DEVELOPMENT OF CHEMICAL ANALYSIS TECHNIQUES
FOR ADVANCED MATERIALS

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ABSTRACT

Primary emphasis during this quarter was directed toward hydroxyl determination in brucite, $Mg(OH)_2$, employing a graphite-crucible high-temperature induction furnace; thermogravimetric analysis was also used to study brucite. Chemical techniques were used to standardize several elements in Fisher M-300 samples. The carbon content of MgO was studied as a function of heating and storage conditions.

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DEVELOPMENT OF CHEMICAL ANALYSIS TECHNIQUES
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by

E. R. Blosser

INTRODUCTION

During the first quarter of this research program, several MgO samples, previously analyzed by other investigators, were analyzed at Battelle by chemical, emission spectrographic, and mass spectrographic techniques.

The second quarter work described in this report was aimed at two problem areas. First, several elements present in Fisher M-300 MgO were analyzed chemically. Several Fisher M-300 samples can now be considered standardized for F, Si, S, Cl, and Ca. Second, the determination of hydroxyl was studied by thermogravimetric analysis and by the reaction of MgO with graphite crucibles heated by induction.

EXPERIMENTAL WORK

Mass Spectrograph

Analysis of MgO Materials

Nine MgO samples were analyzed mass spectrographically for all detectable impurities. One of these, BMI-3 #3, was rerun because certain impurities appeared at quite high levels compared with impurities in the other samples in that series. The rerun showed that this sample was less pure than the other BMI-3 samples, but that some elements were apparently not as high as had been reported on the first analysis. The results for the BMI-3 series and JPL #63 are given in Table 9[*].

JPL MgO 49, previously analyzed mass spectrographically by Dr. Leipziger of Sperry Rand under an earlier contract and emission spectrographically by Battelle during the first quarter of this contract, was analyzed mass spectrographically during this quarter. The results, shown in Table 10, indicate only fair agreement among the different laboratories and techniques. However, the Sperry Rand mass values agree better with the Battelle emission values than do the Battelle mass values. Results for some elements likely to be picked up from the steel briquetting die are probably false; these elements would include Cr, Mn, Fe, and Ni, Others, such as for H and OH, C, and N may be far too high in the Battelle mass results because neither the source nor the sample was baked prior to the analysis. Still other elements, far higher in the Battelle mass analysis than in the other mass or Battelle emission analysis, remain in doubt as no explanation for the variations is apparent and insufficient sample remains for rechecks.

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[*] Tables follow the text of the report, and are numbered sequentially for all reports produced under this contract.

Four B-series samples were also analyzed; the results are shown in Table 12. The possible effects of die contamination might be observed in this series, but can be confirmed only by reruns. B-63 was sparked using a Au counter electrode versus a chunk of the MgO, while the others were briquetted with Ag. Fe, Cr, Mn, and Ni, as well as F, Si, P, S, Cl, Cu, and Zn are lower in this "direct" analysis than in the briquetted analyses. Arbitrarily assuming that B-63 is not, in fact, purer than the other B-series samples, the alternatives are:

(1) The die introduces more contamination than its nominal composition would indicate.

(2) The Ag binder introduces more impurities than its analysis would indicate.

(3) Impurities in the MgO are released and detected in varying amounts relative to Mg and O, depending on whether the sample is sparked as a briquette or directly.

(4) Some impurities are picked up from the plastic vials used to mix the MgO with the Ag.

These possibilities should be studied soon using Fisher M-300, which is in good supply as powder or as JPL-pressed solid, and which now can be pressed at Battelle with or without Ag binder in a noncontaminating die. The results of these studies should clear up the doubts raised by the data in Table 12.

The die mentioned above is an AEI (Associated Electrical Industries, Ltd.) product recently marketed. The material is pressed isostatically in throw-away polyethylene slugs compressed in a high-strength steel die. The manufacturer claims essentially no contamination from the polyethylene, and certainly none is expected from the steel because it does not touch the sample. In preliminary trials, the die produced strong briquettes from graphite, Ag, and MgO. The briquettes have not yet been sparked in the mass spectrograph to establish the contamination, if any.

Very little additional work has been done since the first quarterly report period to determine C, H, and OH mass spectrographically. Further work on these problems was deferred until more definitive values for these impurities could be obtained by other techniques, which are described in the next sections.

Chemical Analysis

Carbon Determination

Carbon is one of the impurities whose value in the Fisher M-300 MgO varied by more than a magnitude, depending on the technique employed to determine it. The mass results were scattered but as low as 300 ppma was observed in the original powder, Batch 2 (BMI #90940). Combustion carbon determinations were made on all four samples, and several were subjected to various heating and storage conditions. From the data in Table 16 it is apparent that the three powder samples give higher C values than does the hot-pressed material. If the accuracy of the combustion determination can be assumed (i.e., if complete recovery of C is realized with the solid hot-pressed material and if no pickup of C occurs for the powdered material), then it is seen that in no case studied was the powdered material lowered in C content to the level of the as-received solid. Furthermore, the powdered material very quickly picks up C, even when stored for brief periods in an Ascarite-containing desiccator. When stored in a desiccator over solid CO_2, the pickup is even more rapid and shows no sign of leveling off after 3 hours. When stored in air with the usual humidity present, the pickup of C after 21 days is in the percent range.

The conclusion that can be reached from these experiments is that C values obtained in powdered samples, no matter how carefully stored, are meaningless. Only on compact samples such as the hot-pressed material can one expect to get reproducible and valid results. This sample, OP243, will be analyzed mass spectrograhically, with special attention given to the C content.

Other Elements

In Fisher M-300 material the values obtained for certain elements varied widely depending on the laboratory and the technique employed. Several chemical methods have been used to standardize the amounts of these elements in the various batches of Fisher M-300. The results are given in Table 17. Some data have appeared in earlier reports. It is clear that the three powder samples of M-300 either are not identical or that they have been contaminated. F, Si, S, and Cl are nearly the same in the three samples but Na and K are not. Even allowing for an error in the K determination on the original lot (BMI #90940), the Na variations are not within the analytical accuracy. It is also apparent that the JPL treatment of B-3 has introduced some Na, Cl, and Ca as shown by the increase of these elements in the calcined and hot-pressed samples.

The K values reported for the last three samples in Table 17 (the B-3 powders and OP243) were obtained using atomic absorption and synthetic standards closely duplicating the samples. The flame photometer also was used for these samples, and gave values ranging from 55 to 65 ppma. The reason for the variance is not known.

S, Si, F, and Cl have remained sufficiently constant in the several samples to be considered standardized, and if the one low Ca value (as-received B-3) is discarded, the Ca is also standardized. Therefore only H (or OH), C, Na, and K remain in some doubt; future work should at least partially standardize these elements.

Hydroxyl Determination

Hydroxyl (OH) is an important impurity in MgO because it can enter the crystal structure. Low levels of OH are difficult to determine, however, because the last traces of OH appear to be more tightly bonded than would be expected were all the OH present as $Mg(OH)_2$. At the suggestion of Dr. Leipold, brucite [natural $Mg(OH)_2$] was used in preliminary experiments to determine whether quantitative recovery of H_2 or H_2O from $Mg(OH)_2$ is feasible.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was the first technique employed. Small, weighed amounts of fibrous brucite were placed on the pan of the TGA apparatus. TGA thermograms were obtained at a heating rate of 4 C per minute from room temperature to 900 C and at a pressure of about 0.1 torr. Duplicate runs were obtained on the fiber sample. The first sample exhibited a weight loss of 2.0 percent between room temperature and 50 C, undoubtedly due to free or surface water. Dehydration began between 250 C and 300 C and appeared to progress in two steps, with the major dehydration complete by 400 C. Total weight loss at 900 C was 32.5 percent. Correcting for the 2 percent free water, the net loss of 30.5 percent is in excellent agreement with the calculated value of 30.85 percent. The second sample contained 1.3 percent free water, which was removed by heating to 50 C. Again, dehydration began between 250 C and 300 C and progressed in two steps, with the major loss occurring by 400 C. Total weight loss at 900 C was 31.8 percent. Again correcting for the 1.3 percent free water, the net loss of 30.5 percent is in excellent agreement with the calculated value.

A sample of material, supposedly chunk-form bricute, was also run. This sample lost only 0.9 percent by 600 C and only 6.0 percent by 900 C versus the theoretical weight loss of 30.85 percent. This sample almost certainly is not brucite. Figure 1 is a copy of the thermograms obtained from the chunk run and the first run of the fibrous sample; the thermogram of the second run of fibrous brucite is shown in Figure 2.

Vacuum-Fusion Furnace

The second and third approaches to the OH determination were basically similar, differing primarily in the apparatus used. The vacuum-fusion technique will be described first, then the quartz-tube graphite-crucible furnace technique.

The vacuum-fusion furnace used in this work is a Battelle-fabricated unit similar to the Guldner-Beach design excpet that the crucible is supported from the bottom of the furnace instead of being supported by platinum wire from the top. The conventional carbon-crucible, graphite-packing, sample-loading system, and degassing method were used.

A fibrous sample was weighed into a tin capsule to facilitate dropping the sample from the loading arm to the crucible. The open end of the tin capsule was crimped to prevent loss of sample. A tin flux frequently is used in normal vacuum-fusion analysis but in this case the primary purpose was to provide a carrier for dropping the sample.

The encased sample was placed in the loading arm and the system was evacuated and degassed at 2000 C for 3 hours. The loading arm remained at room temperature during this furnace degassing procedure. After a 3-hour degassing period, a blank was obtained on the furnace assembly by collecting the gases released during a 35-minute period, pumping the gases into an evacuated sample bulb, and analyzing the gases by mass spectrometric analysis.

The temperature of the furnace was reduced to lower than 1000 C and the capsule containing the sample was dropped into the crucible. The temperature was then increased to 2000 C, and as the gas evolved from the furnace it was pumped into an evacuated bulb for mass spectrometric analysis. The time required to extract the evolved gas from the furnace was 1 hour. The quantity of components found in the blank was adjusted for the longer extraction time required for the sample.

Run 1 was made using 9.39 mg of fibrous brucite obtained from The Ohio State University Mineralogy Laboratory. The results, indicating that approximately 95 volume percent of the gas evolved was water and carbon monoxide, suggest the following decomposition equation:

$$Mg(OH)_2 + C \xrightarrow{2000\ C} H_2O + CO + Mg.$$
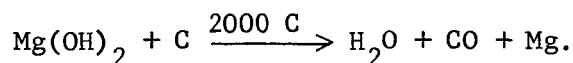
Based on this equation, 9.39 mg of brucite should give 2.9 mg of water and 4.51 mg of carbon monoxide. The actual recovery of water in this experiment was 157 percent and the recovery of carbon monoxide was 59 percent.

The 9.39-mg sample was larger than needed for a good analysis, and the large amount of water was difficult to handle because of possible condensation. Run 2 was made using the same procedure except that the sample weight was reduced to 1.857 mg and the sample and capsule were heated to 100 C in the loading arm during the furnace degassing period to drive off any surface moisture that might have been on the sample.

The extraction time was reduced to 30 minutes for the smaller sample. The extraction time is determined as the point at which no additional gas is being released from the sample. After completion of this extraction the furnace was opened for cleaning and some unreacted sample was found at the base of the furnace compartment. Its weight was subtracted from the original weight, which gave an actual sample weight of 1.658 mg. The most logical explanation for this loss of sample is the "splattering" effect which frequently happens in vacuum fusion if the tin melts too fast and vaporizes, causing turbulence in the carbon crucible.

It again appeared that the decomposition reaction followed the equation given above. Since some hydrocarbons were present, the hydrogen value is probably high. Recovery was 63 percent, based on the amount of water found and 7 percent, based on CO. Some additional unreacted material was found on closer inspection of the furnace interior, but was not weighed. It is not known whether the additional sample found in the bottom of the furnace came from the first or the second run. The vacuum-fusion approach appeared to be giving variable and inaccurate results, and was therefore abandoned.

Quartz-Tube Graphite-Crucible Furnace

The third approach used a specially designed quartz-tube graphite-crucible apparatus differing from the conventional vacuum-fusion furnace in several ways:

(1) The internal volume is much smaller.

(2) There is no graphite packing around the carbon crucible.

(3) The apparatus can be attached directly to the inlet system of the mass spectrometer, eliminating the transfer of the sample through a second sample bulb.

(4) The graphite crucible and support is the same as used in the vacuum-fusion apparatus except that the thickness of the walls has been reduced to produce better coupling with the induction heater and also to reduce the total mass inside the apparatus to improve the degassing characteristics.

The apparatus was assembled, evacuated, and heated by induction to 2200 C for 1.5 minutes. An evacuation system was not available at the site of the induction heating supply; therefore, this procedure was repeated several times until a constant blank was obtained. The apparatus was opened to the air between each heating and evacuation because opening to the air was necessary to load the sample. A 1.625-mg sample of fibrous brucite was weighed into the crucible and the system evacuated. The temperature was increased from room temperature to 2200 C in approximately 1 minute and held for 1.5 minutes. The apparatus was transferred to the mass spectrometer, and the quantity of evolved gas was measured and analyzed. The results obtained are shown in Table 15.

Analysis showed that 96.6 volume percent of the gas formed was hydrogen and carbon monoxide. The formation of H_2 in the quartz tube and H_2O in the vacuum fusion is probably explained by the catalytic effect of the mercury diffusion pumps in the vacuum-fusion apparatus and perhaps by the longer reaction time and the presence of tin in the vacuum-fusion system. The difference in final products suggests that H_2, not H_2O, is the original form of hydrogen, and that the H_2 subsequently reacts with 0 or CO in the vacuum-fusion system to form H_2O.

The weight of sample used should have produced 0.0562 mg of hydrogen. The 0.0563 mg of hydrogen found represents a recovery of 100.2 percent. In addition to the hydrogen present, it would appear that a small portion of the H_2 reacted to form H_2O, which should be added to the amount of H_2 obtained. The total H_2 recovery based on water and hydrogen was 108.6 percent. Calculations based on water and carbon monoxide show an oxygen recovery of only 88.6 percent; however, the analysis for carbon monoxide by the mass spectrometric technique is not sufficiently precise.

The experiment using the quartz-tube graphite-crucible apparatus was repeated using the same procedure except that the quartz tube was cooled by submerging the tube in a beaker of water so that the ground-glass joint was completely covered. This reduced the hydrogen blank from 0.0135 mg to 0.0047 mg. Analysis of the reaction products from a sample of fibrous brucite weighing 1.891 mg showed 44.0 volume percent H_2 and 56 volume percent CO. No significant amounts of H_2O, CO_2, or hydrocarbons were detected. The calculated weights of H_2 and CO were 0.0654 and 0.908 mg whereas the observed weights were 0.0684 and 1.242 mg, representing recoveries of 105 and 137 percent, respectively. These results are also shown in Table 15.

The quartz-tube graphite-crucible technique described above was used to study MgO (Fisher M-300, B-3, Calcined 700 C Vac., BMI #91938). For these experiments the entire reaction vessel and the induction coil were submerged in deionized water to cool the quartz tube. No reduction of the blank was noted, suggesting that the CO and H_2 come from the graphite crucible and its support, not from the walls of the quartz tube.

A sample weighing 0.2012 gram was transferred to the graphite crucible without drying. During evacuation, the pump-down rate was extremely slow; therefore, the gas being pumped off was monitored by the mass spectrometer and found to be water. Not all the water had been pumped off after 30 minutes of pumping.

The temperature was raised gradually but a very small portion of the sample was ejected from the crucible during the low-heat period. No additional loss of sample was observed at the high temperature. The

small amount of water left in the sample may have caused the loss of sample. Analytical results obtained on this run and Runs 2 and 3 are shown in Table 18. The results of this run should not be compared with those of Runs 2 and 3 because this was not a dried sample and some unknown quantity of sample was lost at the start of the heating period.

Two additional analyses were made on a sample that had been dried at 110 C for 75 minutes. The sample was not redried before Run 3 was made, but stood in a silica gel desiccant for 24 hours. These runs were normal in all respects and it was possible to increase the heat without loss of sample. The results of this run are also shown in Table 18.

The gases collected from the blanks and sample were scanned to mass 100, and no compounds except hydrogen and carbon monoxide were detected in any significant amount. Acetylene at the 0.1 percent level was detected in Run 2. Based on the hydrogen evolved from MgO in Runs 2 and 3, the OH content was 3.5 and 3.9 weight percent, respectively. The relative reproducibility is about 11 percent.

These results were encouraging and suggested that the quartz-tube reaction vessel be redesigned to permit degassing and sample loading without opening to the air. This has been done, and a vessel using a much smaller graphite crucible, a swinging funnel for dropping the sample into the crucible, and a side arm to store the sample during degassing is being fabricated. The experiments will use the hot-isostatic-pressed Fisher M-300 MgO to minimize the problems of water adsorption associated with powders.

CONCLUSIONS AND RECOMMENDATIONS

The research described in this report has established fairly well-standardized values for several elements in both powder and compact Fisher M-300 MgO. Using these materials, the mass spectrographic technique can in turn be standardized and relative sensitivity coefficients for these elements can be assigned. Silicon, chlorine, and calcium appear to have

unit sensitivity in Battelle's mass spectrographic analysis, fluorine and sodium are low by a factor of about 10, and potassium and sulfur are high by a factor of about 3. These factors will be reevaluated when compact samples are analyzed.

The decomposition of brucite $[Mg(OH)_2]$ by thermal and chemical means has shown promise for the determination of small amounts of hydroxyl in MgO. Experiments that should establish the feasibility of the graphite-crucible reaction for liberating H_2 are under way. If successful, this would be preferred to the mass spectrographic technique because of many problems with the latter. The mass spectrographic technique still appears to be valid for most other elements, however, and has the advantage of simultaneous detection of all impurity elements.

NEW TECHNOLOGY

No reportable items of new technology have been generated under this contract to date.

FUTURE WORK

Intensive research will be continued on the hydroxyl determination, with prime emphasis on the quartz-tube graphite-crucible reaction. Compact material will be studied almost exclusively because surface absorption of water is a formidable problem for powders.

Carbon in MgO will be studied, using magnesite $(MgCO_3)$ as a reference point, in a manner similar to the brucite studies.

The mass spectrographic technique will be investigated further in an attempt to increase precision and also to correlate mass data for OH and C with the chemically and thermally determined values.

TABLE 9. MASS SPECTROGRAPHIC ANALYSIS OF MgO

(ppma)

	Sample				
	BMI-3				JPL
			#3 (c)		
Element	#1 (a)	#2 (b)	Run 1	Run 2 (d)	#63
H	30,000.	5,000.	1,000.	--	[100,000.]
OH (e)	3,000.	300.	100.	[1,000.]	[30,000.]
Li	0.3	0.3	≤1.	2.	0.5
B	10.	25.	≤1	2.	≤0.1
C	2,000.	1,000.	1,000.	[10,000.]	[5,000.]
N	300.	100.	50.	[1,000.]	[300.]
F	10.	20.	1.	5.	<1.
Na	10.	≤1	<1.	10.	<1.
Al	10.	3.	30.	10.	30.
Si	100.	100.	400.	500.	<3.
P	5.	3.	2.	5.	0.3
S	100.	100.	100.	500.	10.
Cl	100.	12.	40.	130.	40.
K	30.	≤1.	10.	30.	10.
Ca	20.	10.	50.	100.	30.
Ti	<4.	<1.	40.	40.	<10.
Cr	1.	4.	10.	15.	0.2
Mn	≤0.3	1.	10.	3.	0.3
Fe	10.	10.	400.	50.	3.
Co	1.	<0.1	1.	≤1.	<0.1
Ni	1.	1.	15.	15.	≤0.2
Cu	15.	15.	15.	5.	1.
Zn	10.	10.	70.	15.	5.
Pb	1.	3.	1.	<0.4	<0.2

(a) As pressed.
(b) 1010 C.
(c) 1750 C.
(d) From Table 11.
(e) Read at m/e 17.
[ ] Sample not baked.

TABLE 10.   ANALYSIS OF JPL MgO 49

(ppma)

| Element | Battelle | | Sperry Rand Mass[a] |
	Mass	Emission[a]	
H	[100,000.]	--	--
OH (b)	[30,000.]	--	--
Li	0.3	--	--
B	3.	<4.	<1.
C	[200,000.]	--	--
N	[300.]	--	65.
F	30.	--	8.
Na	10.	<50.	<1.
Al	20.	30.	11.
Si	100.	14.	40.
P	10.	--	--
S	200.	--	--
Cl	400.	--	45.
K	30.	--	<0.1
Ca	30.	10.	13.
Ti	40.	--	--
Cr	1.	--	--
Mn	3.	--	--
Fe	30.	3.5	3.
Co	0.3	<2. (T)	--
Ni	10.	--	--
Cu	15.	<3. (T)	--
Zn	20.	--	--
Ag	--	1.	--

(a)   From Table 3.
(b)   Read at m/e 17.
(T)   Trace.
[ ]   Sample not baked.

TABLE 12. MASS SPECTROGRAPHIC ANALYSIS OF MgO

(ppma)

Element	Sample Number			
	B-62	B-63	B-64	B-65
H	[100,000.]	[50,000.]	[100,000.]	[100,000.]
OH (a)	[10,000.]	[50,000.]	[100,000.]	[10,000.]
Li	0.3	0.3	0.3	0.3
B	2.	<0.1	3.	1.
C	[300.]	[3,000.]	[3,000.]	[2,000.]
N	[20.]	[200.]	[30.]	[10.]
F	3.	<1.	20.	2.
Na	1.	<1.	3.	<1.
Al	10.	50.	20.	10.
Si	15.	<4.	300.	10.
P	3.	0.3	10.	3.
S	15.	10.	300.	30.
Cl	300.	100.	1,000.	150.
K	2.	5.	30.	3.
Ca	≤100.	30.	30.	10.
Ti	40.	40.	40.	10.
Cr	1.	0.3	1.	1.
Mn	1.	0.2	3.	1.
Fe	30.	3.	10.	10.
Co	0.3	0.2	1.	0.1
Ni	5.	≤0.1	20.	1.
Cu	1.	0.5	2.	1.
Zn	15.	0.5	20.	2.

(a) Read at m/e 17.
[ ] Sample not baked.

TABLE 15. DECOMPOSITION PRODUCTS OF BRUCITE - QUARTZ TUBE

Compound	Volume Percent	cc Atmospheres*	mg in Sample	mg in Blank	Net mg In Sample
		Run 1			
Water.	3.36	0.063	0.0506	0.0076	0.0430
Carbon monoxide	55.3	1.04	1.300	0.060	1.240
Hydrogen	41.3	0.776	0.0698	0.0135	0.0563
Carbon dioxide	0.06	0.001	0.0020	0.00177	0.00019
		Run 2			
Water	--	--	--	--	--
Carbon monoxide	56.0	1.035	1.295	0.053	1.242
Hydrogen	44.0	0.813	0.0731	0.0047	0.0684
Carbon dioxide	--	--	--	--	--

* Cubic centimeters of gas at standard pressure and ambient temperature (approximately 28 C).

TABLE 16. CARBON IN MgO (FISHER M-300) SAMPLES AS A FUNCTION OF
HEATING AND STORAGE

(ppma)

Treatment	Original[a]	2nd Lot Calcined[b]	2nd Lot As Received[c]	2nd Lot Hot Pressed[d]
As received	27,000	14,000	6,700	170
1 hr @ 100 C	--	14,000	--	170
16 hr @ 140 C	--	14,000	--	170
1 hr @ 1100 C	--	4,700	--	<17
190 hr @ 930 C				
1st sample (1 hr) (e)	--	2,600	--	--
2nd sample (2 hr) (e)	--	4,000	--	--
3rd sample (3 hr) (e)	--	4,700	--	--
21 days in air	--	19,700	--	--
24 hr @ 975 C	--	--	1,000	--
1/4 hr in desiccator	--	--	1,200	--
3/4 hr in desiccator	--	--	1,600	--
3 hr in desiccator	--	--	2,100	--
1/4 hr in CO_2	--	--	4,000	--
1/2 hr in CO_2	--	--	4,000	--
1 hr in CO_2	--	--	4,300	--
2 hr in CO_2	--	--	5,300	--
3 hr in CO_2	--	--	6,000	--

(a) Small plastic bottle, labeled Batch 2; BMI #90940 (powder).

(b) Large plastic bottle, labeled B3 Calcined 700 C Vac; BMI #91938 (powder).

(c) Large plastic bottle, labeled B3 As Received; BMI #91939 (powder).

(d) Small plastic bottle, labeled OP243 (hot pressed); BMI #91940 (solid).

(e) Taken successively from same ignited MgO sample stored in desiccator.

Note: Many of these data appeared in Table 13.

TABLE 17.   COMPARISON OF TECHNIQUES FOR CERTAIN ELEMENTS
IN FOUR SAMPLES OF MgO (FISHER M-300)

(ppma)

	F	Na	Si	S	Cl	K	Ca
Original Lot (BMI #90940)							
Bell & Howell (M)	4700.	61.	1800.	420.	80.	64.	310.
Sperry Rand (M)	110.	100.	2400.	510.	285.	<1.	150.
Sperry Rand (E)	--	500.	>1000.	--	--	<1.	350.
Battelle (M)	100.	30.	350.	300.	400.	30.	2000.
Battelle (E)	--	200.	450.	--	--	<30.	2400.
Battelle (C)	1330.	--	--	100.	200.	300.	--
B-3 Calcined (BMI #91938)							
Battelle (C)	1310.	340.	350.	121.	435.	12.	2750.
B-3 As Received (BMI #91939)							
Battelle (C)	1700.	23.	350.	89.	285.	10.	1450.
OP243 (BMI #91940)							
Battelle (C)	1330.	290.	--	85.	595.	8.	2680.

(M)   Mass spectrographic.
(E)   Emission spectrographic.
(C)   Chemical.

TABLE 18.   PERCENT OH IN MgO (FISHER M-300, B-3, CALCINED 700 C VAC.)
BASED ON EVOLVED HYDROGEN

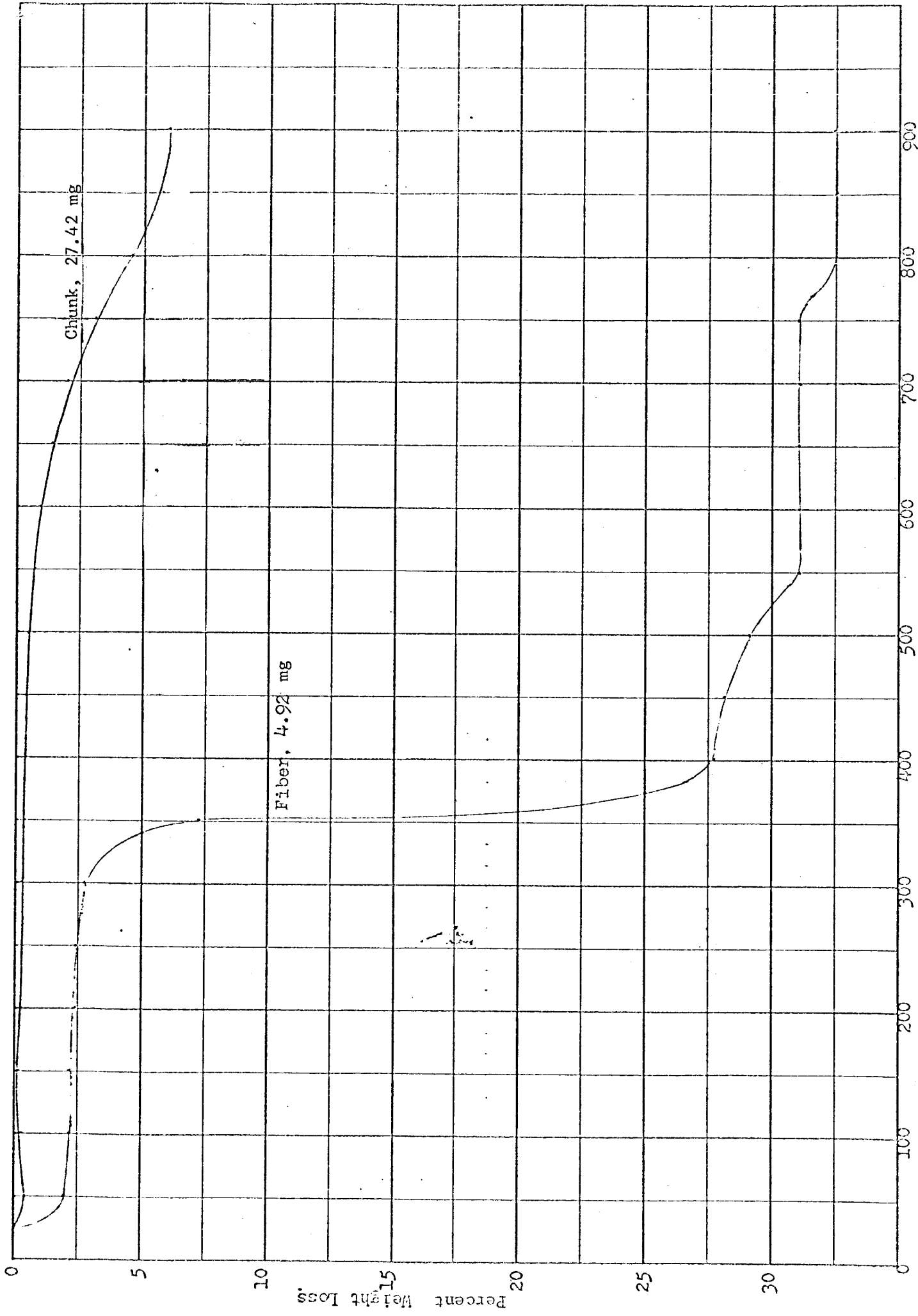| Run	Sample Weight, g	Total Gas, cc-atm.	wt H_2 in Sample, mg	wt H_2 in Blank, mg	Net wt Hydrogen, mg	Weight Percent OH	Weight Percent $Mg(OH)_2$
MgO 1[a]	0.2012	13.75	0.334	0.004	0.330	2.8	4.8
MgO 2	0.2013	16.52	0.420	0.010	0.410	3.5	5.9
MgO 3	0.2003	13.64	0.490	0.020	0.470	3.9	6.7

(a) Not dried, see text.

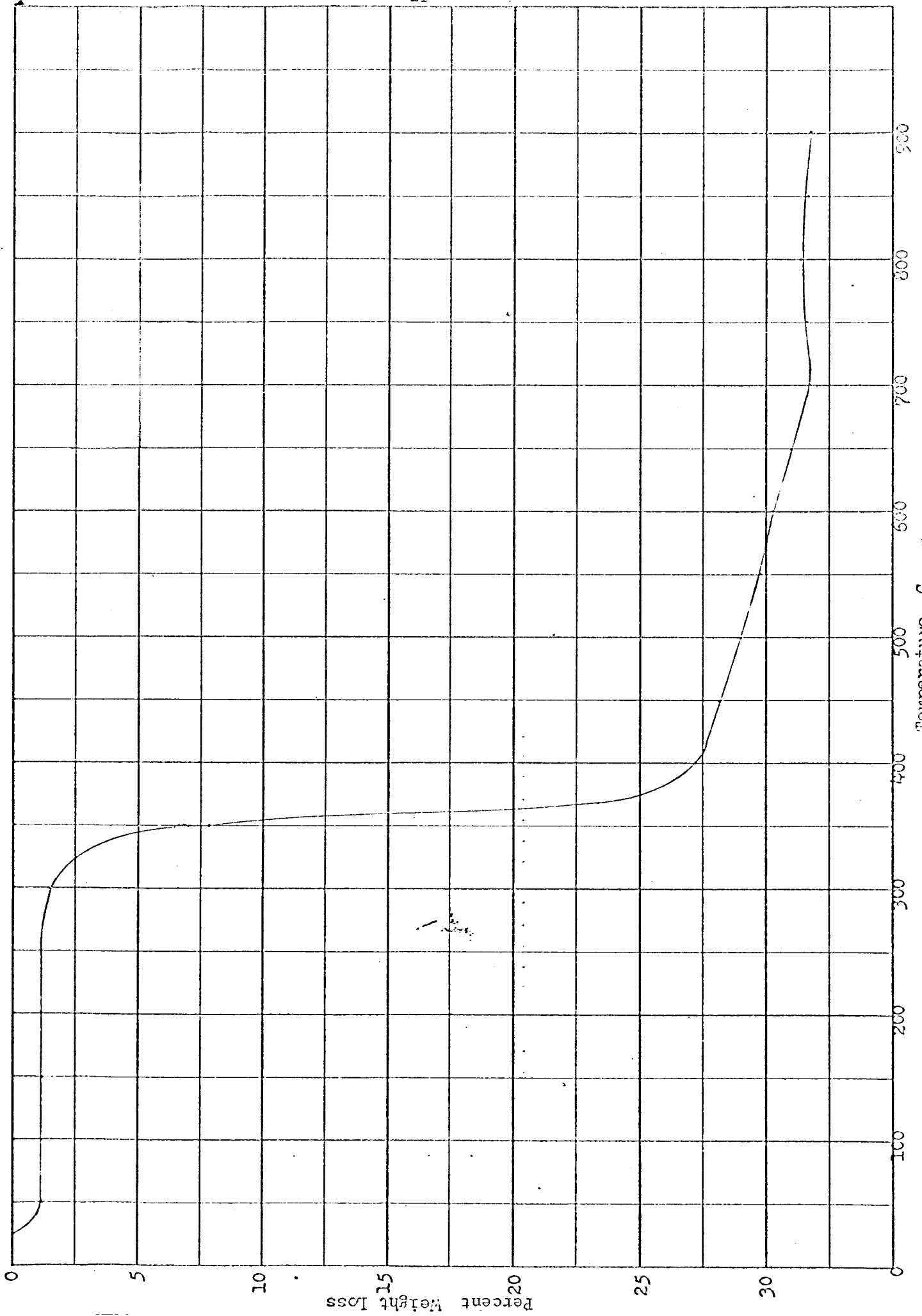FIGURE 1. TGA THERMOGRAMS--BRUCITE, FIBER AND CHUNK

FIGURE 2. TGA THERMOGRAM--BRUCITE FIBER 6.14 mg, 4 C/min., 0.1 mm Hg