

Preliminary Results for the Evaluation of Potential New MgO

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INTRODUCTION

The primary purpose of MgO in WIPP is "to decrease the solubilities of the actinide elements in transuranic (TRU) waste in any brine present in the repository after closure. ... In this report, 'MgO' refers to the bulk, granular material being emplaced in the WIPP to serve as the engineered barrier. MgO comprises periclase (pure, crystalline MgO - the main, reactive constituent of the WIPP engineered barrier) and various impurities. Pure, crystalline MgO is always referred to as periclase in this document. ... MgO will decrease actinide solubilities by consuming essentially all carbon dioxide (CO₂) that would be produced by microbial consumption of all cellulosic, plastic, and rubber (CPR) materials in TRU waste, ... waste containers [and emplacement materials] in the repository. ... Consumption of CO₂ will prevent the acidification of brine or the production of significant quantities of carbonate ion (CO₃²⁻), which could increase actinide solubilities. Another function that MgO will perform in the repository is to consume water (H₂O) in brine, or H₂O vapor in the gaseous phase. ... After the WIPP is filled and sealed, periclase will react with H₂O in the gaseous phase to form brucite (Mg(OH)₂), or with H₂O in brine to form brucite or amorphous or crystalline Mg-Cl-OH-H₂O-bearing solids such as Mg₃Cl(OH)₅·4H₂O or Mg₂Cl(OH)₃·4H₂O. Periclase, brucite, and the Mg-Cl-OH-H₂O phases will react with aqueous or gaseous CO₂ to form solids such as hydromagnesite (Mg₅(CO₃)₄(OH)₂·4H₂O)" (U.S. DOE, 2004, Appendix BARRIERS).

Sandia National Laboratories – Carlsbad Program Group was requested by the DOE/CBFO to evaluate several samples from Martin Marietta Magnesia Specialties LLC (referred to as "Martin Marietta" hereafter) for possible use as the WIPP engineered barrier. If selected, Martin Marietta would replace Premier Chemicals, the current supplier of MgO. We are carrying out this evaluation by performing accelerated hydration tests, and are comparing the amount of brucite formed from the Martin Marietta samples to results obtained from similar tests with Premier MgO in 2001. This report presents the preliminary results of this work. We are also optimizing the parameters used to obtain accurate and meaningful hydration results. This work is carried out under the test plan of Snider et al. (2004), and the results are contained in the notebooks WIPP-MgO-CBD-19 and WIPP-MgO-CBD-20.

WIPP: 1.4.2.5: TD: QA-L^A Engineered Barrier
 DPRP1: NF: 11271051 of 35
 RH

Information Only

It is assumed that the quantity of the reactive constituent (periclase) that reacts with aqueous H₂O in these accelerated experiments is identical to that which will react with aqueous or gaseous CO₂ in the WIPP. Therefore, this experimental work quantifies the extent to which the MgO engineered barrier will consume CO₂, which is in turn used (along with other information) to calculate MgO safety factors.

It should be noted that this work is not a test of the specifications required by WTS (2003). These requirements are:

1. "The sum of magnesium oxide (MgO) plus calcium oxide (CaO) shall be a minimum of 95%, with MgO being no less than 90%. The remainder of the material shall not contain any items considered hazardous to people or the environment.
2. Backfill material shall be of a dry granular form, which shall contain less than 0.5% particles which would be retained on a Tyler 3/8 inch sieve (3/8" × down).
3. Backfill material shall be test for reactivity as outlined in Attachment B, and shall meet the temperature rise listed in the rest procedure.
4. The backfill material shall have a minimum loose bulk density of 87 lb/ft³."

Also, this work does not include any long-term testing (at either high or room temperatures) necessary to develop a kinetic model to predict the carbonation of this MgO. Finally, this work does not include characterization of the Martin Marietta MgO samples to the same extent as was previously done for Premier MgO, which involved X-ray-diffraction analysis and analysis of samples by scanning electron microscopy.

MATERIALS

We received four samples of Martin Marietta MgO on August 27, 2004. Two of the samples did not meet the required density of 87 lb/ft³ specified by WTS (2003); we did not carry out any experiments with these samples. The two other MgO samples, MagChem 10 WTS-30 (referred to as "WTS-30" hereafter) and MagChem 10 WTS-20 (WTS-20), did meet all the WTS specifications. We received another MgO sample from Martin Marietta: MagChem 10 WTS-60 (WTS-60) on September 24, 2004, which meets all WTS specifications, but shows a "marginal 87 lb/ft³ density."

As specified by Martin Marietta, WTS-30, WTS-20, and WTS-60 are hard-burned magnesium oxide containing a minimum of 97% MgO. Sixty to 90% of WTS-30 passes through a U.S. 60 mesh screen sieve, while only 40% of WTS-20 passes through the same sieve. WTS-60 presents even larger size grains than WTS-30 and WTS-20; 95% to 90% pass through 6M mesh sieve, 50% to 80% pass through 16M mesh sieve, 15% to 40% pass through 30M sieve, and 15% to 30% pass through 100M mesh sieve screen. Therefore, the sizes of the three Martin Marietta MgO samples follow the order: WTS-30 < WTS-20 < WTS-60. As a result, the grain size of WTS-30 and WTS-20 could present a higher respiratory hazard for workers than WTS-60.

METHODS

We perform accelerated hydration experiments to compare the amount of brucite formed from the three samples, WTS-30, WTS-20, and WTS-60. Similar experiments had been conducted in the study of Premier MgO.

The accelerated hydration test is performed as follows:

1. Place 5 g of MgO, weighed with a calibrated balance, in a high-density polyethylene or 125-mL polypropylene bottle.
2. Add 100 mL deionized (DI) water.
3. Place bottles in a 90 °C oven. Record the day and time at which the bottles are placed in the oven.
4. Shake the bottle three times a day for the first 10 days; after that, shake the bottles only once a day.
5. At a predetermined and recorded day and time, remove a bottle from the oven.
6. Immediately filter the content of the bottle through a Whatman ashless filter paper #41. Discard the liquid and keep the solid in a labeled container in a dessicator.
7. When the solid is dried, crush it with a mortar and pestle to obtain a fine powder. The powder is kept in the dessicator until loss on ignition (LOI) analysis.
8. The solid is tested by LOI, which is performed as follows:
 - A. Weigh a crucible on a calibrated balance.
 - B. Add about 2 g of MgO powder to be analyzed to the weighed crucible, weigh the crucible containing the MgO.
 - C. Place the crucible and its contents in a 60 °C furnace for 2 hours. This step is intended to remove the loosely bound water molecules, which are not participating in the formation of brucite.
 - D. Let the crucible and its contents cool down in a dessicator.
 - E. Weigh the cooled crucible with its contents.
 - F. Place in a 500 °C (or 750 °C) furnace for 3 hours. This step is intended to convert brucite to periclase.
 - G. Let the crucible and its contents cool down in a dessicator.
 - H. Weigh the cooled crucible with its contents.

The amount of brucite formed at the end of the test is calculated from the loss of water after the sample is dried at 60 °C and heated at 500 °C (or 750 °C).

The LOI-test parameters are also being evaluated. This test follows Steps 1 to 7 of the previous procedure. The last step is a modified version of Step 8 of the previous procedure:

1. Weigh a crucible on a calibrated balance.
2. Add about 2 g of MgO powder to be analyzed to the weighed crucible, weigh the crucible and the MgO.
3. Place the crucible and its contents in the furnace, set at a predetermined temperature, T_1 , for a recorded period of time.
4. Let the crucible and its contents cool down in a dessicator.
5. Weigh the cooled crucible and its contents, and record the mass m_1 .

6. Place the crucible and its contents in the furnace, set at the same temperature T_1 as previously used, for a recorded period of time.
7. Let the crucible and its contents cool down in a desiccator.
8. Weigh the cooled crucible with its contents; record the mass as m_2 .
 - A. If $m_1 - m_2 < 0.0005$ g, place the crucible and its contents in the furnace, set at a predetermined temperature, T_2 (where $T_2 > T_1$), for a recorded period of time.
 - B. If $m_1 - m_2 > 0.0005$ g, place the crucible and its contents in the furnace, set at the same temperature, T_1 , for a recorded period of time.
9. Repeat 1. to 8. until data are obtained for a temperature of 800 °C.

RESULTS AND DISCUSSION

Figures 1 through 3 present the amount of brucite formed, expressed as weight percent (wt %) of generated brucite, as a function of time, calculated from the results obtained after the LOI tests at 500 °C, for WTS-30, WTS-20, and WTS-60. The results obtained by A.C. Snider in 2001 for the test performed on Premier MgO are also included in each figure. On these figures, the different shades of diamond symbols indicate different batch experiments, which show the excellent reproducibility of the experimental results. The data presented in Figures 1 through 3 are also presented in Appendix Tables A1 through A3. These tables present the period of time each MgO sample was allowed to equilibrate with DI-water (age), the weight of the crucible and of the crucible containing the reacted MgO, both before and after heating at 60 °C or 500 °C, and the calculated wt % and mole percent (mol %) of brucite formed. The wt % of generated brucite is calculated from the LOI results, assuming that all the water consumed by the MgO during the hydration experiment converts exclusively to brucite. The mol % of brucite formed is calculated from the LOI results, assuming that all the water consumed by the MgO material during the hydration experiment converts exclusively to brucite and that the original MgO is exclusively periclase (pure crystalline MgO).

Figure 4 shows the superposition of the results (wt % of generated brucite) for WTS-30, WTS-20, WTS-60, and Premier MgO.

Finally, Figure 5 presents the amount of brucite formed as a function of time, calculated from the results obtained after LOI tests at 750 °C, for WTS-30; Figure 5 shows the results obtained by Snider and Xiong (2004) for Premier MgO after a similar LOI test. The data used to generate Figure 5 are presented in Table A1; the column labels are as described above. We also attempted to perform LOI tests at 750 °C for WTS-20, but no useful results could be obtained from these analyses, because of the tendency of this material to decrepitate when heated at such a high temperature. We have not attempted to run any LOI tests at 750 °C for WTS-60 to date, because we expect that we will have the same problem for WTS-60 at 750 °C as we have for WTS-20. Figures 1, 2, and 5 present the amount of brucite (wt %) in the unreacted materials (i.e., for the materials prior to any contact with water).

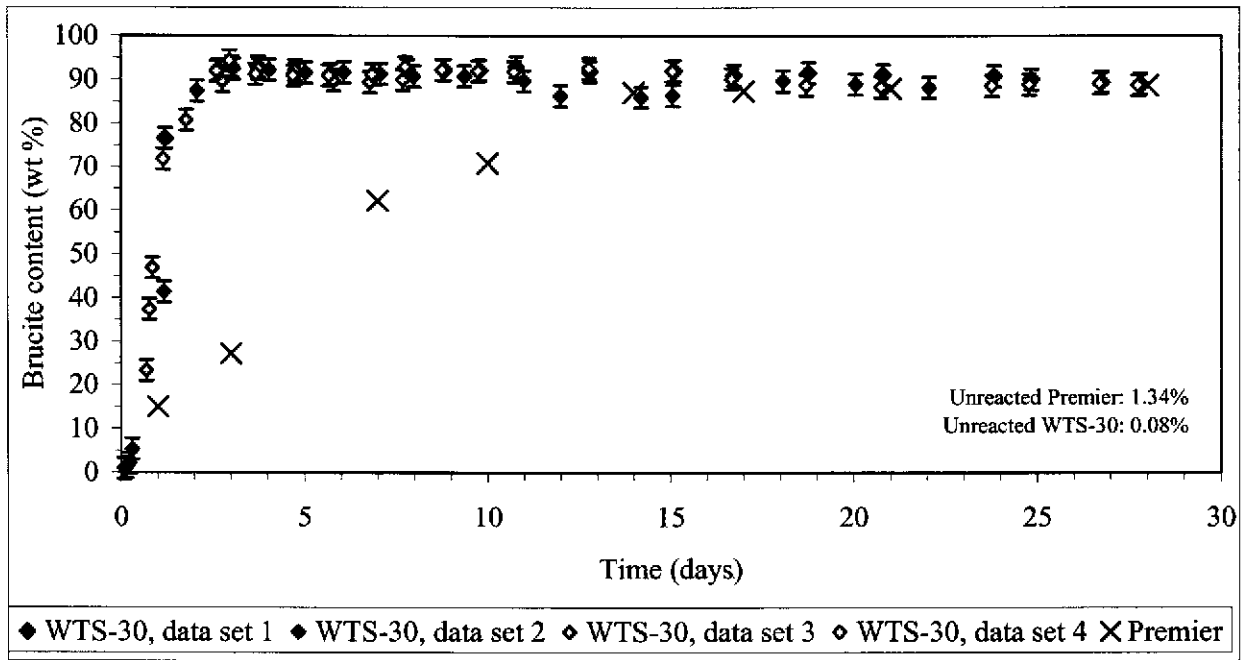


Figure 1. Comparison of the hydration of WTS-30 and Premier MgO in DI water at 90 °C. LOI at 500 °C.

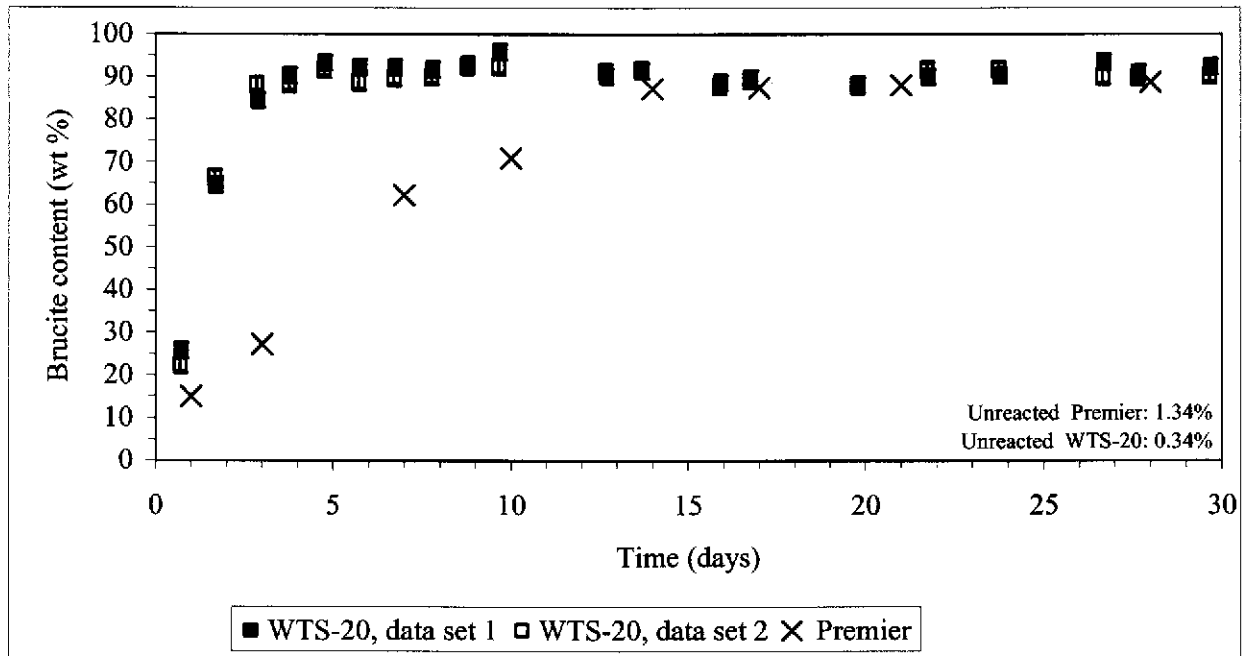


Figure 2. Comparison of the hydration of WTS-20 and Premier MgO in DI water at 90 °C. LOI at 500 °C.

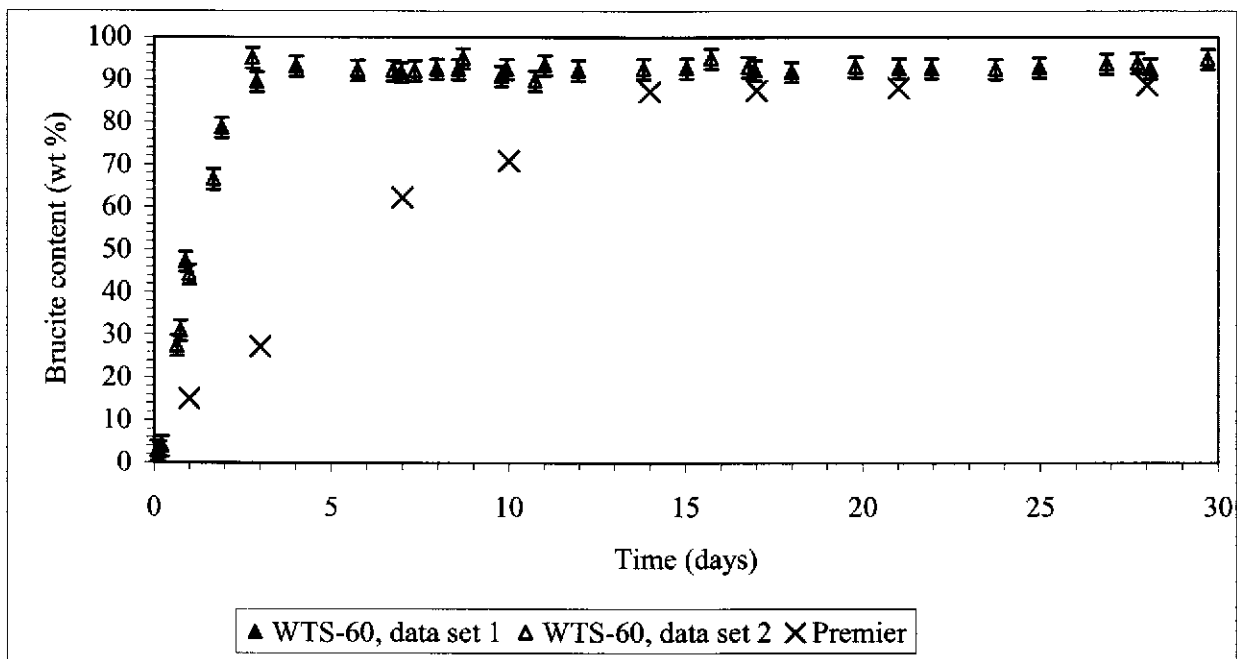


Figure 3. Comparison of the hydration of WTS-60 and Premier MgO in DI water at 90 °C. LOI at 500 °C.

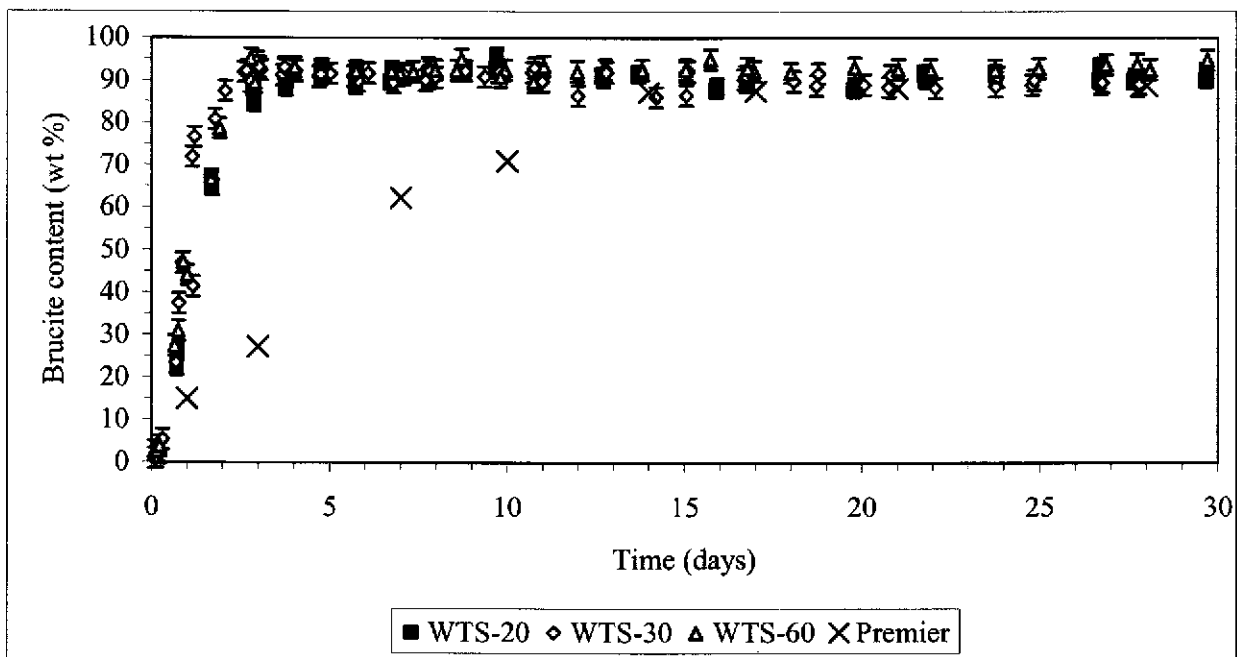


Figure 4. Comparison of the hydration of WTS-30, WTS-20, WTS-60, and Premier MgO in DI water at 90 °C. LOI at 500 °C.

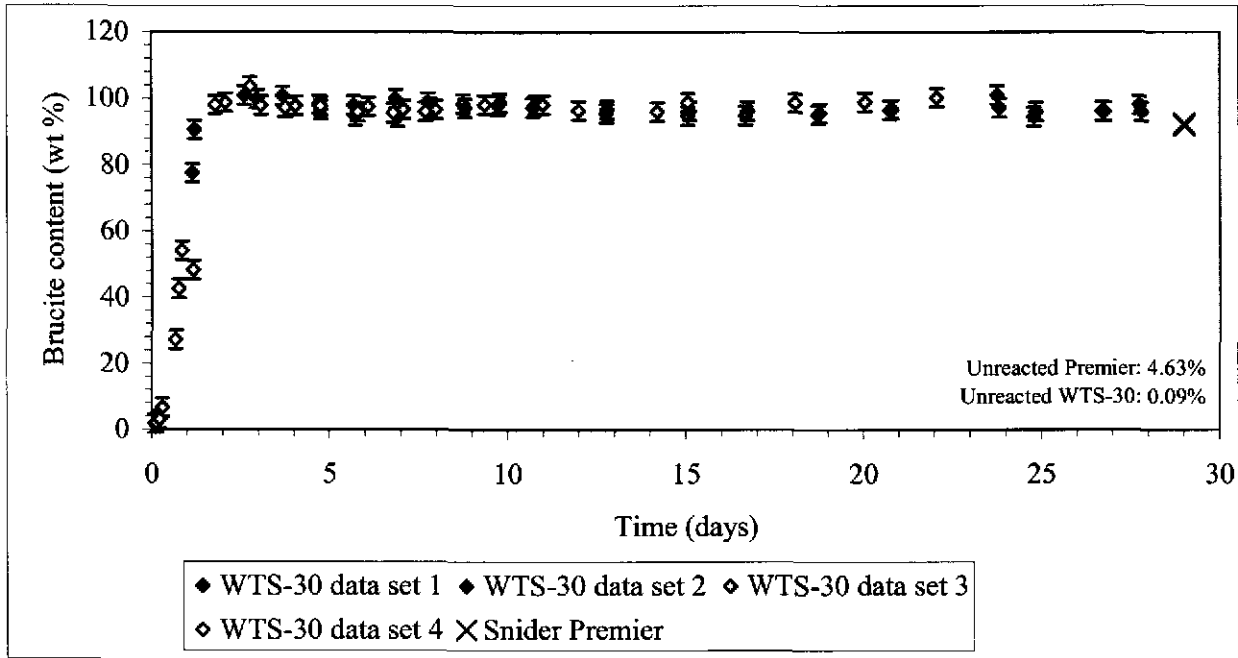


Figure 5. Comparison of the hydration of WTS-30 and Premier MgO in DI water at 90 °C. LOI at 750 °C.

Table 1 presents the average maximum wt and mol percents of brucite generated from each MgO sample (Premier MgO or Martin Marietta MgO), based on the LOI tests performed at 500 °C or 750 °C.

Table 1: Summary of Reactivity Data (mol% or wt % brucite produced from periclase)

LOI Temperature:	500 °C		750 °C	
	Mol %	Wt %	Mol %	Wt %
Premier MgO ⁽¹⁾	84.6	88.8	89	92
WTS-30 ⁽²⁾	87 +/- 5	91 +/- 4	96 +/- 5	97 +/- 3
WTS-20 ⁽²⁾	87 +/- 5	91 +/- 4	-	-
WTS-60 ⁽²⁾	90 +/- 3	93 +/- 2	-	-

(1): From U.S. DOE (2004, Appendix BARRIERS) and Snider and Xiong (2004).

(2): This work.

Figures 1 through 4 and Table 1 show that the three Martin Marietta MgO samples hydrate similarly when subjected to the accelerated hydration test: the average wt % of brucite formed at equilibrium, based on the LOI test at 500 °C, from either WTS-30 or WTS-20 is $91\% \pm 4$ (2σ) and it is $93\% \pm 2$ (2σ) for WTS-60. All three Martin Marietta MgO samples react faster than Premier does. A maximum amount of brucite is generated from WTS-30, WTS-20, and WTS-60 after only 3 to 4 days, while the maximum amount of brucite generated from Premier MgO occurs after 15 days.

Figure 5 and Table 1 show that the LOI test at 750 °C yields higher calculated amounts of generated brucite than the results from an LOI at 500 °C. The average wt % of brucite formed at equilibrium from WTS-30 is $97\% \pm 3$ (2σ) based on the LOI test at 750 °C, while it is $91\% \pm 4$ (2σ) for tests run at 500 °C. This is also observed for Premier MgO: the average wt % of brucite formed at equilibrium from Premier, from LOI tests at 500 °C and 750 °C are 88.8% and 92%, respectively (Snider and Xiong, 2004). The data obtained for the unreacted MgO materials shows that the material left in the original container produces almost no brucite (i.e., periclase does not hydrate appreciably to brucite during storage).

In addition to the reactivity tests we perform, we are also optimizing the parameters used to obtain accurate and meaningful hydration results. A slight modification of the parameters might improve the LOI results; for example, a slow increase of temperature might avoid the decrepitation we observe for WTS-20 and WTS-60 at 750 °C. The experiments are currently being performed. In the current LOI test method, it is assumed that the samples should be dried for 2 hours at 60 °C to remove the loosely bound water molecules, which are not participating in the formation of brucite. Also, it is assumed that the samples brought to 500 °C for 3 hours, after the 60 °C drying step, should convert all the brucite to periclase. The present experiment is performed to (1) ensure that the period of time used to heat the samples at 60 °C and 500 °C is sufficient, (2) ensure that 60 °C is high enough to dry the sample, (3) ensure that 500 °C is appropriate to convert all and only brucite to periclase, (4) evaluate the type of minerals which decompose when heated for 3 hours at 750 °C, and (5) evaluate whether a gradual heating to 750 °C would prevent decrepitation of the material in the furnace.

The data are presented in Figure 6; they are plotted as the percent of mass lost between two measurements, after completion of drying at 60 °C. The experimental data are also presented in the Appendix, in Tables A4 to A6. Three replicates of each Martin Marietta MgO samples are being tested. The time necessary to obtain stable sample masses at 60 °C, 100 °C, 150 °C, and 200 °C are 2, 7, 2, and 6 hours, respectively. The tests at 300 °C have been performed for 45 hours and are still underway. The Martin Marietta sample WTS-30 has reached equilibrium and could be tested at a higher temperature, but the samples WTS-20 and WTS-60 have still not reached equilibrium for the 300 °C test after 45 hours. The data show that the rate at which a constant mass is obtained is a function of the grain size of the material; the larger the original grain size, the longer the sample must be heated. Similarly, the mass loss during the heating is a function of the original grain size; the larger the grain size, the more mass is lost in the furnace. The data also show that a 2 hour period is sufficient to obtain a constant mass for the 60 °C test. Moreover, additional calculations show identical results whether the percent of mass lost is calculated using the data obtained after the 60 °C, the 100 °C, or the

150 °C tests. Therefore, heating a sample at 60 °C for 2 hours is sufficient to remove the loosely bound water from the reacted MgO samples.

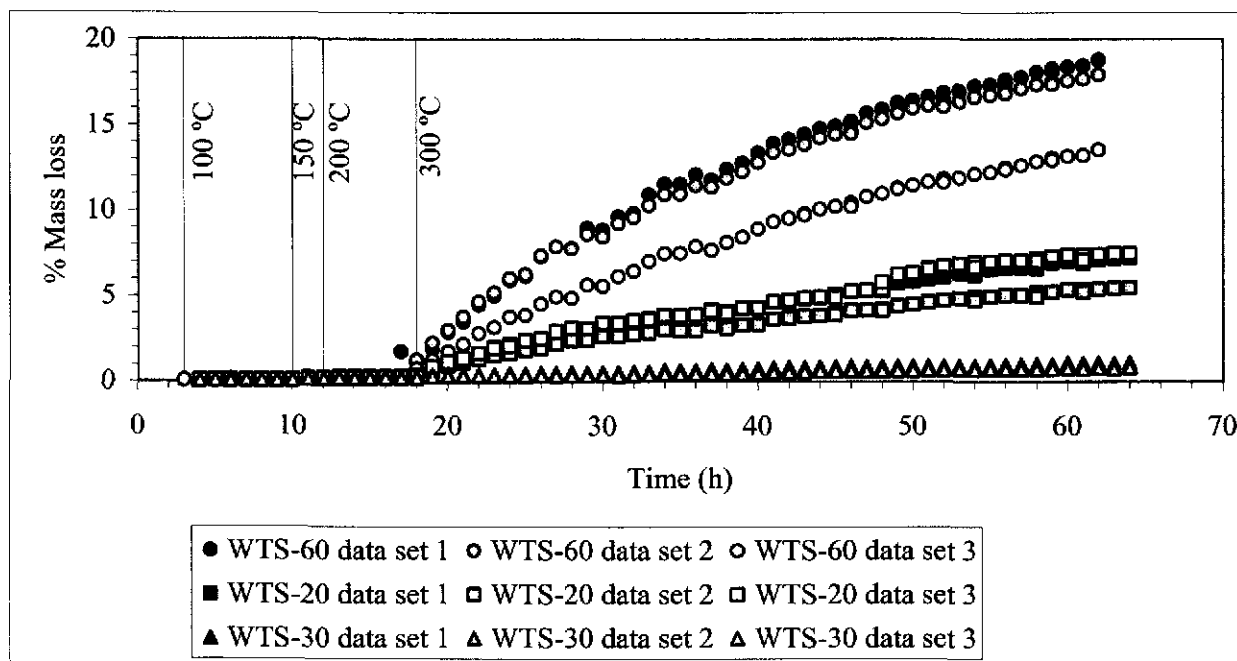


Figure 6. Mass loss of WTS-30, WTS-20, and WTS-60 hydrated in DI water at 90 °C. Samples dried at 60 °C for 2 hours.

CONCLUSIONS

The preliminary results, presented in this report, show that the MgO materials supplied by Martin Marietta react faster than Premier MgO, and they produce slightly higher quantities of brucite (which will consume CO₂ in the WIPP) than Premier MgO does. The data from the 500 °C LOI test show that: (1) for WTS-30 and WTS-20, 87 ± 5 mol % and 91 ± 4 wt % brucite form; (2) for WTS-60, 90 ± 3 mol % and 93 ± 2 wt % brucite form; and (3) for Premier, 88.8 mol % and 94.6 wt % brucite form. Experiments with a LOI at 750 °C were performed for Premier MgO and WTS-30. The calculated results show larger amounts of generated brucite than for the 500 °C LOI tests and larger amounts of generated brucite for WTS-30 than for Premier MgO. The data from the 750 °C LOI showed that for WTS-30, 96 ± 5 mol % and 97 ± 3 wt % of brucite form; and for Premier, 89 mol % and 92 wt % brucite form. Although the 750 °C LOI could not be performed with WTS-20 and WTS-60, we expect results similar to those for WTS-30, based on the similarity of results obtained for the three Martin Marietta MgO samples for the 500 °C LOI tests. These preliminary results suggest that any of the three Martin Marietta MgO materials, WTS-30, WTS-20, and WTS-60, would be an acceptable to replace Premier MgO. The choice between any of the three Martin Marietta MgO materials can be based on other considerations, such as ES&H requirements.

However, additional data will be needed. The new MgO material will have to be tested to ensure it follows all the specifications required by WTS (2003). Long-term testing of the new MgO will be necessary, after it is selected, to develop a kinetic model to predict the carbonation of the new MgO material selected for the WIPP engineered barrier. Also, we must characterize the new MgO, to the same extent as was done for Premier MgO. Finally, the testing of LOI parameters needs to be completed to generate meaningful data for the calculation of the safety factor.

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APPENDIX: TABLES OF EXPERIMENTAL RESULTS

Table A1. LOI Results – WTS-30.

sample #	age (d)	LOI 500 °C						LOI 750 °C					
		before heating		2 h. at 60 °C	3h at 500 °C	wt %	mol %	before heating		2 h. at 60 °C	3h at 750 °C	wt %	mol %
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)			Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-9.1	1.21	20.1522	22.1480	22.1430	21.6721	76.6	69.3	20.8743	22.8898	22.8854	22.3228	90.6	86.9
MgO 19-9.2	2.67	18.4654	20.5538	20.5476	19.9540	92.3	89.2						
MgO 19-9.3	3.02	18.8569	20.6492	20.6439	20.1300	93.1	90.3						
MgO 19-9.4	3.74	19.6041	21.7200	21.5790	21.0113	93.1	90.3	11.6681	13.7411	13.6113	13.0276	97.2	96.1
MgO 19-9.5	4.75	17.4133	19.4450	19.1902	18.6887	91.4	88.0	11.7010	13.7711	13.5194	12.9770	96.6	95.1
MgO 19-9.6	5.73	19.9203	21.9746	21.7389	21.2261	91.3	87.9	11.6808	13.7463	13.5187	12.9811	94.7	92.5
MgO 19-9.7	6.91	19.0332	21.0367	20.5927	20.1537	91.1	87.7	11.2668	13.4189	12.9569	12.4646	94.3	92.0
MgO 19-9.8	7.82	20.1524	22.1800	21.8650	21.3770	92.3	89.2	12.2191	14.2951	13.9782	13.4500	97.2	96.0
MgO 19-9.9	8.81	18.4652	20.4337	20.2880	19.7686	92.3	89.2	11.6680	13.6768	13.5489	12.9844	97.2	95.9
MgO 19-9.10	9.78	18.8568	20.8177	20.6338	20.1282	92.1	89.0	11.6159	13.6244	13.4396	12.8834	98.7	98.2
MgO 19-9.11	10.77	12.2191	14.4683	14.3597	13.7453	92.9	90.1	11.5215	13.5245	13.4292	12.8515	98.0	97.2
MgO 19-9.12	12.79	20.1529	22.1711	21.7496	21.2964	91.9	88.7	11.7716	13.7530	13.3515	12.8796	96.7	95.3
MgO 19-9.13	15.10	11.7706	13.8666	13.3967	12.9340	92.1	89.0	11.7012	13.7612	13.2980	12.8233	96.2	94.7
MgO 19-9.14	16.73	18.4655	20.4924	19.9760	19.5514	91.0	87.5	11.7249	13.7650	13.2624	12.8057	96.2	94.5
MgO 19-9.15	18.77	11.6807	13.7237	13.0993	12.6980	91.6	88.3	11.9440	13.9820	13.3560	12.9389	95.6	93.8
MgO 19-9.16	20.80	11.7711	13.7989	13.6049	13.0888	91.1	87.6	11.8638	13.8672	13.6747	13.1336	96.7	95.3
MgO 19-9.17	23.82	11.0430	13.0654	12.8875	12.3700	90.8	87.3	11.2668	13.2704	13.1011	12.5506	97.2	95.9

Table A1. LOI Results – WTS-30 (cont.)

sample #	age (d)	LOI 500 °C						LOI 750 °C					
		before heating		2 h. at 60 °C	3h at 500 °C	wt %	mol %	before heating		2 h. at 60 °C	3h at 750 °C	wt %	mol %
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)			Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-9.18	24.84	12.2192	14.2276	13.8741	13.4139	90.0	86.2	11.8230	13.8450	13.4977	13.0011	96.0	94.3
MgO 19-9.19	26.75	11.7000	13.7074	13.5176	13.0153	89.5	85.4	11.6811	13.7293	13.5347	12.9849	96.0	94.4
MgO 19-9.20	27.80	11.8634	13.8879	13.8858	13.3293	89.1	84.9	11.6811	13.7293	13.5347	12.9849	96.0	94.4
MgO 19-11.1	1.15	11.2669	13.6047	13.6002	13.0822	1.68	71.87	19.1634	21.1832	21.1779	20.6954	1.56	77.54
MgO 19-11.2	2.61	11.9440	14.0915	14.0744	13.4693	1.96	91.95	33.5906	35.6004	35.5943	34.9690	2.02	101.03
MgO 19-11.3	2.96	11.0743	13.1499	13.1463	12.5424	1.96	94.36	33.5743	35.5429	35.5394	34.9330	1.96	99.90
MgO 19-11.4	3.68	11.6812	13.8081	13.5428	13.0179	1.70	91.28	33.1170	35.1103	34.8930	34.3400	1.79	100.81
MgO 19-11.5	4.70	11.8236	13.7790	13.5233	13.0460	1.55	90.91	20.2523	22.2400	21.9809	21.4564	1.70	98.23
MgO 19-11.6	5.67	19.0330	21.0797	20.6574	20.2011	1.48	90.94	11.5212	13.5711	13.1474	12.6554	1.59	97.95
MgO 19-11.7	6.85	11.5217	14.0958	13.6335	13.0383	1.93	91.25	17.4128	19.7099	19.2975	18.7159	1.88	99.90
MgO 19-11.8	7.76	20.2523	22.2078	22.0760	21.5526	1.69	92.91	11.864	13.7485	13.6218	13.0852	1.74	98.83
MgO 19-11.9	8.75	11.6148	13.7563	13.4780	12.9469	1.72	92.28	20.6506	22.8704	22.5775	21.9912	1.90	98.51
MgO 19-11.10	9.73	11.0429	13.0820	12.7450	12.2614	1.57	91.98	11.7011	13.5735	13.2592	12.7882	1.52	97.87
MgO 19-11.11	10.72	11.8637	13.8977	13.7724	13.2308	1.75	91.86	11.0755	13.0978	12.9752	12.4043	1.85	97.29
MgO 19-11.12	12.76	11.2663	13.3865	12.7440	12.3215	1.37	92.56	11.7239	13.759	13.1459	12.7263	1.36	95.53
MgO 19-11.13	15.05	11.8226	13.8556	13.3442	12.9118	1.40	92.00	11.8637	13.9042	13.3942	12.9461	1.45	94.79
MgO 19-11.14	16.67	18.8567	20.8818	20.2319	19.8487	1.24	90.21	11.5217	13.565	12.9161	12.5075	1.32	94.87
MgO 19-11.15	18.71	12.2190	14.3715	13.7477	13.3287	1.36	88.74	11.7708	13.831	13.2404	12.8089	1.40	95.06
MgO 19-11.16	20.74	11.0742	13.0811	12.9505	12.4385	1.66	88.34	11.8231	13.9363	13.7997	13.2108	1.91	96.46
MgO 19-11.17	23.76	11.5217	13.5365	13.5341	12.9834	1.78	88.59	11.9441	13.9454	13.9426	13.3187	2.02	101.07

Table A1. LOI Results – WTS-30 (cont.)

sample #	age (d)	LOI 500 °C						LOI 750 °C					
		before heating		2 h. at 60 °C	3h at 500 °C		before heating		2 h. at 60 °C	3h at 750 °C			
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)	wt %	mol %	Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)	wt %	mol %
MgO 19-11.18	24.78	11.7709	13.7943	13.3935	12.9479	1.44	88.91	11.0432	13.0708	12.6642	12.1913	1.53	94.45
MgO 19-11.19	26.70	11.9440	13.9821	13.7187	13.2301	1.58	89.13	11.0432	13.1928	12.9229	12.365	1.81	96.09
MgO 19-11.20	27.74	11.6680	13.6623	13.6025	13.0723	1.72	88.73	11.0745	13.0975	13.0375	12.443	1.92	98.05
MgO 19-22.1	0.08	20.1518	22.1610	22.1598	22.1537	0.02	0.98	11.2667	13.2679	13.2673	13.2559	0.04	1.84
MgO 19-22.2	0.14	18.4651	20.4715	20.4702	20.4630	0.02	1.16	11.9442	13.9457	13.9447	13.9322	0.04	2.02
MgO 19-22.3	0.22	18.8564	20.8651	20.8643	20.8511	0.04	2.13	11.0745	13.0767	13.0757	13.0565	0.06	3.11
MgO 19-22.4	0.30	19.6036	21.6096	21.6085	21.5750	0.11	5.41	11.8234	13.8241	13.8224	13.7808	0.13	6.74
MgO 19-22.5	1.17	19.6036	21.6525	21.5587	21.3084	0.81	41.45	19.6037	21.608	21.5317	21.2445	0.93	48.23
MgO 19-22.6	2.08	19.6041	21.6298	21.2651	20.8163	1.45	87.48	11.8624	13.8782	13.8694	13.2569	1.98	98.80
MgO 19-22.7	3.07	17.4139	19.3983	19.0329	18.5706	1.50	92.44	20.1519	22.1711	21.8499	21.3364	1.66	97.91
MgO 19-22.8	4.04	19.9202	21.6383	21.4850	21.0391	1.44	92.25	11.6163	13.665	13.3943	12.8571	1.74	97.82
MgO 19-22.9	5.03	20.6511	22.6673	22.2127	21.7708	1.43	91.61						
MgO 19-22.10	6.09	20.2522	22.2892	21.6902	21.2830	1.32	91.68	11.5215	13.5336	13.32	12.7782	1.75	97.53
MgO 19-22.11	7.08	17.4137	19.4222	18.8714	18.4601	1.33	91.35	11.7709	13.7867	13.1224	12.7188	1.31	96.68
MgO 19-22.12	7.98	19.9208	21.9251	21.3747	20.9673	1.32	90.72	11.668	13.6779	13.0358	12.6279	1.32	96.55
MgO 19-22.13	9.36	19.0338	21.0440	20.4310	20.0390	1.27	90.83	11.2666	13.2737	12.9888	12.4663	1.69	98.22
MgO 19-22.14	10.99	19.8695	21.8907	21.2522	20.8687	1.24	89.79	11.944	13.9541	13.7284	13.187	1.75	98.23
MgO 19-22.15	11.99	11.7241	13.8330	13.3378	12.9074	1.39	86.35	18.4654	20.4692	20.0091	19.5496	1.49	96.37
MgO 19-22.16	14.20	11.9441	13.9884	13.3848	13.0019	1.24	86.04	18.8571	20.8622	20.2975	19.8704	1.38	96.00
MgO 19-22.17	15.06	20.1521	22.1568	22.1540	21.6195	1.73	86.44	19.6035	21.6496	21.6481	21.0237	2.02	98.87

Table A1. LOI Results – WTS-30 (cont.)

sample #	age (d)	LOI 500 °C						LOI 750 °C					
		before heating		2 h. at 60 °C	3h at 500 °C	wt %	mol %	before heating		2 h. at 60 °C	3h at 750 °C	wt %	mol %
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)			Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-22.18	18.08	11.6808	13.7564	13.5295	13.0172	1.66	89.71	19.1633	21.181	21.0024	20.442	1.81	98.65
MgO 19-22.19	20.05	11.7240	13.7842	13.5814	13.0711	1.65	88.95	19.9023	21.9056	21.7364	21.1761	1.81	98.90
MgO 19-22.20	22.06	11.8229	13.8222	13.8036	13.2641	1.75	88.18	19.0332	21.0597	21.0582	20.4308	2.03	100.31
MgO 19-24.1	0.68	20.1518	22.1672	22.1644	22.0189	0.47	23.41	20.1516	22.1527	22.1498	21.9816	0.54	27.25
MgO 19-24.2	0.77	18.4647	20.5069	20.4221	20.1956	0.73	37.46	18.4652	20.4679	20.3961	20.1418	0.82	42.64
MgO 19-24.3	0.85	18.8564	20.8873	20.6960	20.4294	0.86	46.92	18.8565	20.8584	20.6883	20.3826	0.99	54.03
MgO 19-24.4	1.79	19.0334	20.9304	20.7494	20.3213	1.39	80.77	11.0778	13.0783	12.7625	12.2521	1.65	98.08
MgO 19-24.5	2.78	19.8690	21.7220	21.5534	21.0869	1.51	89.66						
MgO 19-24.6	3.75	11.0431	12.9555	12.6836	12.2146	1.52	92.56	11.043	13.0577	12.571	12.1117	1.49	97.31
MgO 19-24.7	4.74	11.7008	13.7356	13.5079	12.9933	1.67	92.19	11.8235	13.8444	13.4225	12.9398	1.56	97.73
MgO 19-24.8	5.80	18.8564	20.8876	20.2238	19.8444	1.23	89.83	11.7009	13.7088	13.1718	12.7358	1.41	95.96
MgO 19-24.9	6.79	18.4653	20.6306	19.9230	19.5204	1.30	89.41	19.8695	21.8697	21.2868	20.8679	1.36	95.69
MgO 19-24.10	7.69	20.2510	22.2552	21.9543	21.4807	1.53	90.02	19.9205	21.931	21.3539	20.9287	1.38	96.04

Table A2. LOI Results – WTS-20.

sample #	age (d)	LOI 500 °C				wt %	mol %
		before heating		2 h. at 60 °C	3h at 500 °C		
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-15.1	0.73	19.8693	21.8906	21.8872	21.7260	0.52	25.86
MgO 19-15.2	1.70	11.5212	13.6759	13.6053	13.1889	1.35	64.68
MgO 19-15.3	2.88	19.8691	21.9193	21.5463	21.1088	1.42	84.45
MgO 19-15.4	3.79	19.9204	21.8260	21.6462	21.1650	1.56	90.27
MgO 19-15.5	4.78	19.811	21.7128	21.539	21.0411	1.61	93.28
MgO 19-15.6	5.76	19.0331	20.75	20.6726	20.2054	1.51	92.26
MgO 19-15.7	6.75	19.9028	21.8717	21.6651	21.1638	1.62	92.09
MgO 19-15.8	7.80	18.8565	20.7619	20.3028	19.8933	1.33	91.66
MgO 19-15.9	8.79	11.6807	13.8094	13.5234	12.9945	1.71	92.92
MgO 19-15.10	9.69	11.9441	13.952	13.9485	13.3554	1.92	95.80
MgO 19-15.11	12.70	19.6033	21.6284	21.5415	21.0014	1.75	90.22
MgO 19-15.12	13.71	19.1627	21.1796	21.0777	20.5373	1.75	91.36
MgO 19-15.13	15.91	11.6158	13.7110	13.4723	12.9641	1.65	88.62
MgO 19-15.14	16.77	11.8229	13.9790	13.8982	13.3231	1.86	89.72
MgO 19-15.15	19.79	11.2667	13.5146	13.2857	12.7363	1.78	88.10
MgO 19-15.16	21.76	11.6158	13.6682	13.6266	13.0675	1.81	90.02
MgO 19-15.17	23.77	11.5216	13.546	13.466	12.9238	1.76	90.28
MgO 19-15.18	26.69	11.2666	13.2311	13.2249	12.6598	1.83	93.42
MgO 19-15.19	27.67	11.6808	13.67	13.1358	12.7273	1.32	90.89

Table A2. LOI Results – WTS-20 (cont.).

sample #	age (d)	LOI 500 °C				wt %	mol %
		before heating		2 h. at 60 °C	3h at 500 °C		
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-15.20	29.68	11.0744	13.0055	12.8124	12.3167	1.60	92.34
MgO 19-17.1	0.70	11.0428	13.0873	13.0730	12.9332	0.45	22.29
MgO 19-17.2	1.67	11.6804	13.7849	13.5926	13.2003	1.27	66.42
MgO 19-17.3	2.85	19.1631	21.2853	21.0187	20.5132	1.64	88.19
MgO 19-17.4	3.76	19.8693	21.8897	21.5617	21.1006	1.49	88.21
MgO 19-17.5	4.75	18.4656	20.5256	20.3132	19.7901	1.69	91.66
MgO 19-17.6	5.73	19.6038	21.4966	21.3322	20.859	1.53	88.63
MgO 19-17.7	6.72	20.1521	22.1857	21.8107	21.3517	1.49	89.59
MgO 19-17.8	7.77	17.4137	19.7829	19.3802	18.8341	1.77	89.90
MgO 19-17.9	8.76	11.668	13.7884	13.5149	12.9879	1.71	92.38
MgO 19-17.10	9.66	11.7239	13.969	13.5433	13.0239	1.68	92.42
MgO 19-17.11	12.67	19.9021	21.9632	21.3998	20.9778	1.37	91.22
MgO 19-17.12	13.68	19.8104	21.8197	21.3667	20.9261	1.43	91.65
MgO 19-17.13	15.88	11.6682	13.6722	13.1815	12.7715	1.33	87.71
MgO 19-17.14	16.74	11.8638	13.9648	13.9612	13.3844	1.87	89.03
MgO 19-17.15	19.76	11.7009	13.8733	13.6194	13.0993	1.68	87.77
MgO 19-17.16	21.73	11.0733	13.0746	12.8939	12.3784	1.67	91.67
MgO 19-17.17	23.74	11.6803	13.3886	13.6423	13.0866	1.80	91.69
MgO 19-17.18	26.66	19.8105	21.809	21.8067	21.2521	1.80	89.95

Table A2. LOI Results – WTS-20 (cont.).

sample #	age (d)	LOI 500 °C				wt %	mol %
		before heating		2 h. at 60 °C	3h at 500 °C		
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-17.19	27.64	11.6683	13.6824	13.1924	12.7698	1.37	89.77
MgO 19-17.20	29.65	11.6162	13.6799	13.4478	12.9379	1.65	90.13

Table A3. LOI Results – WTS-60.

sample #	age (d)	LOI 500 °C				wt %	mol %
		before heating		2 h. at 60 °C	3h at 500 °C		
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-59.1	0.08	11.2666	13.2876	13.1076	13.0923	0.05	2.69
MgO 19-59.2	0.15	11.0744	12.9990	12.9628	12.9478	0.05	2.57
MgO 19-59.3	0.22	11.0431	13.0648	13.0137	12.9904	0.08	3.83
MgO 19-59.4	0.89	11.5216	13.5382	13.2263	12.9781	0.80	47.14
MgO 19-59.5	1.92	11.7711	13.8463	13.7085	13.2381	1.52	78.61
MgO 19-59.6	2.91	11.8635	13.9208	13.4422	13.0062	1.41	89.41
MgO 19-59.7	4.01	11.7009	13.8713	13.8698	13.2458	2.02	93.14
MgO 19-59.8	6.97	11.8226	13.8319	13.3372	12.9084	1.39	91.66
MgO 19-59.9	7.97	11.0430	13.0495	12.7762	12.2813	1.60	92.44
MgO 19-59.10	8.57	11.7708	13.7927	13.6065	13.0829	1.70	92.34
MgO 19-59.11	9.93	11.6158	13.6528	13.4304	12.9124	1.68	92.42
MgO 19-59.12	11.02	11.6681	13.6795	13.6757	13.0970	1.87	93.32
MgO 19-59.13	11.97	12.2192	14.8847	13.8636	13.3958	1.51	92.10
MgO 19-59.14	15.04	11.6807	13.6872	13.4038	12.9112	1.59	92.55
MgO 19-59.15	16.95	11.0431	13.0438	13.0417	12.4729	1.84	92.14
MgO 19-59.16	17.99	11.6158	13.6509	13.0706	12.6584	1.33	91.73
MgO 19-59.17	21.01	11.5215	13.5605	13.4802	12.9201	1.81	92.58
MgO 19-59.18	21.93	11.7242	13.7738	13.4660	12.9679	1.61	92.58
MgO 19-59.19	24.97	11.8630	13.8777	13.8745	13.2981	1.87	92.77

Table A3. LOI Results – WTS-60 (cont.).

sample #	age (d)	LOI 500 °C				wt %	mol %
		before heating		2 h. at 60 °C	3h at 500 °C		
		Crucible (g)	Cruc +MgO (g)	Cruc +MgO (g)	Cruc +MgO (g)		
MgO 19-59.20	28.08	11.7714	13.7950	13.4927	13.0008	1.59	92.52
MgO 19-61.1	0.65	11.9447	13.9145	13.8922	13.7271	0.53	27.45
MgO 19-61.2	0.75	11.8231	13.8185	13.8082	13.6183	0.61	30.97
MgO 19-61.3	1.00	12.2194	14.2429	14.1066	13.8494	0.83	44.12
MgO 19-61.4	1.68	11.0433	13.0763	13.0671	12.6514	1.35	66.50
MgO 19-61.5	2.79	11.7010	13.7858	13.7845	13.1727	1.98	95.06
MgO 19-61.6	5.74	11.9440	14.0319	13.6644	13.1748	1.59	92.13
MgO 19-61.7	6.75	11.7240	13.7368	13.4148	12.9341	1.56	92.04
MgO 19-61.8	7.34	11.5216	13.6011	13.3055	12.7984	1.64	92.03
MgO 19-61.9	8.71	11.2666	13.2970	13.0897	12.5552	1.73	94.92
MgO 19-61.10	9.80	11.8634	13.8745	13.8711	13.3084	1.82	90.74
MgO 19-61.11	10.75	19.8104	22.0786	21.2944	20.8836	1.33	89.62
MgO 19-61.12	13.81	11.9441	13.9910	13.6845	13.1874	1.61	92.47
MgO 19-61.13	15.72	11.6680	13.7026	13.7007	13.1049	1.93	94.89
MgO 19-61.14	16.77	11.8230	13.8363	13.6239	13.1073	1.67	92.87
MgO 19-61.15	19.78	11.0743	13.0891	13.0503	12.4832	1.84	92.91
MgO 19-61.17	23.74	11.2667	13.2678	13.2653	12.6948	1.85	92.41
MgO 19-61.18	26.86	12.2199	14.2280	13.9757	13.4676	1.64	93.69
MgO 19-61.19	27.73	11.7009	13.7166	13.7153	13.1314	1.89	93.84
MgO 19-61.20	29.69	11.7242	13.7229	13.7213	13.1369	1.89	94.74

Table A4. LOI Parameter Tests - WTS-30.

Sample: MgO 19-50.4 Sample age: 6.32 days Cruc: g Cruc + MgO: g				Sample: MgO 19-50.5 Sample age: 6.32 days Cruc: 11.6809 g Cruc +MgO: 13.7113 g				Sample: MgO 19-50.6 Sample age: 6.32 days Cruc: 11.0745 g Cruc + MgO: 13.0906 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
60	1	13.2766		60	1	13.3640		60	1	13.0896	
60	1	12.9563		60	1	12.9605		60	1	13.0892	
60	1	12.9560		60	1	12.9602		60	1	13.0890	
100	1	12.9550	0.07	100	1	12.9588	0.11	100	1	13.0878	0.06
100	1	12.9546	0.10	100	1	12.9585	0.13	100	1	13.0874	0.08
100	1	12.9533	0.19	100	1	12.9591	0.09	100	1	13.0879	0.05
100	1	12.9547	0.09	100	1	12.9585	0.13	100	1	13.0868	0.11
100	1	12.9551	0.06	100	1	12.9588	0.11	100	1	13.0877	0.06
100	1	12.9550	0.07	100	1	12.9587	0.12	100	1	13.0877	0.06
100	1	12.9546	0.10	100	1	12.9584	0.14	100	1	13.0872	0.09
150	1	12.9539	0.15	150	1	12.9578	0.19	150	1	13.0862	0.14
150	1	12.9537	0.16	150	1	12.9576	0.20	150	1	13.0860	0.15
200	1	12.9532	0.20	200	1	12.9571	0.24	200	1	13.0854	0.18
200	1	12.9533	0.19	200	1	12.9574	0.22	200	1	13.0856	0.17
200	1	12.9530	0.21	200	1	12.9570	0.25	200	1	13.0854	0.18
200	1	12.9528	0.22	200	1	12.9566	0.28	200	1	13.0849	0.20
200	1	12.9528	0.22	200	1	12.9568	0.27	200	1	13.0851	0.19

Table A4. LOI Parameter Tests - WTS-30 (cont.).

Sample: MgO 19-50.4 Sample age: 6.32 days Cruc: g Cruc + MgO: g				Sample: MgO 19-50.5 Sample age: 6.32 days Cruc: 11.6809 g Cruc +MgO: 13.7113 g				Sample: MgO 19-50.6 Sample age: 6.32 days Cruc: 11.0745 g Cruc + MgO: 13.0906 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
200	1	12.9526	0.24	200	1	12.9566	0.28	200	1	13.0848	0.21
300	1	12.9512	0.33	300	1	12.9555	0.37	300	1	13.0837	0.26
300	1	12.9509	0.36	300	1	12.9554	0.38	300	1	13.0833	0.28
300	1	12.9505	0.38	300	1	12.9550	0.41	300	1	13.0830	0.30
300	1	12.9503	0.40	300	1	12.9550	0.41	300	1	13.0828	0.31
300	1	12.9500	0.42	300	1	12.9548	0.42	300	1	13.0827	0.31
300	1	12.9496	0.45	300	1	12.9546	0.44	300	1	13.0824	0.33
300	1	12.9494	0.46	300	1	12.9548	0.42	300	1	13.0824	0.33
300	1	12.9489	0.49	300	1	12.9543	0.46	300	1	13.0817	0.36
300	1	12.9489	0.49	300	1	12.9543	0.46	300	1	13.0820	0.35
300	1	12.9484	0.53	300	1	12.9539	0.49	300	1	13.0815	0.37
300	1	12.9488	0.50	300	1	12.9542	0.47	300	1	13.0817	0.36
300	1	12.9481	0.55	300	1	12.9535	0.52	300	1	13.0812	0.39
300	1	12.9483	0.54	300	1	12.9541	0.48	300	1	13.0818	0.36
300	1	12.9480	0.56	300	1	12.9538	0.50	300	1	13.0813	0.38
300	1	12.9476	0.59	300	1	12.9536	0.52	300	1	13.0809	0.40
300	1	12.9463	0.68	300	1	12.9528	0.58	300	1	13.0798	0.46

Table A4. LOI Parameter Tests - WTS-30 (cont.).

Sample: MgO 19-50.4 Sample age: 6.32 days				Sample: MgO 19-50.5 Sample age: 6.32 days				Sample: MgO 19-50.6 Sample age: 6.32 days			
Cruc: g		Cruc + MgO: g		Cruc: 11.6809 g		Cruc +MgO: 13.7113 g		Cruc: 11.0745 g		Cruc + MgO: 13.0906 g	
T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C	
		Cruc + MgO (g)	% mass lost			Cruc + MgO (g)	% mass lost			Cruc + MgO (g)	% mass lost
300	1	12.9465	0.66	300	1	12.9530	0.56	300	1	13.0803	0.43
300	1	12.9464	0.67	300	1	12.9530	0.56	300	1	13.0800	0.45
300	1	12.9458	0.71	300	1	12.9523	0.62	300	1	13.0794	0.48
300	1	12.9462	0.68	300	1	12.9527	0.59	300	1	13.0797	0.46
300	1	12.9456	0.72	300	1	12.9526	0.59	300	1	13.0795	0.47
300	1	12.9453	0.75	300	1	12.9523	0.62	300	1	13.0793	0.48
300	1	12.9444	0.81	300	1	12.9515	0.68	300	1	13.0783	0.53
300	1	12.9443	0.82	300	1	12.9513	0.70	300	1	13.0781	0.54
300	1	12.9444	0.81	300	1	12.9517	0.66	300	1	13.0775	0.57
300	1	12.9437	0.86	300	1	12.9509	0.73	300	1	13.0768	0.61
300	1	12.9436	0.86	300	1	12.9509	0.73	300	1	13.0764	0.63
		12.9437	0.86			12.9509	0.73			13.0766	0.62
300	1	12.9432	0.89	300	1	12.9506	0.75	300	1	13.0761	0.64
300	1	12.9435	0.87	300	1	12.9508	0.73	300	1	13.0765	0.62
300	1	12.9435	0.87	300	1	12.9508	0.73	300	1	13.0764	0.63
		12.9436	0.86			12.9508	0.73			13.0760	0.65

Table A4. LOI Parameter Tests - WTS-30 (cont.).

Sample: MgO 19-50.4 Sample age: 6.32 days				Sample: MgO 19-50.5 Sample age: 6.32 days				Sample: MgO 19-50.6 Sample age: 6.32 days			
Cruc: g		Cruc + MgO: g		Cruc: 11.6809 g		Cruc +MgO: 13.7113 g		Cruc: 11.0745 g		Cruc + MgO: 13.0906 g	
T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C	
		Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost
300	1	12.9431	0.90	300	1	12.9506	0.75	300	1	13.0756	0.67
300	1	12.9432	0.89	300	1	12.9507	0.74	300	1	13.0755	0.67
300	1	12.9430	0.91	300	1	12.9506	0.75	300	1	13.0754	0.68
		12.9428	0.92			12.9500	0.80			13.0748	0.70
300	1	12.9430	0.91	300	1	12.9503	0.77	300	1	13.0751	0.69
300	1	12.9428	0.92	300	1	12.9498	0.81	300	1	13.0747	0.71
300	1	12.9426	0.93	300	1	12.9498	0.81	300	1	13.0747	0.71
		12.9430	0.91			12.9501	0.79			13.0750	0.69
300	1	12.9422	0.96	300	1	12.9496	0.83	300	1	13.0744	0.72
300	1	12.9418	0.99	300	1	12.9496	0.83	300	1	13.0745	0.72
300	1	12.9417	1.00	300	1	12.9495	0.84	300	1	13.0744	0.72
300	1	12.9414	1.02	300	1	12.9492	0.86	300	1	13.0738	0.75
		12.9415	1.01			12.9493	0.85			13.0741	0.74
300	1	12.9418	0.99	300	1	12.9491	0.87	300	1	13.0739	0.75
300	1	12.9404	1.09	300	1	12.9490	0.88	300	1	13.0734	0.77
300	1	12.9408	1.06	300	1	12.9491	0.87	300	1	13.0732	0.78

Table A4. LOI Parameter Tests - WTS-30 (cont.).

Sample: MgO 19-50.4 Sample age: 6.32 days				Sample: MgO 19-50.5 Sample age: 6.32 days				Sample: MgO 19-50.6 Sample age: 6.32 days			
Cruc: g		Cruc + MgO: g		Cruc: 11.6809 g		Cruc + MgO: 13.7113 g		Cruc: 11.0745 g		Cruc + MgO: 13.0906 g	
		after 60 °C				after 60 °C				after 60 °C	
T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost
		12.9418	0.99			12.9489	0.88			13.0737	0.76
300	1	12.9405	1.08	300	1	12.9485	0.91	300	1	13.0731	0.79
300	1	12.9408	1.06	300	1	12.9489	0.88	300	1	13.0738	0.75
300	1	12.9399	1.12	300	1	12.9483	0.93	300	1	13.0726	0.81

Table A5. LOI Parameter Tests – WTS-20.

Sample: MgO 19-50.1 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 13.7668 g				Sample: MgO 19-50.2 Sample age: 6.32 days Cruc: 11.7236 g Cruc +MgO: 11.7236 g				Sample: MgO 19-50.3 Sample age: 6.32 days Cruc: 11.6157 g Cruc + MgO: 13.6198 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
60	1	13.3784		60	1	13.5447		60	1	13.2526	
60	1	13.1050		60	1	13.1775		60	1	13.0399	
60	1	13.1048		60	1	13.1775		60	1	13.0398	
100	1	13.1036	0.09	100	1	13.1762	0.10	100	1	13.0384	0.10
100	1	13.1030	0.13	100	1	13.1756	0.14	100	1	13.0380	0.13
100	1	13.1033	0.11	100	1	13.1759	0.12	100	1	13.0383	0.11
100	1	13.1036	0.09	100	1	13.1760	0.11	100	1	13.0381	0.12
100	1	13.1032	0.12	100	1	13.1757	0.14	100	1	13.0385	0.09
100	1	13.1038	0.07	100	1	13.1760	0.11	100	1	13.0384	0.10
100	1	13.1033	0.11	100	1	13.1756	0.14	100	1	13.0380	0.13
150	1	13.1025	0.17	150	1	13.1747	0.21	150	1	13.0373	0.18
150	1	13.1022	0.19	150	1	13.1744	0.24	150	1	13.0369	0.20
200	1	13.1013	0.25	200	1	13.1736	0.30	200	1	13.0361	0.26
200	1	13.1013	0.25	200	1	13.1736	0.30	200	1	13.0363	0.25
200	1	13.1013	0.25	200	1	13.1736	0.30	200	1	13.0361	0.26
200	1	13.1010	0.28	200	1	13.1735	0.30	200	1	13.0359	0.27
200	1	13.1012	0.26	200	1	13.1734	0.31	200	1	13.0358	0.28

Table A5. LOI Parameter Tests – WTS-20 (cont.).

Sample: MgO 19-50.1 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 13.7668 g				Sample: MgO 19-50.2 Sample age: 6.32 days Cruc: 11.7236 g Cruc +MgO: 11.7236 g				Sample: MgO 19-50.3 Sample age: 6.32 days Cruc: 11.6157 g Cruc + MgO: 13.6198 g			
T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C	
		Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost
200	1	13.1007	0.30	200	1	13.1731	0.33	200	1	13.0357	0.29
300	1	13.0960	0.64	300	1	13.1694	0.62	300	1	13.0277	0.85
300	1	13.0910	1.00	300	1	13.1655	0.91	300	1	13.0235	1.14
300	1	13.0863	1.34	300	1	13.1630	1.10	300	1	13.0206	1.35
300	1	13.0831	1.57	300	1	13.1604	1.30	300	1	13.0164	1.64
300	1	13.0771	2.01	300	1	13.1575	1.52	300	1	13.0119	1.96
300	1	13.0749	2.16	300	1	13.1556	1.67	300	1	13.0103	2.07
300	1	13.0722	2.36	300	1	13.1537	1.81	300	1	13.0056	2.40
300	1	13.0708	2.46	300	1	13.1520	1.94	300	1	13.0039	2.52
300	1	13.0653	2.86	300	1	13.1484	2.21	300	1	12.9980	2.94
300	1	13.0620	3.10	300	1	13.1460	2.40	300	1	12.9950	3.15
300	1	13.0626	3.06	300	1	13.1456	2.43	300	1	12.9955	3.11
300	1	13.0579	3.40	300	1	13.1426	2.66	300	1	12.9909	3.43
300	1	13.0575	3.42	300	1	13.1424	2.67	300	1	12.9913	3.41
300	1	13.0550	3.61	300	1	13.1409	2.78	300	1	12.9887	3.59
300	1	13.0535	3.71	300	1	13.1401	2.85	300	1	12.9878	3.65
300	1	13.0508	3.91	300	1	13.1372	3.07	300	1	12.9846	3.88

Table A5. LOI Parameter Tests – WTS-20 (cont.).

Sample: MgO 19-50.1 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 13.7668 g				Sample: MgO 19-50.2 Sample age: 6.32 days Cruc: 11.7236 g Cruc +MgO: 11.7236 g				Sample: MgO 19-50.3 Sample age: 6.32 days Cruc: 11.6157 g Cruc + MgO: 13.6198 g			
after 60 °C				after 60 °C				after 60 °C			
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
300	1	13.0515	3.86	300	1	13.1382	2.99	300	1	12.9847	3.87
300	1	13.0506	3.92	300	1	13.1378	3.02	300	1	12.9838	3.93
300	1	13.0465	4.22	300	1	13.1344	3.28	300	1	12.9805	4.16
300	1	13.0480	4.11	300	1	13.1364	3.13	300	1	12.9826	4.02
300	1	13.0456	4.29	300	1	13.1338	3.32	300	1	12.9783	4.32
300	1	13.0455	4.29	300	1	13.1334	3.36	300	1	12.9784	4.31
300	1	13.0405	4.66	300	1	13.1292	3.67	300	1	12.9729	4.70
300	1	13.0408	4.63	300	1	13.1286	3.72	300	1	12.9718	4.77
300	1	13.0376	4.87	300	1	13.1272	3.83	300	1	12.9698	4.92
300	1	13.0366	4.94	300	1	13.1267	3.86	300	1	12.9693	4.95
300	1	13.0345	5.09	300	1	13.1249	4.00	300	1	12.9671	5.10
		13.0358	5.00			13.1261	3.91			12.9695	4.94
300	1	13.0318	5.29	300	1	13.1227	4.17	300	1	12.9646	5.28
300	1	13.0308	5.36	300	1	13.1225	4.18	300	1	12.9637	5.34
300	1	13.0289	5.50	300	1	13.1216	4.25	300	1	12.9627	5.41
		13.0314	5.31			13.1228	4.16			12.9568	5.83

Table A5. LOI Parameter Tests – WTS-20 (cont.).

Sample: MgO 19-50.1 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 13.7668 g				Sample: MgO 19-50.2 Sample age: 6.32 days Cruc: 11.7236 g Cruc +MgO: 11.7236 g				Sample: MgO 19-50.3 Sample age: 6.32 days Cruc: 11.6157 g Cruc + MgO: 13.6198 g			
T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C	
		Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost
300	1	13.0250	5.78	300	1	13.1189	4.46	300	1	12.9505	6.27
300	1	13.0236	5.88	300	1	13.1180	4.53	300	1	12.9492	6.36
300	1	13.0203	6.12	300	1	13.1157	4.70	300	1	12.9451	6.65
		13.0217	6.02			13.1164	4.65			12.9460	6.59
300	1	13.0201	6.13	300	1	13.1147	4.78	300	1	12.9435	6.76
300	1	13.0171	6.35	300	1	13.1134	4.88	300	1	12.9417	6.89
300	1	13.0149	6.51	300	1	13.1122	4.97	300	1	12.9405	6.97
		13.0187	6.23			13.1149	4.76			12.9443	6.71
300	1	13.0138	6.59	300	1	13.1121	4.98	300	1	12.9399	7.01
300	1	13.0128	6.66	300	1	13.1111	5.05	300	1	12.9393	7.06
300	1	13.0123	6.70	300	1	13.1111	5.05	300	1	12.9392	7.06
300	1	13.0102	6.85	300	1	13.1089	5.22	300	1	12.9369	7.23
		13.0136	6.60			13.1119	4.99			12.9395	7.04
300	1	13.0087	6.96	300	1	13.1085	5.25	300	1	12.9358	7.30
300	1	13.0074	7.05	300	1	13.1068	5.38	300	1	12.9345	7.39
300	1	13.0066	7.11	300	1	13.1064	5.41	300	1	12.9344	7.40
		13.0091	6.93			13.1085	5.25			12.9385	7.11

Table A5. LOI Parameter Tests – WTS-20 (cont.).

Sample: MgO 19-50.1 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 13.7668 g				Sample: MgO 19-50.2 Sample age: 6.32 days Cruc: 11.7236 g Cruc + MgO: 11.7236 g				Sample: MgO 19-50.3 Sample age: 6.32 days Cruc: 11.6157 g Cruc + MgO: 13.6198 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost
300	1	13.0061	7.15	300	1	13.1060	5.44	300	1	12.9339	7.44
300	1	13.0049	7.23	300	1	13.1055	5.48	300	1	12.9333	7.48
300	1	13.0046	7.25	300	1	13.1050	5.52	300	1	12.9333	7.48

Table A6. LOI Parameter Tests – WTS-60.

Sample: MgO 19-61.18 Sample age: 26.86 days Cruc: 11.9439 g Cruc + MgO: 13.9520 g				Sample: MgO 19-61.19 Sample age: 27.73 days Cruc: 11.0432 g Cruc +MgO: 13.0722 g				Sample: MgO 19-61.20 Sample age: 29.69 days Cruc: 11.2668 g Cruc + MgO: 13.2710 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
60	1	13.7008		60	1	13.0714		60	1	13.2703	
60	1	13.7004		60	1	13.0710		60	1	13.2703	
100	1	13.6996	0.05	100	1	13.0697	0.06	100	1	13.2686	0.08
100	1	13.6995	0.05	100	1	13.0694	0.08	100	1	13.2685	0.09
100	1	13.6998	0.03	100	1	13.0698	0.06	100	1	13.2690	0.06
100	1	13.6993	0.06	100	1	13.0692	0.09	100	1	13.2683	0.10
100	1	13.6997	0.04	100	1	13.0695	0.07	100	1	13.2687	0.08
100	1	13.6996	0.05	100	1	13.0693	0.08	100	1	13.2686	0.08
100	1	13.6993	0.06	100	1	13.0689	0.10	100	1	13.2682	0.10
150	1	13.6987	0.10	150	1	13.0681	0.14	150	1	13.2675	0.14
150	1	13.6985	0.11	150	1	13.0679	0.15	150	1	13.2671	0.16
200	1	13.6981	0.13	200	1	13.0674	0.18	200	1	13.2667	0.18
200	1	13.6982	0.13	200	1	13.0675	0.17	200	1	13.2669	0.17
200	1	13.6981	0.13	200	1	13.0672	0.19	200	1	13.2664	0.19
200	1	13.6979	0.14	200	1	13.0671	0.19	200	1	13.2666	0.18
200	1	13.6979	0.14	200	1	13.0672	0.19	200	1	13.2664	0.19
200	1	13.6698	1.74	200	1	13.0671	0.19	200	1	13.2663	0.20

Table A6. LOI Parameter Tests – WTS-60 (cont.).

Sample: MgO 19-61.18 Sample age: 26.86 days Cruc: 11.9439 g Cruc + MgO: 13.9520 g				Sample: MgO 19-61.19 Sample age: 27.73 days Cruc: 11.0432 g Cruc +MgO: 13.0722 g				Sample: MgO 19-61.20 Sample age: 29.69 days Cruc: 11.2668 g Cruc + MgO: 13.2710 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
300	1	13.6806	1.13	300	1	13.0558	0.75	300	1	13.2451	1.26
300	1	13.6671	1.90	300	1	13.0457	1.25	300	1	13.2250	2.26
300	1	13.6498	2.88	300	1	13.0356	1.75	300	1	13.2100	3.01
300	1	13.6389	3.50	300	1	13.0270	2.17	300	1	13.1948	3.77
300	1	13.6219	4.47	300	1	13.0138	2.82	300	1	13.1769	4.66
300	1	13.6124	5.01	300	1	13.0068	3.17	300	1	13.1663	5.19
300	1	13.5964	5.92	300	1	12.9946	3.77	300	1	13.1496	6.02
300	1	13.5920	6.17	300	1	12.9924	3.88	300	1	13.1444	6.28
300	1	13.5719	7.32	300	1	12.9787	4.55	300	1	13.1229	7.36
300	1	13.5618	7.89	300	1	12.9703	4.97	300	1	13.1124	7.88
300	1	13.5633	7.81	300	1	12.9719	4.89	300	1	13.1145	7.78
300	1	13.5425	8.99	300	1	12.9563	5.66	300	1	13.0976	8.62
300	1	13.5445	8.88	300	1	12.9577	5.59	300	1	13.1009	8.46
300	1	13.5305	9.67	300	1	12.9458	6.17	300	1	13.0847	9.26
300	1	13.5276	9.84	300	1	12.9400	6.46	300	1	13.0778	9.61
300	1	13.5081	10.95	300	1	12.9280	7.05	300	1	13.0638	10.31
300	1	13.4970	11.58	300	1	12.9189	7.50	300	1	13.0511	10.94

Table A6. LOI Parameter Tests – WTS-60 (cont.).

Sample: MgO 19-61.18 Sample age: 26.86 days Cruc: 11.9439 g Cruc + MgO: 13.9520 g				Sample: MgO 19-61.19 Sample age: 27.73 days Cruc: 11.0432 g Cruc + MgO: 13.0722 g				Sample: MgO 19-61.20 Sample age: 29.69 days Cruc: 11.2668 g Cruc + MgO: 13.2710 g			
T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C		T (°C)	time (h)	after 60 °C	
		Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost			Cruc + MgO (g)]	% mass lost
300	1	13.4973	11.56	300	1	12.9186	7.52	300	1	13.0502	10.99
300	1	13.4876	12.12	300	1	12.9106	7.91	300	1	13.0405	11.47
300	1	13.4930	11.81	300	1	12.9149	7.70	300	1	13.0427	11.36
300	1	13.4823	12.42	300	1	12.9057	8.15	300	1	13.0322	11.88
300	1	13.4755	12.80	300	1	12.8999	8.44	300	1	13.0241	12.29
300	1	13.4656	13.37	300	1	12.8899	8.93	300	1	13.0134	12.82
300	1	13.4557	13.93	300	1	12.8812	9.36	300	1	13.0016	13.41
300	1	13.4518	14.15	300	1	12.8773	9.55	300	1	12.9980	13.59
300	1	13.4461	14.48	300	1	12.8722	9.80	300	1	12.9911	13.94
		13.4475	14.40			12.8736	9.73			12.9924	13.87
300	1	13.4406	14.79	300	1	12.8666	10.08	300	1	12.9847	14.26
300	1	13.4378	14.95	300	1	12.8631	10.25	300	1	12.9806	14.46
300	1	13.4329	15.23	300	1	12.8591	10.45	300	1	12.9766	14.66
		13.4363	15.04			12.8637	10.22			12.9798	14.50
300	1	13.4242	15.72	300	1	12.8514	10.83	300	1	12.9665	15.16
300	1	13.4209	15.91	300	1	12.8480	11.00	300	1	12.9623	15.37

Table A6. LOI Parameter Tests – WTS-60 (cont.).

Sample: MgO 19-61.18 Sample age: 26.86 days Cruc: 11.9439 g Cruc + MgO: 13.9520 g				Sample: MgO 19-61.19 Sample age: 27.73 days Cruc: 11.0432 g Cruc +MgO: 13.0722 g				Sample: MgO 19-61.20 Sample age: 29.69 days Cruc: 11.2668 g Cruc + MgO: 13.2710 g			
		after 60 °C				after 60 °C				after 60 °C	
T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost	T (°C)	time (h)	Cruc + MgO (g)]	% mass lost
300	1	13.4140	16.31	300	1	12.8414	11.32	300	1	12.9547	15.75
		13.4158	16.20			12.8424	11.27			12.9557	15.70
300	1	13.4117	16.44	300	1	12.8382	11.48	300	1	12.9499	15.99
300	1	13.4079	16.65	300	1	12.8341	11.68	300	1	12.9467	16.15
300	1	13.4039	16.88	300	1	12.8308	11.85	300	1	12.9434	16.32
		13.4086	16.57			12.8339	11.64			12.9470	16.07
300	1	13.4015	16.98	300	1	12.8283	11.91	300	1	12.9413	16.35
300	1	13.3961	17.29	300	1	12.8237	12.14	300	1	12.9355	16.64
300	1	13.3949	17.35	300	1	12.8217	12.24	300	1	12.9333	16.75
300	1	13.3896	17.66	300	1	12.8161	12.51	300	1	12.9275	17.04
		13.3931	17.46			12.8185	12.40			12.9315	16.84
300	1	13.3870	17.80	300	1	12.8135	12.64	300	1	12.9255	17.14
300	1	13.3820	18.09	300	1	12.8088	12.87	300	1	12.9212	17.35
300	1	13.3786	18.28	300	1	12.8046	13.08	300	1	12.9180	17.51
		13.3814	18.12			12.8071	12.96			12.9207	17.38
300	1	13.3766	18.40	300	1	12.8026	13.18	300	1	12.9164	17.59

Table A6. LOI Parameter Tests – WTS-60 (cont.).

Sample: MgO 19-61.18 Sample age: 26.86 days Cruc: 11.9439 g Cruc + MgO: 13.9520 g				Sample: MgO 19-61.19 Sample age: 27.73 days Cruc: 11.0432 g Cruc +MgO: 13.0722 g				Sample: MgO 19-61.20 Sample age: 29.69 days Cruc: 11.2668 g Cruc + MgO: 13.2710 g			
			after 60 °C				after 60 °C				after 60 °C
T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost	T (°C)	time (h)	Cruc + MgO (g)	% mass lost
300	1	13.3754	18.47	300	1	12.8014	13.24	300	1	12.9145	17.69
300	1	13.3695	18.80	300	1	12.7947	13.57	300	1	12.9094	17.94