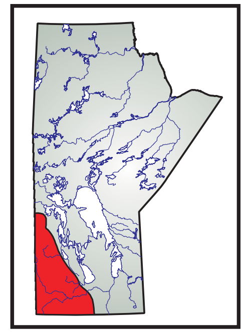
GS-13 Geochemistry and mineralogy of Cretaceous shale, southwestern Manitoba (parts of NTS 62F, G, J, K, N, 63C): phase 2 results

by M.P.B. Nicolas and J.D. Bamburak



Nicolas, M.P.B. and Bamburak, J.D. 2011: Geochemistry and mineralogy of Cretaceous shale, southwestern Manitoba (parts of NTS 62F, G, J, K, N, 63C): phase 2 results; *in* Report of Activities 2011, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 143–149.

Summary

The Shallow Unconventional Shale Gas (SUSG) Project is a multi-year investigation of the shale gas potential of the Late Cretaceous shale strata of southwestern Manitoba. The formations being evaluated as potential gas targets are the Ashville, Favel, Carlile and Pierre Shale. Phase 2 samples were collected in 2009 and represent each of these formations, as well as most of the constituent members. The sampling sites are located along the Manitoba Escarpment in the Riding Mountain, Duck Mountain, and Swan River Valley and Porcupine Hills regions. Based on the geochemical and mineralogical information presented herein, the Favel and Carlile formations are identified as the best shale gas targets, with some potential in the Gammon Ferruginous Member of the Pierre Shale.

Introduction

The SUSG Project is a multi-year investigation of the shale-gas potential of the Late Cretaceous shale sequences of southwestern Manitoba. The formations included in this study are the Ashville, Favel, Carlile and Pierre Shale (Figure GS-13-1). The goal of the project is to gather basic geoscientific information on the Late Cretaceous shale strata in southwestern Manitoba as a means of better understanding these units potential as a shale gas resource for the future. This project, first introduced by Nicolas (2008) as a response to an identified lack of geoscientific information on these shale sequences, was a direct result of inquiries from industry seeking to identify new shale gas prospects at a time when emerging drilling and completion technologies were changing the face of the unconventional gas industry. Historical gas shows from shallow sources had been documented in some areas (Bamburak, 2008), further adding to the interest from the natural gas industry.

The project was initially subdivided into two phases based on geographic area (Figure GS-13-2). Phase 1 included information south of Township 13, while phase 2 included information north of and including Township 13. Fieldwork for phase 1 was conducted in 2008, with results reported by Fedikow et al. (2009), Nicolas and Bamburak (2009), Nicolas and Grasby (2009), and Nicolas et al. (2010). The results of the geochemical and mineralogical evaluation of the samples collected in 2009 in the phase 2 area are discussed herein, together with some results on new samples collected in the phase 1 area.

During the 2009 field season, several localities were visited along the Manitoba Escarpment in the areas of Riding Mountain, Duck Mountain, Swan River Valley and the Porcupine Hills. Initial plans were to sample key localities in these regions with the goal of collecting shale samples from the Late Cretaceous shale sequences exposed in these areas. High water levels and local flooding in creeks and rivers, along with erosion and slumping of many previously known outcrops, resulted in few samples being collected (Nicolas and Bamburak, 2009). Samples collected represented horizons from the Belle Fourche Member of the Ashville Formation up to the Assiniboine Member of the Favel Formation. To compensate for the lack of samples collected in 2009, archived samples collected during an earlier sampling campaign of the area and discussed in Bamburak (1999) were added to the dataset to ensure a wide stratigraphic coverage.

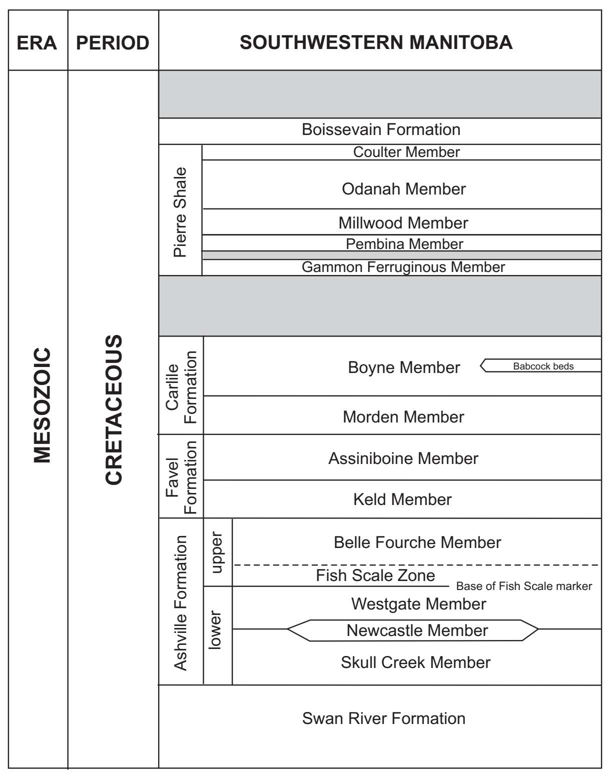


Figure GS-13-1: *Cretaceous stratigraphy of southwestern Manitoba.*

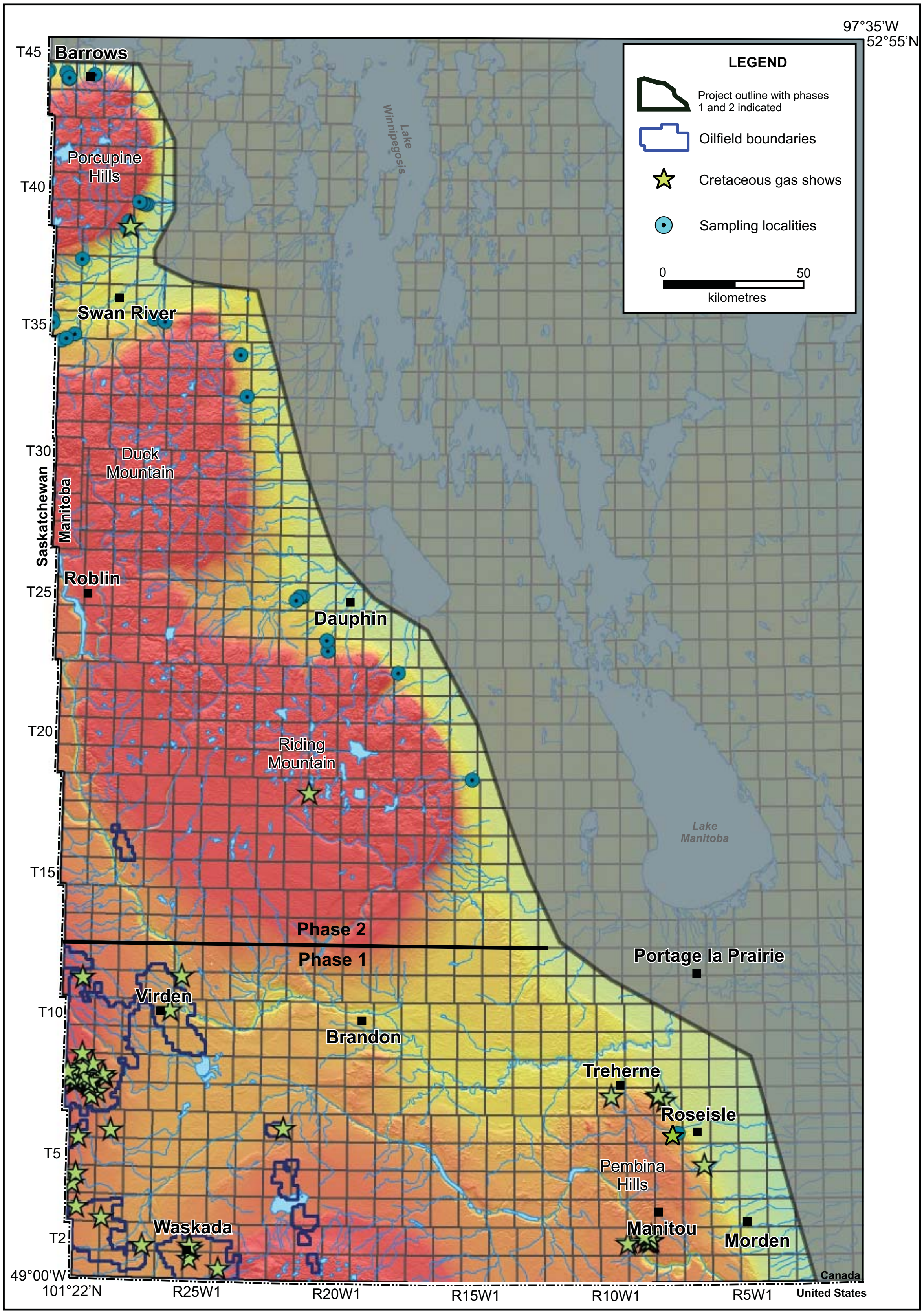


Figure GS-13-2: *Digital elevation model map of the study area for the Shallow Unconventional Shale Gas Project, southwestern Manitoba. Shown are the areas of phases 1 and 2 of the project, locations of Cretaceous gas shows in relation to existing oilfields, and field station sites of the 2009 sampling program and collection sites of archival samples.*

Phase 2 results

Organic geochemistry

Rock Eval™ 6 and total organic carbon (TOC) analysis was conducted on a total dataset of 79 samples consisting of the 34 outcrop samples collected in 2009 and, to create a larger working dataset and include horizons that could not be sampled in 2009, on archival samples collected in 1997 and 1999 for the project discussed in Bamburak (1999). The Rock Eval™/TOC analytical method measures hydrocarbons, carbon dioxide and carbon monoxide released during heating of a sample. Nicolas and Bamburak (2009) described in detail the parameters measured and their use in understanding the organic evolution of a sample.

The data from the raw Rock Eval™/TOC results were subjected to filtering to eliminate false readings and unreliable results, as described in Nicolas and Bamburak (2009). Figure GS-13-3 and Figure GS-13-4 show the organic geochemistry results, subdivided by formation and/or member. For some samples, it was uncertain if the sample was collected from a particular member; in those cases, the sample was grouped under the formation without a specific member designation. Most of the Favel samples are thought to belong to the Keld Member, whereas most of the Ashville samples are thought to come from the Belle Fourche Member, thus the vertical position they occupy on the graphs in Figure GS-13-3 is that closest to their assumed members.

The raw Rock Eval™/TOC results are presented in MGS Data Repository Item DRI2011003[1].

Figure GS-13-3a is a plot of TOC for samples of each formation and/or member in stratigraphic order; after data filtering this graph includes 75 samples. A general guideline followed in industry is that a TOC of 2 wt. %, as indicated by the green dashed line, is required for a sample to be classified as a 'good' source rock, whereas a sample with a TOC value of >10 wt. % is classified as an 'excellent' source rock. Samples with TOC values between 1 and 2 wt. % are classified as 'moderately good' source rock, whereas with a value <1 wt. %, a sample is considered an uneconomic candidate. Since TOC is a good measure to initially evaluate if a shale sample contains enough organic matter to create natural gas, it serves as a useful tool to pinpoint potential target horizons. The highest TOC values in the presented sample suite were obtained from the Assiniboine and Morden members of the Favel Formation, which yielded an organic geochemical signature for a moderately good source rock. The Gammon Ferruginous Member has not been studied much in this project due to rare outcrop exposures; this is because the Gammon Ferruginous is bounded at the bottom and at the top by disconformities, and pinches and swells over short distances. In outcrop, it is common for the member to be completely absent in a section, with the Pembina Member of the Pierre Shale directly overlying the Carlile Formation.

The general trend in the Cretaceous shale sequence shows increasing TOC values upsection, to a peak in the Assiniboine and Morden members and, from that point, decreasing values further upsection. A similar trend is observed in the results presented in Nicolas and Bamburak

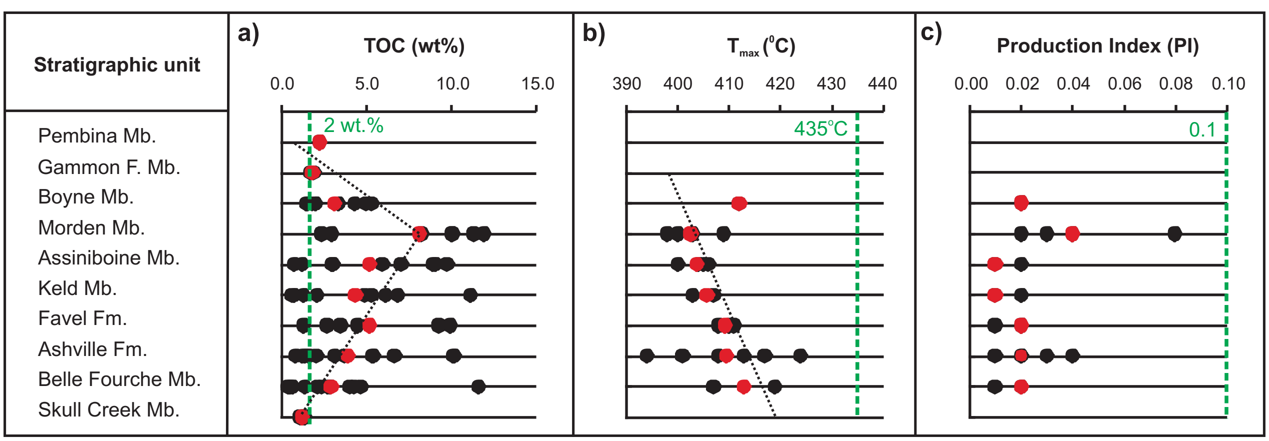


Figure GS-13-3: *New Rock Eval™ and total organic carbon (TOC) data grouped by formation and/or member (Cretaceous shale, Manitoba) and placed in order of stratigraphic depth; an average value for each member is shown in red;* ***a)*** *green dashed line indicates where TOC = 2 wt. %;* ***b)*** *green dashed line indicates where T_{max} = 435°C; and* ***c)*** *green dashed line indicates where production index (PI) = 0.1; trends are indicated by the dashed black lines. Abbreviations: F., Ferruginous, Fm., Formation; Mb., Member; TOC, total organic carbon.*

[1] MGS Data Repository Item DRI2011003, containing the data or other information sources used to compile this report, is available online to download free of change at http://www2.gov.mb.ca/itm-cat/web/freedownloads.html, or on request from minesinfo@gov.mb.ca or Mineral Resources Library, Manitoba Innovation, Energy and Mines, 360–1395 Ellice Avenue, Winnipeg, Manitoba R3G 3P2, Canada.

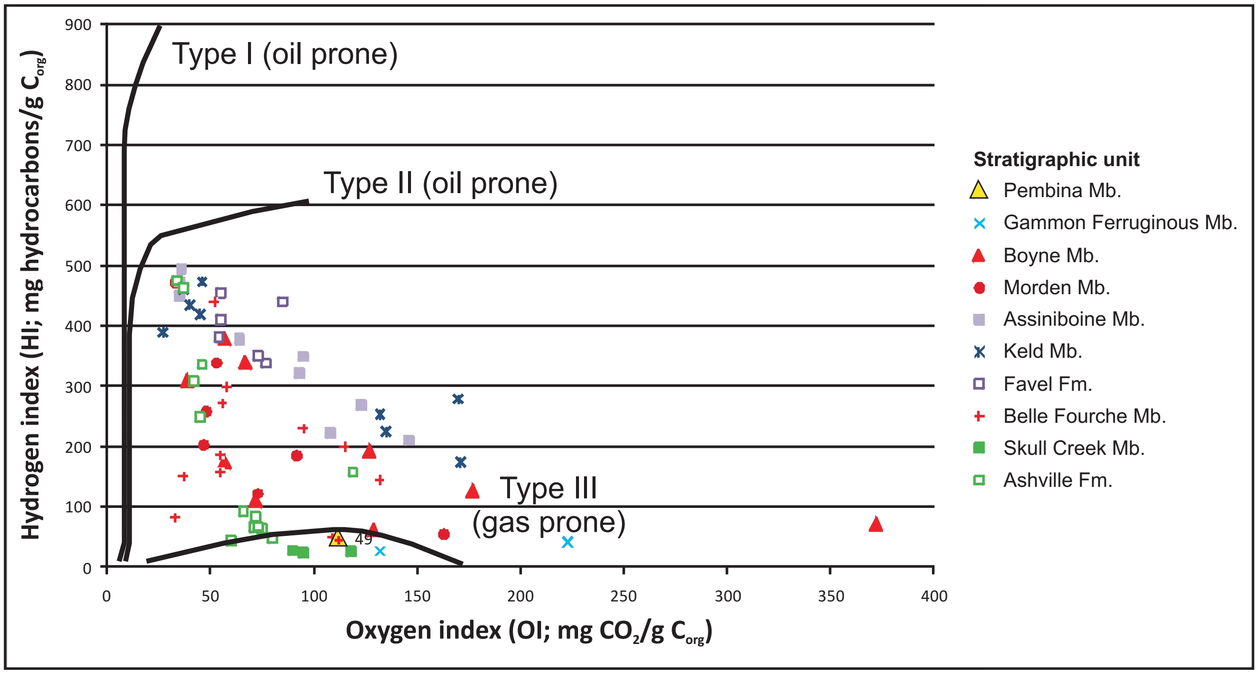


Figure GS-13-4: *Modified van Krevelen diagram of new Rock Eval™ data grouped by formation and/or member (Cretaceous shale, Manitoba). Abbreviations: Fm., Formation; Mb., Member.*

(2009, Figure GS-17-3a), which represent a much larger database.

Figure GS-13-3b is a plot of T_{max} for each member in stratigraphic order; after data filtering, this graph includes 26 samples. The green dashed line indicates the lower temperature limit of the Rock Eval™ oil-generation window for mature oils, which corresponds to 435–465°C, as measured on the Rock Eval™ instrumentation. All analyzed samples plot far below the oil window, indicating the sedimentary rocks are thermally immature and have not been subject to high burial temperatures. A general trend of increasing thermal maturity with increasing stratigraphic depth is apparent on Figure GS-13-3b, as would be expected for a sequence of this thickness.

Figure GS-13-3c is a plot of the production index (PI), which is a measure of the hydrocarbon generation potential of a source rock, where samples from a thermogenically derived hydrocarbon system would plot between 0.1 and 1.0 (Zhang, 2008). PI is the ratio of $S_1/(S_1+S_2)$, where the parameter S_1 is a measure of free or adsorbed hydrocarbons volatilized during heating of the sample up to 300°C, and S_2 a measure of the hydrocarbons released during gradual heating from 300 to 550°C at increments of 25°C/min. All 26 samples in the filtered dataset in Figure GS-13-3c plot well below 0.1, the thermogenic minimum indicated by the green dashed line; this is expected for a thermally immature shale sequence, in which gas is considered to be formed by biogenic processes (Nicolas and Grasby, 2009). Based on the established abundance of organic matter in most of the studied formations, the low PI values indicate that there is a significant amount of free and/or adsorbed natural gas present in the samples.

A modified van Krevelen diagram (Figure GS-13-4) uses the hydrogen index (HI) and the oxygen index (OI) derived from Rock Eval™ analysis to distinguish kerogen types in the samples. In this diagram, samples that contain type I or type II kerogen are indicative of variably oil-prone sediments, whereas samples that contain type III kerogen are indicative of gas-prone sediments. Samples represented here plot in the fields for type II and type III kerogen. Their distribution is similar to that observed in the larger dataset of Nicolas and Bamburak (2009) and is consistent with the overall immaturity of the sediments. It should be noted that all samples from the Favel Formation plot closer to the type II line, which is consistent with the fact that this formation is classified as an oil shale.

Inorganic geochemistry

Instrumental neutron activation analysis (INAA) and inductively coupled plasma–emission spectrometry (ICP-ES) results from Cretaceous black shale and other sedimentary rocks from outcrops and oilwell core and chips collected in southwestern Manitoba for the SUSG Project were previously reported by Bamburak (2008), Nicolas and Bamburak (2009), Bamburak and Nicolas (2010a, b) and Nicolas et al. (2010).

Figure GS-14-2 of Nicolas et al. (2010) shows that there is a relative enrichment of Th, U and rare earth elements (REE) in the Gammon Ferruginous Member of the Pierre Shale and in the Babcock beds of the Boyne Member of the Carlile Formation compared with other Cretaceous stratigraphic units, suggesting that there may be potential for economic Th, U and/or REE mineralization in these rocks. In addition, it should be noted that the Babcock beds also show a corresponding increase in TOC and petroleum potential (S_1+S_2).

Bamburak and Nicolas (2010a, b) reported on trace-element composition of the Gammon Ferruginous Member at 12 sample localities in the Pembina Hills area of southwestern Manitoba and northeastern North Dakota. In September 2010, an additional outcrop locality was sampled at the Pembina Gorge, in North Dakota (Bamburak and Nicolas, 2010a, Table GS-15-1, locality 13; Figure GS-15-2), but the analytical results (shown in MGS Data Repository Item DRI2011004[2]) were not received until February 2011 and therefore not included in their report. These results from locality 13 proved relatively anomalous compared to other Cretaceous outcrop samples contained in the chemostratigraphic database, yielding lower values than the maximum values shown in Bamburak and Nicolas (2010a, Table GS-15-2).

Testing of the Th, U and REE mineral potential of the Gammon Ferruginous Member and of the Babcock beds in the southwestern corner of the province (Figure GS-13-2) by means of analysis of chip samples collected over 5 m intervals from the vertical portion (330 to 455 m in depth) of a petroleum development well spudded in L.S. 16, Sec. 30, Twp.7, Rge. 29, W 1st Mer. (abbreviated 16-30-7-29W1) was described by Bamburak and Nicolas (2010a). As a first approach, the chip samples were sent unwashed for chemical analysis, to avoid risking the loss of any mineralized fine fraction. The results of this preliminary test indicated that the chip samples collected from the Gammon Ferruginous Member and Babcock beds are only slightly elevated in Th, U and REE relative to background, as shown in MGS Data Repository Item DRI2011004. The chip samples have since been washed and will be resubmitted for inorganic geochemical analysis, as well as Rock Eval™/TOC analysis, to test whether or not the results against background can be improved.

Mineralogy

Understanding the mineralogy of gas-bearing shale is crucial when planning a well drilling, stimulation and completion program. This is due to the sensitivity of the minerals to the fluids used at every stage, as well as to the response of the rock to hydraulic fracturing. For example, the higher the quartz content, the better the rock will respond to fracturing since the rock is more brittle; similarly, the less swelling clays there are in the target zone, the less chance there is of formation damage during drilling. Analysis by X-ray diffraction (XRD) was used as the method of bulk mineral identification for the samples.

A suite of 68 out of the 79 samples analysed by Rock Eval™ were selected for XRD. The average mineral abundance for Cretaceous shale samples from the phase 2 area is shown in Figure GS-13-5. For the purposes of this representation, the Favel Formation samples without member designation were included in the Keld Member averages based on the striking similarity in XRD results shown by samples from these two groups. Three main shale categories can be seen in the figure: 1) calcareous, 2) quartz-rich, and 3) siliceous. The Keld and Assiniboine members of the Favel Formation are calcareous shales due to their high (>75%) calcite content, whereas the Odanah Member of the Pierre Shale is a siliceous shale that consists dominantly (>80%) of quartz, cristobalite and opal. All the other members represented have quartz as their dominant mineralogical component, commonly exceeding 55% of the bulk mineralogy for each member, with the remainder of the minerals being a mix of calcite, gypsum, siderite, jarosite and clay minerals. The Gammon Ferruginous Member, where it has been identified in outcrop, is a rusty to brown, quartz-rich, silty to sandy shale, siltstone and sandstone; its rich brown colour is due to iron-bearing minerals, such as siderite.

While it is understood that mineralogical variations within the Cretaceous shale may occur over very short vertical and horizontal distances, the samples described in this report represent a wide range of stratigraphic positions within the same member. Each of the Cretaceous shale members sampled in this area is therefore well represented by the average of the results shown here.

The complete XRD results discussed in this report are presented in MGS Data Repository Item DRI2011005[3].

Discussion

The results presented herein, combined with the findings published in previous years, provide a good basic geochemical and mineralogical understanding of the Late Cretaceous shale strata in southwestern Manitoba.

The information they provide support the following observations:

- The best targets for shale gas based on geochemical and mineralogical criteria are the Favel and Carlile formations, and the Belle Fourche Member of the Ashville Formation. However, the Gammon Ferruginous

[2] MGS Data Repository Item DRI2011004, containing the data or other information sources used to compile this report, is available online to download free of change at http://www2.gov.mb.ca/itm-cat/web/freedownloads.html, or on request from minesinfo@gov.mb.ca or Mineral Resources Library, Manitoba Innovation, Energy and Mines, 360–1395 Ellice Avenue, Winnipeg, Manitoba R3G 3P2, Canada.

[3] MGS Data Repository Item DRI2011005, containing the data or other information sources used to compile this report, is available online to download free of change at http://www2.gov.mb.ca/itm-cat/web/freedownloads.html, or on request from minesinfo@gov.mb.ca or Mineral Resources Library, Manitoba Innovation, Energy and Mines, 360–1395 Ellice Avenue, Winnipeg, Manitoba R3G 3P2, Canada.

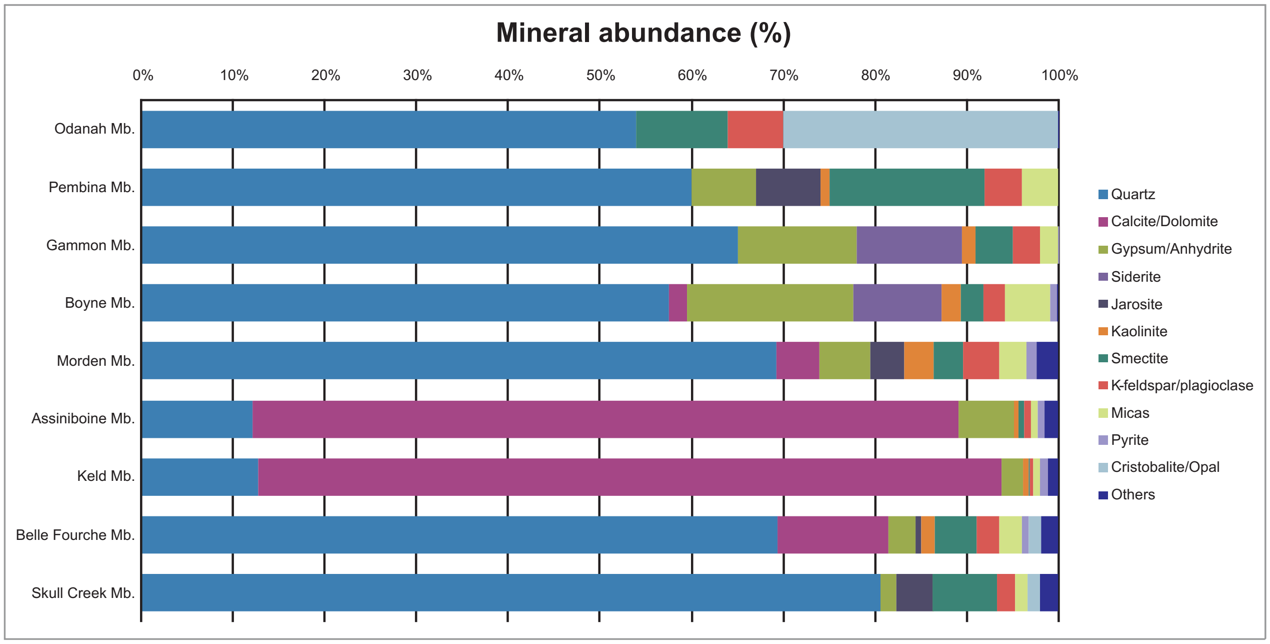


Figure GS-13-5: *Average mineral abundance for 68 Cretaceous shale samples from southwestern Manitoba, as determined by XRD. Abbreviations: Mb., Member.*

Member should also be considered, since it is identified as a potential unconventional reservoir on the southwestern rim of the Williston Basin (Spencer et al., 2010) and, in Manitoba, its organic chemistry signature classifies it as a moderately good source rock. The combination of its unique geochemistry with its silty and sandy composition makes the Gammon Ferruginous a target worthy of further study.

- Within the Carlile Formation, the silty to sandy Babcock beds in the Boyne Member make the latter a better target for shale gas than the Morden Member (Nicolas and Bamburak, 2009; Nicolas et al., 2010). The Morden Member, despite its high organic content and relatively high concentration of clay, micaceous and alteration minerals (such as gypsum and jarosite), rarely contains porous silty or sandy beds.
- The siliceous character of the Odanah Member makes it an attractive option as a reservoir since it is more likely to undergo brittle fracturing, but its organic content is very low (<0.5 wt. %), as reported in Nicolas and Bamburak (2009).

Economic considerations

The geochemistry and mineralogical information that has been gathered about the Late Cretaceous shale strata is important to providing a geoscientific knowledge base for further study and exploration. Using this information to identify units in Manitoba's Late Cretaceous shale sequences that have the right geochemical and mineralogical characteristics is an essential step toward expanding the potential pay-zone thickness at any given location, making exploratory drilling more attractive.

Acknowledgments

The authors would like to thank K. Osadetz of the Geological Survey of Canada-Calgary (GSC-Calgary) for his interest in this project and his assistance with the processing of the samples. The authors would like to acknowledge the GSC-Calgary Laboratories, particularly the organic geochemistry and mineralogy laboratories, for analyzing the samples and providing reliable technical support.

The authors would also like to acknowledge G. Benger, R. Unrau, V. Varga and the summer students of the Midland Core Facility for their assistance in preparing the samples for geochemical analysis. Thank you to B. Duncan from the Department of Geological Sciences at the University of Manitoba for filtering and preparing the data for interpretation.

The authors would further like to thank S. Anderson for the thorough review of this manuscript and constructive comments.

References

Bamburak, J. D. 1999: Cretaceous black shale investigations in the northern part of the Manitoba Escarpment (parts of NTS 62J/W, 62K/N, 62N/E and 63C/W); *in* Report of Activities 1999, Manitoba Industry, Trade and Mines, Geological Services, p. 120–122.

Bamburak, J. D. 2008: Geochemistry of Upper Cretaceous shale in southwestern Manitoba (NTS 63F, G, H4): potential reservoir rocks for shallow unconventional shale gas; *in* Report of Activities 2008, Manitoba Science, Technology, Energy and Mines, Manitoba Geological Survey, p. 180–184.

Bamburak, J.D. and Nicolas, M.P.B. 2010a: Gammon Ferruginous Member of the Cretaceous Pierre Shale in southwestern Manitoba: distribution and mineral potential (parts of NTS 62F, G, J, K, N, O, 63C); *in* Report of Activities 2010, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 170–177.

Bamburak, J.D. and Nicolas, M.P.B. 2010b: Selected analyses of the Gammon Ferruginous Member of the Cretaceous Pierre Shale, southwestern Manitoba (parts of NTS 62F, G, J, K, N, O, 63C); Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, Data Repository Item DRI2010001, URL <http://www.gov.mb.ca/stem/mrd/info/libmin/DRI2010001.zip>, Microsoft® Excel® file, ESRI® shape files.

Fedikow, M. A. F., Bezys, R. K., Nicolas, M.P.B. and Prince, P. 2009: Preliminary results of soil geochemistry surveys in support of shallow gas exploration, Manitou area, Manitoba (NTS 62G2); *in* Report of Activities 2009, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 193–206.

Nicolas, M.P.B. 2008: Summary report on petroleum and stratigraphic investigations, southwestern Manitoba; *in* Report of Activities 2008, Manitoba Science, Technology, Energy and Mines, Manitoba Geological Survey, p. 171–179.

Nicolas, M.P.B. and Bamburak, J.D. 2009: Geochemistry and mineralogy of Cretaceous shale, Manitoba (parts of NTS 62C, F, G, H, J, K, N): preliminary results; *in* Report of Activities 2009, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 165–174.

Nicolas, M.P.B. and Grasby, S.E. 2009: Water and gas chemistry of Cretaceous shale aquifers and gas reservoirs of the Pembina Hills area, Manitoba (parts of NTS 62G); *in* Report of Activities 2009, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 175–182.

Nicolas, M.P.B., Edmonds, S.T., Chow, N. and Bamburak, J.D. 2010: Shallow unconventional Cretaceous shale gas in southwestern Manitoba: an update (parts of NTS 62C, F, G, H, J, K, N); *in* Report of Activities 2010, Manitoba Innovation, Energy and Mines, Manitoba Geological Survey, p. 159–169.

Spencer, R.J., Pedersen, P.K., Clarkson, C.R. and Aguilera, R. 2010: Shale gas series: Part 1 – Introduction; Reservoir, no. 8, p. 47–51.

Zhang, S. 2008: New insights into Ordovician oil shales in Hudson Bay Basin: their number, stratigraphic position, and petroleum potential, Bulletin of Canadian Petroleum Geology, v. 56, p. 300–324.