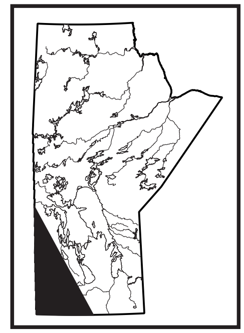
GS-12 Radiometric dating of Late Cretaceous bentonite beds in southwestern Manitoba

by J.D. Bamburak, M.P.B. Nicolas and J. Hatcher[1]



Bamburak, J.D., Nicolas, M.P.B. and Hatcher, J. 2013: Radiometric dating of Late Cretaceous bentonite beds in southwestern Manitoba; *in* Report of Activities 2013, Manitoba Mineral Resources, Manitoba Geological Survey, p. 129–136.

Summary

This report presents the preliminary results of a study to determine the feasibility of dating Late Cretaceous bentonite beds in southwestern Manitoba using U-Pb zircon geochronology. Radiometric dating of bentonite beds in southwestern Manitoba in the 1980s employed the K-Ar dating method, which had many inherent problems due to large analytical errors. The present study will evaluate newer methods that have proven to be more reliable, such as U-Pb dating of zircon and $^{40}Ar/^{39}Ar$ dating of sanidine or biotite. The results of these investigations should help to resolve cross-sectional modelling problems and provincial 'boundary faults', which result from the time-transgressive nature of many stratigraphic units in the Western Canada Sedimentary Basin[2]. The objective is to develop a simplified and standardized nomenclature, which can be used across western Canada and the western United States to make target selection easier and less expensive for petroleum and industrial mineral exploration. This study has determined that a bentonite bed near the top of the Boyne Member of the Carlile Formation at Spencer's Ditch has a U-Pb zircon age of 81.5 ±0.5 Ma and that a bentonite bed near the base of the Pembina Member of the Pierre Shale at Mount Nebo has an age of 78.8 ±1.0 Ma. These ages form the approximate lower and upper age limits, respectively, of the intervening Gammon Ferruginous Member of the Pierre Shale.

Introduction

According to Gill and Cobban (1965, p. A7) and Bannatyne and Watson (1982, p. 45), bentonite beds present in the Upper Cretaceous Pembina Member of the Pierre Shale (Figure GS-12-1) are formed of altered volcanic ash derived from volcanic eruptions in the Elkhorn Mountains of western Montana and deposited within the former Western Interior Seaway in southwestern Manitoba.

Previous attempts in the 1970s and 1980s to determine the ages of bentonite beds from outcrops and quarries in the Pembina Hills area, near Morden, and in a shaft being sunk in Saskatchewan near the Manitoba boundary did not provide dates sufficiently precise for the purpose of stratigraphic correlation (see 'Previous work' section). However, more precise methods of dating, such as U-Pb dating of zircon and $^{40}Ar/^{39}Ar$ dating of sanidine or biotite, have since been developed. In late 2010, the Manitoba Geological Survey (MGS) entered into discussions with L. Heaman of the University of Alberta (UofA) Radiogenic Isotope Facility on the possibility of dating Cretaceous bentonite samples using U-Pb zircon geochronology. These discussions led to the initiation of a Phanerozoic dating subproject (funded under an existing MGS geochronology project) in April 2012 to undertake a geochronology and isotopic study of bentonite beds. The objective of the subproject is to help resolve cross-sectional modelling problems and provincial 'boundary faults', which result from stratigraphic units that are time transgressive from west to east across the Western Canada Sedimentary Basin. In addition, a separate collaborative study has been initiated with the Alberta Geological Survey to possibly undertake

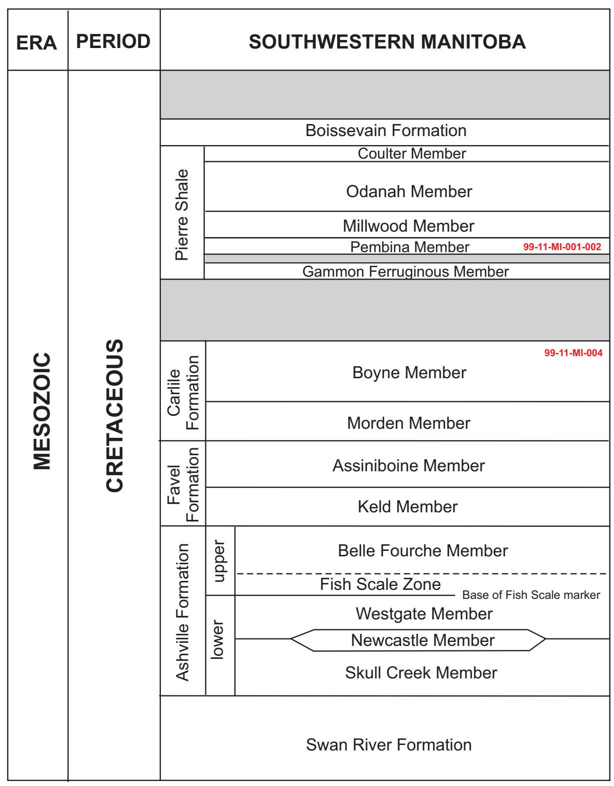


Figure GS-12-1: *Cretaceous stratigraphy of southwestern Manitoba with bentonite samples (99-11-MI-004 and 99-11-MI-001-002) collected in 2011 (modified from Nicolas and Bamburak, 2009).*

[1] Canadian Fossil Discovery Centre, 111-B Gilmour Street, Morden, MN R6M 1N8

[2] For the sake of consistency, the Manitoba Geological Survey has opted to make a universal change from capitalized to noncapitalized for the generic part of lithostructural feature names (formal stratigraphic and biostratigraphic nomenclature being the exceptions).

$^{40}Ar/^{39}Ar$ dating of sanidine and biotite in some of the bentonite samples from Manitoba, as a check on the U-Pb dates.

Previous work

Radiometric dating studies of Late Cretaceous bentonite beds have been carried out only once in Manitoba, in the Pembina Hills. However, another study was initiated during the sinking of a potash shaft in western Saskatchewan, close to the provincial boundary, and is thus relevant to the current study.

Pembina Hills

In 1985, six 50 kg samples of Upper Cretaceous Pembina Member bentonite from two localities situated northwest and south of Morden, Manitoba, were submitted for K-Ar dating at the Geological Survey of Canada (GSC) geochronology lab (Nicholls, 1988). At Spencer's Ditch (described below), an age of 81 ±3 Ma was reported from the uppermost bentonite bed. Units 5, 11 and 13 (in ascending order) in the former Hoeppner quarry (south of Morden) also yielded ages of 88 ±3, 80 ±3 and 82 ±3 Ma, respectively (Nicholls, 1988). The method of sampling of the bentonite beds and the analytical procedures were not discussed in the Nicholls' report; an attempt to find further information through the GSC was not successful. Unfortunately, the results of this work are not sufficiently precise for the purpose of stratigraphic correlation.

Sylvite No. 2 shaft, Saskatchewan

The Sylvite No. 2 mine is situated only 5 km west of the Manitoba-Saskatchewan boundary, 16 km northeast of Rocanville, Saskatchewan (de Korompay, 1971). According to Price (1985), during the sinking of Sylvite No. 2 shaft, C.E. Wright of the GSC noted and photographed stratigraphic units in the shaft wall and collected fossils and rock specimens from the excavated material. Price (1985) produced lithological logs of the shaft based on these observations, including preliminary microscopic examination of material excavated from the shaft. In addition, Price used geophysical logs and drillcore descriptions from pilot holes drilled for both Sylvite No. 1 and 2 shafts to help define stratigraphic units (de Korompay, 1971).

Fourteen mineral separates for K-Ar age determination were recovered from nine bentonite beds between 146.3 m (Millwood Member?) and 257.3 m (Favel Formation) below surface in the Sylvite No. 2 shaft. According to Williams and Baadsgaard (1975, Table I, Figure 3), 12 'valid' dates ranging from 91.9 to 78.6 Ma were recorded from biotite and sanidine in these separates. However upon further reflection, H. Baadsgaard (formerly of the UofA) stated that the bentonite samples from the Sylvite No. 2 shaft were likely contaminated by overlying and underlying shale and, as a consequence, the results of K-Ar dating were likely inaccurate, especially with regards to the sanidine dates (H. Baadsgaard, pers. comm., 2013).

Current investigations

Sample locations

Two samples of bentonite were collected for U-Pb zircon dating in 2011.One sample was collected from the Boyne Member of the Carlile Formation (Figure GS-12-1) at Spencer's Ditch and the other from the Pembina Member of the Pierre Shale at Mount Nebo. In addition to acquiring dates for the two respective members, the purpose of the sampling was to bracket the age of the intervening Gammon Ferruginous Member (Figure GS-12-1). At Spencer's Ditch, this member contains a high concentration of fossils, informally known as the *Xiphactinus* Kill Zone quarry, described by Bamburak et al. (2012a, p. 145, 146). The results of the zircon U-Pb dating are described below.

In 2012, a sample of bentonite (the X-bentonite) was collected from the Belle Fourche Member of the Ashville Formation (Figure GS-12-1) along the Wilson River. The sample location is situated about 1 km southwest of Ashville in L.S. 14, Sec. 14, Twp. 25, Rge. 21, W 1st Mer. (abbreviated 14-14-25-21W1; UTM Zone 14U, 408242E, 5669335N; NTS 62N1NW), a short distance east of outcrop section 67 of McNeil and Caldwell (1981), which also contains the X-bentonite. The X-bentonite, in association with the *Ostrea beloiti* beds, has been recognized as one of the most widespread isochronous lithostratigraphic, biostratigraphic and chronostratigraphic markers in the entire Western Interior Seaway (McNeil and Caldwell, 1981, p. 50, 51, 79). The sample is currently being analyzed at the UofA; initial reports indicate that although the zircon crystals are very tiny, they are of excellent quality. A duplicate of this sample may also undergo $^{40}Ar/^{39}Ar$ dating by the Alberta Geological Survey in the near future.

In 2013, seven additional bentonite samples were collected (Table GS-12-1), for possible dating in the future, from the

- Odanah Member of the Pierre Shale at the Treherne quarry;
- Odanah Member of the Pierre Shale at 108th Northeast roadcut, North Dakota;
- Pembina Member of the Pierre Shale at Vermilion River streamcut;
- Pembina Member of the Pierre Shale at the Friesen roadcut;
- Pembina Member of the Pierre Shale at Pembina gorge roadcut, North Dakota;

Table GS-12-1: Details of 2013 bentonite sample sites.

Formation/Member	Site name	Sample no.	Easting[1]	Northing[1]	NTS	Twp. and Rge.
Pierre Shale/Odanah - bentonite	Treherne quarry	99-13-TR-001	520969	5495817	62G10SE	12-25-07-10W1[2]
Pierre Shale/Odanah - bentonite	108th Northeast roadcut, North Dakota	99-13-WA-002	564496	5425616		SW35-163N-58W[3]
Pierre Shale/lower Pembina - uppermost thick bentonite bed (Q4?)	Vermilion River streamcut	99-13-VR-002	416427	5649592	62K16NE	15-15-23-20W1
Pierre Shale/lower Pembina - Q3 bentonite	Friesen roadcut	99-13-MO-001	563579	5439339	62G01SE	04-07-01-05W1
Pierre Shale/lower Pembina - Q3 bentonite	Pembina gorge roadcut, North Dakota	99-13-WA-001	567221	5421884		NE13-163N-58W
Pierre Shale/Gammon Ferruginous - 8 cm bentonite	"Wozniak" creek streamcut	99-13-MO-002	553470	5429237	62G01SW	01-01-07W1
Favel/Assiniboine - uppermost thick bentonite bed	Ochre River streamcut	99-13-OR-002	442837	5642134	62J13NW	09-30-22-17W1

[1] NAD 83, Zone 14
[2] Abbreviated form of L.S. 12, Sec. 25, Twp. 7, Rge. 10, W 1st Mer.
[3] Abbreviated form of SW Sec. 35, Twp. 163N, Rge. 58W

- Gammon Ferruginous Member of the Pierre Shale at Wozniak creek streamcut; and
- Assiniboine Member of the Favel Formation at Ochre River streamcut.

U-Pb dating methodology

The two 2011 samples underwent mineral separation, processing and analysis at the UofA Radiogenic Isotope Facility in Edmonton, Alberta. Heavy minerals were separated using standard crushing, pulverizing and heavy liquid techniques, followed by sorting of the concentrates using a Frantz isodynamic separator. Final selection of mineral separates was done by hand under a binocular microscope.

Zircon separates underwent conventional U-Pb dating by isotope dilution–thermal ionization mass spectrometry (ID-TIMS), generally following the procedures outlined in Heaman et al. (2002). Exterior surfaces of intact zircon crystals were generally removed by air abrasion, whereas crystal fragments were mostly unabraded. All analyses were performed on a VG 354 thermal ionization mass spectrometer operated in single Faraday or Daly (analogue) collector peak-hopping mode, and were corrected for mass discrimination based on replicate measurement of the NBS 981 and U500 standards. In addition, all measurements obtained with the Daly photomultiplier detector were adjusted for detector bias. The isotopic composition of common Pb in excess of an analytical blank (2 picograms [pg] of Pb per analysis) was calculated using the two-stage model of Stacey and Kramers (1975).

U-Pb dating results

Spencer's Ditch (Pembina Hills)

The Boyne Member of the Carlile Formation bentonite sample (99-11-MI-004) was provided by the Canadian Fossil Discovery Centre from the east wall of Spencer's Ditch (Figure GS-12-2; 15-31-3-6W1; UTM Zone 14U, 553843E, 5457258N; NTS 62G8SW). The 8 cm thick bentonite bed is labelled as the thicker bentonite in Figure GS-12-3. Care was taken not to contaminate the sample with any of the overlying and underlying calcareous shale.

The analytical results for this sample are listed in Table GS-12-2 and depicted in Figure GS-12-4. All errors reported in Table GS-12-2 and Figure GS-12-4 are quoted at 1σ and were calculated by numerical propagation of all known sources of uncertainty. Regression calculations and age uncertainties were determined using Isoplot version 3.0 (Ludwig, 2003).

Four colourless zircons were recovered from sample 99-11-MI-004 (Table GS-12-2). The U contents of the zircons ranged from 138 to 192 ppm and the measured total common Pb ranged from 3 to 4 pg per analysis. It should be noted that the model U/Th ratios are somewhat variable, perhaps indicating the zircons were derived from more than one source. According to L. Heaman (pers. comm., 2012), the best constraint on depositional age is to report the youngest analysis for nonconcordant detrital zircon grains (i.e., the youngest detrital zircon will provide the maximum depositional age of the sample). The youngest of the four U-Pb zircon ages obtained was 81.5 ±0.5 Ma (Table GS-12-2). The older zircons may be

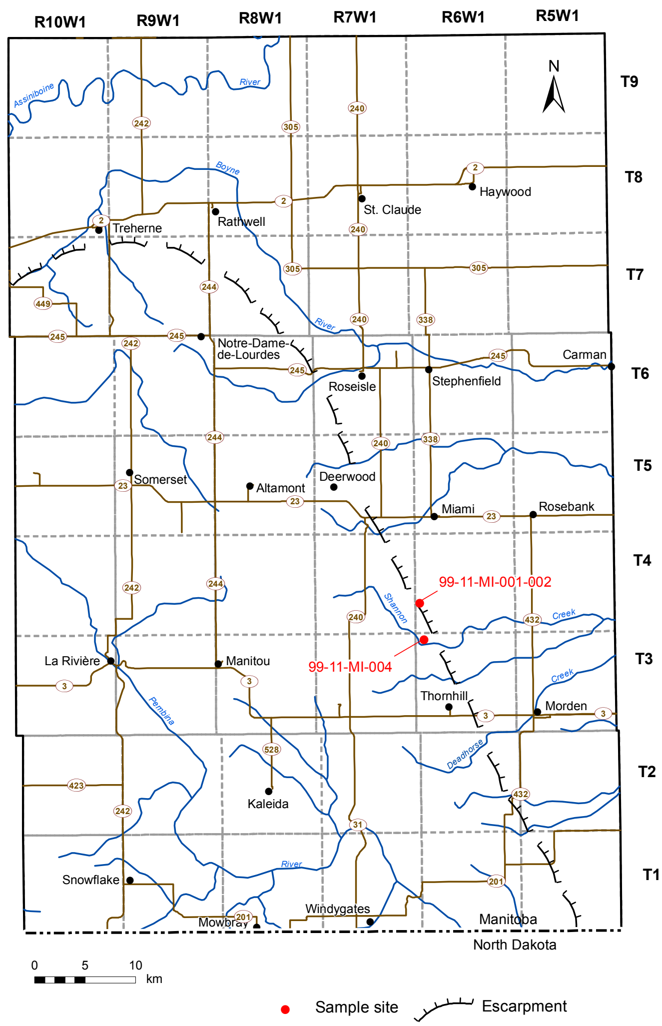


Figure GS-12-2: *Location of 2011 bentonite sample sites in the Pembina Hills area of southwestern Manitoba (modified from Bamburak and Nicolas, 2010).*

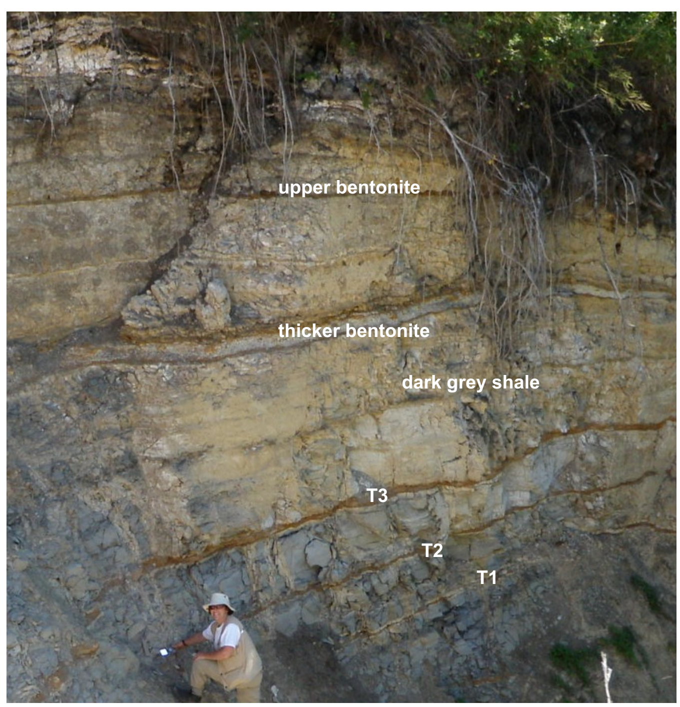


Figure GS-12-3: *Spencer's Ditch east wall with bentonite bed annotations.*

Table GS-12-2: U-Pb isotope dilution–thermal ionization mass spectrometry (ID-TIMS) analytical data for zircon from Spencer's Ditch (sample 99-11-MI-004) and Mount Nebo (sample 99-11-MI-001-002). All errors are quoted at 1σ.

									Isotopic ratios[2]			Ages (Ma)			
	Description[1]	Weight (mg)	U (ppm)	Th (ppm)	Pb (ppm)	Model U/Th	^{204}Pb (pg)	^{206}Pb/^{204}Pb	^{206}Pb/^{238}U	^{207}Pb/^{235}U	^{207}Pb/^{206}Pb	^{206}Pb/^{238}U	^{207}Pb/^{235}U	^{207}Pb/^{206}Pb	Disc. %
	Sample 99-11-MI-001-002														
1z	larger colourless 3:1 prisms MIH (7)	11.4	290	91	4.3	0.32	7	410	0.01302 ±0.00002	0.0857 ±0.0006	0.04775 ±0.00030	83.4 ±0.1	83.5 ±0.5	86.9 ±14.8	4.1
2z	colourless 2:1 prisms MIH (11)	8.6	127	54	2.3	0.43	6	175	0.01355 ±0.00004	0.0913 ±0.0018	0.04885 ±0.00091	86.8 ±0.2	88.7 ±1.6	140.4 ±43.3	38.5
3z	smaller colourless 2:1 prisms MIH (11)	5.5	28	35	0.8	1.22	3	69	0.01330 ±0.00031	0.1945 ±0.0128	0.10608 ±0.00651	85.2 ±2.0	180.5 ±10.8	1733.1 ±108.6	95.7
4z	colourless equant small MIH (14)	7.6	99	86	3.8	0.86	19	49	0.01231 ±0.00015	0.0766 ±0.0054	0.04515 ±0.00318	**78.8 ±1.0**	75.0 ±5.1	-47.3 ±162.9	268.3
	Sample 99-11-MI-004														
1z	clear and colourless prism MIH (1)	1.7	192	187	4	0.97	3	123	0.01408 ±0.00032	0.1892 ±0.0315	0.09742 ±0.01435	90.1 ±2.1	175.9 ±26.5	1575.3 ±253.1	94.9
2z	small colourless prism MIH (1)	1.0	138	96	5	0.69	4	50	0.01331 ±0.00017	0.0777 ±0.0102	0.04236 ±0.00540	85.2 ±1.1	76.0 ±9.5	-205.2 ±292.3	142.5
3z	small clear colourless prism MIH (1)	1.3	173	30	4	0.17	3	96	0.01514 ±0.00008	0.1120 ±0.0055	0.05364 ±0.00252	96.9 ±0.5	108.0 ±5.0	356.1 ±102.9	73.3
4z	small clear colourless prism MIH (1)	0.9	164	41	4	0.25	3	63	0.01272 ±0.00008	0.0884 ±0.0084	0.05043 ±0.00461	**81.5 ±0.5**	86.0 ±7.8	215.1 ±199.2	62.5

[1] Numbers in parentheses indicates number of grains included in analysis.
[2] Isotopic ratios and ages are corrected for fractionation, blank, spike and initial common Pb (Stacey and Kramers, 1975).
Abbreviations: disc., discordance relative to concordia; MIH, methylene iodide heavy fraction; pg, picogram.

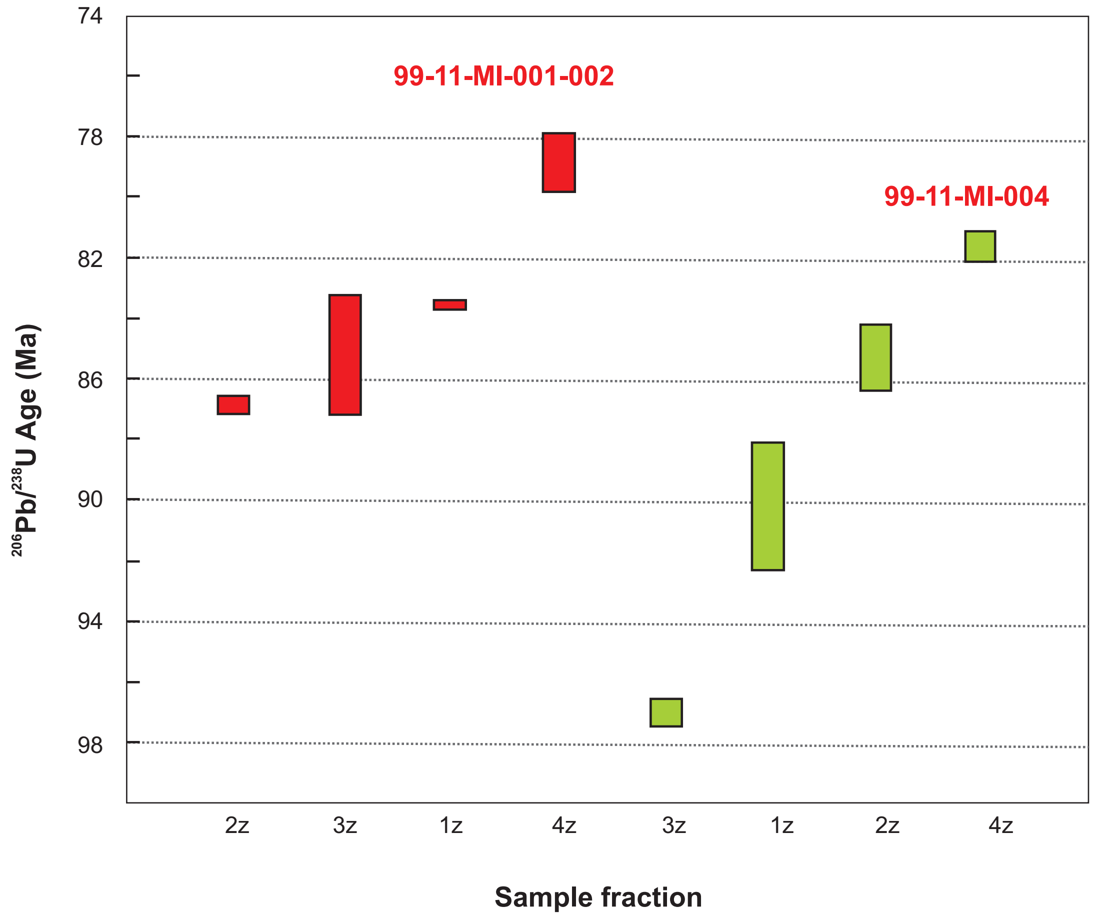


Figure GS-12-4: *Plot of U-Pb ($^{206}Pb/^{238}U$) zircon ages and uncertainties of bentonite samples from Spencer's Ditch (sample 99-11-MI-004) and Mount Nebo (sample 99-11-MI-001-002). All errors are quoted at 1σ.*

inherited from earlier eruptions or are possibly contaminated by the enclosing shale.

Mount Nebo, east flank quarry (Pembina Hills)

In 2011, one of the lowest bentonite beds in the former Mount Nebo quarry was sampled (99-11-MI-001-002, Figure GS-12-2) for U-Pb zircon dating. The bentonite bed is situated on the east flank of Mount Nebo in 4-18-4-6W1 (UTM Zone 14U, 553031E, 5460947N; NTS 62G8SW). It was designated as Spl 1 by Hatcher and Bamburak (2010) and was noted to overlie 'Ianto', a fossil plesiosaur skeleton (CFDC specimen P.088.01.04). The bed has also been identified as L1A (Bamburak et al., 2012b) as depicted in Figure GS-12-5.

Sample 99-11-MI-001-002 was collected from an 8 cm thick L1A bed. Although care was taken not to collect any overlying and underlying black shale, small inclusions of in situ shale were noted in the sample. The bentonite sample was submitted for U-Pb zircon dating in 2011 and the results are shown in Table GS-12-2. In contrast to the sample from Spencer's Ditch, the four dated fractions from this sample included multiple zircon grains. The U content of the zircon fractions ranged from 28 to 290 ppm and the measured total common Pb ranged from 3 to 19 pg per analysis. It should be noted that the model U/Th ratios may indicate a non-unique source. The youngest of four U-Pb dates is 78.8 ±1.0 Ma, which provides the maximum depositional age of the sample (Table GS-12-2). The older dates likely reflect inherited grains, either from earlier eruptions or contamination from the shale inclusions. It is interesting to note that Obradovich and Cobban (1975) reported two biotite K-Ar dates of 77.9 ±0.8 and 78.1 ±1.6 Ma from a sample collected from an 85 cm thick bentonite bed in the Claggett shale (equivalent to the Pembina Member of the Pierre Shale) in Fergus County, Montana (SW¼ SE¼ NW¼ Sec. 14, Twp. 22N, Rge. 16E), perhaps indicating that the youngest U-Pb zircon age closely approximates the depositional age of the sample.

As a result of the above investigations, the age of the Gammon Ferruginous Member, and the enclosed *Xiphactinus* Kill Zone quarry (Bamburak et al., 2012a, p. 145, 146), has now been roughly bracketed between 78.8 ±1.0 and 81.5 ±0.5 Ma.

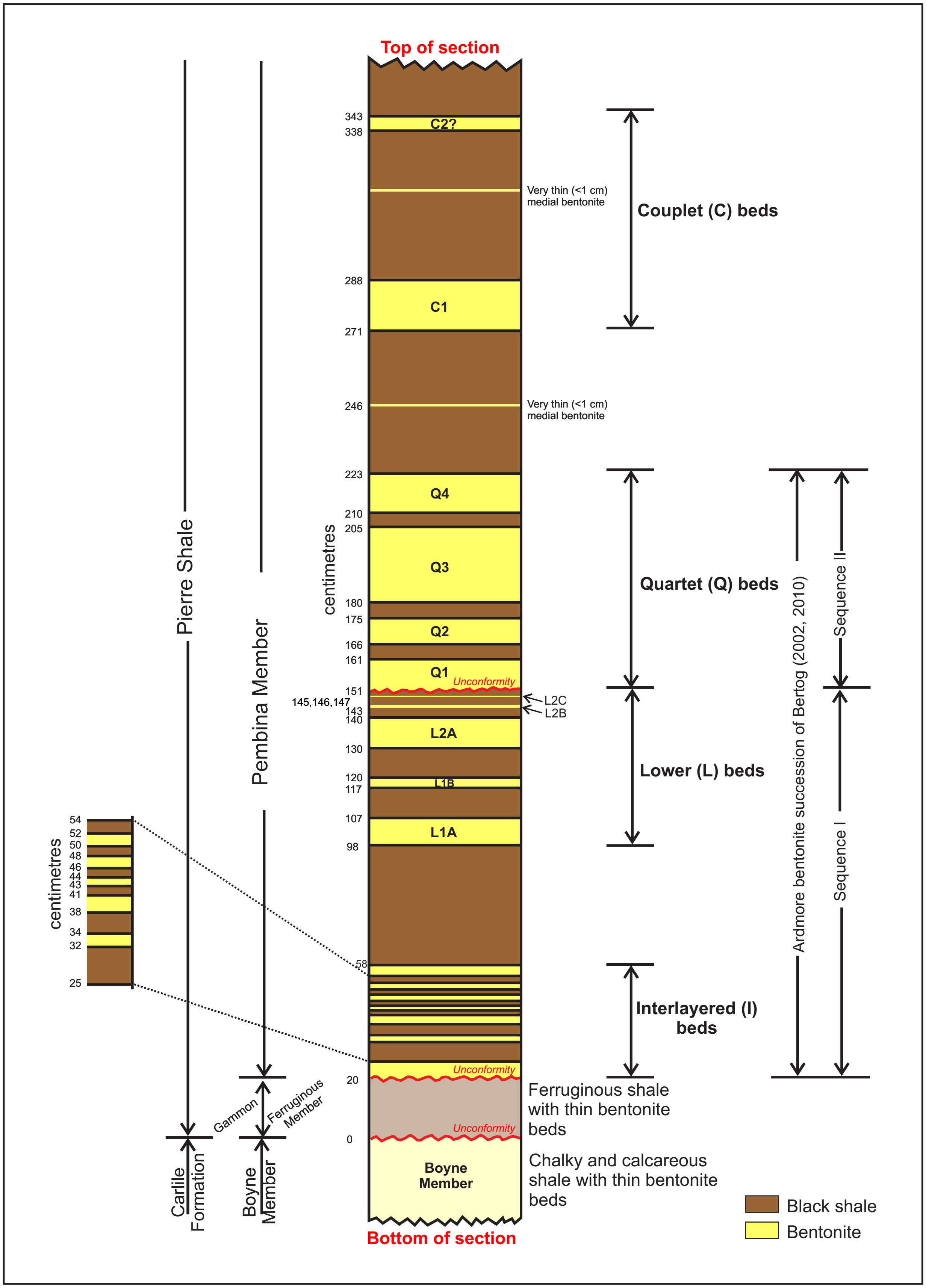


Figure GS-12-5: *Schematic representation of the lowermost bentonite beds in the Pembina Member and underlying units in the Pembina Hills area, Manitoba and North Dakota (from Bamburak et al., 2012b).*

Economic considerations

Development of a chronological framework, based upon the dating of bentonite beds, for formations and members across the Western Canada Sedimentary Basin will help to resolve cross-sectional modelling problems and provincial 'boundary faults', which result from stratigraphic units that are time transgressive from west to east. For the petroleum and Phanerozoic industrial mineral industries, this is anticipated to result in a simpler and less expensive approach to targeting potential resources.

Acknowledgments

The authors would like to thank and acknowledge D. Rheault, former Treherne quarry manager, for providing assistance in sampling at the quarry and to J. Spencer for allowing access to her land. B. Duncan is also thanked for her field assistance during 2011 and S. Anderson is gratefully acknowledged for providing a valuable and constructive peer review of the paper.

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