	0.0162(1)
O15	0.30454(20)	-0.15453(36)	0.03169(55)	0.0162(1)
O16	0.09435(29)	-0.06679(30)	0.41359(30)	0.0162(1)
O17	-0.10124(31)	0.13341(23)	0.42042(38)	0.0162(1)
O18	0.19412(31)	-0.15396(35)	-0.06973(28)	0.0162(1)
O19	0.08153(33)	0.05044(18)	0.33099(58)	0.0162(1)
O20	-0.07044(48)	0.250000	0.34780(55)	0.0162(1)
O21	-0.12669(30)	0.16071(36)	0.23438(44)	0.0162(1)
O22	-0.09407(29)	0.00295(23)	0.41618(44)	0.0162(1)
O23	0.24436(31)	0.15376(43)	0.25959(49)	0.0162(1)
O24	0.09087(35)	0.16622(27)	0.41891(33)	0.0162(1)
O25	0.29847(16)	0.06129(31)	0.02097(50)	0.0162(1)
O26	0.31310(47)	0.04987(17)	0.31466(61)	0.0162(1)
		MTBE1		
Site	x/a	y/b	z/c	Uiso
C1a	-0.0397	-0.2500	0.0789	0.264
C2a	-0.1056	-0.2500	0.1220	0.264
C3a	0.0093	-0.2500	0.1577	0.264
O1a	-0.0312	-0.1907	0.0179	0.264
C5a	0.0229	-0.1478	0.0033	0.264
		MTBE2		
Site	x/a	y/b	z/c	Uiso
C1b	0.0509	-0.2500	0.2414	0.172
O1b	0.1064	-0.2500	0.3108	0.172
C3b	0.4885	0.7500	0.2039	0.172
C4b	0.6083	0.7499	0.0846	0.172
C5b	0.5534	0.6903	0.3206	0.172
		W		
Site	x/a	y/b	z/c	Uiso
W	-0.0383	-0.1216	0.0339	0.151
				Frac
				0.223

Table S3. Fractional atomic coordinates of ZSM-5 loaded with MTBE at 400 °C.

Site	ZSM-5-MTBE 400°C			
	x/a	y/b	z/c	Uiso
T1	0.22798(26)	-0.17153(17)	0.03597(37)	0.0208(8)
T2	-0.07681(22)	-0.05705(23)	0.33738(33)	0.0208(8)
T3	0.19023(27)	-0.12897(24)	-0.18048(37)	0.0208(8)

T4	0.07105(26)	0.12979(26)	0.31797(41)	0.0208(8)
T5	-0.07622(25)	0.17196(17)	0.32202(36)	0.0208(8)
T6	0.21940(22)	0.06105(24)	0.03252(33)	0.0208(8)
T7	0.12273(22)	-0.06485(24)	0.52745(36)	0.0208(8)
T8	-0.11800(27)	-0.17376(18)	0.47001(40)	0.0208(8)
T9	0.31067(27)	-0.03041(23)	0.31324(35)	0.0208(8)
T10	0.18786(24)	-0.05953(26)	0.17800(37)	0.0208(8)
T11	0.18831(27)	0.17205(18)	0.18359(38)	0.0208(8)
T12	0.07146(25)	-0.02775(25)	0.31720(36)	0.0208(8)
O1	-0.11001(54)	-0.250000	0.43464(56)	0.0397(1)
O2	0.11585(25)	-0.05407(35)	0.22713(43)	0.0397(1)
O3	0.19726(38)	0.12912(26)	0.08485(41)	0.0397(1)
O4	0.19421(55)	0.250000	0.15730(77)	0.0397(1)
O5	0.19453(37)	-0.13087(25)	0.12500(38)	0.0397(1)
O6	-0.00462(21)	-0.04336(34)	0.29273(38)	0.0397(1)
O7	0.21906(52)	-0.250000	0.05436(74)	0.0397(1)
O8	0.24251(30)	-0.05303(49)	0.26337(56)	0.0397(1)
O9	-0.00487(24)	0.14430(42)	0.29099(48)	0.0397(1)
O10	0.19711(40)	-0.00147(28)	0.09812(54)	0.0397(1)
O11	0.11694(30)	0.15750(48)	0.23038(45)	0.0397(1)
O12	-0.07690(30)	-0.12646(21)	0.39669(44)	0.0397(1)
O13	0.31428(30)	-0.05681(41)	0.42501(36)	0.0397(1)
O14	-0.12895(31)	-0.06099(36)	0.24865(45)	0.0397(1)
O15	0.30543(22)	-0.15375(42)	0.03185(63)	0.0397(1)
O16	0.09210(31)	-0.06481(36)	0.41763(35)	0.0397(1)
O17	-0.10233(35)	0.13249(26)	0.41726(44)	0.0397(1)
O18	0.19394(34)	-0.15120(41)	-0.06672(32)	0.0397(1)
O19	0.08168(37)	0.05100(21)	0.32988(70)	0.0397(1)
O20	-0.07070(53)	0.250000	0.34789(61)	0.0397(1)
O21	-0.12658(35)	0.16215(38)	0.23144(52)	0.0397(1)
O22	-0.09580(33)	0.00210(27)	0.41172(51)	0.0397(1)
O23	0.24439(36)	0.15270(48)	0.26245(55)	0.0397(1)
O24	0.08968(40)	0.16642(31)	0.41954(36)	0.0397(1)
O25	0.29823(17)	0.06054(35)	0.02009(54)	0.0397(1)
O26	0.31568(52)	0.04935(19)	0.31283(70)	0.0397(1)