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CANADA

THE EFFECT OF VARIOUS FACTORS
ON THE PROTECTION OF MOLTEN
MAGNESIUM METAL BY MIXED
HALIDE FLUXES

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A. Hubert Webster* and Norman F.H. Bright**

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SYNOPSIS

The losses of magnesium from small molten samples, protected by a flux cover under a variety of conditions, have been determined experimentally. When argon was substituted for air as the ambient atmosphere the magnesium loss increased, but when oxygen was substituted for air there was no significant change in magnesium loss. When a "refining" type of flux was substituted for a "protective" type of flux, no significant change in the magnesium loss was observed. The presence of moisture in the flux and in the atmosphere was shown to increase the magnesium loss slightly. The flux appeared to protect the metal from rapid oxidation up to temperatures as high as the boiling point of the metal (1107°C).

Evidence is discussed for the protective action of an oxide layer, formed in conjunction with the flux.

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EFFET DE DIVERS FACTEURS SUR LA PROTECTION
DU MAGNÉSIUM MÉTALLIQUE FONDU À L'AIDE D'UN
MÉLANGE DE FONDANTS HALOGÉNÉS

par

A. Hubert Webster* et Norman F.H. Bright**

RÉSUMÉ

Des essais ont permis de déterminer les pertes de magnésium subies par de petits échantillons fondus qui se trouvaient protégés de diverses façons à l'aide d'un enduit de fondant. Quand on prenait l'argon au lieu de l'air en tant que milieu ambiant, on constatait une perte accrue de magnésium, mais en remplaçant l'air par l'oxygène, on ne notait aucune perte sensiblement plus forte de magnésium. Il en était de même quand on remplaçait un fondant "protecteur" par un fondant "propre à l'affinage". Il a été démontré que la présence d'humidité dans le fondant et dans le milieu augmentait légèrement les pertes de magnésium. Il a semblé que le fondant protégeait le métal contre l'oxydation rapide jusqu'à des températures aussi élevées que le point d'ébullition du métal (1, 107°C).

On traite des preuves de l'action protectrice que peut avoir une couche d'oxyde formée conjointement avec le fondant.

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CONTENTS

	<u>Page</u>
Synopsis	i
Résumé	ii
Introduction	1
Experimental Procedures	2
Experimental Results	6
Discussion	16
Acknowledgements	19
References	19
Appendix - Statistical Treatment of Results	20-25

LIST OF TABLES

<u>No.</u>		<u>Page</u>
1(a).	Results of Factorial Experiment to Investigate the Effects of Magnesium Sample Weight, of Flux Sample Weight and of Air Flow Rate on Magnesium Loss	7
1(b).	Results of Additional Experiments on Magnesium Held under Pouring Flux at 720°C in Dry Air for 3 Hours	7
2.	Magnesium Weight Losses of Samples Heated in Argon to 720°C and then Cooled in Argon without Holding at Peak Temperature	8
3.	Magnesium Weight Loss of Sample Held at 720°C for 3 Hours in Dry Air	9
4.	Magnesium Weight Losses of Samples Held in Dry Oxygen at 720°C for 3 Hours	10
5.	Results of Experiments to Show the Effect of Moisture on Magnesium Loss	11
6.	Magnesium Weight Losses of Samples Held under Charging Flux for 3 Hours in Oxygen Atmosphere (Flow Rate - 0.16 litre/min) at 720°C	12
7.	Magnesium Weight Losses of Samples Exposed to Air and to Argon for Various Periods of Time ...	13
8.	Ignition Points of Magnesium in Oxygen, With and Without Flux Cover	15

FIGURE

1.	Furnace for Holding Magnesium Samples under Controlled Atmospheres	3
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INTRODUCTION

It is known that magnesium must be protected from oxidation during melting operations. This protection is generally accomplished by the application of a flux, composed principally of molten halide salts, to the surface of the magnesium (1, 2). The present investigation was undertaken in an attempt to determine the effects of various conditions on the magnesium losses that occur when the metal is held in the molten state, and also, if possible, to determine the process(es) whereby these losses occur. This investigation has formed part of a comprehensive study of magnesium fluxes conducted at the Mines Branch on behalf of a Canadian magnesium-smelting company.

In the standard procedure of the present investigation, a small piece of magnesium was held in the molten state for three hours at 720°C, with a cover of a "pouring"-type flux, under a dry air atmosphere. The effects of the following principal variables on the loss of magnesium, expressed in weight per cent, were investigated:

- (1) the substitution of oxygen for the air atmosphere,
- (2) the addition of moisture to the atmosphere and to
the flux,

- (3) the replacement of the "pouring"-type flux by a "charging"-type flux,
- (4) the substitution of argon for the air atmosphere,
- and (5) the variation in time of exposure to air, and to argon.

EXPERIMENTAL PROCEDURES

The experiments were performed in a vertical tube furnace in which a controlled atmosphere could be maintained (see Figure 1). The magnesium sample, usually about 1.5 to 2 grams in weight, was cleaned with emery paper, dipped in dilute hydrochloric acid, and washed in distilled water and, finally, in acetone. The sample was then weighed and placed in an MgO crucible. These crucibles were, unfortunately, porous and could absorb the flux, but they contained the molten metal satisfactorily without absorption. In a typical experiment, the magnesium sample of known weight, protected by the flux, was heated in argon to 720°C, the desired atmosphere was introduced, and the system was maintained at the necessary temperature for the appropriate time. Argon was then re-introduced and the sample was cooled. The solidified boule of magnesium metal was recovered.

The amount of metallic magnesium remaining in the boule was determined by measurement of the hydrogen displaced from

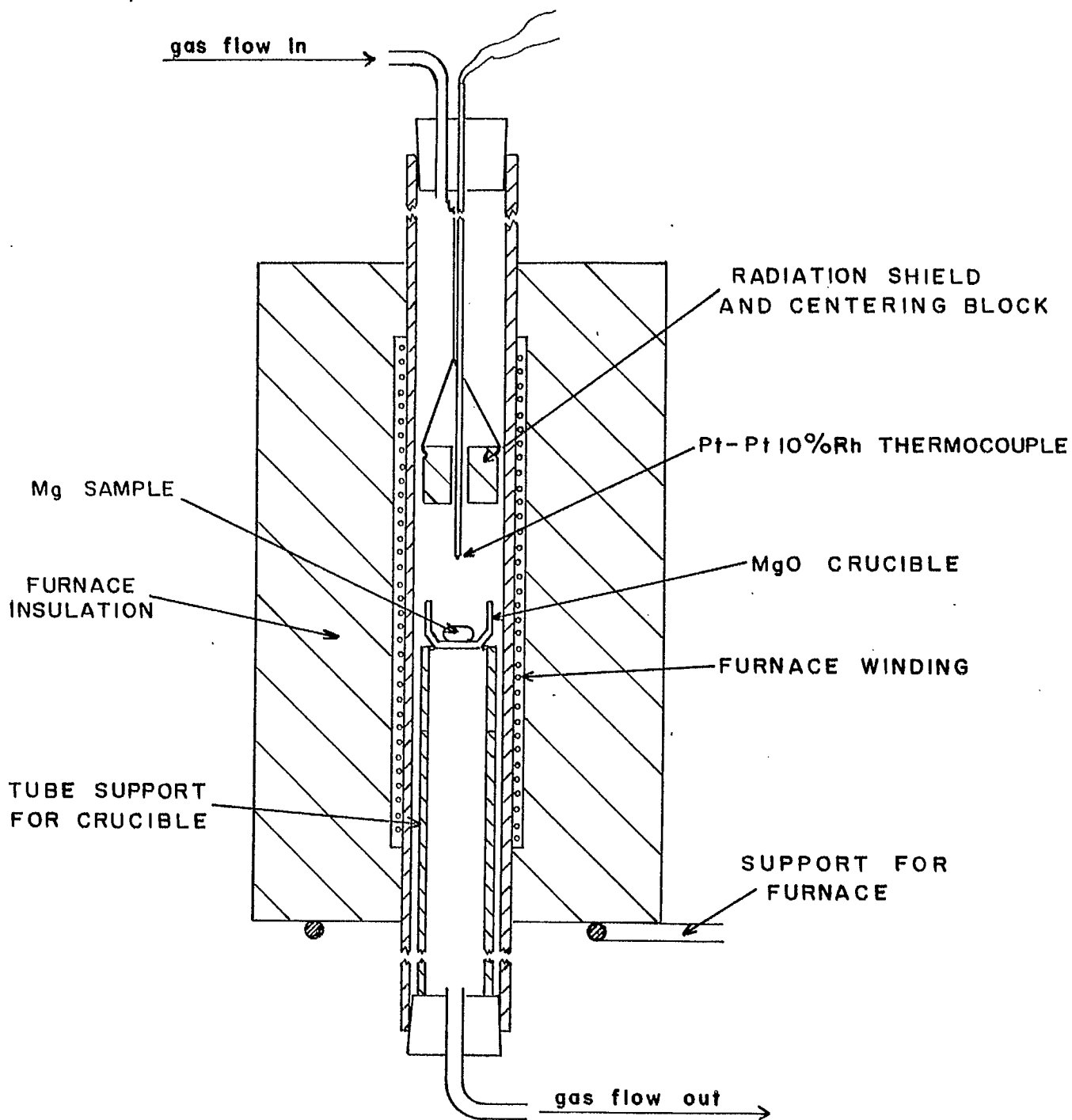


Figure 1. Furnace for Holding Magnesium Samples under Controlled Atmospheres.

sulphuric acid by the magnesium. In most cases the method was modified so that the weight of water displaced by the hydrogen was determined instead of the volume of the hydrogen itself. The calculated results of the magnesium determinations were all reduced by 0.26% on the basis of determinations which had been made on cleaned samples of magnesium taken from the stock that was used in the experiments. These determinations indicated the apparent presence of 100.26% Mg. The standard deviation in the ratio, $(\text{wt magnesium used}) / (\text{wt magnesium found})$, was 0.2%, as determined on three unmelted samples. This was regarded as an adequate degree of precision of analysis for the present purposes.

The crucibles were washed and dried after each experiment, and were heated to about 700°C just prior to use, in order to expel any residual moisture. The addition of the flux to the crucible containing the magnesium was done in a dry box containing desiccated air. The crucible and contents were subsequently handled in a closed container, with only a brief exposure to the room atmosphere during transfer to the furnace.

Temperatures were measured by the thermocouple placed above the crucible. The temperature measurement was calibrated by replacement of the sample in the crucible with a second thermocouple.

Flux samples* were supplied by the company on whose behalf the work was done, and the magnesium metal samples were cut from a bar supplied by Mr. B. Lagowski, Non-Ferrous Metals Section, Physical Metallurgy Division, Mines Branch. The argon was deoxidized over hot copper and the gases were dried over Drierite, where necessary.

Ignition point determinations were also made with the apparatus shown in Figure 1. The temperature was increased by periodically raising the power input to the furnace on an arbitrary schedule which was used for all determinations. This schedule produced the temperature-time relationship given in the following table:-

<u>Temperature</u> (°C)	<u>Time</u> (min)
Room Temperature	0
500	8 [±] 1
850	40 [±] 3
940	50 [±] 1
1000	56 [±] 2
1080	62 [±] 3
1150	69 [±] 3

Ignition was indicated by a sudden increase in temperature above that indicated by the schedule. Magnesium samples used in these

* The compositions of the fluxes were:-

Pouring Flux: 60 wt % $MgCl_2$, 40 wt % KCl

Charging Flux: 52 wt % $MgCl_2$, 35 wt % KCl, 13 wt % CaF_2 .

determinations were generally about 0.2 g in weight.

EXPERIMENTAL RESULTS

(a) Oxidation of Magnesium in the Absence of Flux

A sample of magnesium (1.88 g) was held in an MgO crucible to which no flux had ever been added, and was exposed to a dry oxygen atmosphere for 1 hour at 720°C. The magnesium was completely oxidized to a white powder. It is probable that the reaction took place entirely within the first few minutes, as a sharp rise in temperature was observed shortly after the introduction of the oxygen at 720°C.

(b) The Effects of Variations in Magnesium Sample Weight, in Flux Weight, and in Air Flow Rate

The effects of changes in magnesium weight, in flux weight and in air flow rate, on the weight loss of magnesium protected by the pouring flux, were examined at 720°C with 3 hour exposures to a flowing dry air atmosphere. The results are shown in Table 1(a). Statistical analysis of these results* indicated that the variations in the factors investigated produced no changes in weight loss that were significant (at the 10% level) when compared with changes in weight loss produced by uncontrolled factors.

* Outlines of the statistical analyses are given in the Appendix; see pages 20 to 25.

TABLE 1(a)

Results of Factorial Experiment to Investigate the Effects of
Magnesium Sample Weight, of Flux Sample Weight and
of Air Flow Rate on Magnesium Loss
 Samples were held under pouring flux at 720°C in dry air
 for 3 hours. Heating and cooling were under argon.

Wt Mg (g)	Wt flux (g)	Air flow rate (litre/min)	Mg loss (wt %)
1.754	1.56	0.17	0.2 ₆
2.039	1.59	0.17	0.5 ₄
1.744	2.53	0.17	1.1 ₄
1.987	2.68	0.17	1.3 ₁
1.766	1.68	0.22	1.9 ₈
2.095	1.67	0.22	0.3 ₈
1.688	2.58	0.22	0.9 ₅
2.019	2.44	0.22	0.8 ₄

TABLE 1(b)

Results of Additional Experiments on Magnesium Held
under Pouring Flux at 720°C in Dry Air for 3 Hours
 Heating and cooling were under argon.

Wt Mg (g)	Wt flux (g)	Air flow rate (litre/min)	Mg loss (wt %)
1.709	1.61	0.11	1.0 ₅
2.062	1.67	0.22	3.1 ₀
1.439	1.30	0.17	1.7 ₄
1.710	1.49	0.17	0.3 ₅
1.704	1.65	Not determined	0.5 ₈

Mean weight loss for samples listed in Tables 1(a) and 1(b):-
 1.0₉ wt %.

The results of five additional experiments, made under similar general conditions, are reported in Table 1(b). The combined results of Tables 1(a) and 1(b) gave a mean magnesium weight loss of $1.0_9\%$ for molten metal under the pouring flux exposed to dry air for 3 hours at 720°C .

Magnesium samples, protected by the pouring flux, were heated to 720°C and cooled in a flowing argon atmosphere without holding at the peak temperature or exposure to any other atmosphere. The magnesium weight losses observed are reported in Table 2; the mean weight loss under these conditions was $0.5_4\%$. The difference between this value and the mean weight loss for samples exposed to dry air for 3 hours is 0.5_5 weight %. This difference is significant at the 10% level.

TABLE 2

Magnesium Weight Losses of Samples Heated in Argon to 720°C and then Cooled in Argon without Holding at Peak Temperature
Pouring flux used.

Wt Mg (g)	Wt flux (g)	Mg loss (wt %)
1.880	1.58	0.4_3
1.640	2.07	0.5_5
1.712	1.57	0.6_4

Mean Mg loss:- 0.5_4 wt %.

(c) The Effect of Residual Flux in the MgO Crucible

A magnesium sample was placed in a crucible which had held the pouring flux during previous experiments, but to which no further addition of flux was made. The result of exposure of this sample to flowing dry air for 3 hours is given in Table 3.

TABLE 3

Magnesium Weight Loss of Sample Held at 720°C for 3 Hours
in Dry Air

No flux was added, but crucible had been used previously with pouring flux. Heating and cooling were in argon.

Wt Mg (g)	Air flow rate (litre/min)	Mg loss (wt %)
1.748	0.17	0.6 ₃

This result was not significantly different at the 10% level from the mean result found when flux was added directly to the sample. A similar indication was given by the results obtained with oxygen (see Table 4). It appears that the flux held in the pores of the magnesia crucible wetted the metal sample and was sufficient to provide protection against rapid oxidation.

(d) Effect of Oxygen Atmosphere

Magnesium samples, protected by the pouring flux (either added, or present in the pores of the crucible), were exposed to dry oxygen for 3-hour periods at 720°C. The results of these experiments are shown in Table 4. The difference between the

TABLE 4

Magnesium Weight Losses of Samples Held in Dry Oxygen
at 720°C for 3 Hours

Pouring flux added or used previously in the crucible.
Heating and cooling were in argon.

Wt Mg (g)	Wt flux (g)	O ₂ flow rate (litre/min)	Mg loss (wt %)
1.771	1.378	0.16	1.1 ₃
1.596	None added	0.16	0.5 ₆

Mean Mg loss:- 0.8₅ wt %.

mean weight losses observed after exposure to air (Table 1) and after exposure to oxygen (Table 4) was not significant at the 10% level.

(e) Effect of Moisture

Experiments were conducted in which magnesium samples were held for 3 hours at 720°C, with moisture added either to the pouring flux or to the air atmosphere. Since the scatter in the results was large, the effect of the moisture was not found to be significant. Further experiments were accordingly conducted under conditions in which the scatter would be reduced.

The results of this further investigation of the effect of moisture are reported in Table 5. Samples of magnesium protected by the pouring flux, with and without added water, were heated to 720°C and cooled. Both dry air and wet air atmospheres were used. It was found that both the addition of moisture to the flux and the addition of moisture to the atmosphere increased the magnesium

TABLE 5

Results of Experiments to Show the Effect of Moisture
on Magnesium Loss

Samples were heated to 720°C and cooled without holding at 720°C.
Pouring flux, with and without added water, was used. The
atmosphere was either dry air or air saturated with water
at 25°C, and was passed at 0.17 litre/min.

Wt Mg (g)	Wt flux (g)	Wt water added to flux (g)	% water added to flux	Atmosphere	Mg loss (wt %)
1.474	1.24	None added	--	Dry air	0.3 ₄
2.130	1.31	None added	--	Dry air	0.0 ₉
1.889	1.21	None added	--	Dry air	0.0
1.737	1.13	0.14	12	Dry air	0.4 ₆
2.015	1.21	0.20	16	Dry air	0.7 ₉
1.710	1.32	0.24	18	Dry air	0.5 ₈
1.673	0.99	None added	--	Wet air	0.4 ₈
1.660	1.53	None added	--	Wet air	0.5 ₀
1.745	1.39	None added	--	Wet air	0.3 ₆
1.493	1.54	0.25	16	Wet air	1.3 ₀
1.481	1.52	0.23	15	Wet air	0.8 ₉
1.467	1.25	0.18	15	Wet air	1.5 ₉

Mean Mg loss: Flux as received, dry air - 0.1₄ wt %
Wet flux, dry air - 0.6₁ wt %
Flux as received, wet air - 0.4₅ wt %
Wet flux, wet air - 1.2₆ wt %

loss. At the levels investigated, the effects of moisture in the air and in the flux appear to be additive.

(f) Use of a Charging Flux

A charging-type flux (either added or present in the pores of the crucible) was used to protect magnesium samples exposed to oxygen for 3 hours at 720°C. The results are reported in Table 6.

TABLE 6

Magnesium Weight Losses of Samples Held under Charging Flux
for 3 Hours in Oxygen Atmosphere
(Flow Rate - 0.16 litre/min) at 720 °C
Flux was added, or had been used previously in the crucible.
Heating and cooling were in argon.

Wt Mg (g)	Wt flux (g)	Mg loss (wt %)
1.247	1.91	1.0 ₄
1.810	None added	0.0 ₆

Mean Mg loss:- 0.5₅ wt %.

The difference between the mean weight loss of samples protected by the charging flux and that of samples protected by the pouring flux (see Table 1) was not significant at the 10% level.

(g) Magnesium Losses in Air and in Argon

Magnesium weight losses from samples exposed to air or to argon atmospheres for periods of 1.5, 3.0 or 4.5 hours are reported in Table 7. The two results for 3 hour exposure to air were chosen at random from Table 1. In all these experiments the samples were protected by the pouring flux. An analysis of variance

TABLE 7

Magnesium Weight Losses of Samples Exposed to Air and to
Argon for Various Periods of Time

The samples were held at 720°C under pouring flux, either added or used previously in the crucible. Heating and cooling were in argon.

Wt Mg (g)	Wt flux (g)	Atmosphere	Time of exposure to atmosphere (hr)	Flow rate of atmosphere (litre/min)	Mg loss (wt %)
2.122	2.05	Air	1.5	Not determined ^{**}	0.4 ₇
1.350	1.25	Air	1.5	0.17	0.3 ₃
* 1.987	2.68	Air	3.0	0.17	1.3 ₁
* 1.709	1.61	Air	3.0	0.11	1.0 ₅
1.608	2.33	Air	4.5	Not determined	1.6 ₈
1.602	1.56	Air	4.5	0.17	2.0 ₆
1.721	None added	Argon	1.5	0.17	2.5 ₆
1.721	2.20	Argon	1.5	Not determined	3.9 ₅
1.700	1.55	Argon	3.0	Not determined	1.0 ₀
1.867	None added	Argon	3.0	0.17	6.5 ₄
1.939	1.87	Argon	4.5	Not determined	10.3 ₇
1.631	1.30	Argon	4.5	0.17	4.7 ₆

* From Table 1.

** Early experiments with uncontrolled flow rate. The data from the factorial experiment recorded in Table 1(a) and discussed statistically in the Appendix, Section (1), showed that the effect of flow rate was not significant at the 10% level.

of the results obtained indicates that the effect of exposure time was significant at the 10% level, but not at the 5% level, and that the effect of the change in atmosphere was significant at the 5% level.

An increase in magnesium loss with increased time of exposure might be expected. The increase in magnesium loss when argon was substituted for air, however, is somewhat surprising. This effect may be attributed to the formation, when the magnesium is heated in air, of an oxide crust over or within the flux, which retards the diffusion of the magnesium through the flux. Some support for this conjecture may be found in the observation that a magnesium sample exposed for 0.5 hour to air, and then exposed for 3 hours to argon lost only 0.8% by weight. X-ray diffraction of the crust, recovered after cooling, indicated the presence of MgO and KCl.

(h) Ignition Point Determinations

Approximate ignition points for magnesium samples, contained in MgO crucibles under an oxygen atmosphere, are reported in Table 8. The presence of flux increases the ignition point under the conditions used, from a temperature slightly below the melting point of magnesium metal (650°C) to a temperature apparently slightly above the normal boiling point of magnesium (1107°C).

TABLE 8

Ignition Points of Magnesium in Oxygen, With and Without Flux Cover

Flux	Ignition point (°C)
None	644 632 631 644 Mean $\frac{638}{}$
Pouring flux - added to Mg sample - used previously in crucible	1141 $\frac{1138}{}$ Mean $\frac{1140}{}$
Charging flux - added to Mg sample - used previously in crucible	1124 $\frac{1111}{}$ Mean $\frac{1118}{}$

DISCUSSION

Some difficulty is introduced into the interpretation of the results because of their relatively large scatter. The attempt to attribute this scatter to small variations in the gas flow rate, in the weight of magnesium and in the weight of flux used, was not successful. Part of the scatter can be attributed to uncertainties in the magnesium determinations, but other factors, which have not been recognized in this study, are probably also involved in most of the experiments.

It is quite clear that an increase in the oxygen partial pressure by a factor of five, from 0.2 atmosphere (air) to 1 atmosphere (pure O_2), does not produce a significant increase in the loss of magnesium. Further, the losses are significantly increased when argon is substituted for air. These results, together with the observations on the sample exposed to air prior to exposure to argon, support the hypothesis that an oxide layer is formed in, or adjacent to, the flux layer. This oxide probably serves to inhibit the diffusion of magnesium through the flux. Once the oxide layer has formed, therefore, the loss of magnesium from the sample will be retarded. However, if the layer is not formed, magnesium will be able to diffuse through the flux and be lost to the inert atmosphere by volatilization.

The presence of moisture, added as moist air or as moist flux, appeared to increase the magnesium losses slightly. The increase, however, was not found to be significant for samples held in air for 3 hours. When the scatter in the observed weight losses was reduced by omission of the holding period, and by heating and cooling in air, a significant increase in the weight loss was observed when moisture was added either to the flux or to the air atmosphere or to both. The deleterious effects of about 15% moisture in the flux and of air with a dew point of 25°C have been demonstrated. Under some circumstances, however, these effects may be obscured by other factors.

The residual flux left in the porous wall of the crucible was capable of providing protection for the magnesium sample without the need for additional flux. It appears, therefore, that the flux flowed and wetted the magnesium surface. The area over which such a flow could extend was not investigated in this study.

The substitution of a charging type or refining type of flux for a pouring or protective type produced no significant change in magnesium loss. Any distinction which does exist between two such fluxes may become apparent only when the flux has to flow over quite large distances to give full protection to the metal. For the small size of sample used in this investigation, such a distinction would not be apparent.

The magnesium losses appear to increase with time both in air and in argon, but the experimental results are not precise enough to draw any conclusions with respect to rates.

The ignition point found for unprotected magnesium in oxygen, $638 \pm 6^\circ\text{C}$, is somewhat higher than the result reported by Fassell et al. (3), namely 623°C . This may be attributed to differences in the procedures used. It is apparent that both the pouring flux and the charging flux will prevent ignition up to a temperature close to the boiling point of the metal.

It is important to note, in consideration of a study of this type, that any quantitative extrapolation of the results to larger scale industrial processes cannot be fully justified. For example, the surface-to-volume ratios of the metal may be quite different. In the experiments described in this report agitation of the metal was avoided, and any interaction with the container was minimized by the use of magnesium oxide crucibles. The results, however, can probably be applied qualitatively to larger-scale operations if due care is observed. It is probable that the use of a non-oxidizing atmosphere, in conjunction with a flux, would not reduce magnesium losses, but would, instead, increase such losses as a result of diffusion of the magnesium through the flux layer, followed by volatilization of the metal into the inert atmosphere. Any difference observed between the use of the pouring flux and of the charging flux for the protection of magnesium would not be a result of a difference

in inherent protection against oxidation, but might perhaps be a result of a difference in ability to spread on the molten metal surface. The presence of moisture in the atmosphere or in the flux appears to be deleterious, as had been suspected from the behaviour of the flux under plant conditions.

ACKNOWLEDGEMENTS

Certain of the magnesium analyses were performed by Mr. L.G. Ripley, Analytical Chemistry Subdivision, Mineral Sciences Division, who also undertook a further investigation of the reliability of his method of metallic magnesium analysis in connection with the present study.

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(An Appendix follows,
on pages 20 to 25.)

APPENDIX

STATISTICAL TREATMENT OF RESULTS

(1) Analysis of factorial experiment reported in Table 1(a).

Variables -

A - Mg weight:- lower level 1.73 \pm 0.04 g
 upper level 2.04 \pm 0.05 g
 B - Flux weight:- lower level 1.62 \pm 0.06 g
 upper level 2.56 \pm 0.12 g
 C - Air flow rate:- lower level 0.17 \pm 0.01 litre/min
 upper level 0.22 \pm 0.01 litre/min

Source of estimate	Sum of squares	df	Mean square	F ratio
Effect of A	0.1985	1	0.1985	0.452
Effect of B	0.1458	1	0.1458	0.332
Effect of C	0.1012	1	0.1012	0.230
Residual	1.7573	4	0.4393	
Total	2.2028	7		

$$F_{0.10}(1, 4) = 4.54$$

. . . None of the effects of A, B, or C is significant at the 10% level*.

* When it is concluded that a difference or effect is significant at the 10% level, there is, on the basis of the experimental data, 1 chance in 10 (or less than 1 chance in 10) that an error will be made by rejecting the hypothesis that there is no difference or effect. When a difference or an effect is stated not to be significant at the 10% level, the probability of making an error by rejecting the hypothesis that there is no effect or difference would be greater than 1 in 10. The corresponding probability for the 5% level is, of course, 1 in 20.

(2) Comparison of means from Table 1 (Mg held in dry air at 720°C for 3 hours) and from Table 2 (Mg heated and cooled in argon without holding at 720°C).

		<u>Table 1</u>	<u>Table 2</u>
Mean % wt loss	\bar{x}	1.09	0.54
Sample variance	S^2	0.641	0.0111
Number of observations	n	13	3

Hypothesis:- Means of populations from which samples in Table 1 and in Table 2 were drawn are equal.

$$\text{Statistic } t = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{S_1^2/n_1 + S_2^2/n_2}} \text{ is distributed as } t$$

with degrees of freedom ν ,

$$\text{where } \frac{1}{\nu} = \frac{1}{(n_1 - 1)} \left(\frac{S_1^2/n_1}{S_1^2/n_1 + S_2^2/n_2} \right)^2 + \frac{1}{(n_2 - 1)} \left(\frac{S_2^2/n_2}{S_1^2/n_1 + S_2^2/n_2} \right)^2,$$

$$\text{whence } \nu = 13.4$$

and $|t| = 2.39$, which is greater than $t_{0.10}(13.4) = 1.77$

. . Hypothesis is rejected;

i.e., there is a significant difference between the mean Table 1 and the mean from Table 2 at the 10% level.

* C.A. Bennett and N.L. Franklin, "Statistical Analysis in Chemistry and the Chemical Industry", published by John Wiley and Sons Inc., New York (1954).

- (3) Comparison of means from Table 1 (Mg held in air, pouring flux added)
and value in Table 3 (Mg held in air, flux used previously in crucible).

From Table 1:- Mean % wt loss $\bar{x} = 1.09$ $S^2 = 0.641$ $n = 13$
 From Table 3:- wt loss 0.63

Assume equal variances for populations from which samples reported in Table 1 and in Table 3 were drawn.

Hypothesis:- Means of populations from which samples in Table 1 and in Table 3 were drawn are equal.

$$t = \frac{\bar{x}_1 - \bar{x}_3}{S \sqrt{\frac{1}{n_1} + \frac{1}{n_3}}} \text{ is distributed as } t, \text{ with } n_1 + n_3 - 2 = 12 \text{ degrees of freedom,}$$

whence $|t| = 0.55$, which is less than $t_{0.10}(12) = 1.78$.

. . . No reason to reject hypothesis at 10% level.

Also, 90% confidence limits for $\bar{d} = \bar{x}_1 - \bar{x}_3$ are 0.46 ± 1.5

- (4) Comparison of means from Table 1 (Mg held in air at 720°C)
and from Table 4 (Mg held in O₂ at 720°C).

		<u>Table 1</u>	<u>Table 4</u>
Mean % wt loss	\bar{x}_2	1.09	0.84
Sample variance	S^2	0.641	0.162
Number of observations	n	13	2

Hypothesis:- Means of populations from which samples in Table 1 and in Table 4 were drawn are equal.

As in (2) above, $\nu = 2.5$

and $|t| = 0.69$, which is less than $t_{0.10}(2.5) = 2.5$

. . . No reason to reject hypothesis at 10% level.

Also, 90% confidence limits for $\bar{d} = \bar{x}_1 - \bar{x}_4$ are 0.25 ± 0.9

(5) Analysis of variance on results reported in Table 5.

Note: Bartlett's test (Bennett and Franklin, p. 197) indicates that there is no reason to reject (at the 10% level) the hypothesis that the variance of the 4 groups of data in Table 5 are homogeneous.

Group Totals

Moisture in flux	Moisture in atmosphere		Totals
	No	Yes	
No	0.43	1.34	1.77
Yes	1.83	3.87	5.61
Totals	2.26	5.12	7.38

Source of estimate	Sum of squares	df	Mean square	F ratio
Row Means (effect of moisture in flux)	1.2288	1	1.2288	26.1
Column Means (effect of moisture in atmosphere)	0.6816	1	0.6816	14.5
Interaction	0.0902	1	0.0902	1.91
Subtotal	2.0006	3		
Within groups	0.3767	8	0.0471	
Total	2.3773	11		

$$F_{0.10}(1, 8) = 3.46$$

$$F_{0.05}(1, 8) = 5.32$$

$$F_{0.01}(1, 8) = 11.3$$

. . Effect of moisture in flux is significant at 1% level.

Also, effect of moisture in the atmosphere is significant at 1% level.

(6) Comparison of means from Table 1 (Mg held under pouring flux) and from Table 6 (Mg held under charging flux).

		<u>Table 1</u>	<u>Table 6</u>
Mean % wt loss	\bar{x}	1.09	0.55
Sample variance	S^2	0.641	0.480
Number of observations	n	13	2

As in (2) above, $\bar{x} = 1.45$

and $t = 1.0$, which is less than $t_{0.10}(1.45) = 3.8$

. . . No significant difference at 10% level.

(7) Analysis of variance on results presented in Table 7.

Note: Bartlett's test indicates that there is reason to reject (at the 10% level) the hypothesis that the variances of the 6 groups of data in Table 7 are homogeneous. After a logarithmic transformation the variances were more nearly equal, and Bartlett's test was satisfied at the 10% level. The analysis of variance was therefore done on the transformed data.

Group Totals

Time of exposure (hr)	Atmosphere		Totals
	Air log % wt loss	Argon log % wt loss	
1.5	-0.8094	1.0048	0.1954
3.0	0.1385	0.8156	0.9541
4.5	0.5392	1.6934	2.2326
Totals	-0.1317	3.5138	3.3821

Source of estimate	Sum of squares	df	Mean square	F ratio
Between rows (effect of time)	0.53003	2	0.2650	3.72
Between columns (effect of atmosphere)	1.10747	1	1.1075	15.53
Interaction	0.16302	2	0.0815	1.14
Subtotal	1.80052	5		
Within groups	0.42788	6	0.0713	
Total	2.22840	11		

$$F_{0.10}(2, 6) = 3.46$$

$$F_{0.10}(1, 6) = 3.78$$

$$F_{0.05}(2, 6) = 5.14$$

$$F_{0.05}(1, 6) = 5.99$$

. : Effect of time is significant at the 10% level.

Also, effect of atmosphere is significant at the 5% level.

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AHW:NFHB/DV

