1979OA194-07

Mineralogical Analyses by X-Ray Diffraction of Potential Gas Shales - Molopo - Wells A166, A184 and A194

Mineralogical Analyses

by X-Ray Diffraction

of Potential Gas Shales

Molopo

Well 166
Well 194
Well 184

November 24, 2009

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WELL SUMMARY

Well Name:	Various
Well Locations:	A166 A194 A184
Formations:	Utica and Lorraine
# of Zones Sampled:	**3**
# of Sample Collected:	11
Sample Type:	cuttings
Analyses Completed:	X-ray diffraction analysis, Rietveld quantification

EXECUTIVE SUMMARY

The mineralogical composition of 12 cutting samples from three Quebec wells (A166, A184, A194) was determined and then quantified using Rietveld refinement methods. Quartz, feldspar, carbonates, clay minerals and pyrite were detected and quantified. In most samples, quartz (27-47%), clays (14-33%) and carbonate minerals (10-48%) make up the majority of the composition. Albite (feldspar) ranges from 9-12% except in well 184 where it is 19% (2350), while pyrite ranges between 1-3.5%. In core 166, quartz, clays and albite decrease downcore whilst calcite increases from 2% at 4060 m depth to 35% at 4270 m depth. There, illite constitutes between 73-90% of the clay fraction with chlorite making up the remainder. In well 194, the seven samples of Lorraine A and B Members show very comparable mineralogical compositions and no real downhole trends are observed. Quartz (43-47%), albite (10-12%), carbonates (10-14%), clays (31-33%) and pyrite (1-2.6%) all show very narrow ranges. Well 184 had one sample analysed (Fm: Dolomite) which did not contain any carbonate minerals. Overall, no swelling clay minerals were identified in any of the samples.

DISCLAIMER

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INTRODUCTION

X-ray diffraction (XRD) analyses of **11** shale samples of three Quebec wells were carried out in order to determine and quantify the mineralogical composition. This report summarizes the methods and analytical results. XRD is generally the fastest and most reliable method used in the identification and quantification of crystalline materials. The technique utilises the diffraction (reflection) of X-rays from the unique arrangement of atoms in a crystal structure. The technique is particularly useful for materials with grain sizes too small for microscopic identification (ie. clay minerals, soils minerals, dusts etc.). The XRD trace data were analysed by Rietveld methods to quantify the mineralogy.

We would like to point out that Rietveld analysis only performs "quantitative" analysis of mineralogical phases identified from the XRD data. Mineral identification by ***CBM Solutions*** is done by reference to the International Centre for Diffraction Data database (*http://www.icdd.com/)*. Most minerals can be unambiguously identified except for clays and complex solid solutions such as feldspars, amphiboles or even carbonates. Feldspars, which make up ~60% of the Earth's crust contains the alkali feldspar (orthoclase/microcline–albite) and plagioclase (albite-anorthite) solid solution series where certain minerals have such similar X-ray diffraction patterns that they are essentially interchangeable for phase analysis. Carbonates on the other hand easily allow elemental substitution (i.e. Mg, Fe, Ni, Sr for Ca) which in small amounts will result in peak changes which are not present in the ICDD database. These carbonate phases will then be most likely slightly underestimated by the Rietveld refinement method. In addition, traces of some minerals may be present in the rock that produce only subtle diffraction patterns or the patterns are underlying others, more dominant phase patterns and hence their quantification may be difficult or impossible. Only if other data such as petrography or chemical analyses are available, better distinctions and mineral quantifications are possible. In order to characterize properly the mineralogical composition of rock samples, other methods need to be incorporated, such as petrographic thin sections, chemical (XRF) or image (SEM/TEM) analyses, etc.

METHODS AND SAMPLE DETAILS

Eleven shale samples were investigated for their mineralogy. The samples were ground to a homogenous particle size of ~2 micrometres using a micronising mill. Using ethanol as the transfer medium, smear slides of the ground material were made on glass disks and dried. X-ray diffraction was conducted between 3 and 70° 2Theta using a Cu X-ray tube with a Siemens D500 Kristalloflex instrument. The instrument features a 39 position sample changer, fixed divergence slit geometry, an anti-scatter slit and a curved graphite crystal diffracted beam monochromator.

After the samples were analysed, they were glycolated overnight in a glass desiccator at 60°C in order to determine expandable water sensitive clays (i.e. smectites or illite/smectite mixed-layers). These samples were then analysed between 3-27° 2Theta.

The results of the x-ray diffraction analyses were analysed using the PDF-4 Minerals database 2009 (peak identification) and then quantified using Jade 9 software, a commercial Rietveld analyses program for quantification of the mineralogy. For further information on Rietveld theory refer to http://en.wikipedia.org/wiki/Rietveld_refinement.

The standard patterns in the PDF-4 minerals database are used for both phase identification and quantification. Prior to all analysis, XRD pattern shifts were corrected using the Quartz 101 or Quartz 100 peak. For all samples, refinement of the scale factor and instrument zero was performed first and the background determination checked. The background was not corrected or subtracted prior to the Rietveld analysis.

In samples in which the mineralogy is composed of discrete minerals with fixed cell dimensions, such as calcite, quartz, gypsum and pyrite, the accuracy of the analyses by Rietveld is considered to be ±3-5% relative. Samples with disordered clays (or solid solution series), such as some samples in this study, the accuracy of the data is less (sometimes >±10%), the amount of which varies with the amount of disordered minerals. In samples in where substantial disordered clays are present Rietveld analyses does not fit the mineral patterns unless standard specifics to the samples are developed.

RESULTS AND INTERPRETATION

The quantitative mineralogy is tabulated in Table 1 and 2. Selected trace patterns and major phase peaks are provided in Fig. 1, 2 and Fig. 3. Please note that only the major peaks are labelled and many peaks have subordinate or underlying peaks of other mineral phases. The x-ray traces with mineral identification are provided on the CD as Appendix A Rietveld analyses.

Figure 1 shows the XRD pattern (only 3-34° 2Theta degrees) of sample MO2300 from MOLOPO Well 194. The figure illustrates the peak positions of the major minerals identified and the abundance of peaks identified in XRD analysis. Please be aware that most peaks have subordinate and underlying peaks of other mineral phases (i.e. the calcite 104 has an underlying illite peak, illite 112). ***No swelling clay minerals*** were identified in any of the samples from these wells. Sample MO2300 (Fig. 1) shows that quartz, calcite, albite, dolomite and clay minerals with small amounts of pyrite are dominating the mineralogical composition in this sample. Chlorite and illite make up the clay mineral assemblage. Well 194 has a similar mineral assemblage (Fig. 2) whilst the only sample of well 184 lacks carbonates (Fig. 3). It must be noted that the Rietveld quantification of clays can be difficult because of their overlying peak patterns (i.e. chlorite and kaolinite have overlying peak patterns, whilst illite has overlying patterns with other minerals, including calcite). Hence, quantification may not reflect the true percentage of these mineral phases.

Fig. 1: Example of typical mineral phases identified in the rock samples from Lorraine A in well A194 showing sample MO2300 (2300 m depth). Quartz, illite, chlorite, albite, calcite, dolomite, and pyrite are present. Scan length: 3-35° 2Theta.

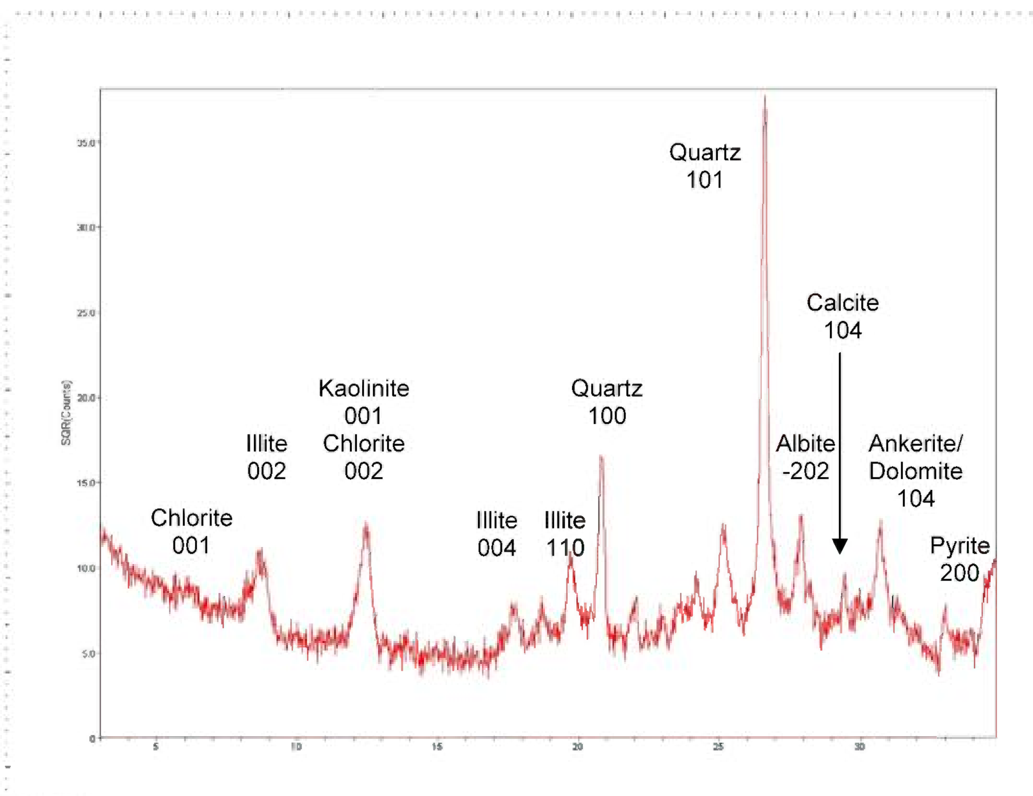


Fig. 2: Carbonate mineral phases identified in the rock samples from Molopo Well A166 (MO4270) showing the presence of quartz, illite, some chlorite, albite, calcite, dolomite, and pyrite.

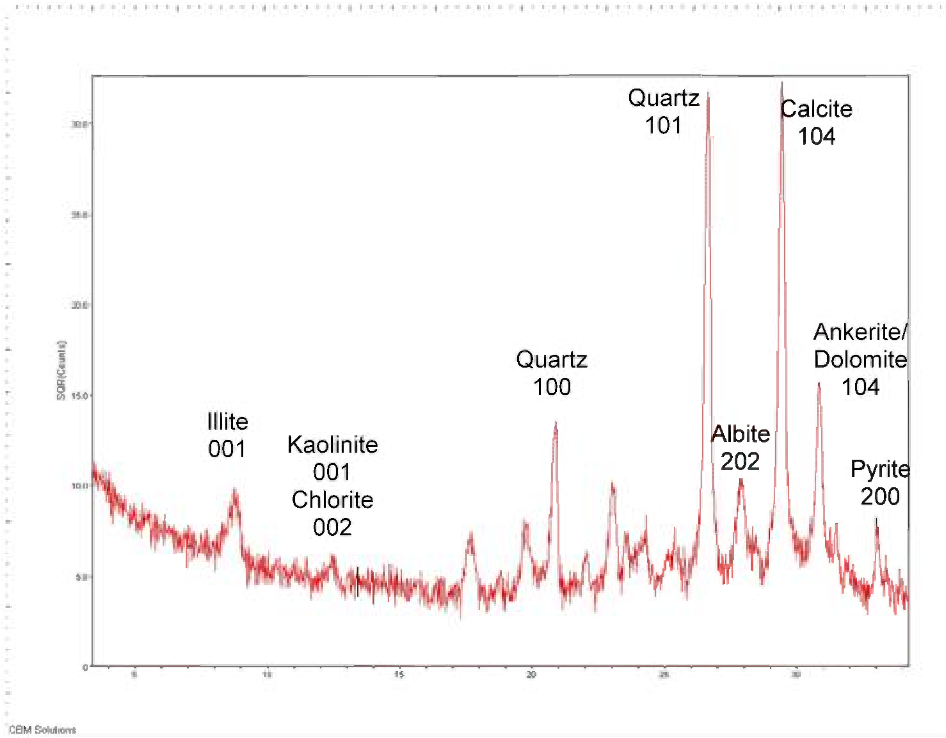
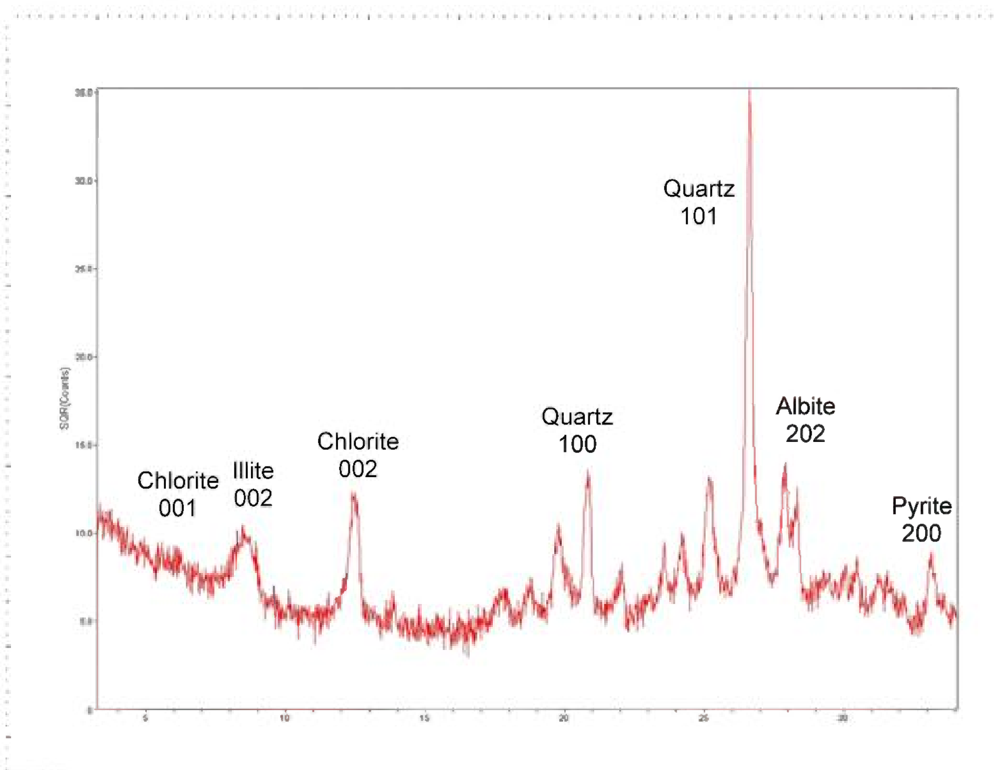


Fig. 3: Example of the mineral phases identified in the rock samples from well A184 showing sample MO2350 (Dolomite). Please note absence of both calcite and dolomite peaks around the 30° 2Theta range. Scan length: 3-35° 2Theta.



Mineralogy of the MOLOPO well A166

The mineralogical composition of the samples from MOLOPO well A166 are listed in Table 1. The samples belong to the Utica Formation and are composed of quartz, feldspars, carbonates (calcite and Fe-dolomite = ankerite), clay minerals (illite and chlorite) and pyrite. Across the four samples, quartz makes up between 27% (4270) and 44% (4060) (Fig. 4). Feldspars are dominated by albite that ranges between 8.5% (4270) and 12% (4060). The carbonate fraction contains calcite ranging between 2% (4060) and 35% (4270), whilst dolomite (mainly Fe-dolomite) makes up between 12-14%. The ankerite/dolomite peak position in all samples suggests that no pure end-members of the dolomite-ankerite solid solution are present but rather a mixture of the solid solution spectra.

The clay fraction of the samples from Well A166 is composed of mainly illite (12-20%), with 2-7% of chlorite and therefore, the total clay content ranges between 14 and 27%. ***No kaolinite or swelling clays were identified.*** Pyrite is present in all samples and ranges between 2-3%.

Table 1. Mineralogy of the MOLOPO well A166 quantified using Rietveld Analyses. Please note that there is a poor fit for illite, chlorite and occasionally calcite/dolomite peaks due to poor crystallinity and not pure endmembers (i.e. calcite with small % of Mg substitutions) which could result in underestimations of these mineral phases.

Molopo - Well166								
Depth m	Formation	Quartz	Feldspar	Carbonate		Clays		Pyrite
			Albite	Calcite	Ankerite	Illite	Chlorite	
4060	Upper Utica	44.2	12.4	1.6	12.1	19.7	7.3	2.7
4150	Upper Utica	36	9.7	18.6	14.2	15.6	3.4	2.5
4200	Upper Utica	31	8.9	29.5	12.8	13.9	1.6	2.3
4270	Upper Utica	27.3	8.5	34.7	13.1	11.9	2.5	2.1

In Well A166, a general downhole trend in the mineralogical composition can be observed through the transition from lowermost Lorraine to uppermost Utica (Fig. 4, 5). Quartz decreases from 44% at 4060 m depth to 27% at 4270 m depth. A similar decrease is observed within the clay fraction that decreases from 27% to 14% over the same interval. In contrast, calcite steadily increases from 2% at 4060 m depth to 35% at 4270 m depth (Fig. 5).

The clay fraction is composed of illite and chlorite (Table 1, Fig. 4) and shows a general downhole decrease. The relative percentage of the two clay phases shows that the top samples has a higher percentage of chlorite (27%) compared to the three samples further down which have chlorite fractions of 10-18% (Fig. 6).

PLEASE NOTE: the general mineralogical trends described in the section of Well A166 may be reflective of the entire section and thus represent the true trends of the lithology but it could also be the result of having only four data points!

Fig. 4: Downhole mineralogical composition of MOLOPO well A166 illustrating the mineralogical changes with depth in the transition from lowermost Lorraine to uppermost Utica.

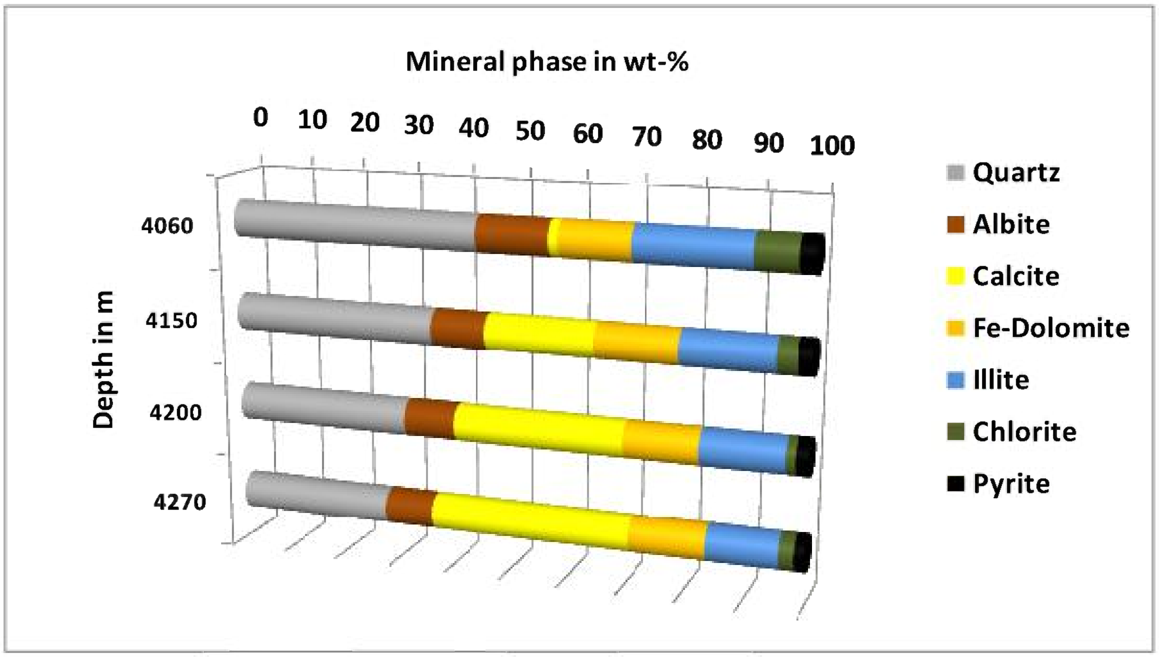


Fig. 5: Left: Quartz and carbonate content variations with depth. Right: Albite and total clay content variations with depth in well A166. Note the decreasing trends of quartz and clays (and to a small degree albite) and the increasing trend of the carbonates (mainly the calcite fraction).

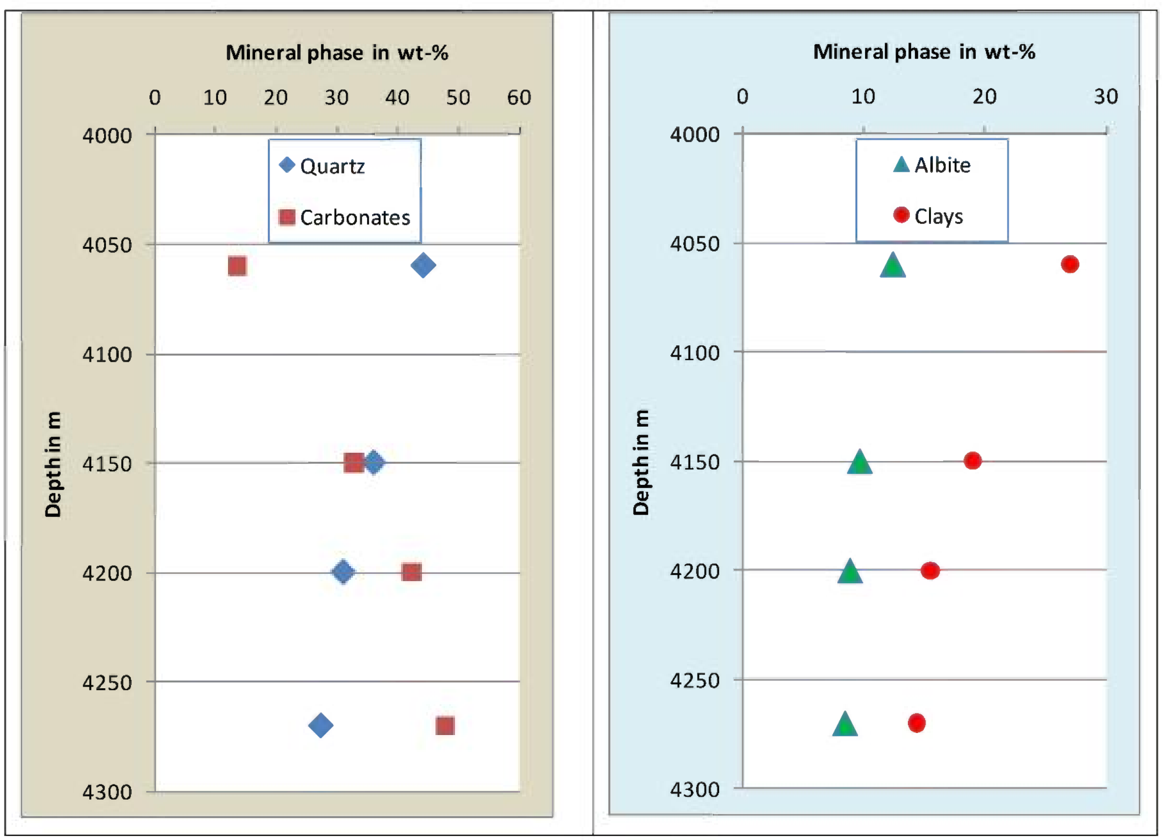
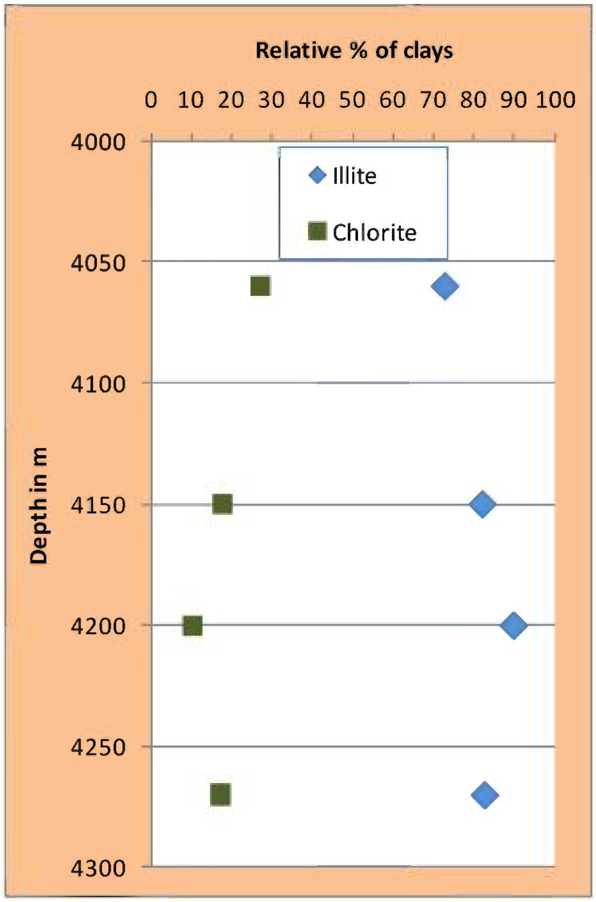


Fig. 6: Relative clay percentage of the MOLOPO well A166 samples quantified using Rietveld Analyses. The top sample has a slightly higher chlorite/illite ratio than the three samples further down.



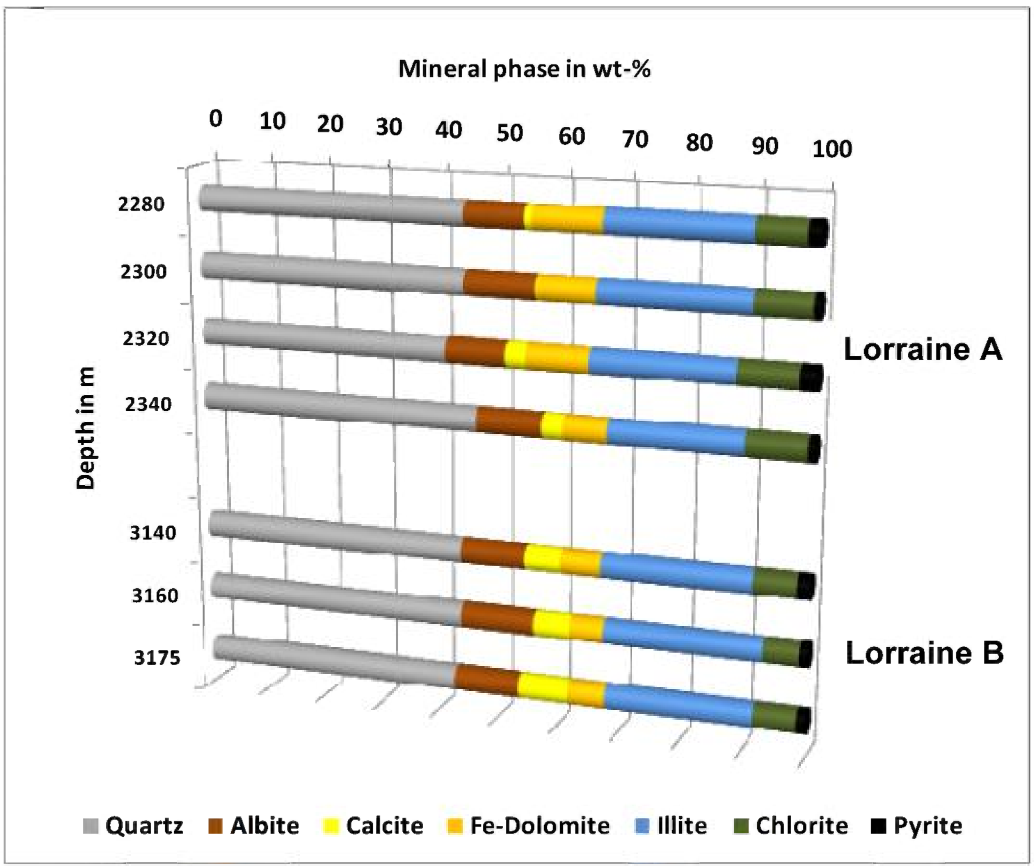
Mineralogy of the core MOLOPO Well A194

The mineralogical composition of the samples from MOLOPO well A194 are listed in Table 2. The samples belong to the Lorraine A and Lorraine B Members and are composed of quartz, feldspars, carbonates (calcite and Fe-dolomite = ankerite), clay minerals (illite and chlorite) and pyrite. The samples of Lorraine A and Lorraine B have a very similar composition. Across the seven samples, quartz makes up between 42-47% (Fig. 4). Feldspars, dominated by albite, range between 10-12%. The carbonate fraction contains calcite up to 8% (3175), whilst dolomite (mainly Fe-dolomite) makes up between 5% (3160, 3170) and 11% (2280). The ankerite/dolomite peak position in all samples suggests that no pure end-members of the dolomite-ankerite solid solution are present but rather a mixture of the solid solution spectra.
The clay fraction of the samples from Well A194 is similar to the samples of Well A166 composed of mainly illite and chlorite. Illite content ranges from 22 (2340) to 25% (3160), whilst the chlorite content ranges from 6% (3160) to 10% (2320, 2340). Total clay content of these seven sample is very consistent and ranges from 31-33%. Pyrite is present in all samples and ranges between 1-2.6%.

Table 2. Mineralogy of the MOLOPO well A194 quantified using Rietveld Analyses. Please note that there is a poor fit for illite and occasionally calcite/dolomite peaks due to poor crystallinity and not pure endmembers (i.e. calcite with small % of Mg substitutions) which could result in underestimations of these mineral phases.

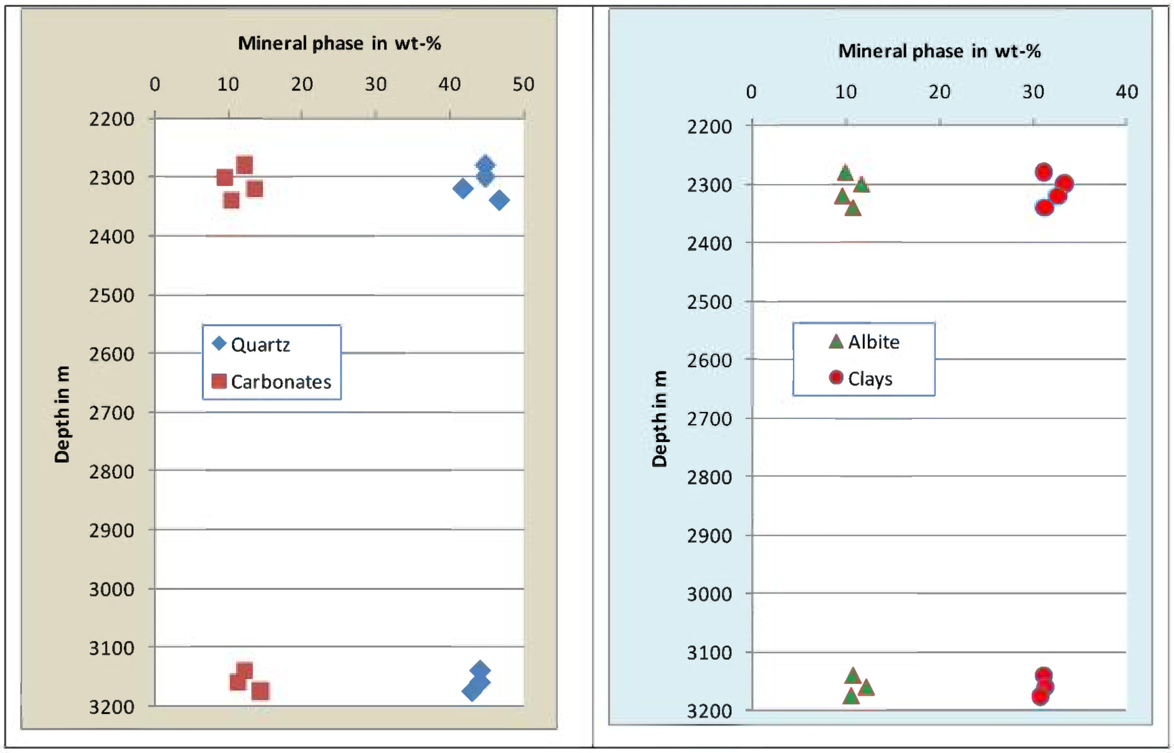
Molopo - Well194								
Depth m	Formation	Quartz	Feldspar	Carbonate		Clays		Pyrite
			Albite	Calcite	Ankerite	Illite	Chlorite	
2280	Lorraine A	44.7	10	1.1	11.1	23.3	7.8	2.1
2300	Lorraine A	44.7	11.7	0.3	9.2	24.4	8.9	0.9
2320	Lorraine A	41.7	9.7	3.5	10	22.9	9.7	2.6
2340	Lorraine A	46.6	10.8	3.3	7.1	21.5	9.7	1
3140	Lorraine B	44	10.8	5.7	6.5	24.3	6.8	1.9
3160	Lorraine B	44	12.2	6	5.3	25.5	5.8	1.2
3175	Lorraine B	42.9	10.6	8.4	5.9	24	6.8	1.5

Fig. 7: Downhole mineralogical composition of MOLOPO well A194. Note the similarity of mineralogical composition amongst all samples across both Lorraine A and Lorraine B Members.



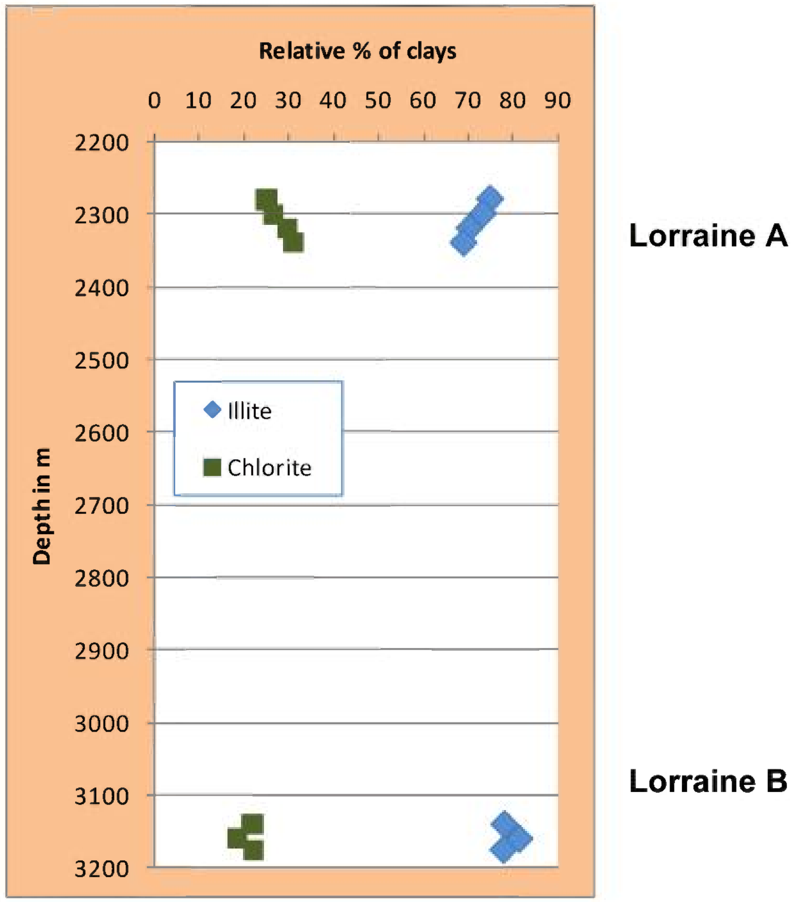
In contrast to the mineralogical downhole trends of Well A166, the mineralogical composition across the samples of Well A194 appears much more consistent and little trends occur (Fig. 7, 8). a general downhole trend in the mineralogical composition can be observed (Fig. 4, 5). In fact, the mineralogical composition of both Lorraine A and Lorraine B samples are almost identical with the exception of slightly higher calcite contents of the Lorraine B samples (6-8%) compared to 0.3-3.5% of the Lorraine A samples (Fig. 7). However, most of the other minerals have a very narrow range, such as the quartz (42-47%), albite (10-12%), total carbonates (10-14%), total clay contents (6-10% and pyrite (1-2.6%) (Table 2, Fig. 7, 8).

Fig. 8: Left: Quartz and carbonate content variations with depth. Right: Albite and total clay content variations with depth in well A166. Note the decreasing trends of quartz and clays (and to a small degree albite) and the increasing trend of the carbonates (mainly the calcite fraction).



The clay fraction is these samples are also composed of illite and chlorite (Table 2, Fig. 7). There, both samples suits are also similar to each other. The Lorraine A samples average about 32.5% clays whilst the Lorraine B samples average about 31% (Fig. 8). Comparing the relative clay composition, i.e. chlorite versus illite ratio, the Lorraine A samples have a slightly higher chlorite percentage (~28%) compared to the Lorraine B samples (~21%) (Fig. 9). The remainder is composed of illite. No kaolinite or swelling clays were identified.

Fig. 9: Relative clay percentage of the MOLOPO well A194 samples quantified using Rietveld Analyses.



Mineralogy of the core MOLOPO well A184

The only sample of MOLOPO well A184 contains quartz, feldspar, clay minerals (illite, chlorite) and pyrite (Table 3). No carbonate mineral were identified in the XRD trace (Fig. 3). Quartz makes up 40%, albite 19%, illite 27%, chlorite 10% and pyrite 3.5%. No water-sensitive (i.e. expandable) clay minerals were identified.

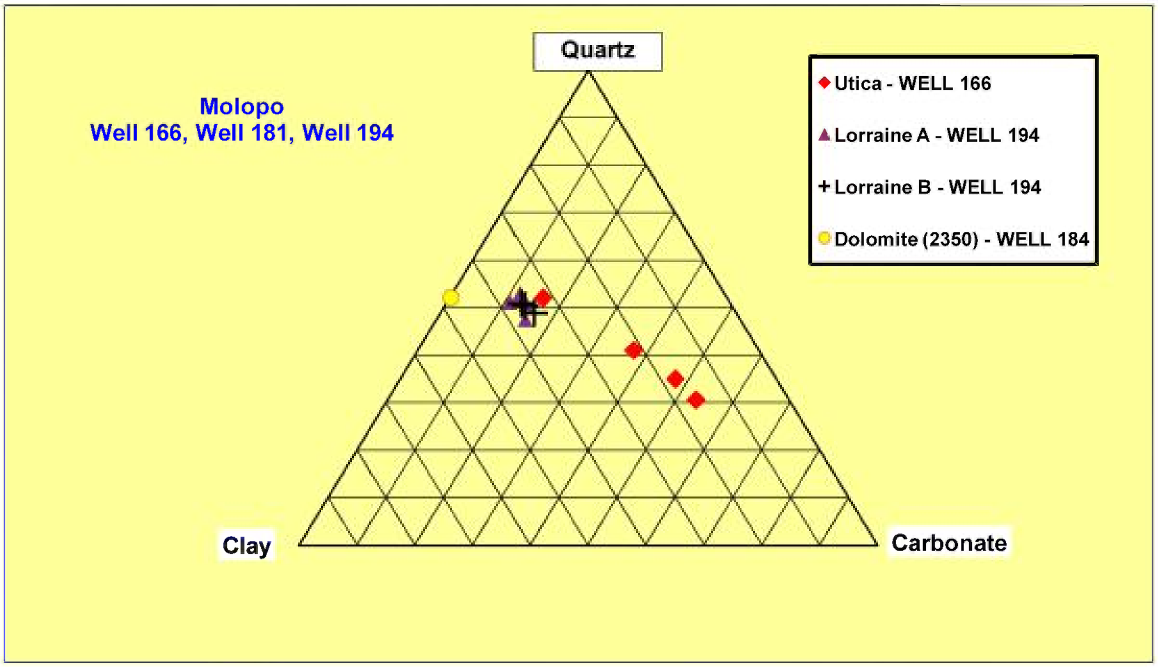
Table 3. Mineralogy of the MOLOPO well A184 quantified using Rietveld Analyses.

Molopo - Well184								
Depth m	Formation	Quartz	Feldspar	Carbonate		Clays		Pyrite
			Albite	Calcite	Ankerite	Illite	Chlorite	
2350	Dolomite	40.4	19.3	0.1	0.1	26.7	10.1	3.5

Ternary Diagram – quartz – clays – carbonates – of all wells (A166, A184, A194)

All samples were plotted on a ternary plot with the end members of quartz, clays and carbonates to illustrate the population distribution according to the individual units (Formation, Members) (Fig. 10). The samples of Well A194 and A184 plot close to the center of the quartz-clay line whilst only the top sample of well A166 (4060) plots closely there too. The other samples of Well A166 plot closer to the carbonate endmember due to their higher carbonate contents.

Figure 10: Ternary Diagram of the samples from the various MOLOPO wells (A166, A184, A194) showing the quartz, clay and carbonate contents.



CONCLUSIONS

The mineralogical composition of 12 cutting samples from three MOLOPO wells (A166, A184, A194) was determined and then quantified using Rietveld refinement methods. Quartz, feldspar, carbonates, clay minerals and pyrite were detected and quantified. In most samples, quartz (27-47%), clays (14-33%) and carbonate minerals (10-48%) make up the majority of the composition. Albite (feldspar) ranges from 9-12% except in well 184 where it is 19% (2350), while pyrite ranges between 1-3.5%. In core A166, quartz, clays and albite decrease downcore whilst calcite increases from 2% at 4060 m depth to 35% at 4270 m depth. There, illite constitutes between 73-90% of the clay fraction with chlorite making up the remainder. In well 194, the seven samples of Lorraine A and B Members show very comparable mineralogical compositions and no real downhole trends are observed. Quartz (43-47%), albite (10-12%), carbonates (10-14%), clays (31-33%) and pyrite (1-2.6%) all show very narrow ranges. Well A184 had one sample analysed (Fm: Dolomite) which did not contain any carbonate minerals. Overall, no swelling clay minerals were identified in any of the samples.

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