Triclinic Labs

Data Summary

XRPD Measurements for a Clinoptilolite Zeolite

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Overview

Two clinoptilolite zeolite powder samples were received from KMI Zeolite Inc. for X-ray powder diffraction (XRPD) measurement under standard conditions. A single aliquot was taken from one of the samples and mounted using a low background silicon sample holder. The powder was placed into the holder to achieve a flat and level surface. Information concerning the samples are presented in Table 1.

Table 1. Samples and Materials as Provided

TCL#	Sample
TCL6923	clinoptilolite zeolite
TCL6924	clinoptilolite zeolite

X-ray Powder Diffraction (XRPD) Parameters

Instrument measurement parameters and resulting XRPD data are shown in Tables 2 and 3.

Table 2. XRPD Parameters for Rigaku SmartLab system

Parameters	Value
Geometry	Reflection B-B
X-ray Tube	copper
Monochromatization	beta-filter
Detector	D'teX PSD
Voltage (kV)	40.00
Current (mA)	44.00
Start Angle (2θ)	2.00
End Angle (2θ)	80.00
Step Size (2θ)	0.02
Scan Speed (2θ/min)	1.00
Slits (S0deg, S1deg, S3mm)	1/3, 4, 13
Measurement Type	symmetric θ:2θ
Sample Holder	Si low background
Sample Rotation (RPM)	50.00

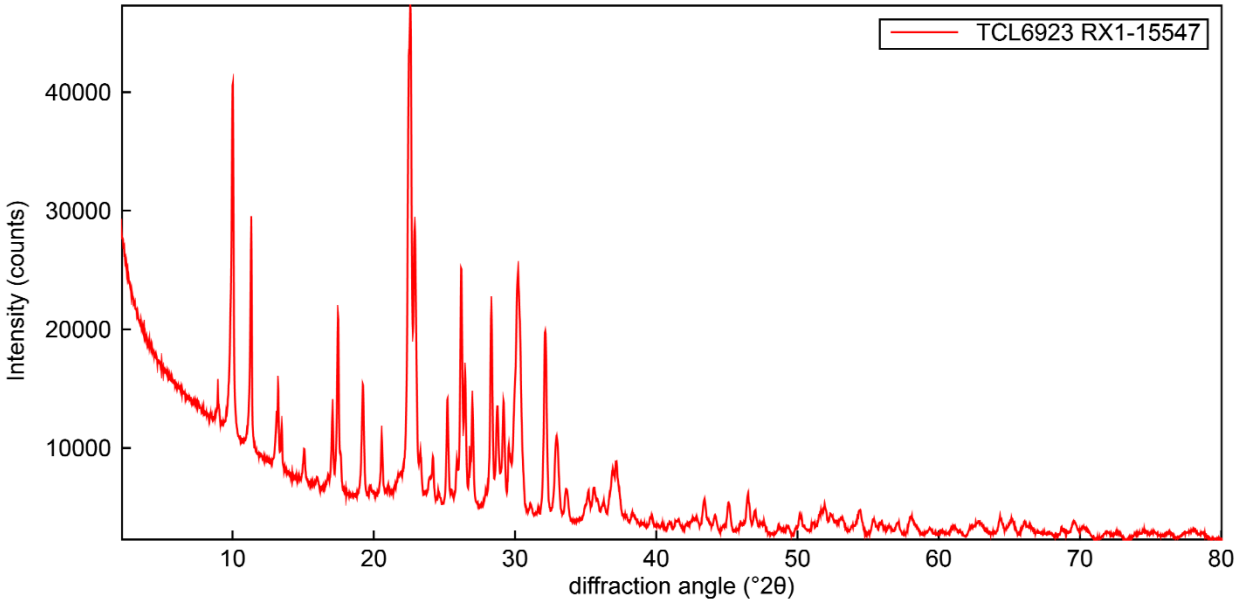
Table 3. XRPD Data Collected for General Powder Measurement

TCL#	Sample	XRPD#
TCL6923	clinoptilolite zeolite	RX1-15547

Measurements

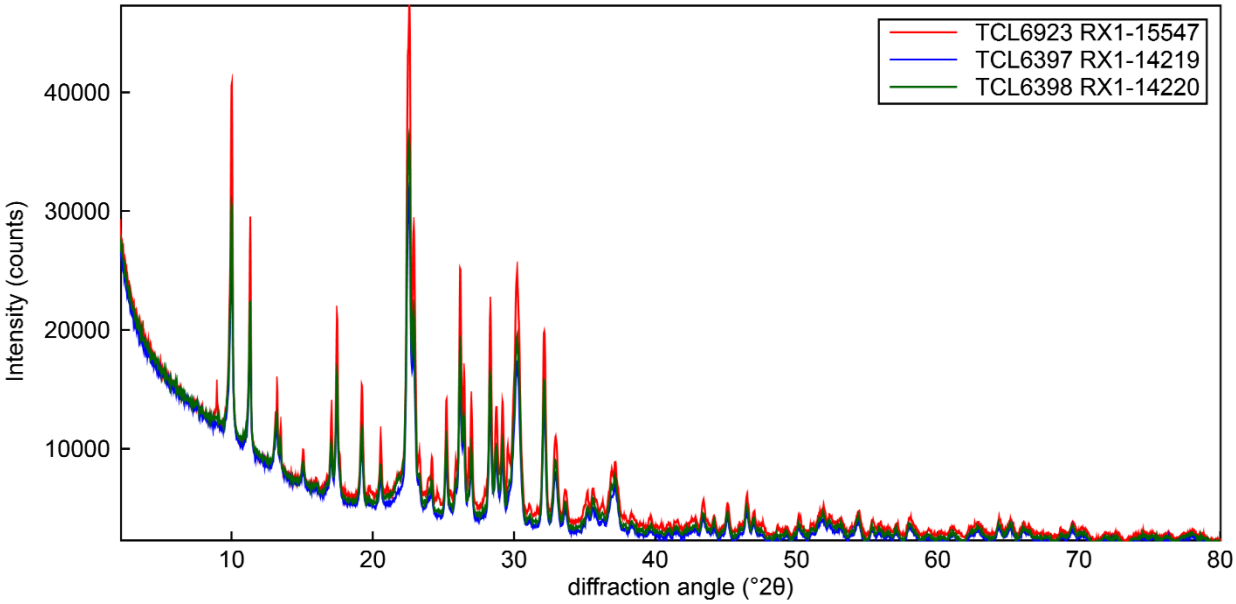
Data as measured displayed in Figure 1.

Figure 1: Measured XRPD data for powder sample.



In Figure 2, the data are compared with two previous measurements of clinoptilolite zeolite samples (taken from Triclinic Labs report to KMI Zeolite R2017116.02).

Figure 2: Measured XRPD data for powder sample compared with previous data



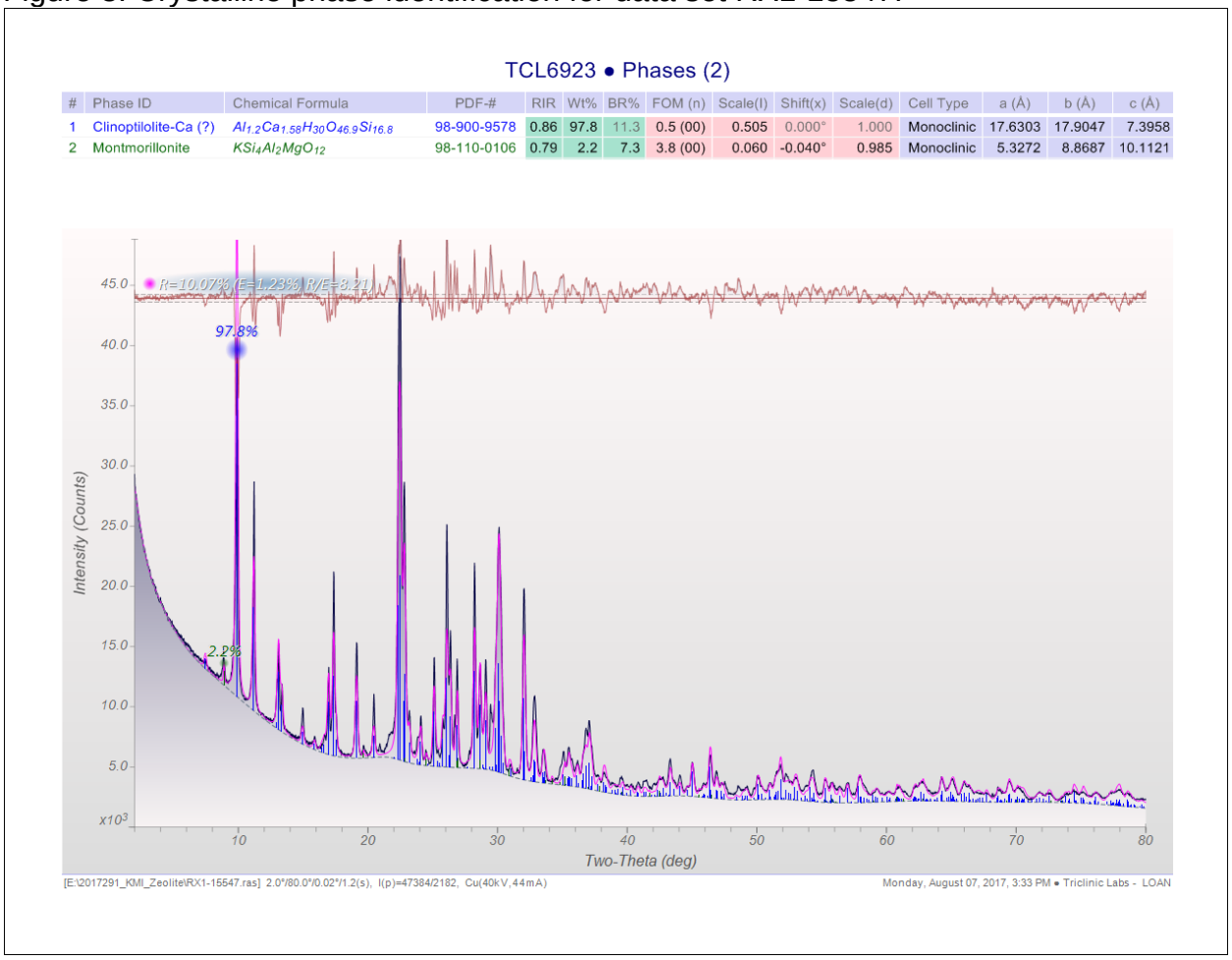
Visual inspection of the data overlay in Figure 2 indicates that the new powder sample is essentially the same as the previous samples with respect to the X-ray powder diffraction response. Some addition weak peaks were observed in the latest XRPD data.

Analysis

Phase Identification

A crystalline peak search was performed for the observed data file and the resulting peak list matched against the COD crystal structure data base using the software MDI-Jade2010 (V 5.0.0). The crystalline phase identification results are presented in Figure 3. Clinoptilolite (PDF# 98-900-9578) was identified as being the primary crystalline phase with Montmorillonite (PDF# 98-110-0106) identified as a possible minor phase.

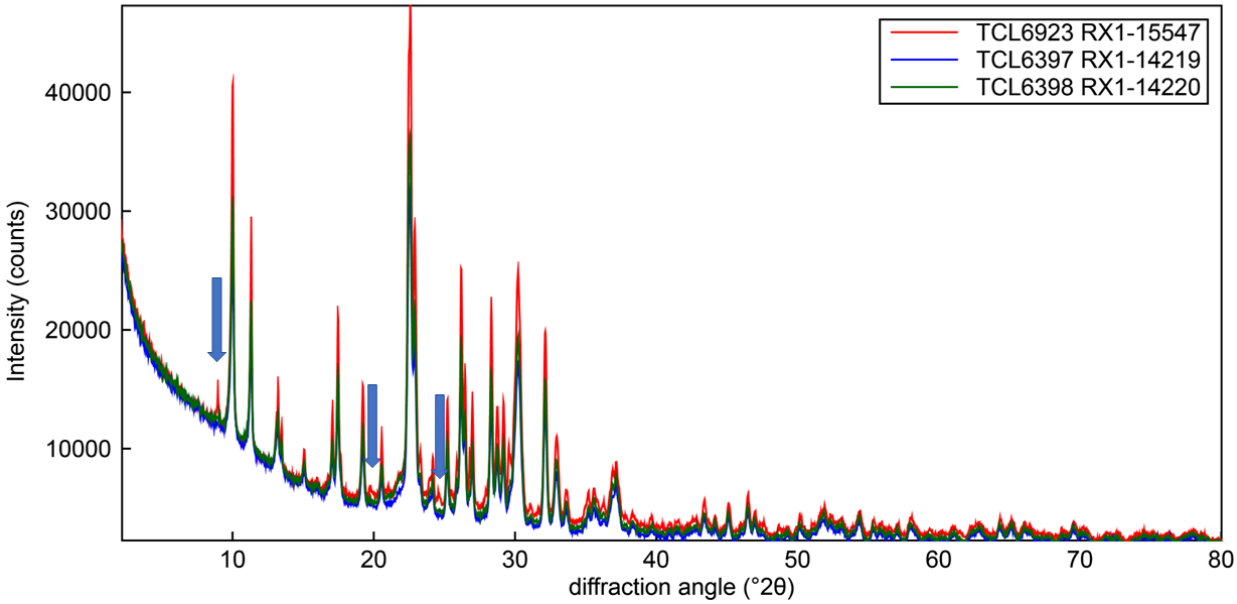
Figure 3: Crystalline phase identification for data set RX1-15547.



Semi-Quantitative analysis

Phase identification returned clinoptilolite-Ca as the primary crystalline phase with montmorillonite being a potential minor crystalline phase. A significant majority of the observed peaks are associated with the clinoptilolite crystalline form. Three small peaks not identified by clinoptilolite are described by the mineral phase montmorillonite; see Figure 4.

Figure 4: Small unidentified peaks described by the montmorillonite mineral



A previous X-ray diffraction study performed on similar clinoptilolite samples (LUCAS report to KMI Zeolite, 2010) had identified the presence of the mineral form Phlogopite as a minor phase along with Clinoptilolite. One of the phlogopite crystalline forms describes the unidentified low angle peak assigned to montmorillonite by the JADE search engine. An additional mineral phase that may appear along with the clinoptilolite-heulandite zeolites is Stilbite. To determine whether these mineral forms were present along with the clinoptilolite and to determine whether the unidentified peaks were best described by montmorillonite or phlogopite, a Rietveld semi-quantitative analysis was performed using Jade2010. For each Rietveld analysis, the crystalline minerals Clinoptilolite-Ca, Montmorillonite, Phlogopite and Stilbite-Ca were included within the starting model. The results of the Rietveld semi-quantitative analysis are presented in Figure 5.

For routine XRPD analysis, the detection limit for crystalline phases using Rietveld semi-quantitative analysis is generally around 1% w/w. For data set RX1-15547, the Rietveld analysis returned quantitative values for both Phlogopite and Stilbite-Ca that were below detection limits. The semi-quantitative results are summarized in Table 4.

Table 4. Results of Rietveld semi-quantitative analysis

TCL#	Sample	XRPD#	Mineral phase	Quantity (w/w%)
TCL6923	clinoptilolite zeolite	RX1-15547	clinoptilolite-Ca	96.90%
			montorillonite	2.30%
			phlogopite	<< 1%
			stilbite-Ca	<< 1%

Figure 5: Rietveld semi-quantitative WPF analysis for data set RX1-15547.