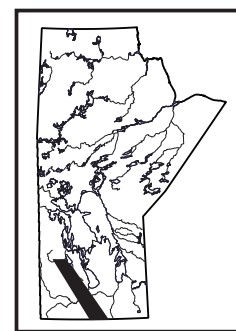


GS-16 Geochronology of Late Cretaceous bentonite beds in southwestern Manitoba: 2016 update

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Summary

This paper summarizes the results of U-Pb age determinations of zircon grains from Upper Cretaceous bentonite beds from various lithological units in southwestern Manitoba. The study was undertaken to resolve cross-sectional modelling issues and provincial ‘boundary faults’ that result from the time-transgressive nature of many stratigraphic units in the Western Canada Sedimentary Basin. The chronostratigraphic framework of the Late Cretaceous in southwestern Manitoba now has five ages for the following formations and members:

- 67.2 ±0.4 Ma for the Odanah Member of the Pierre Shale;
- 80.04 ±0.11 Ma for the Pembina Member of the Pierre Shale;
- 81.5 ±0.5 Ma for the Boyne Member of the Carlile Formation;
- 92.54 ±0.28 Ma for the Assiniboine Member of the Favel Formation; and
- 96.0 ±0.2 Ma for the Belle Fourche Member of the Ashville Formation.

The objective of a simplified and standardized nomenclature (for use across western Canada and the western United States) is to make target selection easier and less expensive for petroleum and industrial mineral exploration.

Introduction

In April 2012, the Manitoba Geological Survey (MGS) initiated a Phanerozoic geochronology project to determine the age of bentonite beds. The objective of the project is to help resolve cross-sectional modelling discrepancies and provincial ‘boundary faults’ that result from stratigraphic units that are time transgressive from west to east across the Western Canada Sedimentary Basin (Bamburak et al., 2013). The sampled bentonite beds (including two sampled prior to the start of the project) are present in the Upper Cretaceous Belle Fourche Member of the Ashville Formation upward to the Odanah Member of the Pierre Shale (Figure GS-16-1). The beds were formed from altered volcanic ash (derived

from volcanic eruptions in the Elkhorn Mountains of western Montana), which was deposited within the former Western Interior Seaway in southwestern Manitoba (Gill and Cobban, 1965, p. A7; Bannatyne and Watson, 1982, p. 45).

Previous work

Bamburak et al. (2013) documented the radiometric dating of two bentonite samples from the Cretaceous shale present in southwestern Manitoba. A bentonite bed near the top of the Boyne Member of the Carlile Formation (sample 99-11-MI-004, Figure GS-16-1) at Spencer’s ditch (Figure GS-16-2) yielded a U-Pb zircon age of 81.5 ±0.5 Ma (Table GS-16-1), and a bentonite bed near the base of the Pembina Member of the Pierre Shale (sample 99-11-MI-001-002, Figure GS-16-1) at Mount Nebo was dated at 78.8 ±1.0 Ma (Bamburak and Heaman, 2016³). However, it is possible that the latter sample may have been contaminated and that the date is incorrect. Subsequent geochronological studies of similar strata have produced significantly different ages, and the Mount Nebo site is known to have been disturbed by quarrying in the past. This is further discussed in the ‘Sample 99-13-WA-001’ section.

U-Pb age results (2014–2016)

The following six Late Cretaceous bentonite samples (collected in 2012–2013) underwent mineral separation, processing and analysis at the University of Alberta (UofA) Radiogenic Isotope Facility in Edmonton, Alberta (described in Bamburak et al., 2013), or, beginning in 2015, at the University of Toronto (UofT) Jack Satterly Geochronology Laboratory, Toronto, Ontario. The latter facility used generally the same methods as the UofA, beginning with crushing, pulverizing, sorting, magnetic separation and heavy liquid techniques. First, a portion of the bentonite sample was immersed in water to form a slurry. The slurry was then placed in an industrial blender to reduce the grain size to that suitable for shaking on a Wilfley table. The resultant heavy mineral concentrate

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³ MGS Data Repository Item DRI2016008, containing the data or other information sources used to compile this report, is available online to download free of charge at <http://www2.gov.mb.ca/itm-cat/web/freedownloads.html>, or on request from minesinfo@gov.mb.ca or Mineral Resources Library, Manitoba Growth, Enterprise and Trade, 360–1395 Ellice Avenue, Winnipeg, MB R3G 3P2, Canada.

ERA	PERIOD	SOUTHWESTERN MANITOBA			
MESOZOIC	CRETACEOUS	Boissevain Formation			
		Coulter Member			
		Pierre Shale	Odanah Member	99-13-TR-001A	
			Millwood Member		
			99-13-WA-001; 99-13-MO-001A Pembina Member	99-11-MI-001-002	
			Gammon Ferruginous Member	99-13-MO-002	
			Swan River Formation		
		Carille Formation	Boyne Member	99-11-MI-004	
			Morden Member		
		Favel Formation	Assiniboine Member	99-13-OR-002	
			Keld Member		
		Ashville Formation	upper	Belle Fourche Member	99-12-WR-001C
				Fish Scale Zone	Base of Fish Scale marker
			lower	Westgate Member	
				Newcastle Member	
				Skull Creek Member	
		Swan River Formation			

Figure GS-16-1: Cretaceous stratigraphy of southwestern Manitoba with bentonite samples collected from 2011 to 2013 (modified from Nicolas and Bamburak, 2009).

was dried and magnetically separated. This was followed by heavy liquid (methylene iodide) separation to recover the zircon grains.

At the UofT, zircon separates underwent conventional U-Pb dating by isotope dilution–thermal ionization mass spectrometry (ID-TIMS). The grains were dissolved individually in a HF-HNO₃ solution at 195°C for 120 hours and then an anion exchange chromatography procedure was used to isolate Pb and U from the dissolved zircon samples (Krogh, 1973). The purified sample-spike solutions were loaded onto single Re filaments with silica gel and the isotopic ratios were measured with a Micro-mass VG 354 Solid Source mass spectrometer using a Daly detector in pulse-counting mode.

Sample 99-12-WR-001C

Sample 99-12-WR-001C (Figure GS-16-1) was collected from the X-bentonite bed within the Belle Fourche Member of the Ashville Formation along the Wilson River, about 1 km southwest of Ashville (Figure GS-16-3). The sample location is situated in L.S. 14, Sec. 14, Twp. 25, Rge. 21, W 1st Mer. (abbreviated 14-14-25-21W1; UTM NAD83, Zone 14U, 408242E, 5669335N;

NTS northwest corner of 62N1). The X-bentonite (in association with the *Ostrea beloiti* beds) was recognized by McNeil and Caldwell (1981, p. 50, 51, 79) as one of the most widespread isochronous lithostratigraphic, biostratigraphic and chronostratigraphic markers in the entire Western Interior Seaway.

The sample was processed at the UofA Radiogenic Isotope Facility. Although preliminary visual analysis of two very tiny zircons in 2013 had indicated that they were of excellent quality (Bamburak et al., 2013), they were not used for radiometric dating, probably due to their small size. Instead four other zircons were identified, which provided dates of 82.3 ±0.4, 91.9 ±0.9, 95.9 ±0.2 and 96.1 ±0.2 Ma (Bamburak and Heaman, 2016). The stratigraphic position of the X-bentonite bed in the Belle Fourche Member of the Ashville Formation shows that the first two dates are inaccurate. Erosion along the stream bank at this sample site may have led to sample contamination. However, the latter two dates, which average 96.0 ±0.2 Ma (Table GS-16-1), show good correspondence with the 95.87 ±0.10 Ma age for the X-bentonite bed sampled within the Sunkay Member of the Blackstone Formation at site 3, Burnt Timber Creek in Alberta

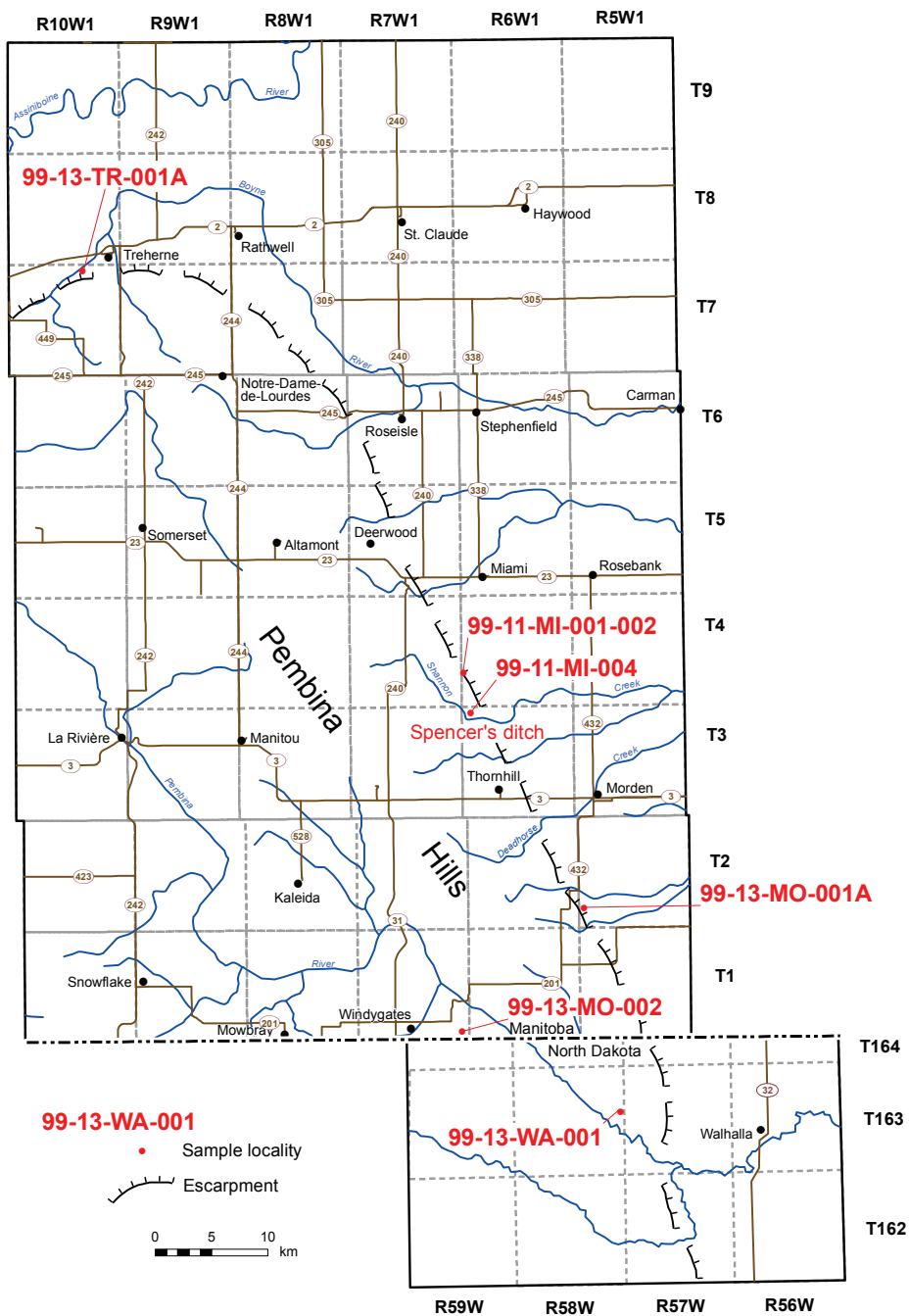


Figure GS-16-2: Bentonite sample localities in the Pembina Hills area of southwestern Manitoba.

Table GS-16-1: The U-Pb zircon age summary of bentonite samples collected in southwestern Manitoba from 2011 to 2013.

Formation	Member	Bed	Bentonite	Sample	Location	Age
Pierre Shale	Odanah			99-13-TR-001A	Treherne quarry	67.2 ±0.4 Ma
Pierre Shale	Pembina		Q3	99-13-WA-001	Pembina gorge roadcut	80.04 ±0.11 Ma
Carlile	Boyne	Chalky		99-11-MI-004	Spencer's ditch	81.5 ±0.5 Ma
Favel	Assiniboine	Marco Calcarenite	Uppermost thick	99-13-OR-002	Ochre River streamcut	92.54 ±0.28 Ma
Ashville	Belle Fourche		X	99-12-WR-001C	Wilson River	96.0 ±0.2 Ma

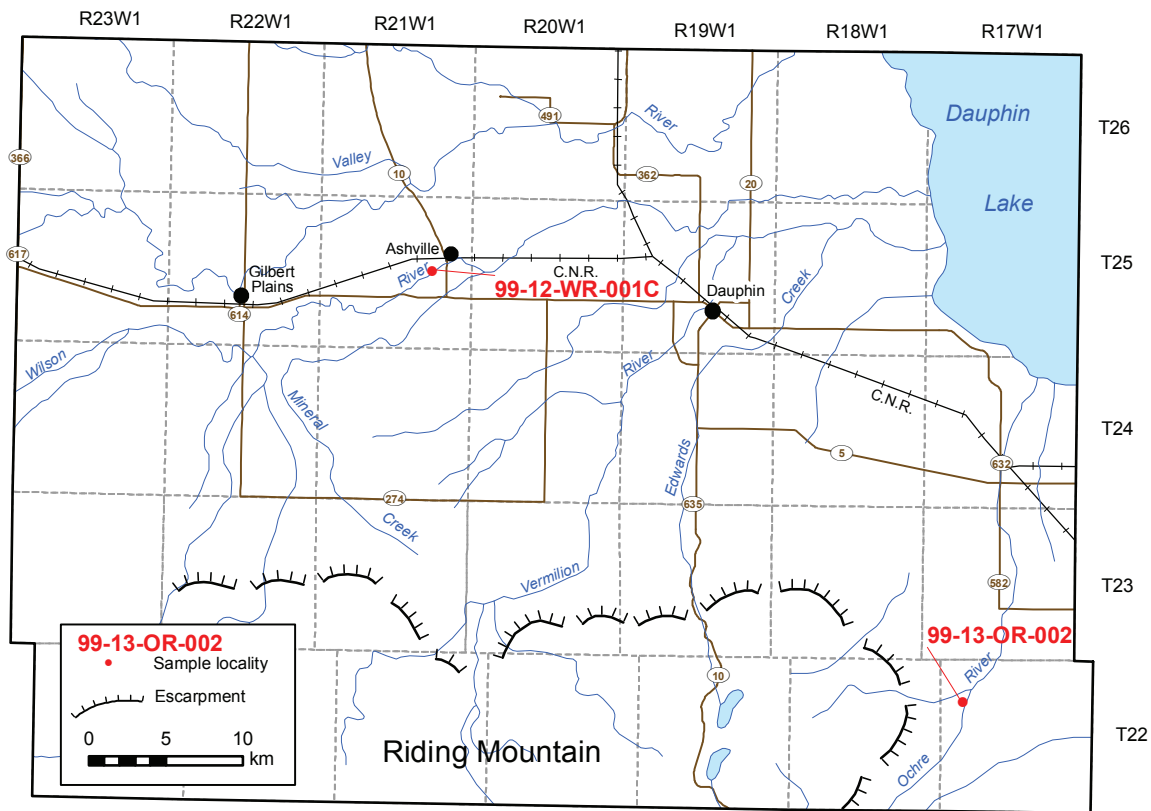


Figure GS-16-3: Location of bentonite samples collected along the north flank of Riding Mountain, west and south of Dauphin, southwestern Manitoba.

by Barker et al. (2011). It should be noted that the Burnt Timber Creek sample was extracted using a 2.54 cm coring tube and the outer ~1 cm portion of the sample was discarded. Then the core exteriors were visually inspected to ensure homogeneity. These sampling procedures likely reduced the potential for contamination, which is suspected in the Wilson River sampling. The Burnt Timber Creek sample was processed at the UofT Jack Satterly Geochronology Laboratory.

Sample 99-13-TR-001A

Sample 99-13-TR-001A was collected from a bentonite bed within the Odanah Member of the Pierre Shale (Figure GS-16-1) at the Treherne quarry, a few kilometres south of Treherne (Figure GS-16-3). The sample location is situated in 12-25-7-10W1 (UTM NAD83, Zone 14U, 520969E, 5495817N; NTS southeast corner of 62G10).

The sample was processed at the UofA Radiogenic Isotope Facility. Three dates were reported from five zircon crystals (Bamburak and Heaman, 2016). Only the youngest date, 67.2 ± 0.4 Ma, is shown in Table GS-16-1 because it provides the maximum depositional age of the sample. The older dates are likely from inherited grains, either from earlier eruptions or contamination from shale inclusions.

Sample 99-13-MO-001A

Sample 99-13-MO-001A was collected from the Q3 bentonite bed of Bamburak et al. (2013) within the Pembina Member of the Pierre Shale (Figure GS-16-1) at the Friesen roadcut, 10 km south of Morden (Figure GS-16-2). The sample location is situated in 4-7-2-5W1 (UTM NAD83, Zone 14U, 563579E, 5439339N; NTS southeast corner of 62G1).

The sample was processed at the UofA Radiogenic Isotope Facility. No stratigraphically relevant radiometric ages were recovered from the four zircon crystals tested in sample 99-13-MO-001A (Bamburak and Heaman, 2016), probably due to contamination of the site from road construction.

Sample 99-13-WA-001

Sample 99-13-WA-001 was also collected from the Q3 bentonite bed of Bamburak et al. (2013) within the Pembina Member of the Pierre Shale (Figure GS-16-1) at Pembina gorge roadcut, west of Walhalla in North Dakota (Figure GS-16-2). The sample location is situated in the northeast portion of Sec. 13, Twp. 163N, Rge. 58W (UTM NAD83, Zone 14U, 567221E, 5421884N).

The sample was processed at the UofT Jack Satterly Geochronology Laboratory. Despite this sample being collected in a roadcut, it provided five well clustered zircons with a Concordia age of 80.04 ± 0.11 Ma (Figure GS-16-4, Table GS-16-1). This is in marked contrast to sample 99-13-MO-001A, where the Q3 bentonite bed at the Friesen roadcut (18 km to the north) did not provide any stratigraphically relevant radiometric ages. It should also be noted that the Concordia age of 80.04 ± 0.11 Ma from the Pembina gorge roadcut compares well with the Ar/Ar determinations of 80.54 ± 0.55 and 80.04 ± 0.4 Ma obtained from sanidine of the Ardmore bentonite in the Elk Basin of Wyoming and near Redbird, Wyoming, respectively (Martin et al. 2007, p. 14).

The 80.04 ± 0.11 Ma age for the Q3 bed (Figure GS-16-4) at Pembina gorge and the Ardmore bentonite ages of Martin et al. (2007) contradict the 2013 interpretation by Bamburak et al. (2013) that the L1A bentonite bed (Figure GS-16-5) at the Mount Nebo sample site (which is substantially lower in the stratigraphic section) had an age of 78.8 ± 1.0 Ma. It should be noted that Bamburak et al. (2013) had commented that small inclusions of in situ shale were contained in the Mount Nebo bentonite sample, despite care having been taken not to collect overlying and underlying black shale. The

L1A bentonite bed had initially been selected for dating (despite previous quarrying of the site and the potential for contamination) because of its close proximity to a high concentration of fossils (Bamburak et al., 2012, 2013). However, it is concluded that the 78.8 ± 1.0 Ma age reported for the L1A bentonite bed is much too young and it should be discarded.

Sample 99-13-MO-002

Sample 99-13-MO-002 was collected from a bentonite bed within the Gammon Ferruginous Member of the Pierre Shale (Figure GS-16-1) at Wozniak creek streamcut in Pembina Valley Provincial Park, a short distance away from the United States border (Figure GS-16-2). The sample location is situated in 16-1-1-7W1 (UTM NAD83, Zone 14U, 553470E, 5429237N; NTS southwest corner of 62G1).

The sample was processed at the UofT Jack Satterly Geochronology Laboratory. Fraction Z5 from sample 99-13-MO-002 provided a $^{206}\text{Pb}/^{238}\text{U}$ age of 91.3 ± 0.3 Ma. However, this does not agree with the other results from the bentonite beds contained within the Upper Cretaceous chronostratigraphic framework and must be discounted as being due to contamination at the sample site, probably due to erosion along the stream bank.

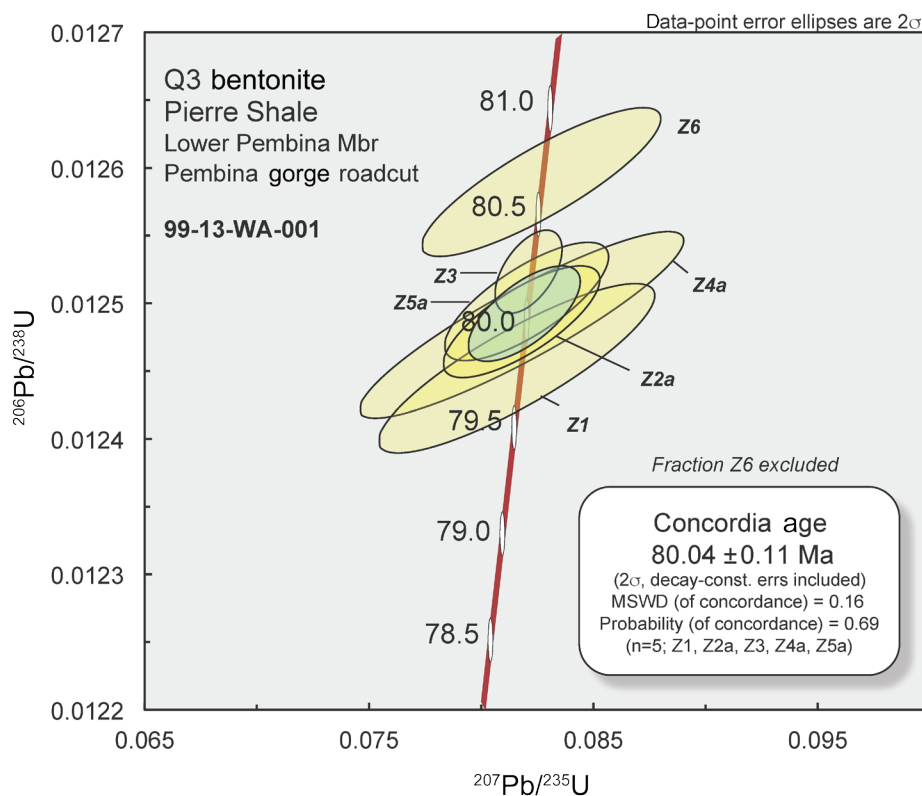


Figure GS-16-4: Concordia diagram for five zircon fractions (Z1, Z2a, Z3, Z4a, Z5a; ages in Ma) recovered from sample 99-13-WA-001 at the Pembina gorge roadcut, west of Walhalla in North Dakota (University of Toronto Jack Satterly Geochronology Laboratory, Toronto, Ontario). Abbreviations: const., constant; errs, errors; MSWD, mean square of weighted deviates.

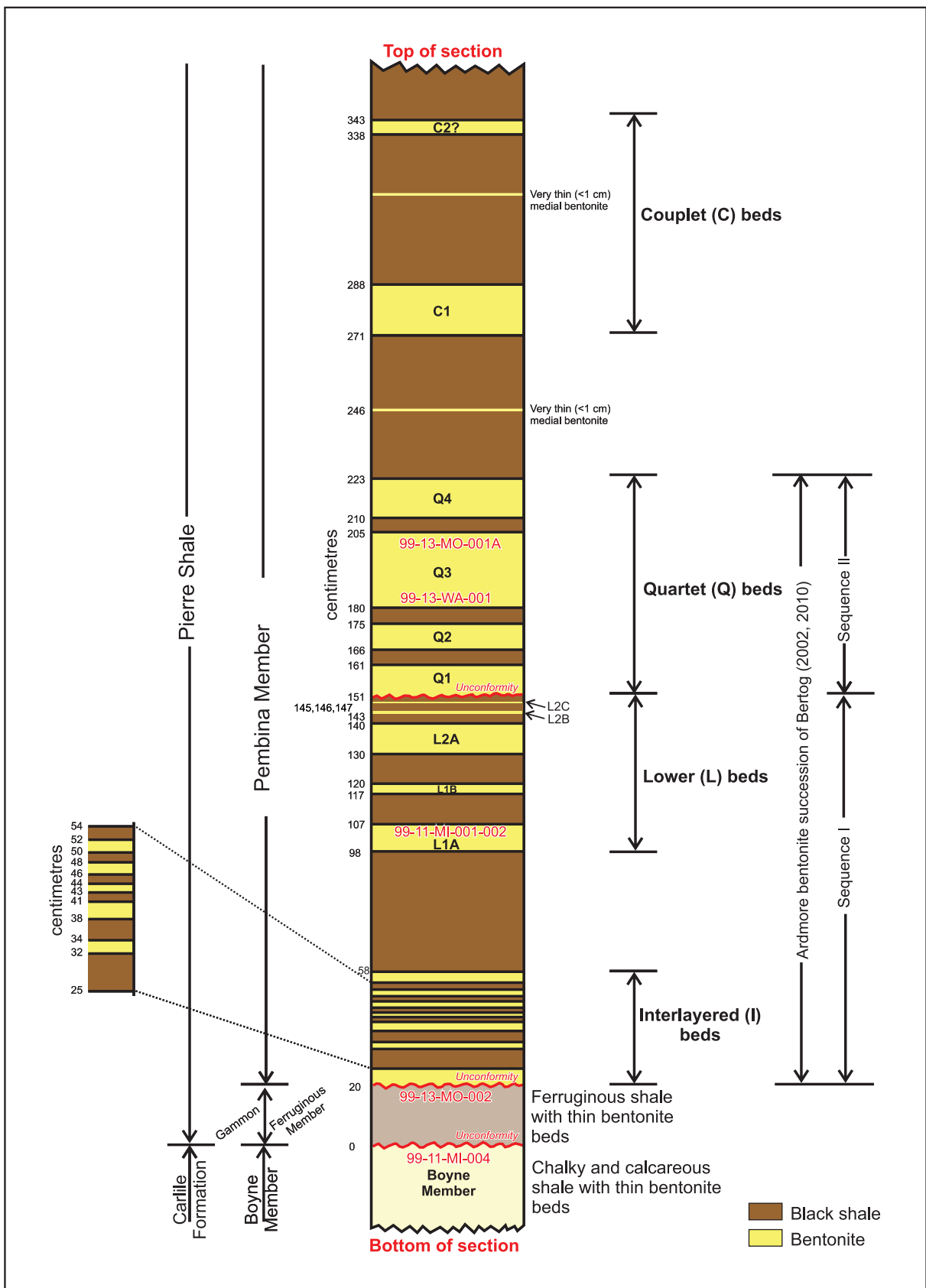


Figure GS-16-5: Schematic representation of the lowermost bentonite beds in the Pembina Member and underlying units in the Pembina Hills area, Manitoba and North Dakota (modified from Bamburak et al., 2012), including Ardmore bentonite succession of Bertog (2002, 2010). Location of samples numbers (in red) within the units is only approximate.

Sample 99-13-OR-002

Sample 99-13-OR-002 was collected from a bentonite bed within the Assiniboine Member of the Favel Formation (Figure GS-16-1) at Ochre River streamcut, southeast of Dauphin (Figure GS-16-3). The sample location is situated in 9-30-22-17W1 (UTM NAD83, Zone 14U, 442837E, 5542134N; NTS northwest corner of 62J13).

The sample was processed at the UofT Jack Satterly Geochronology Laboratory. A Concordia age of 92.54 ± 0.28 Ma (Figure GS-16-6, Table GS-16-1) was calculated from three zircon fractions (Z1a, Z3a, Z6a). A Late Cretaceous (Turonian) volcanic event is indicated from the age result of this bentonite sample.

Discussion

From 2012 to 2016, the MGS bentonite dating project has yielded reliable ages for five Upper Cretaceous bentonite beds (Table GS-16-1), mostly within southwestern Manitoba, using U-Pb zircon geochronology. During this time, a total of eight samples (Figure GS-16-1) were submitted for radiometric dating but the results for three of the samples had to be discarded because contamination introduced inaccuracies. These were due to inherited grains from earlier eruptions, shale inclusions or sites that

had been subjected to surface disruption—quarrying, road construction or stream erosion. For future sampling of bentonite beds for dating, it is recommended that extreme care must be taken at sites where surface disruption may have occurred and every effort must be made to try to obtain a clean sample free from contamination, possibly through the use of a coring tube as described by Barker et al. (2011).

Future work

Future work on the project will include collecting new bentonite samples and sending them to the UofT Jack Satterly Geochronology Laboratory for U-Pb zircon dating. One of these samples will be from the X-bentonite bed (site of sample 99-12-WR-001C, Figure GS-16-3) within the Belle Fourche Member of the Ashville Formation along the Wilson River, about 1 km southwest of Ashville. This will be done to reconfirm the 96.0 ± 0.2 Ma date indicated in this report.

Economic considerations

Creating a chronological stratigraphic framework, based upon dating of bentonite beds for Upper Cretaceous strata in Manitoba, will greatly benefit future exploration activities. Stratigraphic units are time transgressive

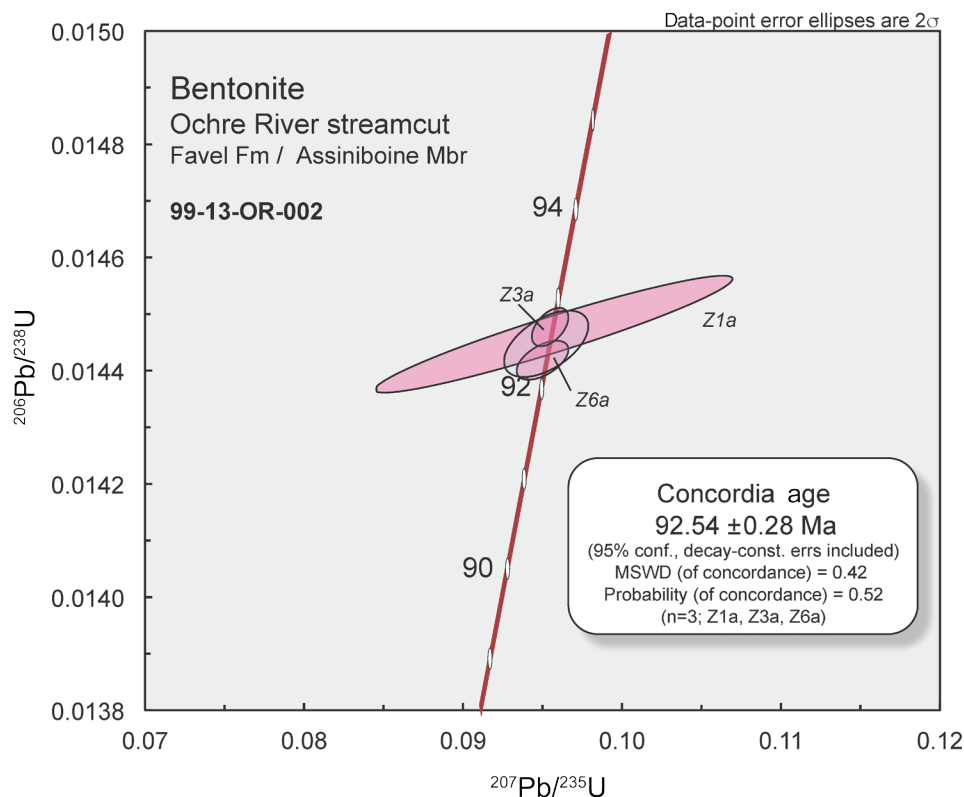


Figure GS-16-6: Concordia diagram for three zircon fractions (Z1a, Z3a, Z6a; ages in Ma) recovered from sample 99-13-OR-002 at the Ochre River streamcut southeast of Dauphin (University of Toronto Jack Satterly Geochronology Laboratory). Abbreviations: conf., confidence; const., constant; errs, errors; MSWD, mean square of weighted deviates.

from west to east across the Western Canada Sedimentary Basin, resulting in a need to resolve cross-sectional modelling problems and provincial ‘boundary faults’. The Upper Cretaceous stratigraphy of Manitoba contains petroleum and industrial mineral commodities; resolution of conflicting stratigraphic units will result in a simpler and less expensive approach to targeting potential resources.

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