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**A Round Robin Evaluation of the
Corrosiveness of Wet Residential
Insulation by Electrochemical
Measurements**

E. E. Stansbury

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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A ROUND ROBIN EVALUATION OF
THE CORROSIVENESS OF WET RESIDENTIAL INSULATION
BY ELECTROCHEMICAL MEASUREMENTS

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Part of
The National Program
for
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FOREWORD

This is one of a series of reports to be published describing research, development, and demonstration activities in support of the National Program for Building Thermal Envelope Systems and Materials. The National Program involves several federal agencies and many other organizations in the public and private sectors who are addressing the national objective of decreasing energy wastes in the heating and cooling of buildings. Results described in this report are part of the National Program through delegation of management responsibilities for the DOE lead role to the Oak Ridge National Laboratory.

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RESIDENTIAL BUILDING THERMAL INSULATION BY
ELECTROCHEMICAL MEASUREMENTS

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ABSTRACT

The results of a round robin evaluation of the use of an electrochemical method of calculating the corrosion rate of low carbon steel in environments related to cellulosic building insulations are reported. Results are compared for six of eight participants involved in the test program. Environments included the leachate from a wet cellulosic insulation and solutions based on pure and commercial grades of borax, ammonium sulfate and aluminum sulfate. The pH values of these environments were in the range of 2.5 to 9.5. Electrochemical measurements were made using a direct reading corrosion rate instrument. The calculated corrosion rates were compared with those determined directly by weight loss measurements. Electrochemical measurements were made over a period of 48 hours and weight loss exposures were for two weeks.

Poor agreement was observed for the corrosion rates determined electrochemically and the values were consistently larger than those based on weight loss. Reasons proposed for these results included the complex nature of the corrosion product deposits and the control these deposits have on oxygen diffusion to the metal interface. Both factors influence the validity of the calculation of the corrosion rate by the direct reading instrument. It was concluded that development of a viable electrochemical method of general applicability to the evaluation of the corrosiveness of wet residential building thermal insulations was doubtful.

Because of the controlling influence of dissolved oxygen on the corrosion rate in the insulation leachate, an alternate evaluation method is proposed in which a thin steel specimen is partially immersed in wet insulation for three weeks. The corrosiveness of the wet insulation is evaluated in terms of the severity of attack near the metal-air-wet insulation interface. With thin metal specimens, complete penetration along the interface is proposed as a pass/fail criterion. An environment of sterile cotton wet with distilled water is proposed as a comparative standard.

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BACKGROUND

ASTM Standard C665 (1) on Mineral-Fiber Blanket Thermal Insulation, C739 (2) on Cellulosic Loose-Fill Thermal Insulation and C764 (3) on Mineral-Fiber Loose-Fill Thermal Insulation contain procedures for evaluating the wet corrosiveness of these products on contact with low carbon steel, aluminum and copper. These procedures have been evaluated critically with respect to method and reproducibility of results (4,5). A general limitation is that the procedures differ sufficiently to question their ability to provide information on the relative corrosiveness of different types of insulation. These problems led to the formation of an ASTM C16.31.001 Task Group on the Corrosiveness of Residential Building Thermal Insulations. In response to the discussions of this Task Group, Weil and co-workers (6,7) initiated an investigation to assess use of electrochemical methods for evaluating the corrosiveness of wet insulations. The method had the potential of providing information in a short time, hours rather than days, as required by the ASTM methods, and would be of general applicability to several types of insulation.

Weil (7) made potentiodynamic polarization measurements in wet insulation and wet insulation leachates over the potential range from the open-circuit corrosion potential to +2000 mV (SHE)¹. Satisfactory estimates of the corrosion rates at the open-circuit potential could not be made by an analysis of the polarization curves. As an alternate measure of corrosiveness, it was proposed to determine the limiting or maximum current density attained on the potentiodynamic upscan. His curves tended to increase continuously, with no pronounced tendency for passivation, and became almost vertical at the higher potential. Since Weil observed differences in limiting current densities for low carbon steel in different wet insulation environments, he proposed that determination of this limiting value might be a measure of the corrosiveness of the wet insulation. To further investigate the feasibility of

¹SHE Standard Hydrogen Electrode potential scale

the method, a round robin test program to involve six participants was established by Weil in cooperation with the ASTM C16.31.001 Task Group. The test program was initiated in 1987 with the intent to circulate an inexpensive potentiostat and selected test insulations to the participants who would evaluate the corrosiveness based on the relative characteristics of the polarization curves. It was recognized that the results would be qualitative but the objective was to examine the extent of agreement as to the relative corrosiveness of the insulations. Because of problems in procedure and maintenance of the potentiostat, the round robin program was not completed.

During 1989-1990 Stansbury, in cooperation with ORNL, re-examined the possibility of using electrochemical methods for evaluation of the corrosiveness of wet insulations and conducted extensive measurements on low carbon steel exposed to both wet insulations and filtered insulation leachates. Results of this investigation were presented in two reports (8,9). It was shown that potentiodynamic measurements can give useful information on the general corrosion behavior, including very approximate corrosion rates and tendencies for localized corrosion, particularly pitting. It was concluded however that development of a standard method for evaluating corrosiveness based on potentiodynamic measurements was not feasible. Reasons for this conclusion included: (1) the procedures based on use of a conventional potentiostat, with the associated electrochemical cell, are too involved; (2) calculations for conversion of the electrochemical measurements to corrosion rates are too uncertain because of the complex availability of dissolved air and variable corrosion product formation in the insulation environments; and, (3) personnel knowledgeable in interpreting electrochemical measurements in terms of corrosion behavior would frequently be unavailable.

In view of the above limitations, but recognizing the value of an electrochemical method, a program was initiated to investigate the use of a direct reading corrosion rate meter to evaluate the corrosiveness of wet insulations. The instrument selected was the Corrater 9030 (Rohrback Cosasco Systems, Santa Fe Springs, CA) which provides a direct digital display of a calculated corrosion rate based on electrochemical measurements processed by a

micro-processor within the instrument. The Corratrater was circulated during the period December 1989 - December 1990 to eight round robin participants along with a set of materials from which environments were to be prepared for immersion of low carbon steel specimens. Direct evaluations of corrosion rates for comparison with those calculated from the electrochemical measurements were to be determined by weight-loss measurements.

The following report is an expansion of a progress report covering the round robin evaluation program presented to the ASTM 16.31.001 corrosion task group for their consideration for further action. This meeting was held in New Orleans in October, 1990. Six of eight initial participants to whom the equipment was circulated reported their results. Some of these did not conduct the weight-loss tests or make measurements of the pH of the environments.

OBJECTIVE OF THE ROUND ROBIN TEST EVALUATION PROGRAM:

The round robin program was conducted over a ten month period starting December 1, 1989, with eight participants. The program was initiated with the following objectives:

1. Determine if electrochemical measurements using a direct reading corrosion rate meter (Corratrater 9030, manufactured by Rohrbach Cosasco Systems) might be used to evaluate the corrosiveness of residential building thermal insulation.
2. Conduct electrochemical measurements on low carbon steel exposed to environments based on or related to those produced by wet cellulosic insulation.
3. Conduct direct determinations of the corrosion rate by weight loss measurements for comparison to the rates determined electrochemically.

4. Use the round robin evaluation to develop a consensus as to the viability of the method and to establish the reproducibility of the corrosion rates determined by both electrochemical and weight loss measurements.
5. Determine the extent to which corrosion rates calculated by the electrochemical method were direct measures of the corrosion rate or could be acceptably correlated with rates determined by weight loss.
6. Conduct the measurements in a filtered leachate from a commercial cellulosic insulation and in environments based on selected additives that may be used in the production of cellulosic insulations.

SUMMARY OF TEST PROCEDURES.

The direct reading corrosion rate meter was circulated to the participants along with low carbon steel specimens and materials for preparing the test environments. The latter included:

A cellulosic insulation from which a leachate is prepared.

Pure and commercial grades of borax from which saturated and 5% saturated solutions are prepared.

Pure and commercial grades of ammonium sulfate from which saturated and 5% saturated solutions are prepared.

Pure and commercial grades of aluminum sulfate from which saturated and 5% saturated solutions are prepared.

Participants prepared their own corrosion test environments. The cellulosic insulation leachate was prepared by contacting 100 grams of insulation with 1000 mL of distilled water, holding for three days, and filtering. The three chemicals represented additives that industry has used to produce cellulosic

insulations to meet performance standards, particularly resistance to smolder combustion and radiant heat flux. Participants were requested to report the pH of each environment.

The Corratrater utilizes a probe to which a pair of identical low carbon steel specimens (0.54 cm in diameter and 4.06 cm long) is attached. The electrochemical instrument is programmed to apply +20 mV and then -20 mV between the probes and to calculate the average resulting current. The data are passed to an internal micro-processor and a calculated corrosion rate in mpy (mils per year) is displayed digitally by the instrument. An output voltage proportional to the corrosion rate is available for continuously recording the rate. Values were reported by the participants following 1, 24, and 48 hours exposure to show any time-dependence of the indicated corrosion rate. Corrosion rates based on weight loss measurements of steel specimens, 5.08 cm x 5.08 cm x 0.053 cm, totally immersed for two weeks were also requested. Corrosion rates were to be reported in mils per year (mpy).

RESULTS OF THE ROUND ROBIN EVALUATION

The direct results of the measurements by participants in the round robin evaluation are reproduced in an Appendix to this report. Corrosion rates calculated from electrochemical measurements using the Corratrater are reported following 1, 24 and 48 hours immersion. The values listed under "IMBALANCE" are obtained from the instrument as a measure of the extent to which the two low carbon steel specimens attached to the test probe are corroding identically. For a valid corrosion rate measurement, this reading should be less than the displayed corrosion rate with a value of zero indicating best performance. Also recorded are values of the pH of the test environments and the corrosion rate by weight loss in the insulation leachate and in the 5% saturated solutions.

The 13 environments to which plain carbon steel specimens were exposed for electrochemical Corratrater and for weight loss

measurements to determine corrosion rates are listed in Table 1. The Ph of these environments as reported by four of the participants are also listed. While the values are reasonably consistent for most cases, large differences occur for some of the environments. The values for the cellulose insulation leachate are in the range of near neutral to very slightly acid. The borax solutions are consistently more alkaline, having Ph values generally above 9. The ammonium sulfate hydrolyzes to give solutions which are more acid. Excluding the three reports of Ph > 7, the average Ph of the ammonium sulfate solutions is 5.2. Aluminum sulfate hydrolyzes more strongly resulting in solutions in the approximate Ph range of 2.5-3.5.

The results of the corrosion rate measurements using the Corratrater and by direct weight loss are given in Table 2. There is significant scatter in the values reported by the several participants for the same environment. Following are summary statements directed to each of the environment types. Included for each case, are comments based on extensive investigations at the University of Tennessee by E. E. Stansbury (8). It is emphasized that comparisons are being made of rates determined directly by weight loss and rates calculated from electrochemical measurements which are sensitive to both the measurement technique and the procedures for conversion of data to corrosion rates.

Corrosion rates in cellulosic insulation leachates.

DATA: Reference to Table 2 shows that corrosion rates determined by the Corratrater extend from the one low value of 0.7 mpy to the range of 3.0-9.0 mpy. Of the five weight loss rates reported, two are <0.5 mpy, one at 0.99 mpy and two at 1.6 mpy.

Comments: Participants reported rates indicated by the Corratrater following 1, 24, and 48 hours exposure. In general there was a

TABLE 1

ASTM 16.31 ROUND ROBIN CORROSIVENESS TEST PROGRAM

ENVIRONMENTS AND REPORTED VALUES OF pH*

Environment	pH			
Cellulose insulation leachate	7.41	7.0	6.05	5.8
Borax				
Saturated CP**	9.48	10.0	9.0	9.4
5% saturated CP	9.08	8.5	9.96	9.0
Saturated comm.***	9.38	10.0	9.18	9.4
5% saturated comm.	9.1	8.5	10.02	8.9
Ammonium sulfate				
Saturated CP	4.77	8.0	6.91	4.8
5% saturated CP	4.86	6.5	4.75	4.9
Saturated comm.	4.11	8.5	7.8	5.0
5% saturated comm.	5.11	6.5	4.97	4.9
Aluminum sulfate				
Saturated CP	2.76	1.0	1.64	1.1
5% saturated CP	3.35	3.0	3.22	3.2
Saturated comm.	2.57	3.0	3.08	2.4
5% saturated comm.	3.47	3.5	3.68	3.6

* Ph values were reported by four of eight participants.

** CP = chemically pure grade

*** comm. = commercially pure grade

TABLE 2

ASTM 16.31 ROUND ROBIN CORROSIVENESS TEST PROGRAM

Corrosion Rates Determined by Electrochemical Corraters
and Weight Loss Measurements

All values for low carbon steel at 75±5 F
 Rates indicated by Corraters at 48 hours
 Rates by weight loss based on 14 day exposure
 Results from 6 participants

Environment	Corrosion rate in mils per year					
Cellulose Ins Leachate						
Corrater	9.0	7.5	0.7	3.0	7.0	6.0
Weight loss	0.46	0.99		0.45	1.6	1.6
Sat CP borax						
Corrater	0.0	0.0	0.0	<0.1	0.0	0.0
5% sat CP borax						
Corrater	0.0	0.0		0.05	0.0	0.0
Weight loss	0.01	0.13		0.03	0.36	nil
Sat Comm Borax						
Corrater	0.0	0.10	0.0	0.06	0.0	0.0
5% sat Comm Borax						
Corrater	0.0	0.0		0.07	0.0	0.0
Weight loss	0.03	0.10		<0.01	0.43	nil
Sat CP Am sulfate						
Corrater	42.	38.	14.	7.	17.	42.
5% sat CP Am sulfate						
Corrater	23.	19.		7.	16.	17.
Weight loss	4.67	6.1		3.3	5.23	7.1
Sat Comm Am sulfate						
Corrater	107.	94.	103.	78.	67.	86.
5% sat Comm Am sulfate						
Corrater	40.	33.		20.	28.	37.
Weight loss	6.56	8.1		5.8	7.05	7.0

Table 2 (continued)

ASTM 16.31 ROUND ROBIN CORROSIVENESS TEST PROGRAM

Corrosion Rates Determined by Electrochemical Corrater
and Weight Loss Measurements

All values for low carbon steel at 75±5 F
 Rates indicated by Corrater at 48 hours
 Rates by weight loss based on 14 day exposure
 Results from 6 participants

Environment	Corrosion rate in mils per year					
Sat CP Al sulfate Corrater	9.	22.	7.	83.	10.	9.1
5% sat CP Al sulfate Corrater	41.	81.		70.	69.	111.
Weight loss	18.1	20.4		28.	26.4	28.0
Sat Comm Al sulfate Corrater	18.2		63.	48.	58.	95.
5% sat Comm Al sulfate Corrater	41.	175.		59.	64.	75.
Weight loss	9.15	16.6		13.6	10.7	13.5

decrease in rate with increase in time. For example, the specimen showing a rate of 0.7 mpy at 48 hours was reported to have a rate of 7 mpy at 24 hours. Changes of this order have been observed on longer times (72+ hours) at the University of Tennessee. Also careful weight loss measurements following 2 rather than 14 days exposure showed a higher short-time corrosion rate. These observations confirm that the formation of corrosion product deposits on the steel surface significantly and progressively reduce the corrosion rate. This is an inherent aspect of the corrosion process and is probably the major contributor to both the scatter in values within a test procedure and between the two test methods.

Although not requested in the round robin program, corrosion rates determined in Knoxville water under comparable conditions were in the range of 3-4 mpy by Corratel and about 2 mpy by weight loss (8). In view of the observations that the longer time rates in the particular cellulosic insulation leachate used in the round robin test are <1 mpy, chemical species used as additives in this cellulosic insulation may have a mild inhibiting effect to the corrosion of low carbon steel.

Corrosion rates in borax solutions.

Data: Reference to Table 2 shows, with two exceptions, that the corrosion rates in either saturated or 5% saturated solutions of both CP and commercial grades of borax were recorded as 0.0 or <.13 mpy.

Comments: Borax solutions, partially because of $pH > 9$ and also the presence of the tetraborate ion, have a strong tendency to produce highly protective passive oxide films on steel. Saturated solutions of both the CP and commercial grades maintain bright surfaces on low carbon steels for long times. The border line concentration for maintaining this passive condition appears to

be near 5% saturation. Of triplicate specimens immersed in the 5% saturated solutions for two weeks, two specimens remained bright with nil weight loss while the third developed a flocculent red corrosion product (8). The variable corrosion rates, including the large values of 0.36 and 0.43 mpy reported in Table 2, demonstrate the uncertainty in forming a passive film and, if formed, the transition from passive to local active corrosion that can accompany film breakdown.

Corrosion rates in ammonium sulfate solutions.

Data: The scatter in Table 2 is large among the corrosion rates (7 to 42 mpy) indicated by the Corrater for the saturated and 5% saturated CP ammonium sulfate solutions. The scatter is less but the indicated rates (20 to 107 mpy) are much higher in the saturated and 5% saturated commercial ammonium sulfate.

In both the CP and commercial grade 5% saturated environments, the corrosion rates determined by weight loss (3.3 to 8.1 mpy) show less scatter among participants and are significantly lower than the rates indicated by the Corrater. The rates are slightly lower in the 5% saturated CP ammonium sulfate (average, 5.28 mpy) as compared to the rate in 5% saturated commercial grade ammonium sulfate (average, 6.9 mpy).

Comments: The scatter in the rates indicated by the Corrater is partially related to the time dependence of the rate. For example, the indicated rate of 7 mpy (at 48 hours) for the 5% saturated CP grade was recorded as 20 mpy at one hour and 16 mpy at 24 hours. Another participant reported 31, 15 and 14 mpy at 1, 24 and 48 hours for the saturated CP ammonium sulfate. These values confirm a variable time dependence of the indicated Corrater values to which the scatter in the values is attributed.

Again the difference in corrosion rates indicated by the Corrater

and determined from weight loss is related to the difference in time of exposure (48 hours versus 14 days) and to difference in method of measurement.

The cause for the higher corrosion rates indicated by both the Corratel and weight loss evaluations for solutions based on the commercial versus CP grades of ammonium sulfate is uncertain. Impurities in the commercial grade are probably responsible but these have not been identified. Again independent of the round robin program, measurements were made at the University of Tennessee on solutions based on another source of commercial ammonium sulfate (8). In this second source the corrosion rates of the low carbon steel were lower than in the initial source, particularly when determined by weight loss.

Corrosion rates in aluminum sulfate solutions.

Data: Very large differences (7 to 83 mpy) are indicated in Table 2 for the corrosion rate based on Corratel measurements in saturated CP aluminum sulfate. Generally larger (41 to 111 mpy) but more consistent values are reported for the 5% saturated CP solution. The rates in the saturated and 5% saturated solution of commercial grade aluminum sulfate are more consistent and, within the scatter of values, do not differ significantly from those of the 5% saturated CP grade environment.

The corrosion rates based on weight loss are reasonably consistent. The average rate for the 5% saturated CP aluminum sulfate is 21.4 mpy; for the 5% saturated commercial grade, the average rate is 12.7 mpy.

Comments: The aluminum sulfate hydrolyzes to quite acid environments ($\text{pH} = 2.5-3.5$). At these pH values, significant corrosion is due to the acidity resulting in observable formation of hydrogen gas bubbles. Particularly in the saturated solutions,

the corrosion product is black, probably a hydrated magnetite, and a white flocculent precipitate tends to form on the specimen and in the solution. This precipitate is presumed to be aluminum hydroxide resulting from the increase in the Ph of the solution with time of exposure. Both the black and white corrosion products tend to adhere irregularly to the specimen which is a factor contributing to variable corrosion rate.

DISCUSSION.

The results reported by six of the eight round robin participants are summarized as follows:

1. The 13 environments exhibited Ph values extending from approximately 2.5 to 9.
2. The values of the corrosion rate of a given environment determined by weight loss are in reasonable agreement among the participants. The averaged rates are:

Cellulosic insulation leachate	1.0 mpy
5% saturated CP borax	0.10
5% saturated commercial borax	0.11
5% saturated CP ammonium sulfate	5.28
5% saturated commercial ammonium sulfate	6.90
5% saturated CP aluminum sulfate	24.2
5% saturated commercial aluminum sulfate	12.7
3. Corrosion rates calculated by the Corrater are consistently higher than those determined directly by weight loss. The values of the ratio, (Corrater rate) / (weight loss rate), ranged from 2.1 to 6.7 with a mean value of 4.2. (One very large ratio was excluded; also excluded were the ratios for the borax solutions because of the very small corrosion rates). The corrosion rates calculated by the Corrater are plotted

versus the weight loss rates in Figure 1. The least squares relationship is:

$$R(\text{Corrater}) = 4 + 4.2 R(\text{weight loss}) \quad \text{Eq. 1}$$

4. Corrosion rates decreased with time due to progressive

Figure 1. Relationship between direct measurement of corrosion rates by weight loss and rates indicated by the Corrater electrochemical measurement. Rates in mils per year (mpy). Data from five participants in seven environments.

build up of corrosion product deposits.

Except for the borax environments which form passive films and result in extremely low corrosion rates, the major variables controlling the corrosion are the formation of corrosion product deposits and the accompanying diffusion through these deposits of

species supporting the corrosion. The major cause of the corrosion is dissolved oxygen at $\text{pH} > 4$; as the pH decreases below 4, acidity becomes increasingly important such that at pH values in the range 2-3 and smaller, corrosion due to dissolved oxygen becomes negligible compared to that due to hydrogen ions. In the cellulosic insulation leachate and the ammonium sulfate environments, the major cause of corrosion is dissolved oxygen. In the aluminum sulfate environments, corrosion is largely due to the low pH resulting from the hydrolysis of the aluminum sulfate. Since insulation leachates have $\text{pH} = 4-9$, dissolved oxygen is the major cause of their corrosiveness. Also, in this pH range, solid corrosion products consisting of hydrated iron oxides form on the surface. The structure (adherence and porosity) of these deposits govern the diffusion of oxygen to the metal surface and hence control the corrosion rate. There are several significant consequences of these influences:

1. Short-time (hours to days) corrosion rates can vary because of statistical variations in the early stages of formation and growth of the corrosion product deposits. As a consequence, the results of the Corrater measurements (determined at 1, 24 and 48 hours) will be difficult to reproduce and will be higher than determined by weight loss measurements over two weeks. Also, as the films form, the rate of corrosion decreases as has been confirmed by rates determined by weight loss over two days compared to two weeks.
2. The higher corrosion rates indicated by the Corrater measurements are also related to the validity of the model of the corrosion process programmed into the instrument to convert the electrochemical measurement to a corrosion rate. The model assumes that diffusion of reactants (here oxygen) to or metal ions from the corroding interface are not controlling the corrosion

process. If these are controlling, the Corratel indicates higher corrosion rates than actually exist. The instrument does not provide for correcting for the effects of diffusion. Due to the complex and highly variable diffusion processes, it is difficult to establish quantitatively reliable methods for converting the electrochemical measurements to corrosion rates. This is particularly true when intercomparison of corrosion rates in significantly different environments is being made.

3. Variability of short-time corrosion rates can also be attributed to procedure. Surface condition is obviously significant with respect to both residual interfering films and deposits and to the physical condition resulting from fabrication or subsequent abrasion. Another variable, however, is the effect of oxide films formed on carefully cleaned surface when in contact with air. These air-formed films can influence the early stages of initiation and growth of corrosion deposits. In some cases, the time (minutes to hours) of contact of a clean and dry surface with air prior to immersion into the test environment can be a significant variable in short-time measurements. The non-uniform initiation of corrosion is frequently observed during early exposure of the low carbon steel Corratel and weight loss specimens to the environments used in the round robin investigation.

CONCLUSIONS.

Development of a viable short-time electrochemical method for quantitatively determining the corrosiveness of residential building thermal insulation is doubtful.

Reasons for this conclusion include:

- a. The leachates derived from insulations result in complex and variable corrosion product deposits which make the corrosion rate time-dependent, generally requiring days to perhaps weeks to reach a steady value. The primary cause of the corrosion in wet insulations with pH above 4 is dissolved oxygen and the corrosion rate is dependent on the diffusion of oxygen to the metal interface.
- b. The presence of corrosion product deposits makes it unreasonably complicated to model the corrosion process to establish procedures to convert electrochemical data to corrosion rates with adequate quantitative accuracy.
- c. It is doubtful that a quantitatively acceptable correlation can be developed to allow prediction of the longer time corrosion rates with rates indicated by short time electrochemical measurements.
- d. The Corratrater may provide a method for rapid qualitative evaluation of the upper limit of corrosiveness of wet insulations and find application in quality control.

RECOMMENDATIONS.

Research conducted at the University of Tennessee during 1989-1990 and, in particular, overlapping the period of time during which the ASTM round robin evaluation was conducted established the significance of dissolved oxygen in controlling the corrosion rate of low carbon steel in wet insulation (8). It is evident that maximum corrosion rates will occur near a metal-air-corrosive environment interface. This has been realized for many years and suggests that partial immersion of metal specimens

would provide conditions most conducive to corrosion and offer the potential of providing a simple and direct means of evaluating the relative corrosiveness of insulations.

Metal specimens of the size specified in ASTM C739, 50.8 mm x 50.8 mm x 0.076 mm, were placed vertically in 50 mm deep x 100 mm diameter glass containers and insulation placed around the specimens to a height of 38 mm. Distilled water was added and maintained such that the top of the insulation was continuously wet. Specimens were removed after 14 days and carefully cleaned. Preferential corrosion occurred at and near the air interface. Specimens were examined for any evidence of complete penetration and, if not penetrated, ball-tipped micrometers were used to determine the maximum localized corrosion.

Table 3 shows some of the results from the partial immersion procedure. The pH values of the several environments are listed for reference. Two specimens were penetrated. One of these was in the environment of pH = 4.44 and the other at a pH = 9.24. The appearance of the attack along the interface in the lower pH environment indicated that the acidity was contributing to the interface oxygen concentration sufficiently to result in penetration. The pH of 9.24 is high enough to induce passivity, particularly in the presence of dissolved oxygen near the interface, and the general appearance of this specimen with a thin corrosion product film tends to confirm this. It is well established, however, that in metal-environment systems that create borderline passivity, pitting associated with localized attack can lead to rapid penetration. This is a result of the dominant passive area supporting the cathodic reaction (here dissolved oxygen) concentrating its effect on the small anodic areas where the passive film has broken down.

Other data listed in Table 3 show that the corrosion rates measured in the vicinity of the air interface are much larger

Table 3

CORROSION RATES OF LOW CARBON STEEL PARTIALLY
IMMERSED IN WET INSULATIONS*

Insulation	pH	Rates in mpy Low carbon steel	
		Rate based on maximum local penetration at air interface (24 days)	Rate based on total immersion in leachate (14 days)
Source 1 cellulosic	7.42	6.8	0.45
Source 4 cellulosic	6.48	6.1	1.62
Source 11 cellulosic	4.44	>22.8(1 hole)	.n/d
Source 13 cellulosic	8.08	8.7	.n/d
Source 1 mineral fiber	9.10	7.0	2.0
Source 2 mineral fiber	9.62	9.8	1.1
Source 3 mineral fiber	9.24	>22.8(2 holes)	1.4
Sterile cotton	5.22(a)	9.1	1.8

*Specimens were 50 mm x 50 mm x 0.076 mm immersed in the wet insulation for depth of 38 mm. Containers were 50 mm deep and 100 mm in diameter. Distilled water added to maintain liquid just visible at top of insulation.

(a) pH of liquid at end of exposure.

than the rates based on total immersion in insulation leachate.

The results of these partial immersion studies suggest that a procedure in which a metal specimen is mounted vertically with partial immersion in wet insulation may be a straight-forward, simple and inexpensive procedure to evaluate the corrosiveness of insulations. A tentative configuration for the test is shown in Figure 2. The test specimen (0.076 mm thick) and the pass/fail criterion is similar to that used in ASTM C739. Following exposure, the cleaned specimen is examined for penetration along the metal-wet insulation-air interface. Comparison is made to the response under the same conditions using sterile cotton wet with distilled water. The following test procedure is proposed:

1. Test time: Three weeks at $75 \pm 5^\circ$ F.
2. Test specimen: Low carbon steel, 50 mm x 50 mm x 0.076 mm.
3. Test container: glass crystallizing dish, 50 mm deep x 100 mm diameter and a perforated plastic top with a centered slot
55 mm long and 6 mm wide.
4. Specimen preparation: Carefully clean specimen and handle with protective gloves.
5. Preparation and placement of insulation specimens in glass container:
 - Cellulosic: Hold the sample vertical and place insulation around the metal specimen. Place the perforated top in position to expose 12.5 mm metal above the insulation.
 - Mineral fiber: Form 10 cm diameter cylinder of insulation. Carefully cut the cylinder in half and place around metal specimen. Place the perforated plate in position to expose 12.5 mm of metal above the insulation.
 - Foam insulation: Cut one or more 100 mm diameter cylinders of insulation to provide a total thickness of 38 mm.

Figure 2. Arrangement of metal specimen with partial immersion in wet insulation as a test procedure for the corrosiveness of wet residential building thermal insulation.

Cut the cylinder in half and place the metal specimen between the cut faces. Place the perforated plate in position to hold the insulation with 12.5 mm of metal exposed above the insulation.

6. Addition of water: Add distilled water until visible through the perforated plate. Maintain this level during the test period.
7. Cover: Invert a larger crystallizing dish over the test assembly but with provision for free access of air.
8. Specimen evaluation: Clean specimens using inhibited hydrochloric acid. Examine for perforations. Use ball-faced micrometers to measure minimum thickness.

An informal round robin evaluation of the above procedure involving 3-4 participants is under consideration. Each participant would evaluate three insulations plus sterile cotton using triplicate sets of specimens. If the results are favorable, they would be presented to the ASTM 16.31.001 Corrosion Task Group for further consideration of the procedure as a standard for the evaluation of the corrosiveness of residential building thermal insulating materials.

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APPENDIX

DATA SHEET
ASTM 16.31 ROUND ROBIN TEST PROGRAM
Corrosiveness of Residential Building Insulation

ENVIRONMENT	CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH = 6.05	TIME, hrs	RATE, mpy	IMBALANCE	
	1	12	0.7	
	24	10	0.2	0.466
	48	9	1---	in leachate
COMMENTS: Red rust on electrode after 4 hours. After 48 hours, black/red residue on probes; black residue wipe off. With bubbled air, rate 22 mpy.				

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = 9.0 pH (5% sat) = 9.96	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%	
	1	0.0	0.1	0.0	0.0
	24	0.0	0.0	0.0	0.0
	48	0.0	0.0	0.0	0.0
COMMENTS: Electrodes remained clean. No change weith air bubbling.					

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax pH (sat) = 9.18 pH (5% sat) = 10.02	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%	
	1	0.0	0.1	0.0	0.0
	24	0.0	0.0	0.0	0.0
	48	0.0	0.0	0.0	0.0
COMMENTS: Electrodes remained clean. No change with air bubbled.					

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Ammonium Sulfate	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%		6.563 (in 5% sat)
pH (sat) = 7.80 pH (5% sat) = 4.97	1	104	80	1---	1---	
	24	97	51	1---	0.7	
	48	107	40	1---	0.1	
COMMENTS: Probes had mat black finish, Wipes off with tissue. Probes slightly pitted or etched.						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Aluminum Sulfate	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%		9.152 (in 5% sat)
pH (sat) = 3.08 pH (5% sat) = 3.68	1	154	73	1---	1---	
	24	51	45	0.7	1---	
	48	18	41	0.0	1---	
COMMENTS: Probes have mat black appearance; very little comes off on tissue.						

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Corrosiveness of Residential Building Insulation

ENVIRONMENT	CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH = 7.2	TIME, hrs	RATE, mpy	IMBALANCE	
	1	8.2	1	
	24	6.7	2	.99
	48	7.5	0.7	in leachate
COMMENTS:				

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = 9.03 pH (5% sat) = 9.3	TIME, hrs	RATE, mpy sat 5%	IMBALANCE sat 5%		
	1	0 0	0 0		
	24	0 0	0 0		0.13
	48	0 0	0 0		(in 5% sat)
COMMENTS:					

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax pH (sat) = 9.3 pH (5% sat) = 8.96	TIME, hrs	RATE, mpy sat 5%	IMBALANCE sat 5%		
	1	0 0	0 0		
	24	0.1 0	0 0		0.096
	48	0 0	0 0		(in 5% sat)
COMMENTS:					

ENVIRONMENT		CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Ammonium Sulfate		TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%	
pH (sat) = 4.87		1	100	45	3	8.1 (in 5% sat)
pH (5% sat) = 4.91		24	94	43	0	
		48	94	33	0	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Aluminum Sulfate pH (sat) = 2.4 pH (5% sat) = 3.19	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	50	80	25	49	
	24	90	147	35	69	
48	92	175	41	81	16.6 (in 5% sat)	
COMMENTS:						

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ENVIRONMENT		CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH =	TIME, hrs	RATE, mpy	IMBALANCE		
	1	11	2		
	24	7	2		
	48	0.7	1		
					in leachate
COMMENTS:					

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = pH (5% sat) =	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	0.0		0		
	24	0.0		0		
48	0.0		0		(in 5% sat)	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%		
pH (sat) =	1	0.1		0		
pH (5% sat) =	24	0.0		0		
	48	0.0		0		
						(in 5% sat)
COMMENTS:						

ENVIRONMENT		CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Ammonium Sulfate pH (sat) = pH (5% sat) =	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	152		12		
	24	113		11		
	48	103		3		
					(in 5% sat)	
COMMENTS:						

ENVIRONMENT		CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Aluminum Sulfate pH (sat) = pH (5% sat) =	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	126		1		
	24	68		14		
	48	63		9		
						(in 5% sat)
COMMENTS:						

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ENVIRONMENT	CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH = 7.41	TIME, hrs	RATE, mpy	IMBALANCE	
	1	4.9	0.4	
	24	5.0	0.2	0.45
	48	3.0	0.2	in leachate
COMMENTS:				

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = 9.48 pH (5% sat) = 9.08	TIME, hrs	RATE, mpy sat	5%	IMBALANCE sat	5%
	1	< 0.1	0.1	0	0
	24	< 0.1	0.06	0	0
	48	< 0.1	0.05	0	0
					0.03 (in 5% sat)
COMMENTS:					

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax pH (sat) = 9.38 pH (5% sat) = 9.10	TIME, hrs	RATE, mpy sat	5%	IMBALANCE sat	5%
	1	0.2	0.2	0.1	0.1
	24	0.08	0.08	0	0
	48	0.06	0.07	0	0
					< 0.01 (1.5) (in 5% sat)
COMMENTS: Weight loss: One sample with less than 0.01 mpy					
One sample showed rust with 1.5 mpy					

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
	TIME, hrs	RATE, mpy		IMBALANCE		
Pure Grade Ammonium Sulfate		sat	5%	sat	5%	
pH (sat) = 4.77 pH (5% sat) = 4.86	1	22	20	4	2.5	3.3 (in 5% sat)
	24	13	16	2	1.0	
	48	7	7	0	0.7	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
Commercial Grade Ammonium Sulfate						
pH (sat) = 4.11	1	133	35	4	2	5.8 (in 5% sat)
pH (5% sat) = 5.11	24	80	28	3	3	
	48	78	20	2	1	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Aluminum Sulfate	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%		28 (in 5% sat)
pH (sat) = 2.76 pH (5% sat) = 3.35	1	63	115	7	7.7	
	24	83	70	7	4.1	
	48	83	70	2	6.7	
COMMENTS: Weight loss: One solution turned milky at 10 days 14 mpy One solution turned milky in two days 42 mpy						

ENVIRONMENT		CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Aluminum Sulfate pH (sat) = 2.57 pH (5% sat) = 3.47	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	81	102	10	18	
	24	60	56	16		
48	48	59	12	16		
						13.6 (in 5% sat)
COMMENTS:						

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Corrosiveness of Residential Building Insulation

ENVIRONMENT	CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH = 5.8	TIME, hrs	RATE, mpy	IMBALANCE	
	1	9	1.0	
	24	8	0.0	1.61
	48	7	1.0	in leachate
COMMENTS:				

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = 9.4 pH (5% sat) = 9.0	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%	
	1	0.0	0.1	0.1	0.1
	24	0.0	0.0	0.0	0.0
	48	0.0	0.0	0.0	0.0
COMMENTS:					.36 (in 5% sat)

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax pH (sat) = 9.4 pH (5% sat) = 8.9	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%	
	1	0.0	0.1	0.0	0.0
	24	0.0	0.0	0.0	0.0
	48	0.0	0.0	0.0	0.0
COMMENTS:					0.425 (in 5% sat)

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Ammonium Sulfate pH (sat) = 4.8 pH (5% sat) = 4.9	TIME, hrs	RATE, mpy		IMBALANCE		
		sat	5%	sat	5%	
	1	16	15	3	2	
	24	15	17	6	4	
48	17	16	4	1	5.23 (in 5% sat)	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS
Commercial Grade Ammonium Sulfate pH (sat) = 5.0 pH (5% sat) = 4.9	TIME, hrs	RATE, mpy		IMBALANCE		7.05 (in 5% sat)
		sat	5%	sat	5%	
	1	51	40	6	8.0	
	24	60	38	13	1.0	
	48	67	28	13	5.0	
COMMENTS:						

ENVIRONMENT	CORRATER RESULTS					CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Aluminum Sulfate	TIME, hrs	RATE, mpy sat 5%		IMBALANCE sat 5%		10.7 (in 5% sat)
pH (sat) = 2.4 pH (5% sat) = 3.6	1	33	99	6	6	
	24	51	71	6	2	
	48	58	64	6	4	
COMMENTS:						

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ENVIRONMENT	CORRATER RESULTS			CORROSION RATE FROM WGT. LOSS mpy
Cellulosic Insulation Leachate pH = 7.0	TIME, hrs	RATE, mpy	IMBALANCE	
	1	6	4	
	24	6	2	1.6
	48	6	2	in leachate
COMMENTS: Soln. yellow; specimens somewhat dark(24 hrs), Pickled appearance. (very-fine)-black deposit wipes off easily from wet specimens				

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Pure Grade Borax (sodium tetraborate) pH (sat) = 10.0 pH (5% sat) = 8.5	TIME, hrs	RATE, mpy sat	5%	IMBALANCE sat	5%
	1	0	0.1	0	0.1
	24	0	0	0.1	0.1
	48	0	0	0.1	0.1
COMMENTS: No evidence of attack					

ENVIRONMENT	CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
Commercial Grade Borax pH (sat) = 10.00 pH (5% sat) = 8.5	TIME, hrs	RATE, mpy sat	5%	IMBALANCE sat	5%
	1	0	0.1	0	0.1
	24	0	0	0.1	1
	48	0	0	0.1	0
COMMENTS:					

ENVIRONMENT		CORRATER RESULTS				CORROSION RATE FROM WGT. LOSS mpy
		TIME, hrs	RATE, mpy sat 5%	IMBALANCE sat 5%		
Commercial Grade Ammonium Sulfate pH (sat) = 8.5 pH (5% sat) = 6.5		1	82	24	5	7.0 (in 5% sat)
		24	84	38	5	
		48	86	37	3	
COMMENTS: Sat: Soln nearly opaque (brownish black). @24 hrs spec still shiny Soln has flocculated - still has scum on top. Pickled appearance, no pit 5%: @24 hrs soln is faint orange. Rust flecks over spec, and falling. Spec shiny 48 hrs spec looks dark; many rust flecks falling under spec. No pits.						

COMMENTS: Bubbles covering specs (65 hrs in sat; gone @ 24 hrs in 5%). Spec turned black (brown in 5%) sat. - severe pitting and a ring at top; 5% very fine pitting at top 0.5 mm.

COMMENTS: Bubbles covering spec, gone by 24 hrs. Sat - pickled appearance (black) and ring at top: 5% - smooth (rusty brn) shallow fine pitting on top $\frac{1}{2}$ mm and end.

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119. Smith, G. A., Factory Mutual Research Corp., Norwood, MA
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135. Stern, E., Quebec, Canada
136. Strzepek, B., Dow Chemical USA, Granville, OH
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138. Sullivan, R., National Center for Appropriate Technology, Butte, MT
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140. Tewes, S., Small Homes Council - Building Research Council, Champaign, IL
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151. Williams, D., Dow Chemical USA, Freeport, TX
152. Wilson, K. E., Fiberglas Canada, Inc., Ontario, Canada
153. Wojtechko, B., UC Industries, Tallmadge, OH

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