

## RESPONSE TO COMMENTS BY LANGMUIR (2007)

December 1, 2007

### Comments

The comments made by Langmuir (2007) regarding a draft report on the MgO Excess Factor Planned Change Request by DOE (SCA 2007) can be summarized as follows:

Comment #1 – It is not certain that all MgO will react to completion with CO<sub>2</sub>; DOE assumes that all periclase present in the MgO will react, which may not be adequately supported.

Comment #2 – Physical segregation of MgO from CO<sub>2</sub> may prevent complete reaction; DOE assumes this will not occur.

Comment #3 – It is questionable whether the results of DOE's small-scale laboratory experiments can be extrapolated to conditions in the WIPP repository to support the assumption that all MgO in the repository will be available to react:

- Because of differences in the solution to MgO ratios between the inundated experiments and repository conditions;
- DOE has not performed hydration or carbonation experiments under conditions or at a scale that attempts to reproduce the conditions under which hydration and carbonation reactions will occur in the WIPP repository.
- The environment within a disposal room is likely to be heterogeneous and pockets of unreacted MgO are likely to persist;
- There will be a massive increase in the volume of magnesium solids that will result from the hydration and carbonation of MgO, which will affect the performance and reactivity of the MgO backfill. If the MgO emplaced in the repository becomes coated or armored with Mg(OH)<sub>2</sub>, and that Mg(OH)<sub>2</sub> clogs its porosity because of the large volume increase, then some MgO and some Mg(OH)<sub>2</sub> may be unavailable for further reaction.

### Response

The performance of the MgO backfill is important to the WIPP performance assessment under both humid and inundated conditions. Under humid conditions, the amount of brine present in the repository is insufficient for release of radionuclides by direct brine release. In the absence of direct brine release, the repository chemical conditions and resulting actinide solubilities are not important for repository performance. Consequently, the only important function of the MgO backfill under humid conditions is to control gas pressures by reaction with most of the CO<sub>2</sub>. This issue was specifically addressed by the Conceptual Models Peer Review Panel; the Panel determined that DOE had sufficiently resolved the issue of MgO reactivity for the purposes of the Gas Generation conceptual model by demonstrating that sufficient access of brine and CO<sub>2</sub> to the MgO would occur to substantially remove CO<sub>2</sub> as a pressure source (Wilson et al. 1996b, Section 3.21.3.3).

The questions raised by Langmuir (2007) regarding the effectiveness of the MgO barrier to control chemical conditions are only relevant to inundated conditions, when sufficient brine is available for a direct brine release. Under inundated conditions, the MgO must react to control pH and CO<sub>2</sub> partial pressures and constrain actinide solubilities; these inundated conditions may occur after an initial period of humid conditions. The responses to these comments are organized according to the factors that could affect the availability of the MgO backfill for complete reaction.

Physical Segregation. Langmuir (2007) noted that the issue of physical segregation of MgO was raised by an expert panel, who stated that “local pockets of un-reacted MgO are likely to be present for long periods of time” (RSI 2006, Finding 4, page 63). However, RSI (2006) did not provide the basis for this finding or an explanation of the mechanism by which such pockets of MgO might be isolated from brine and gas in the repository. Physical segregation of MgO as a potential source of uncertainty in the MgO Excess (Safety) Factor was discussed by SCA (2006, page 6-1). SCA (2006) stated that this uncertainty was small because of the methods currently used to emplace MgO in the repository, but that this small remaining uncertainty should be incorporated into the MgO Excess Factor. Vugrin et al. (2007, page 18) addressed the possible physical segregation of MgO by roof collapse, and found that physical segregation was unlikely to occur by intrusion of the roof block into the waste because the roof was likely to lower onto the waste stacks and MgO. In addition, Vugrin et al. (2007) explained that failures of smaller portions of the roof (small blocks or spillings) were unlikely to physically segregate MgO because of the small scale of the blocks and spillings and the likelihood of fractures and high permeability in these smaller failed portions of the roof.

The assumption of significant physical segregation of part of the waste-gas-brine-MgO system is inconsistent with the assumption of chemical homogeneity that was accepted by the Conceptual Models Peer Review Panel (Wilson et al. 1996a). The Conceptual Models Peer Review Panel found that the assumption of chemical homogeneity “should be wholly valid over the time frame involved” (Wilson et al. 1996a, page 3-154). No contradictory evidence has been presented since the Conceptual Models Peer Review, and DOE has adequately explained why physical segregation is unlikely to occur after repository closure (Vugrin et al. 2007). Consequently, the possibility of physical segregation of MgO does not require reconsideration and it does not appear to be a significant source of uncertainty. To improve the explanation of the basis for assuming that physical segregation of MgO would have an insignificant effect on the amount of MgO available for reaction, the SCA (2007) report was revised to include a discussion of the Conceptual Models Peer Review Panel’s acceptance of the assumption of chemical homogeneity (Section 3.4, page 3-9).

Formation of Reaction Rims on MgO Pellets. The possibility that individual MgO pellets could become coated by reaction products and thereby be rendered unavailable for reaction was considered an important source of uncertainty by the Conceptual Models Peer Review Panel (e.g., Wilson et al. 1996b, Section 3.22.3.3). In response to requests for additional information from the Panel, SNL (1997) provided experimental evidence demonstrating that hydromagnesite would nucleate away from the surface of the periclase grains under inundated repository conditions and that isolating reaction rims would not

form. The evidence reviewed by the Panel included experimental results, optical microscopy, scanning electron microscopy, modeling predictions, analogue comparisons, and phase equilibria information. Based on a review of this information, the Conceptual Models Peer Review Panel agreed that the formation of reaction rims on hydrated MgO pellets would not significantly affect the function of the MgO engineered barrier (Wilson et al. 1997b, Section 3.2.3.3); the Panel found the most compelling evidence to be “1) the experimental results indicating that hydrous magnesium carbonate phases could nucleate away from the pellet surface and in the saturated brine, and 2) SEM photographs showing partially dissolved cores remaining within reaction rims.”

DOE has continued investigating the reaction of MgO backfill materials with brine and CO<sub>2</sub> (Bryan and Snider 2001a; Bryan and Snider 2001b; Snider 2001; Zhang et al. 2001; Snider 2002; Snider and Xiong 2002; Snider 2003; Xiong and Snider 2003). Hydration experiments have been conducted under inundated and humid conditions, and carbonation experiments have been conducted under inundated conditions. Several WIPP Test Plans (Bynum 1997, Snider et al. 2004, Deng et al. 2006) have described potential humid MgO/brucite carbonation experiments, but no results have been reported from these experiments; consequently it appears that these carbonation experiments under humid conditions have not been performed. In the humid hydration experiments and in the inundated hydration and carbonation experiments performed since the Conceptual Models Peer Review, there has been no evidence of reaction rim formation on the MgO pellets that would hinder complete reaction.

The SCA (2007) report was modified to include a more detailed explanation of the experimental results that indicate impermeable reaction rims are not expected to form on individual MgO pellet surfaces. Information was also added to indicate that this issue was thoroughly reviewed by the Conceptual Models Peer Review Panel, who found that this issue had been adequately addressed (Section 3.2, pages 3-4 to 3-6).

Reaction Rind Formation on Masses of MgO. The Conceptual Models Peer Review Panel expressed concern that the formation of reaction products on the outside of the MgO backfill packages could seal off a significant amount of the MgO and prevent complete reaction (Wilson et al. 1996a, page 3-155). This concern was attributed to the formation of lower density and lower permeability material upon initial contact with brine on the outside of the packages (Wilson et al. 1996b, Section 3.21.3.3). The final Conceptual Models Peer Review consideration of MgO reactivity (Wilson et al. 1997b) did not explicitly discuss the effects of the volume changes that could occur during the hydration and carbonation of MgO. However, these issues were raised in the Panel’s previous reports (Wilson et al. 1996a, Wilson et al. 1996b, Wilson et al. 1997a). Consequently, the Panel’s conclusion that the MgO barrier would function as designed appears to have included consideration of this issue. The basis of this conclusion was the evidence provided by SNL (1997) that (1) the magnesium carbonate reaction products would not form in place, but would nucleate elsewhere in the repository; and (2) the magnesium carbonate reaction products that formed would remain permeable to brine.

During a review of the MgO backfill performance, EPA accepted DOE’s assertion that the formation of reaction products on the surfaces of the backfill material would not have a significant effect on the ability of the MgO to maintain predicted repository chemical conditions (EPA 1997, pages 44-6 through 44-11). EPA stated that, based on a review of

information in Bynum et al. (1996), “The formation of reaction products on the surfaces of the backfill material do(es) not have a significant, detrimental impact on the ability of the MgO to maintain the predicted chemical conditions.” EPA also noted DOE’s intention to emplace sufficient MgO backfill in the repository to ensure CO<sub>2</sub> consumption would exceed the rate of CO<sub>2</sub> production.

The possible effects of volume changes and the formation of impermeable masses during the hydration and carbonation of the MgO backfill were addressed during EPA’s review of the CCA (EPA 1997, EPA 1998). This issue was raised in Comment 6.W.5 (EPA 1998, page 6-67). EPA stated in their response that the effects of increased reaction product volume as well as the consumption of water were likely to be beneficial to backfill performance. Although EPA stated in their response that the formation of dense, cementitious layers of backfill could limit access of brine to the waste, the evidence reviewed above shows that such cementitious layers are not expected to form. Comments 5.E.2, 5.E.5, 5.E.9, 5.E.10, 5.E.15, 5.E.16, 5.E.18, 5.E.23, and 5.E.24 raised questions regarding the effects of MgO backfill on waste permeability (EPA 1998, pages 5-25 through 5-30). EPA (1998, page 5-31 through 5-33) calculated the change in porosity in a waste disposal room caused by the precipitation of hydromagnesite; the results demonstrated that the overall reduction in porosity caused by hydromagnesite precipitation was likely be only 1.4% of the initial porosity. EPA (1998) related the permeability to the porosity and concluded that the MgO backfill and its reaction products were unlikely to significantly affect permeability in the waste region of the repository.

The possible “lithification” of MgO during hydration under inundated conditions was investigated in a series of experiments with 5 to 15 mm-thick layers of MgO backfill material (Snider 2002). Although there was significant scatter in the results, there was no evidence that an impermeable mass of hydration products formed.

Volume changes will occur as the periclase in the MgO pellets is hydrated and carbonated, as noted by Langmuir (2007). However, these volume changes would be unlikely to significantly limit access of brine and gas to unreacted periclase in the MgO because the carbonation reaction products are expected to nucleate throughout the repository, not just on the MgO (SNL 1997, Wilson et al. 1997b). The dissolution of MgO and the ability of magnesium carbonates to nucleate away from the surface of the MgO pellets were demonstrated by SNL (1997) to the satisfaction of the Conceptual Models Peer Review Panel and EPA, as described above. In addition, evidence has been presented that the reaction products will remain permeable to brine (Bynum et al. 1996, SNL 1997). This evidence was considered and accepted by both the Conceptual Models Peer Review Panel (Wilson et al. 1997b) and the EPA (EPA 1997, 1998). Other factors that may limit the formation of a dense, impermeable mass of MgO carbonation products would be fracturing of the reaction products during room closure or fracturing as a result of the volume changes that would occur as reaction proceeds. No new evidence is available indicating that MgO hydration and carbonation products will form impermeable masses that interfere with the functioning of the MgO engineered barrier. Although DOE originally planned to include a large excess of MgO in the repository, neither the Conceptual Models Peer Review Panel nor the EPA state that this large excess was a critical factor in their determination that the MgO barrier would perform as expected.

Consequently, in the absence of new data, there does not appear to be a basis for revising the assumption that essentially all MgO will be available for reaction. The resolution of this issue was clarified in SCA (2007) by including a discussion of the experimental results reported by Bynum et al. (1996) and SNL (1997) and the consideration of this issue by the Conceptual Models Peer Review Panel and EPA (Section 3.2, pages 3-4 to 3-6). The effects of hydromagnesite precipitation on porosity and permeability were addressed by inserting a discussion of the EPA (1998) calculations in the SCA (2007) report (Section 3.4, page 3-9).

Applicability of MgO Experiments to the WIPP Environment. Experiments have been conducted at Sandia National Laboratories to determine the likely reactions of the MgO backfill in the WIPP repository environment. Some of these experiments were conducted under conditions designed to accelerate reaction rates over those expected in the repository to ensure the experiments could be completed within a reasonable amount of time. Some hydration and carbonation experiments have been conducted at higher temperatures and CO<sub>2</sub> partial pressures than those predicted for the repository environment, with agitation of the samples, and with higher solution to solids ratios than anticipated in the repository. Conducting the hydration and carbonation experiments at conditions different from those expected in the WIPP repository could affect the applicability of the results to repository conditions.

MgO experiments conducted at higher temperature and CO<sub>2</sub> partial pressures (e.g., SNL 1997) have been supplemented by experiments conducted at temperature and CO<sub>2</sub> partial pressure conditions more consistent with expected repository conditions (e.g., Snider 2003, Xiong and Snider 2003). Higher CO<sub>2</sub> partial pressures (5% CO<sub>2</sub>) have resulted in the initial formation of nesquehonite [MgCO<sub>3</sub> • 3H<sub>2</sub>O(s)] in some experiments; however, this nesquehonite was observed to disappear, being replaced by hydromagnesite (Snider and Xiong 2002). If nesquehonite formed and persisted in the repository, predicted CO<sub>2</sub> partial pressures and actinide solubilities would be higher than if hydromagnesite or magnesite formed. However, experiments conducted at lower CO<sub>2</sub> partial pressures (10<sup>-3.5</sup> atm) produced hydromagnesite (Snider and Xiong 2002), demonstrating that nesquehonite is unlikely to form or persist under repository conditions.

The results of experiments designed to determine the effects of sample agitation on the potential formation of reaction rims or impermeable masses were reported by Snider (2002); these “cemented cake” experiments were discussed above. The experiments showed that sample agitation did not have a consistent effect on reaction rates. In addition, because the available evidence shows that impermeable reaction rinds will not form on MgO pellets, sample agitation is likely to have relatively minor effects on the results of the inundated hydration and carbonation experiments.

Many of the MgO backfill hydration and carbonation experiments have included relatively high solution to solid ratios. The larger amounts of solution were used to facilitate solution sampling. However, Bynum et al. (1996) and SNL (1997) reported a series of experiments designed to more closely simulate the lower solution to solids ratios expected in the repository. In these experiments, MgO pellets were placed in a porous bag that was partially suspended in brine through which CO<sub>2</sub> was bubbled. At the end of the experiments, the porous bag was removed from the brine, placed in a dye solution,

removed from the dye, cemented in epoxy, then sectioned and examined to determine the ability of the dye to permeate the reaction products. The experimental results indicated that MgO was likely to continue to react and maintain the required repository chemical conditions. There is relatively little evidence that the solution to solids ratios of the experiments had a significant effect on the nature of the solid reaction products. Bryan and Snider (2001b) reported the results of experiments with varying ratios of GWB brine to solids. In the experiments with GWB brine, a magnesium-chloride-hydroxide-hydrate material was observed to form. The proportions of this phase were found to increase with higher solution to solids ratios in the experiments. Consequently, at the lower solution to solids ratio in the repository, little of this phase is expected to form, and its formation is not expected to have a significant effect on the function of the MgO engineered barrier.

There are a number of inherent difficulties in performing large-scale MgO hydration and carbonation experiments that would more closely reproduce expected repository conditions, especially given the relatively slow reaction rates and long time frame involved. These difficulties led to the review of the Chemical Conditions model by the Conceptual Models Peer Review Panel. Based on a consideration of the experimental data and the conclusions of the Panel, the available data appear to be reasonably representative of repository conditions and additional large-scale experiments do not appear to be required for an understanding of MgO hydration and carbonation reactions in the repository environment. The information incorporated in response to the comments regarding the potential formation of reaction rims on individual periclase granules and of reaction rinds on masses of MgO more fully describe the available experiments and address this issue (Section 3.2, pages 3-4 to 3-6).

Reaction Sequence. During additional review of SCA (2007), it appears that the report may not clearly state that the expected reaction sequence in the repository and in the carbonation experiments is for the initial hydration of periclase in the MgO to brucite and in GWB, magnesium-hydroxide-chloride hydrate, followed by carbonation of these magnesium hydroxide phases. Changes were made on pages 4-1, 4-3, 4-5, and 4-12 of SCA (2007) to make the discussion of this reaction sequence more consistent throughout the report.

**Summary and Conclusions**—The issues raised by Langmuir (2007) in the review of SCA (2007) include issues that were considered by the Conceptual Models Peer Review Panel (Wilson et al., 1996a, 1996b, 1997a, 1997b) and by EPA (1997, 1998) during the review of the CCA and CCA PAVT. These issues relate to the assumption of chemical homogeneity and the possible formation of hydration and carbonation reaction products that could limit access of brine and CO<sub>2</sub> to the interior of the MgO pellets or to the interior of the masses of MgO emplaced in the repository. No new data have been developed since the time of the Conceptual Models Peer Review or the CCA PAVT to contradict the assumptions that were accepted by the Conceptual Models Peer Review Panel or EPA at that time. The available documentation clearly states that the formation of reaction rims on the MgO pellets was not expected to occur and the assumption of chemical homogeneity was reviewed and accepted. It is also clear that the formation of cementitious layers by the hydrated and carbonated MgO was considered by the EPA and Conceptual Models Peer Review Panel and that they determined that formation of

cementitious layers would not prevent the engineered barrier from controlling chemical conditions (EPA 1998).

The documentation of the Peer Review Panel's and EPA's evaluation of the ability of the MgO barrier to perform as expected to control chemical conditions does not provide much information regarding the importance of the large excess of MgO that was originally proposed to be placed in the repository. At the time of the CCA, it was estimated that complete biodegradation of all CPR in the repository would result in the carbonation of only 26% of the MgO backfill because of the large amounts of MgO to be emplaced and the assumption that methanogenesis will be a significant process in CPR degradation (SNL 1997). However, recent evaluation of the available sulfate in the Salado formation resulted in the bounding assumption that all CPR carbon could be transformed into CO<sub>2</sub>. This assumption, combined with the proposed reduction in the MgO Excess Factor from 1.67 to 1.2, makes the assumption that all MgO will be available for reaction with CO<sub>2</sub> more critical. For example, if reaction of only 26% of the MgO was required to maintain chemical conditions in the repository, the segregation of less than 74% of the MgO by reaction product formation would not affect chemical conditions. However, given the theoretical possibility that all CPR degradation could take place by denitrification and sulfate reduction that would transformed the CPR carbon into CO<sub>2</sub>, combined with the proposed smaller proportion of the moles of MgO to moles of CPR carbon to be placed in the repository, even a small amount of MgO segregation, if it occurred, could affect chemical conditions.

Because of the difficulties associated with developing experiments that would simulate the behavior of the MgO in the repository environment over 10,000 years, it would be extremely difficult to design useful, large-scale experiments that would determine whether a significant fraction of MgO could be sequestered by the volume changes associated with MgO hydration and carbonation. In cases where experiments cannot be performed, a peer review must be carried out of the necessary assumptions. The Conceptual Models Peer Review Panel previously reviewed and approved the Chemical Conditions conceptual model (Wilson et al. 1997b). Consequently, because it appears that no new information has been developed that would contradict their conclusions, chemical homogeneity in the WIPP repository and the essentially complete reaction of the MgO engineered barrier can continue to be assumed. On this basis, it appears that sufficient technical information is available for EPA to determine the appropriate Excess Factor for MgO in the WIPP repository.

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