EVALUATION OF CORROSION IN COPPER WATER TUBING

Thesis for the Degree of M. S.
MICHIGAN STATE UNIVERSITY

Arthur L. Plumley

1957

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IN

COPPER WATER TUBING

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ARTHUR L. PLUMLEY

A THESIS

Submitted to the College of Science and Arts
Michigan State University of Agriculture and
Applied Science in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

Department of Chemistry

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Approved Laurence L. Quill

ABSTRACT

Increased use of copper and its alloys during the past quarter century, particularly in domestic and commercial water distribution systems, has placed new emphasis on the problems of copper corrosion. A serious corrosion problem has existed in some of the copper water tubing installations in buildings at Michigan State University. A major study has been undertaken by the Copper and Brass Research Association (CABRA) and the University for the purpose of determining the causes of the copper corrosion.

An experimental water distribution system of copper tubing, in which effects of increasing temperature and velocity could be observed, has been in service. Periodic examinations have been made to observe the progress of corrosion. After nearly two years of service, a part of the system was dismantled and the copper tubing from it sectioned for various observations.

Visual examination of the tubing specimens thus obtained, indicated that a) the deposits on the interior of the tubing varied from a soft, green, powdery layer at 50°F, to a thin, dark, brown adherent film at 200°F, with only scattered islands of deposit at 110° to 140°F; and b) the corrosion was of an impingement type.

The corrosion of this copper tubing was evaluated by determining the wall thickness after corrosion radiographically, gravimetrically and with dial and special pointed micrometers. Agreement between the methods is excellent and probably within the experimental error.

Graphs were made from the results of each measurement method,

showing loss in wall thickness versus temperature and loss in wall thickness versus velocity. From these graphs, it may be concluded that a) after approaching a minimum value at 80°F, loss in wall thickness reaches a maximum prior to the 200°F range. The exact location of this maximum is dependent on the velocity of the water which flowed in the tubing. For the 3/8, 1/2 and 3/4 inch samples this maximum is near 170°F; for the 1 and 1 1/4 inch samples the maximum is nearer to 140°F, and b) loss in wall thickness increases with increasing velocity. However, it is only above 110°F that the velocity increase tends to influence the extent of corrosion. This temperature is also close to the decomposition temperature range for several of the compounds assumed to be formed as initial corrosion products.

From comparison of the results of the three direct measurement methods with the weight loss method, it may be noted that the corrosion in the 3/8 and 1/2 inch samples from 140° through 200°F is probably of an extensive local type. These samples contain a number of very deep pits rather than extensive overall corrosion.

An advantage of the radiographic-microphotometric procedure is that it provides a permanent record of the extent of corrosion, as well as an overall picture of the corrosion.

X-ray powder patterns of the deposits formed on the interior of the various tube specimens indicate the presence of silica, some copper and iron oxides, and some miscellaneous copper compounds.

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INTRODUCTION

During the past quarter century, copper and its alloys, largely because of their inertness, have found increasing use as tubing for both commercial and domestic water distribution systems. In this same period there occurred an upsurge in the chemical treatment of water, particularly for the purpose of reducing hardness. Concurrently, in practice, more frequent instances of corrosion and corrosion failures have been recorded. As a consequence, there has been considerable interest in the determination of the basic causes of such corrosion and in the reduction of their frequency.

The overall study of corrosion in a water distribution system, of which this thesis is but a small part, is the first one in which the progress of the corrosion on the inner surfaces of copper water tubing has been observed continuously under extensive and controlled service conditions. Included in this thesis are: 1) a definition of corrosion and explanations of the various types of corrosive attack; 2) a discussion of some methods of water treatment and their effects on corrosiveness of water; 3) a brief discussion of the corrosion problem at Michigan State University; 4) a report on several methods of evaluating the extent of corrosion; 5) a discussion of x-ray diffraction methods for determination of corrosion product composition; and 6) general comments and conclusions.

Corrosion

Chemical or electrochemical deterioration of metals is known as corrosion, in contrast to erosion, which implies destruction by mechanical wearing. In practice, association of the two may result in acceleration of the deterioration and will be discussed further. A brief resume of the types of corrosion has been compiled from numerous sources.[1]

Direct Chemical Corrosion

Any chemical reaction in which a pure metal may take part with the production of a compound of the metal, would result in corrosion if allowed to proceed on the metal surface. The action of acid on a metal, or the formation of metal oxides or salts due to action of the atmosphere, are typical examples.

Electrochemical Corrosion

Electrochemical corrosion, a deterioration of metals in an electrolyte, involves dissolution of metal at so called anodic areas and deposition of hydrogen from solution at cathodic areas. The finite separation of portions of a metal surface resulting in the anode and cathode areas may be caused by slight differences in metal structure, imperfections in the initial oxide film, hot spots, etc. Electrochemical corrosion will not continue, however, unless hydrogen is evolved or removed by oxidation.

As the greater number of instances of corrosion of metals occur in contact with an electrolyte, a more detailed discussion of the various forms of electrochemical corrosion will be given.

Galvanic-Two Metal Electrochemical Corrosion -- This rapid type of corrosion results when a metal is electrically connected with a less reactive (more electronegative) metal in a single corrosive solution. Corrosion of this type may occur at the juncture of two different metal water pipes, on steel rivets in copper sheeting or the brass bearing on the propeller of a steel ship.

Concentration Cell Corrosion -- A concentration or electrolytic corrosion cell, of which the galvanic cell is one form, may consist of:

1. Two different metals in the same electrolyte. (galvanic cell)
2. Two metals in different electrolytes. (Daniels cell)
3. The same metal in two different concentrations of the same electrolyte. This is the common form of electrolytic corrosion.
4. The same metal in two different electrolytes.

Pitting or Localized Corrosion -- Pitting is the pock-marked form of corrosion of a metal surface due to incomplete protection by a corrosion product or other coating, or to small inhomogeneities in the metal. Corrosion tends to occur more rapidly in the unprotected areas than on the major surface and may be accelerated by the small inclusions. If the inclusions are more reactive than the metal, they will be corroded out, and if less reactive or cathodic, they may induce pitting in their vicinity. A simple concentration cell might also be set up in an inclusion or its vacancy.

Pitting in copper tubing is usually of deposit attack form in which the pits occur under porous deposits of corrosion products or foreign material. This type of pitting is due to the formation of an oxygen-concentration cell. In this concentration cell the area in contact with a solution low in oxygen becomes anodic and is actively corroded, while the area in contact with solution high in oxygen becomes cathodic and is thus protected. The area under a deposit, being shielded from contact with the solution containing oxygen, becomes anodic and is thus subject to corrosion. Pitting is frequently associated with stagnant conditions.

Dezincification Corrosion -- Selective leaching in an alloy is known as dezincification. It is most common in brasses from which zinc may be dissolved, leaving only a surface deposit of copper over a porous and weakened copper base. This type of corrosion is really a specific example of galvanic concentration cell corrosion and is most likely to occur in stagnant or slowly moving water which is relatively high in chloride and low in hardness so that no protective calcium carbonate scale is formed. There are installations of brass tubing now under test; however, this discussion is related primarily to examination of corrosion in copper tubing and dezincification warrants no further discussion.

Stress Corrosion and Corrosion Fatigue -- Corrosion may be accelerated by internal stresses or externally applied stresses. Not only may differential stress produce corrosion, but corrosion of any other type may produce a weakened part which may crack under a stress that would not otherwise have affected it. Separation of grains by some uneven distribution of stresses may lead to pitting. Corrosion fatigue is stress corrosion in which the stresses are fluctuating or cyclic in nature.

Erosion-corrosion or Impingement Corrosion — Erosion is usually regarded as a destructive process involving concurrent removal of material. Impingement or erosion-corrosion is usually characterized by rapid and repeated destruction of protective surface films in the presence of rapidly moving, turbulent liquids. Copper tubing suffers impingement attack in systems where the velocity of the water is unusually high and/or turbulent, and where no protective film is formed or the initially formed film has been loosened. Because the flow is subject to construction irregularities and turbulence, attack usually predominated in local areas or sections near the inlet end of the tubing, but may extend the entire length of an installation. Entrained air bubbles, breaking as they contact the surface of the tubing, act as accelerators of this type of corrosion which may be easily identified by its crescent-shaped, elongated pits, frequently undercut in the downstream direction. (Figure 1)

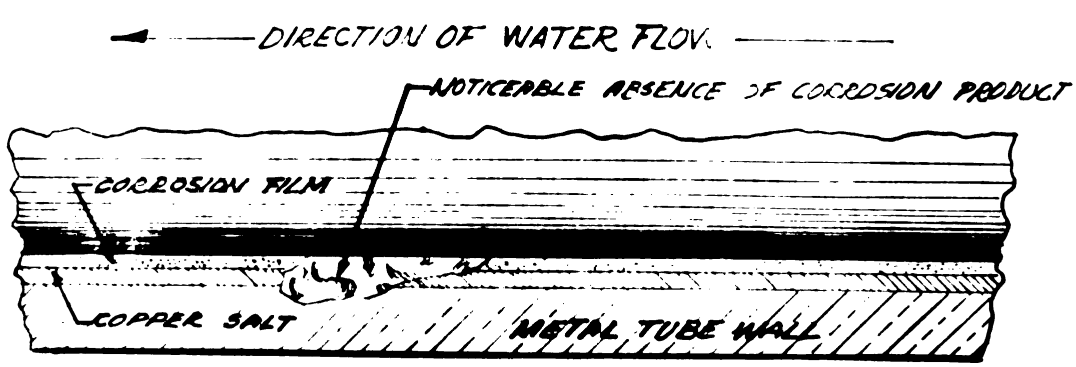


Figure 1. Impingement Type Corrosion

Copper Corrosion

Widespread use of copper and copper alloys is due largely to the excellent resistance to corrosion displayed by these materials in a wide range of environments. Copper itself is not a particularly reactive element and its corrodibility is low, as might be expected, even without a protective film. From the position of copper in the Electromotive Force Series, it is seen that the tendency for formation of hydrogen gas on a copper surface is so slight that the amount of reaction is insignificant. Although the breakdown of a protective film on copper is not apt to lead to rapid attack as with iron or aluminum, the continued resistance of copper to corrosion depends to a considerable extent on the continued maintenance of a protective film of oxide or some other insoluble deposit.

Generally, to be protective, the film must not only be insoluble, but also adherent and continuous. Thin films are more likely to possess these properties than heavy, bulky films which are subject to loosening by flowing water. It seems obvious that formation of soluble salts at the metal surface will result in continued corrosion. Likewise, coatings that are porous, loosely adherent, or easily broken, offer little protection and may result in formation of concentration cells, thus accelerating corrosion.

Many of the corrosion products formed on copper have low solubility and tend to form adherent, relatively impervious films which help make copper resistant to corrosion in many media.

Because of their corrosion resistance in fresh water systems, copper and its alloys find widespread use in cold and hot water systems for both domestic and industrial installation. Tubing thus installed has usually been found to suffer from only a few of the previously indicated forms of corrosion, largely pitting and impingement corrosion.

THE CORROSION PROBLEM AT MICHIGAN STATE UNIVERSITY[2]

Prior to 1951, copper water tubing in use in various buildings on the Michigan State University campus showed few corrosion failures, despite a service record of up to thirty years. In one women's dormitory, Gilchrist Hall, built in 1949, over forty corrosion failures occurred during the interval June 1951 to August 1952. Concern about these failures led to the initiation of a corrosion conference. Attendance at this conference included representatives from the University and from the country's major copper tubing producers, with whom there had been previous correspondence on the problem. As an outcome of this conference, the Copper and Brass Research Association (CABRA), a manufacturer's association, through its corrosion subcommittee and in co-operation with the University, decided to initiate a rather extensive corrosion testing program.

This initial planning comprised two phases of study. First, determination of the causes of the failures in the Michigan State University installations and subsequent reduction of the frequency of these failures and second, establishment under controlled conditions of the type and primary causes of such corrosion. The program has been intended as basic research in the corrosion of copper in active fresh water systems with a corollary study in prevention or retardation of such corrosion.

Specifically, this study has involved determining the effects of variations in temperature, in velocity and in types of waters circulated through a system of copper water tubing. Waters of varied composition were circulated through panels of copper tubing in such a manner that the effects of six different temperatures and five velocities could be observed.

The conditions for experimental work were not arbitrary but were established as a result of observations made over a long period of time. It had been noted that tubing replacements for corrosion failures occurred less frequently in those systems which had been in use for a long time. There were indications that in these older buildings, softeners were run beyond the point of exhaustion, flow rates were relatively low and temperatures moderate. Some protective film apparently built up under these operating conditions.

Newer installations appeared to have practically no protective film and were found on occasions to have high chloride concentrations in these circulating systems, indicating incomplete rinsing of the sodium zeolite softeners before regeneration. Temperatures were found to be in the range of 130° to 200°F. Under these conditions, there appears to be no opportunity for the formation of a protective coating.

Several other observations were made about the installations in which the more frequent failures have occurred. Corrosion of the tubing seems to be prevalent in locations where recirculation systems were in operation, loads were heavy, temperatures were above 150°F in softeners frequently regenerated to maintain zero hardness at all times. Visual examination of the tubes in which failures had occurred indicated pitting-impingement type of erosion. Among the steps which were taken in an effort to reduce the number and severity of failures occurring on campus were (1) lowering of temperatures to below 140°F, (2) blending of soft water and hard water to maintain a slightly scale-forming water, that is, to have a slightly positive Langelier Index for the system, (3) bypassing of recirculation pumps in the distribution system. Recirculation pumps had been installed in the hot water lines to insure prompt delivery of hot water to taps on upper floors of the building, (4) improving regeneration procedures for sodium zeolite softeners with particular attention being paid to the rinsing away of chlorides, (5) controlling the carbon dioxide content at the wells and reservoir, (6) attempting to watch the oxygen concentration in the system.

The above corrective measures have substantially reduced the number of failures on campus.

Water and Water Treatment

As indicated, the composition of the water flowing through the copper tubing is significantly related to the type and degree of corrosion involved. The usual municipal water supply is fresh water which may contain the following ions derived from compounds present in the soil: Na^{+}, K^{+}, NH_4^{+}, Ca^{++}, Mg^{++}, Fe^{+++}, Fe^{++}, Al^{+++}, H^{+}, Cl^{-}, NO_3^{-}, SO_4^{--}, OH^{-}, HCO_3^{-}, CO_3^{--}, PO_4^{---}. There may also be present dissolved gases, such as O_2, CO_2, SO_2, Cl_2, NH_3, along with organic matter of various sorts, as well as suspended material such as silica, clay, etc. This water is usually treated by chlorination, filtration, aeration, etc. to make it potable. In addition, water used for boiler feed, laundry and many domestic purposes may be treated to remove hardness.

The presence of any of the above mentioned ions, or of gases or treatment compounds in sufficient amounts, may alter the corrosiveness of the water toward the tubing through which it flows. Further consideration of the previously indicated corrosion processes shows that the copper ions going into solution at the anodic areas unite with the hydroxyl ions or other ions in solution to form copper hydroxide, oxides or salts. If these corrosion products are capable of forming an adherent, overall coating, they should protect the copper from corrosion, but the general protection depends on the solubility of the corrosion products in their environment.

The calcium, magnesium and iron salts are usually responsible for scale formation in water as the temperature increases, and this scale may also help to retard corrosion through action as a protective coating. These materials are the basis for so called hardness in water.

At present there are two general methods in use for reducing hardness. a) One method, the lime-soda process, involves the removal of the scale forming elements by precipitation. Lime may be added to a water containing calcium bicarbonate and magnesium salts with subsequent precipitation of calcium carbonate and magnesium hydroxide. Soda ash (Na_2CO_3) is frequently used as a supplement to precipitate the remaining calcium salts as calcium carbonate. b) A second method of water softening is by ion exchange. Water to be softened is passed through a bed of insoluble resin, whereupon the calcium and magnesium ions replace the sodium ions of the resin and an effluent water of zero or near zero hardness results. The resin is regenerated by flushing with a brine solution. Some ion exchange resins use hydrogen ions rather than sodium ions and softeners using these resins are regenerated with a mineral acid.

Natural water supplies are usually neutral or slightly acidic from dissolved carbon dioxide and contain sufficient dissolved oxygen to cause and maintain corrosion. Removal of the often objectionable scale and scum-forming calcium and magnesium salts may allow corrosion to proceed freely in the absence of the protective coating which would otherwise form. Additional treatment is sometimes necessary to alleviate this condition.

The Approach to the Problem

During the course of the overall investigation, a number of similar experiments were undertaken using various types of water ranging from zero to well water hardness. This discussion is on observations and evaluations made on copper tubing removed from the first of these series of controlled experiments.

The test panel consisted of six vertical tubing trains constructed from sections of tubing of decreasing diameter (1 1/4 to 3/8") and connected in series by means of 3/4" return lines. These sections were connected in order of decreasing diameters by means of soldered unions for ease in disassembling for inspection. (Figures 2 and 3) (Figure 2 is a photograph of the complete panel, Figure 3 is a diagram of one loop of the panel.) The panel was designed so that the water flowed in the direction of decreasing tube diameters.

The water flowing through each successive loop was heated to a temperature higher than in the previous loop. Specifications for the panel and its operation were determined after consideration of conditions under which copper tubing is used generally, as well as at Michigan State University. The operating temperatures for the various loops of the panel were room temperature, 80, 110, 140, 170 and 200°F. It is noted that the useful temperature range of water is covered. Thermally regulated heat exchangers were used to obtain the desired temperatures. (Figure 4)

The sections were approximately 60 diameters in length, this being the minimum length to allow free flow in the major portion of the tubing, thus minimizing the effects of turbulence at the unions. The water flow was set at 6 gallons per minute in order to obtain a velocity of 1.5 feet/second in the 1 1/4 inch section. This is the critical velocity for initiation of turbulence in the 1 1/4 inch tubing, there being streamline water flow below this velocity. Impingement corrosion should, therefore, be at a minimum in tubing of this diameter. Corresponding velocities in the remaining sections are a function of their respective diameters. The dimensions of the tubing sections are

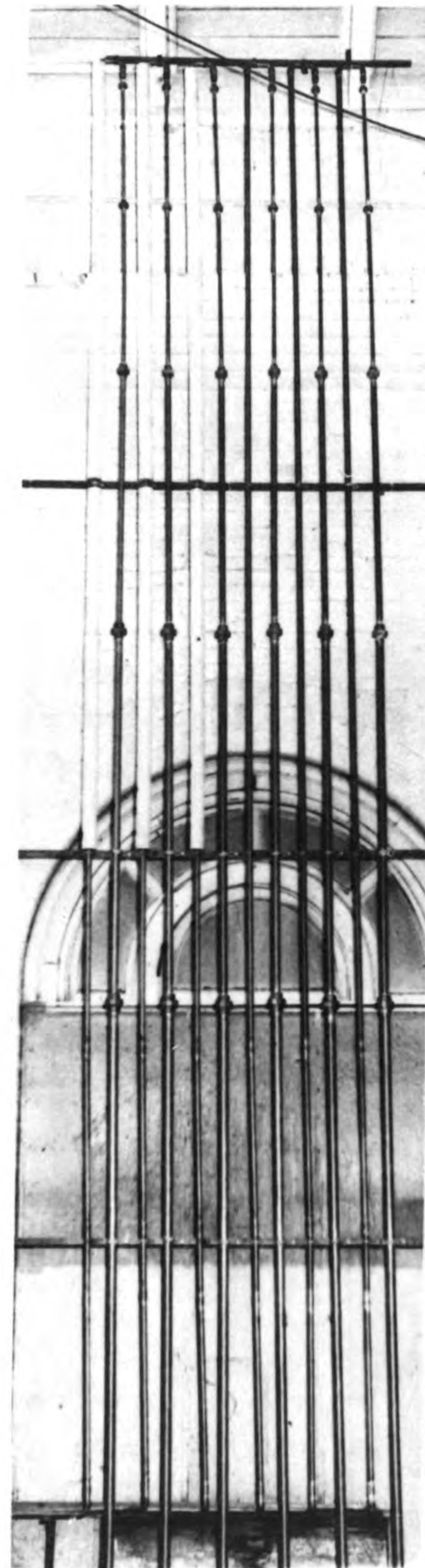
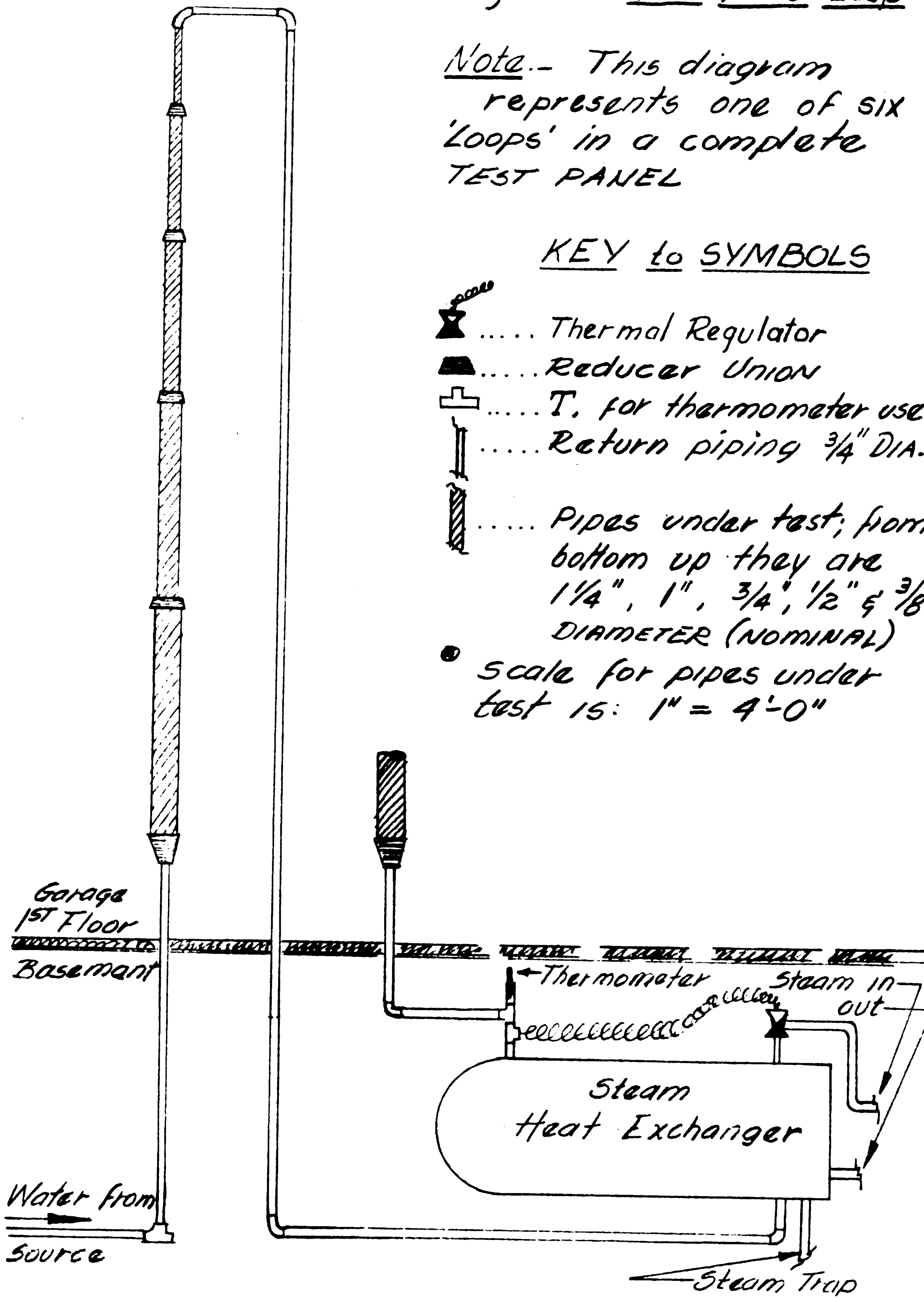


Figure 2. The Panel

Figure 3 Test Panel Loop



included in Table I.

The panel was in actual operation a total of 498 days. The only interruptions in operation occurred at about four month intervals, at which time the panel was completely disassembled for inspection by the committee during the quarterly corrosion conferences. These inspections were visual, primarily by means of a boroscope, in order not to disturb the deposit formed on the interior of the tubing.

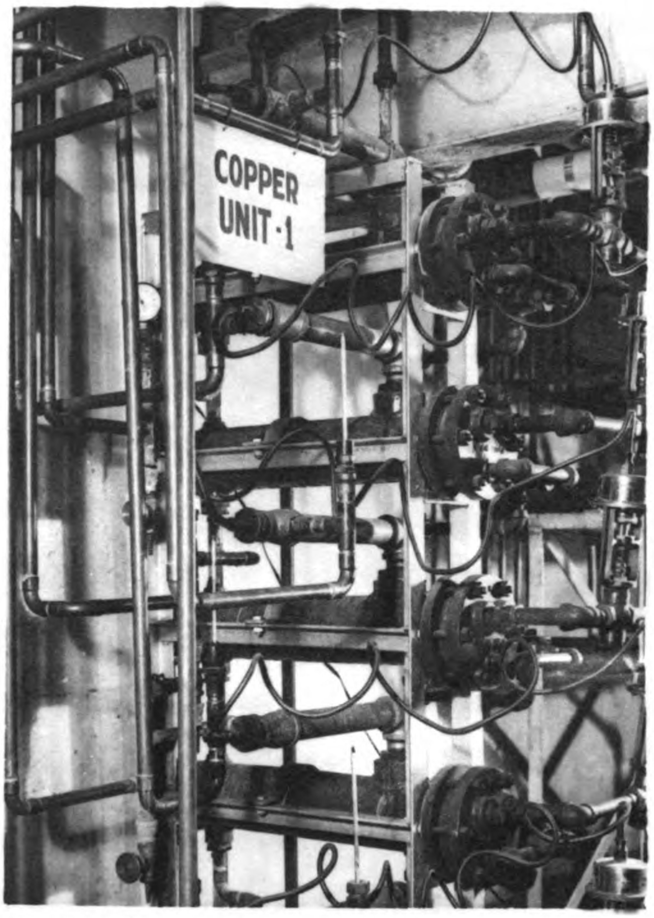


Figure 4. Test Panel Heat Exchangers

Table I

Panel Specifications

Diameter (inches)	1 1/4	1	3/4	1/2	3/8
Length (feet)	6	5	4	3	2
Velocity (ft/sec) at 6 gal/min	1.5	2.3	3.9	8.1	13.1

The panel was removed from operation permanently when it was decided that an additional four months service would probably result in failure somewhere in the system. (This decision was later justified, as the minimum remaining wall thickness at some places was approximately 0.010 inches.)

After the panel had been disassembled, each section was cut transversally in two. The inlet half was put aside for future observation. The outlet half was then cut in two longitudinally, one half being retained for observations, and the other half being shipped to the Copper and Brass Research Association for observation.

For this discussion, observations were on the outlet half-sections of tubing which were retained. From these half-sections, six inch lengths were cut, acid cleaned and used for some of the physical measurements recorded later.

The borescope observations included in this report are the final ones made by the CAHRA corrosion committee on the various lengths of the disassembled panel, prior to sectioning. (Table II)

Other data include microscopic observations on both uncleaned and acid cleaned sections (Table III), wall thickness measurements with micrometers (Tables IV and VIII) and radiographic studies (Tables IX and X). Extent of corrosion was also evaluated by determination of the loss of copper by gravimetric analysis. (Tables XI and XII)

BOROSCOPE OBSERVATIONS

The following are comments on the boroscope observations of copper tubing sections by CANNA numbers at the final inspection December 12, 1956.

MAIN PANEL - CENTER

Temp.	Tube Diameter	Comments
200°	1¼"	+Heavy very dark scale, some slight surface pitting.
	1"	+Inlet attack for about 3/8" then same as above.
	3/4"	+Same, inlet attack in about 1".
	1/2"	+Heavy brown coating, definite but slight erosion +pitting.
	3/8"	+Same, but worse.
170°	1¼"	+Slight velocity effect at entrance, roughened surface with thin brownish film.
	1"	+Same.
	3/4"	+Same, but rougher and some bright etched spots.
	1/2"	+Same, but worse.
	3/8"	+Very rough , with bright bottoms of pits.
140°	1¼"	+Deeply etched and roughened surface throughout tube with some bright spots.
	1"	+Same, but progressively more affect.
	3/4"	+Same.
	1/2"	+Same.
	3/8"	+Definite impingement pitting.
110°	1¼"	+Bright etched surface with islands of brown scale.
	1"	+Same, but more etching.
	3/4"	+Same, but with progressively greater etching.
	1/2"	+Same, with progressively greater etching.
	3/8"	+Deeply etched surface with islands of brown scale.
80°	1¼"	Uniform brown green film, no apparent corrosion.
	1"	Same.
	3/4"	Same.
	1/2"	Same.
	3/8"	Same.
50°	1¼"	Heavy brown scale, no apparent attack.
	1"	Same.
	3/4"	Same.
	1/2"	Spotty brown scale, no apparent attack.
	3/8"	+Bright etched copper surface with small islands of scale.

BINOCULAR MICROSCOPE OBSERVATIONS

The following observations were made with a 15 X binocular microscope.

50°	Observation Deposit	Direction Flow Indicated	Observations Etched Sample
3/8	Lt. Brown Large anodic areas Small cathodic	Yes	Gen. corrosion, stratified islands
1/2	Lt. Brown Large anodic areas Lt. film overall	Not clear	Gen. corrosion, islands
3/4	Lt. Brown Anodic and cathodic areas equal	No	Gen. corrosion, islands
1	Lt. Brown Uniform	No	Slight Gen. corrosion, small islands, minute pits
1¼	Lt. Brown mottled light and dark	No	Very slight Gen. corrosion, very small islands
80°			
3/8	Lt. Red Brown Single anodic line entire length	Not clear	Gen. pitting, islands on effluent end; gouging on effluent side of islands
1/2	Lt. Red Brown Uniform	Not clear	Gen. pitting - some streaked corrosion
3/4	Lt. Red Brown Numerous small streaked anodic areas	Yes	Gen. pitting - streaked
1	Lt. Red Brown some large long anodic areas, numerous small streaked anodic areas	Yes	Gen. corrosion, islands, pitting on islands

1¼	Lt. Brown Mottled light and dark	Not clear	Some Gen. corrosion, few small islands
110°			
1/8	Lt. Brown on islands Large anodic area-cathodic islands, slight streaked deposit in anodic area	Yes	Gen. corrosion, large islands, pitting on islands, gouging on effluent side of islands
1/2	Same-larger areas of light deposit in anodic area	Yes	Small islands, same as 1/8"
3/4	Same-less contrast between anodic	Yes	Gen. corrosion, stratified islands, gouging as above, wrinkling
1	Same-more islands, some wrinkles	Yes	Same as 3/4", choppy wrinkles, large rounded pits
1¼	Stratified deposits varying shades of brown, pits obvious	Not obvious	Gen. pitting, islands beneath level of pitting
140°			
1/8	Very small amounts of Lt. Red Brown	Yes	Gen. corrosion, extreme gouged effect in direction of flow
1/2	Red Brown, streaked light and dark, longitudinal anodic areas	Yes	Gen. corrosion, some deeply gouged places as in 1/8"
3/4	Same-less pronounced	Yes	Gen. corrosion, long shallow clean valleys, blotchy corroded areas under deposits
1	Red Brown, streaked light and dark, slight deposits in anodic areas	Not obvious	Gen. roughening, alternate clean and rough areas
1¼	Same, small anodic areas	Not obvious	Same as 1", but to a lesser degree

170°

3/8	Pits, Purplish Red deposit on cathodic peaks	Yes	Considerable Gen. pitting, gouged effect in direction of flow, rounded pits (clean)
1/2	Same, less pronounced	Yes	Same as 3/8", less pronounced
3/4	Lt. Reddish Brown on islands, similar to 140° 1"	Not clear	Gen. roughening, some smooth rounded areas
1	Same, slight deposit in anodic areas	Not clear	Gen. corrosion, some islands
1¼	Same, less pronounced	No	Gen. corrosion, numerous pin-point islands

200°

3/8	Lt. Reddish and Deep Brown on cathodic peaks, slight deposit in anodic pits	Yes	Gen. corrosion as in 170° 3/8" but not as severe
1/2	Lt. Reddish covered by deep brown - areas of rounded anodic pits	Yes	Gen. elongated pitting, not as severe as 3/8"
3/4	Deep Brown, some anodic areas	Yes	Gen. rounded pitting, large clean gouged areas
1	Deep Brown overall some areas smooth (anodic)	No	Gen. minute pitting
1¼	Same	Not clear	Same, pitting but less pronounced

From the visual, boroscopic and microscopic observations, a few generalizations about the corrosion and the corrosion products may be made. Except for the clean and probably anodic areas, the deposits are overall, quite adherent and become increasingly harder with increasing temperature. Since the pitting consisted of crescent or horse-shoe shaped formations, with the closed end of the crescents always in the same direction, the corrosion can be classified as an impingement type. Furthermore, because the closed ends of the crescents are always in the same direction, it is possible to state the direction in which the water had been flowing. Many studies have shown that the closed ends lie in the direction of flow.

The acid cleaned portions show variation in the degree of this impingement corrosion. There appears to be a transition from directional impingement corrosion in the first four test loops, to ordinary pitting under the deposit in the 170° and 200°F test loops, particularly with the larger diameter tubing. This change in behavior seems to indicate some resistance to velocity effects at higher temperatures, though the deposit is sufficiently porous to allow pitting.

This paper deals with two major studies on the copper tubing removed from the experimental operation. The first of these is an evaluation of the extent of corrosion, while the second deals with the composition of the corrosion products.

Experimental Methods

To determine the extent of corrosion, measurements were made radiographically, gravimetrically and with dial and pointed micrometers.

The second section of this report is on the determination of the corrosion products. X-ray diffraction was used to obtain powder patterns of material from the deposits on the inner walls of the tubing.

REPORT ON THE EXTENT OF CORROSION

Assessment and reporting of corrosion has been plagued with inconsistencies and the lack of a universal language to express the extent of the corrosion.[3] It is felt that no single method of evaluation can adequately describe the attack on a given specimen and, therefore, several approaches have been combined with the hope of providing a broader and more comprehensive picture of the attack.

Each method of measurement is discussed and a comparison of common results made, along with a presentation of the distinctive advantages of each method. The same six-inch, acid-cleaned specimens were used in all of the evaluating measurements except for the gravimetric procedure.

Micrometer Measurements

Dial Micrometer Measurements

The thickness of the metal remaining on each specimen was first determined by dial micrometer measurement. The instrument (Figure 5) was rigidly supported on a frame in contact with a permanently attached ridged block, the zero reading being the contact points of the block. The device was calibrated by measurement of samples of new and unused copper tubing of identical nominal diameters with those of the test specimens. Wall thicknesses of the new tubing were determined by measurement with caliper micrometers. The test specimen readings were then converted to actual inches by comparison with the readings on the new tubing samples.

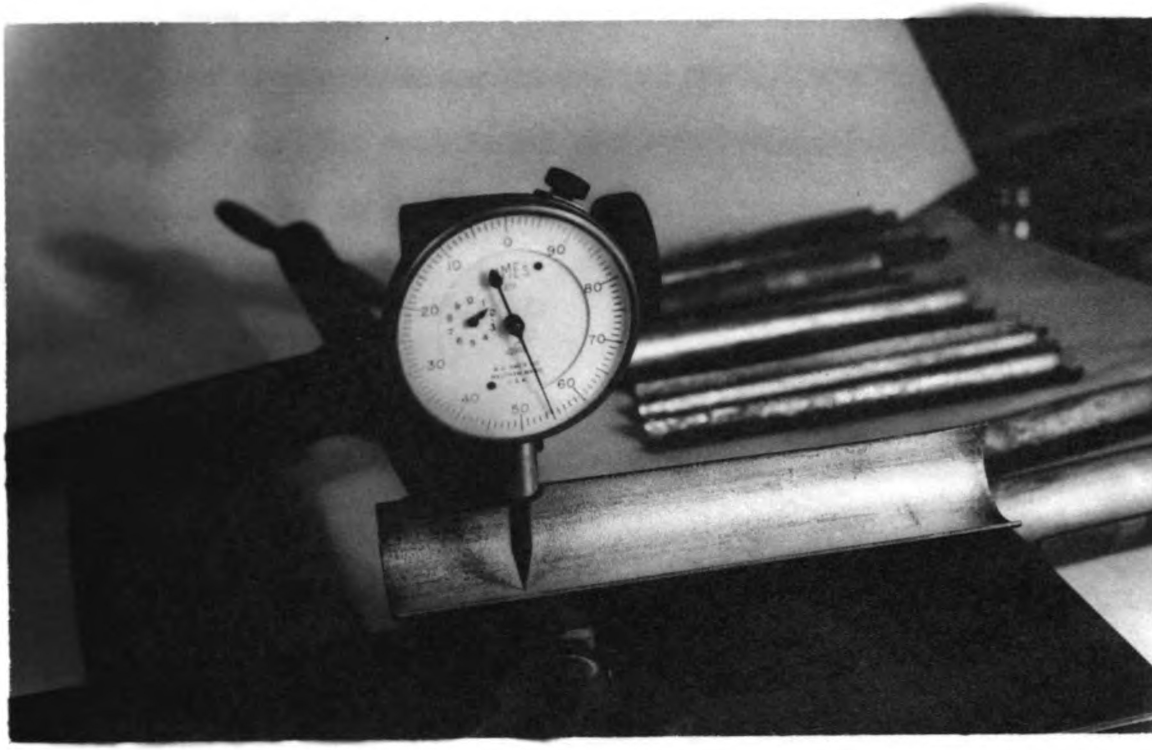


Figure 5. The Dial Micrometer in Use

Figure 6. The Pointed Micrometer in Use

Approximately ten random dial readings were made along the horizontal axis of each corroded sample and the average of each such set was calculated. The maximum and minimum readings found in each set were the extremes in wall thickness remaining.

Pointed Micrometer Measurements

A second set of wall thickness measurements was made with a pair of special caliper micrometers, with measuring points having a 30° taper. (Figure 6) This type of micrometer is in general use by CARRA members for such measurements. In this case, three separate sets of readings were made and averaged. These included ten random readings, ten readings taken in areas of minimum wall thickness and ten readings in areas of maximum wall thickness.

Radiographic Study

Several investigators[4,5] have employed a radiographic technique as an aid in corrosion studies. While their approach does not differ in principle from the one used in this study, certain procedural differences necessitate further discussion.

When an x-ray beam is directed at a corroded metal specimen, which has been placed on a sheet of film (Figure 7), the resulting pattern of blackening on the film is a representation of the degree of pitting. The x-ray functions as a sort of depth gauge, the film recording the variations in intensity of that radiation which penetrates the sample.

The resulting radiograph gives an excellent panoramic view of the corrosion, while quantitative representation of the film blackening can be obtained with the use of a densitometer or microphotometer.

A slight divergence at this point would not be amiss in order to give a brief explanation of the photographic effects of x-rays, radiography and the measurement of intensities of photographic blackening.[6] Even before their exact nature was known, x-rays were observed to cause blackening of photographic plates. These rays are capable of initiating the photochemical change in the photographic emulsion which results in the deposition of silver particles upon development. The degree of blackening or density of the deposit is defined in terms of the fraction of the incident light ultimately transmitted to the film.

$$\text{density} = \log \frac{\text{incident light}}{\text{transmitted light}}$$

In other words, a density of 1 means that only 0.1 of the incident light has been transmitted to the film.

It has also been established that the blackening is proportional to exposure (E), where E = beam intensity x time. This means that a 50 KV beam at 15 ma for one hour would give the same blackening as 1 ma for 15 hours. This relationship, known as the reciprocity law, is valid for the major portion of an exposure range, ceasing to be linear only at the extremes. In order to interpret the blackening of the film quantitatively, some sort of reference scale is necessary, usually a series of graded spots corresponding to known exposures. In view of the great range of validity of the reciprocity relationship, such a reference scale may be made using a beam of constant intensity and changing the time for a series of exposures. This scale should be identical with one using constant time and varying the intensity.

A similar reference scale can be made by radiographing a series of metal sheets of varying thicknesses on the same film plate as the sample. Error is introduced in both methods when conversion between sample and reference is made, not only by graphs or whatever method is used for comparison, but also by variation in handling, exposing and processing the film.

The possibility of such errors was minimized in this study by use of an internal standard. A copper step wedge, milled in 0.003 inch steps over the range of thicknesses of the samples, was radiographed with each sample. The use of the internal step wedge minimizes handling and processing errors, while its use with each exposure eliminates the separate graph.

To make accurate measurements of the film blackening, a recording microphotometer was employed. Basically this instrument measures the fraction of incident light that is transmitted through a slit area of the film by means of a photocell. The photoelectric current thus produced is measured by a galvanometer, the deflection of the galvanometer being proportional to the transmitted intensity. In this study a Jarrell-Ash comparator beam microphotometer was used, together with a Bristol recorder.

This instrument[7] employs a double beam technique in which the second beam, comprising a sample of the incident light source, is fed into the same photomultiplier that measures the main light beam passing through the slit. Operations, then, are independent of any fluctuations in the supply voltage and of any light intensity changes resulting from clouding of the lamp, aging of components, etc.

The recording microphotometer provides a continuous trace of the light transmission through the dark and light spots on the film corresponding to varying degrees of pitting on the samples, since the film sample is moved under the slit light source mechanically at a constant rate. The galvanometer readings are recorded on a continuous chart calibrated from 0 to 100 o/o transmission.

Gravimetric Determination of Corrosion

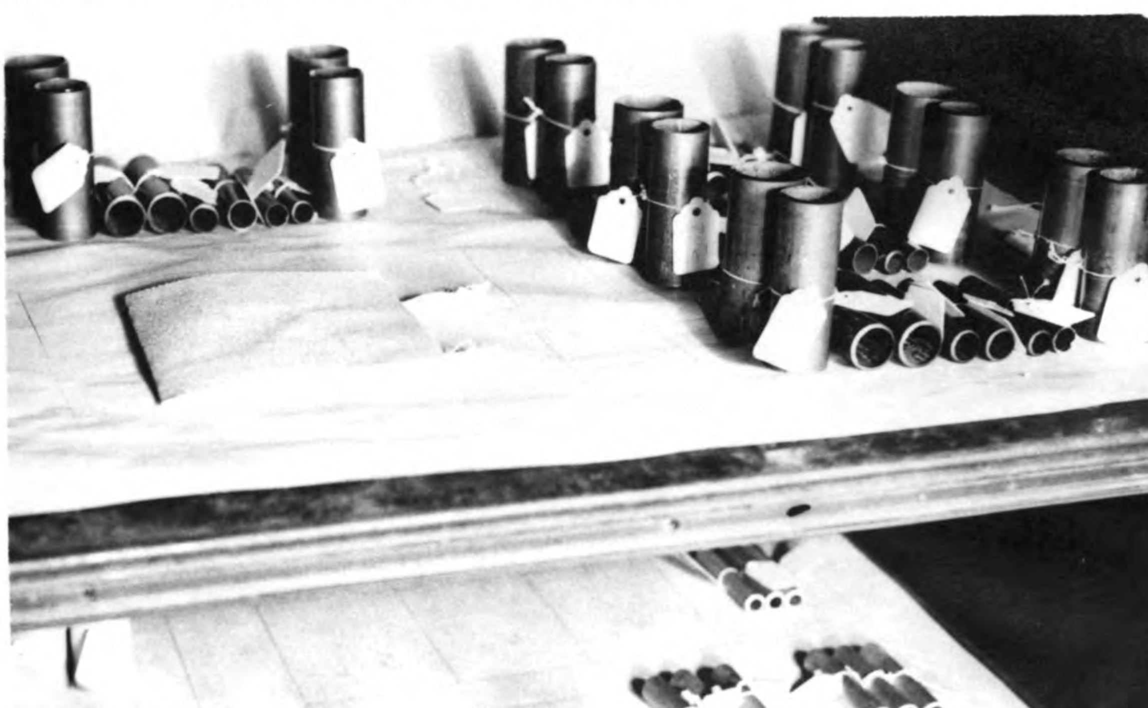
It would appear that the difference in weight of a sample before and after corrosion would be a valid representation of the extent of corrosion. Unfortunately, no preliminary weighing of the tubing sections was made. However, because of the manufacturer's rigid adherence to specifications, tubing of recent manufacture was assumed to be satisfactory as a standard.

The average weight per unit length of each diameter tubing was determined from several three inch lengths of both new and corroded tubing. (Figure 8) Corroded specimens were compared with new tubing specimens before and after cleaning in a sulfuric acid-dichromate solution.

All specimens were cut with a tubing cutter and their lengths determined with a vernier caliper. Weighing was done on a standard analytical balance.

Figure 7. Set-up for Making Radiographs

Figure 8. Samples for Gravimetric Analysis



Qualitative Comparison Between Photography and Radiography

The photograph and radiograph (Figure 9) are prints of the same set of corroded tubing samples. The complementary nature of the two methods is evident; the photograph shows the general shape and location of pitting, whereas the radiograph reveals the extent of corrosion. The light spots on the radiographic print correspond to the greatest depth of penetration by corrosion.

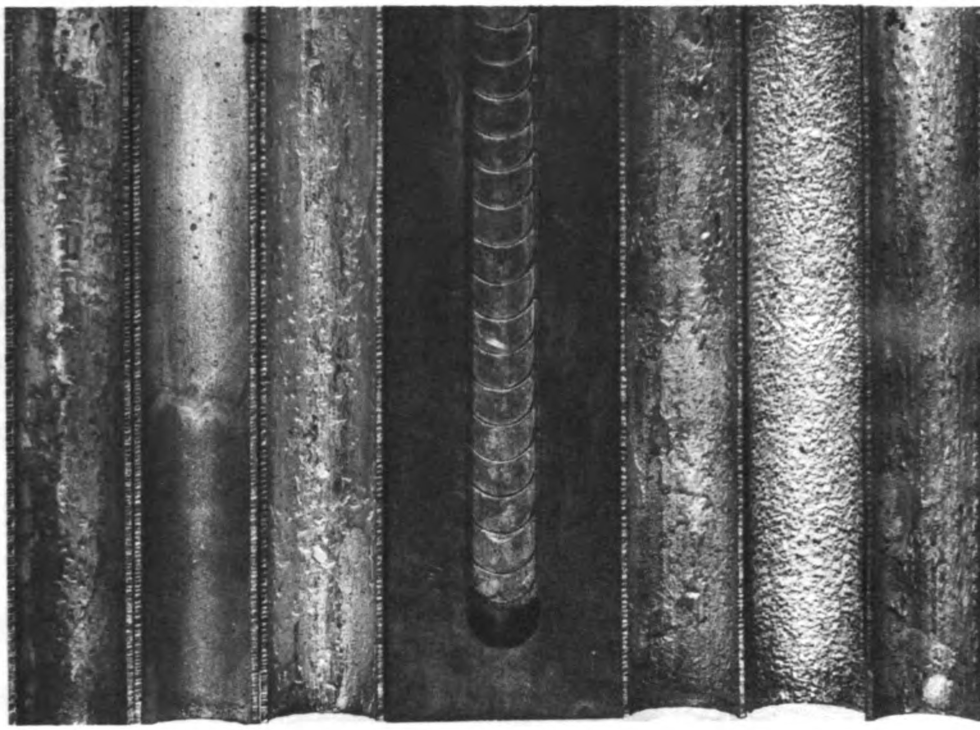
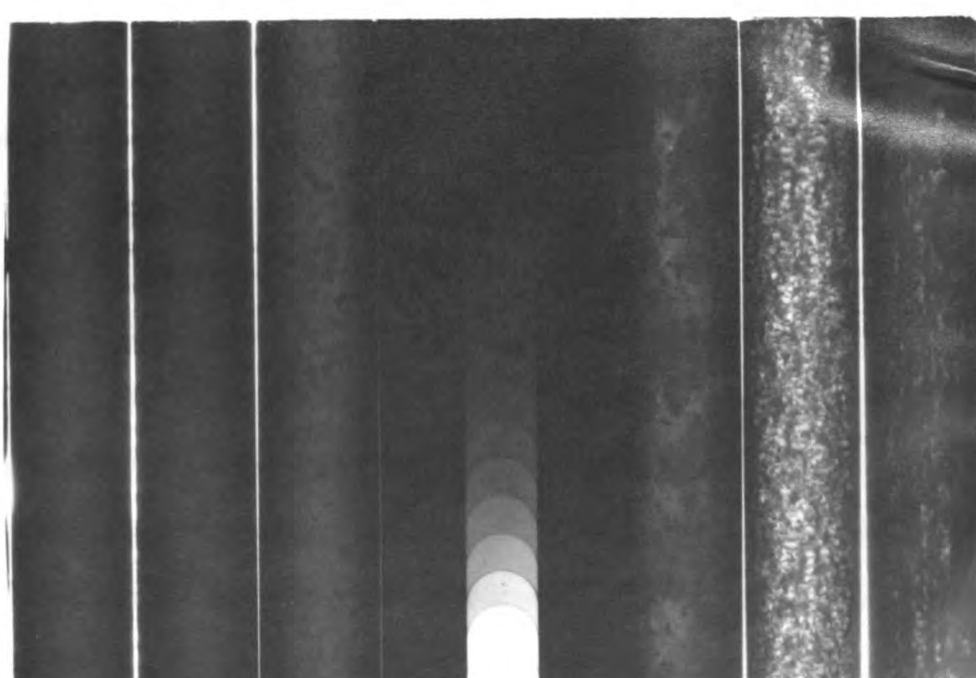


Figure 9. Photograph and Radiograph of Corroded Specimens with Step Wedge



IDENTIFICATION OF DEPOSITS

Early in the course of the corrosion study at Michigan State University (June 1951),[8] microchemical analyses were performed on deposits removed from copper tubing in service at the time. These deposits seemed to be composed largely of cupric oxide, ferric oxide, silica and a basic copper carbonate. Lesser amounts of copper chlorides, sulfates and some calcium salts were found.

It has been assumed that the deposits removed from the panel samples were of a similar nature. The current study was undertaken in an effort to more definitely establish the actual composition of these deposits. Using Norelco equipment with a copper target, Debye-Scherrer powder patterns were made of deposits from the tubing specimens at all temperature ranges. Patterns were also made of a number of pure copper compounds, the presence of which was possible in the deposits.

The "S" distance of all these patterns was measured, and from this, the interplanar "d" spacings determined graphically. These "d" values, along with the approximate intensities of the lines, were checked against tabulated values for a number of copper compounds. The presence of a number of compounds was thus indicated.

At first, difficulties were encountered with fogging of the film. Apparently iron oxide was present in sufficient quantity in the samples to cause x-ray flourescence. The use of an iron target tube lessened the fogging somewhat but required considerally longer (12 to 15 hours) exposure time.

EXPERIMENTAL I — EVALUATION METHODS

Two types of micrometer measurements and the radiographic observations were made on cleaned specimens. For the loss in weight or gravimetric determinations, a set of unsectioned tubing lengths was used.

The cleaned specimens were prepared by immersing six inch sectioned specimens, from the effluent end of each length of tubing used in the panel, in 10 o/o potassium dichromate-sulfuric acid solution for ten minutes, brushing lightly, washing with distilled water and allowing to dry.

Comparative graphs accompany each evaluation method and a table comparing results of the evaluation methods is included following all of the data. (Table XIII)

Dial Micrometer Measurements

For this series of wall thickness measurements a dial micrometer (Ames No. 212 — $\frac{1}{10,000}$ ") (Figure 5), fitted with a special hardened steel pointed contact foot, was used. The instrument was rigidly supported on a frame, with the contact foot coming to rest on a heavy steel base. With the cleaned samples resting on the steel base, initial measurements of the same pit resulted in readings of wide variation. These deviations resulted from a) possibility of transverse rocking of the hemi-cylindrical sample and b) a slight longitudinal bowed effect brought about during sectioning of the tubing.

A special brass 3-point V-block was made to correct these errors, and while they were minimized with this arrangement, new longitudinal rocking was possible on the three points. In addition, the wide variation in tubing diameter made it necessary to adjust the level of the V-block for each set of samples of different diameter.

Finally, a 1-inch block with 1/4 inch slightly elevated, ridged edges was used, so that in making measurements, the cylindrical shaped sample could be rocked to a slight extent in a transverse direction in order that the maximum pit depth as indicated by the minimum micrometer reading, could be obtained.

Readings were also made on samples of new tubing of comparable nominal diameter to determine the amount of copper lost by corrosion. The new tubing wall thicknesses were measured with the special pointed micrometers. The average value of ten random readings was taken as the actual wall thickness of tubing prior to corrosion.

As a basis for comparison, the manufacturer's specifications for tubing of the diameters used in the panels are listed in Table IV.

Table IV

Specifications for Government Type L Copper Water Tubing

Nominal Diameter (inches)	Outside Diameter (inches)	Inside Diameter (inches)	Wall Thickness (Tol = ± 0.004) (inches)	Theoretical Weight (Tol = ± 5 o/o) lb/ft	g/cm
3/8	0.500	0.430	0.035	0.198	0.147
1/2	.625	.545	.040	.285	.212
3/4	.875	.785	.045	.455	.339
1	1.125	1.025	.050	.655	.487
1 1/4	1.375	1.265	.055	.884	.658

NOTE. — Columns 1-5 from Perry, Chemical Engineers' Handbook, 3rd Edition, McGraw (1950) p 427
Column 6 calculated from column 5

Approximately ten random readings were made longitudinally on each sample and the average of each set calculated. The maximum and minimum readings on the corroded samples were assumed to represent pits and islands respectively. (Table V)

The maximum (or pit), minimum (or island) and average thicknesses of metal lost by corrosion, as determined by comparison of these measured values (Table V) with theoretical and average measured values of initial tubing wall thicknesses, are listed in Table VI.

DIAL MICROMETER READINGS

Table V

Readings in this table were made with a dial micrometer on samples of new tubing.

New Tubing - readings inches/10,000

Diameters of tubing sections	3/8	1/2	3/4	1	1 1/4
	515	456	416	335	309
	518	454	419	334	301
	516	458	420	329	303
	518	456	419	334	304
	518	455	418	332	305
	519	453	421	331	304
	521	458	421	332	297
	519	456	420	333	301
	521	456	422	332	302
	517	454	419	331	295
Average	518	456	419	332	303

Below are the average values in inches of 10 readings made on the same new tubing samples with the special pointed caliper micrometers.

.033	.039	.043	.047	.055

The next table contains the average values of the dial readings on the corroded tubing converted into actual inches. This conversion was made on the basis of the foregoing relationship.

NOTE. — In the following tables "i" and "p" refer to island and pit values respectively.

Table V Conversion of Dial Micrometer Readings to Actual Readings

Tubing Diameter		50°F	80°F	110°F	140°F	170°F	200°F
3/8	Average	0.033	0.031	0.023	0.024	0.019	0.021
	Island	.034	.033	.032	.033	.023	.032
	Max. Pit	.030	.029	.025	.020	.012	.015
1/2	Average	.037	.033	.034	.035	.026	.034
	Island	.039	.039	.039	.037	.031	.039
	Max. Pit	.035	.033	.032	.030	.020	.025
3/4	Average	.043	.043	.040	.040	.038	.038
	Island	.045	.043	.041	.042	.040	.041
	Max. Pit	.041	.041	.039	.038	.036	.035
1	Average	.049	.047	.047	.045	.047	.043
	Island	.050	.049	.043	.048	.048	.049
	Max. Pit	.048	.045	.046	.043	.044	.047
1 1/4	Average	.055	.056	.053	.054	.054	.054
	Island	.056	.059	.054	.054	.055	.055

MEASUREMENTS OF PANEL NO. 1 TUBING WALL THICKNESS

Average of 10 Readings

Water Temp	Tube Diameters				
50°F	3/8	1/2	3/4	1	1 1/4
	523	492	418	368	303
	519	478	425	369	305
	523	491	434	371	301
	510I	470	390I	369	310
	522	456I	410I	370	309
	523	485	433	372	310
	521	493	432	364I	312
	527	467	429	350I	305
	513I	451I	405I	369	305
	512I	473	435	375	295I
Average	519	476	421	367	306
Island Avg.	512	453	402	355	296
Max. Pit	527	493	435	375	312
°/o Islands	10	5-10	5	2-5	1

80°F	543	461	415	372	298
	539	465	421	388	312
	526	463	434	380	301
	527	469	430	393	294
	523	456I	410	388	293
	533	453I	415	385	295
	557	464	409	380	294
	540	463	413	368I	304
	521I	465	415	365I	301
	520I	461	420	403	290I
	561				
	553				
Average	537	462	418	382	296
Island Avg.	519	454	no islands	367	290
Max. Pit	553	469	434	403	312
°/o Islands	90	1	----	2-5	1

110°F	1/3	1/2	3/4	1	1 1/4
	511	522	445	335	330
	555	519	451	333	318
	583	519	452	337	325
	572	519	455	334	323
	531	439	451	3751	327
	585	493	452	330	3121
	587	4721	4331	370	2991
	597	4621	4401	370	334
	571	529	4231	333	332
	584	511	4241	3701	3141
	5151				
Average	567	502	444	332	323
Island Avg.	531	460	435	376	308
Max. Pit	597	522	455	390	334
°/o Islands	30	10	5–10	30	70

140°F	1/3	1/2	3/4	1	1 1/4
	629	497	444	412	303
	680	4631	442	391	302
	644	4631	452	395	310
	679	4441	439	399	312
	623	4351	432	410	2931
	5131	4631	463p	392	302
	5191	546	433	395	308
	639	528	4201	399	312
	662	537	432	422	303
	633	4811	453p	417	301
				3751	
				3691	
Average	605	494	443	393	306
Island Avg.	513	473	425	372	301
Max. Pit	652	546	463	422	312
°/o Islands	10–15	75	70–80	40	90

170°F	1/8	1/2	3/4	1	1 1/4
	633	5391	493	3301	310
	703	555	4461	399	3031
	723	535	453	399	315
	650	530	461	401	318
	658	5391	4461	395	321
	691	634	466	402	309
	5431	599	458	400	3021
	5371	593	472	399	321
	709	642	470	410	2371
	654	597	471	3741	305
Average	662	581	464	384	309
Island Avg.	565	535	446	377	298
Max. Pit	723	642	493	410	321
°/0 Islands	2-5	2-5	2-5	60	1 spot isls. 90 slight isls.

200°F					
	5341	4531	4151	370	3051
	653	597p	4481	368	315
	693	475	461	372	318
	678	550	4471	3581	319
	631	4571	495	373	317
	669	432	4451	378	316
	620	433	464	378	313
	5291	515	496	378	3071
	701	430	462	372	313
	665	431	499	375	318
		599p			
Average	642	507	463	372	314
Island Avg.	531	454	439	358	306
Max. Pit	701	599	499	373	319
°/o Islands	30	60-70	90	extremely minute pitting 1	extremely minute pitting 1

Table VI

Depth of Metal Lost by Corrosion (inches)

Nom. Tub. Dia.	Initial Wall Thickness		50°F			80°			110°			140°			170°			200°		
			Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)
3/8	+ Tol	.039	.006	.005	.009	.008	.006	.010	.011	.007	.014	.015	.006	.019	.020	.011	.027	.018	.007	.024
	Theor	.035	.002	.001	.005	.004	.002	.006	.007	.003	.010	.011	.002	.015	.016	.007	.023	.014	.003	.020
	Meas	.033	.000	-.001	.003	.002	.000	.004	.005	.001	.008	.009	.000	.013	.014	.005	.021	.012	.001	.018
1/2	+ Tol	.044	.007	.005	.009	.006	.005	.006	.010	.005	.012	.009	.007	.014	.018	.013	.024	.010	.005	.019
	Theor	.040	.003	.001	.005	.002	.001	.002	.006	.001	.008	.005	.003	.010	.014	.009	.020	.006	.001	.015
	Meas	.039	.002	.000	.004	.001	.000	.001	.005	.000	.007	.004	.002	.009	.013	.008	.019	.005	.000	.014
3/4	+ Tol	.049	.006	.004	.008	.006	.006	.008	.009	.008	.010	.009	.007	.011	.011	.009	.013	.011	.008	.014
	Theor	.045	.002	.000	.004	.002	.002	.004	.005	.004	.006	.005	.003	.007	.007	.005	.009	.007	.004	.010
	Meas	.043	.000	-.002	.002	.000	.000	.002	.003	.002	.004	.003	.001	.005	.005	.003	.007	.005	.002	.008
1	+ Tol	.054	.005	.004	.006	.007	.005	.009	.007	.006	.008	.009	.006	.011	.007	.006	.010	.006	.005	.007
	Theor	.050	.001	.000	.002	.003	.001	.005	.003	.002	.004	.005	.002	.007	.003	.002	.006	.002	.001	.003
	Meas	.047	-.002	-.003	-.001	.000	-.002	.002	.000	-.001	.001	.002	-.001	.004	.000	-.001	.003	-.001	-.002	.000
1 1/4	+ Tol	.059	.004	.003	.005	.003	.000	.005	.006	.005	.007	.005	.005	.006	.005	.004	.006	.005	.004	.006
	Theor	.055	.000	-.001	.001	-.001	-.004	.001	.002	.001	.003	.001	.001	.002	.001	.000	.002	.001	.000	.002
	Meas	.055	.000	-.001	.001	-.001	-.004	.001	.002	.001	.003	.001	.001	.002	.001	.000	.002	.001	.000	.002

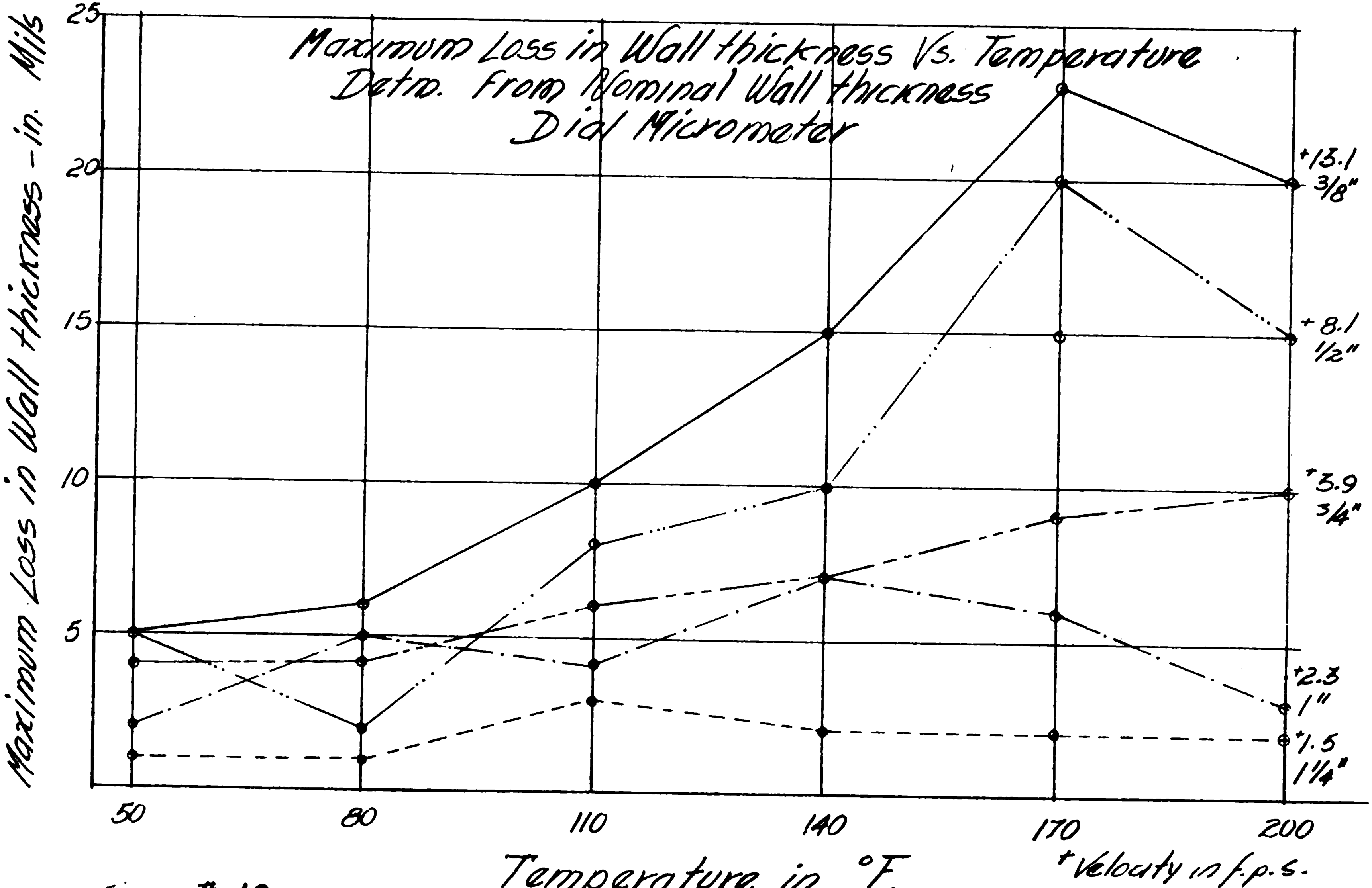
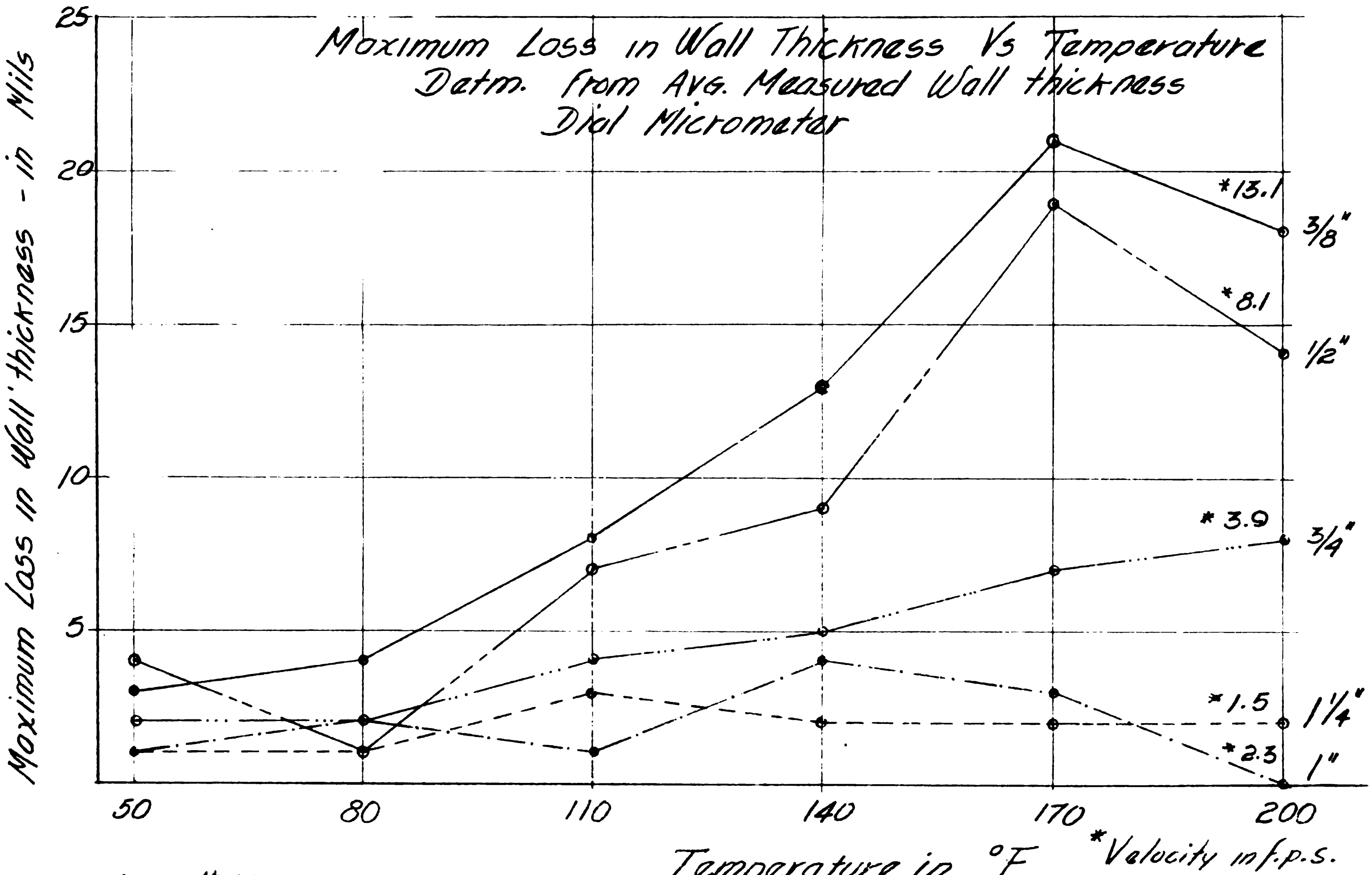

Maximum Loss in Wall thickness Vs. Temperature
Detn. From Nominal Wall thickness
Dial Micrometer
Maximum Loss in Wall thickness - in. Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F.
+Velocity in f.p.s.
+13.1
3/8"
+8.1
1/2"
+3.9
3/4"
+2.3
1"
+1.5
1¼"

Figure # 10



Maximum Loss in Wall Thickness Vs Temperature
Datm. from Avg. Measured Wall thickness
Dial Micrometer
Maximum Loss in Wall thickness - in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F.
*Velocity in f.p.s.
*13.1
3/8"
*8.1
1/2"
*3.9
3/4"
*1.5
1 1/4"
*2.3
1"

Figure # 11

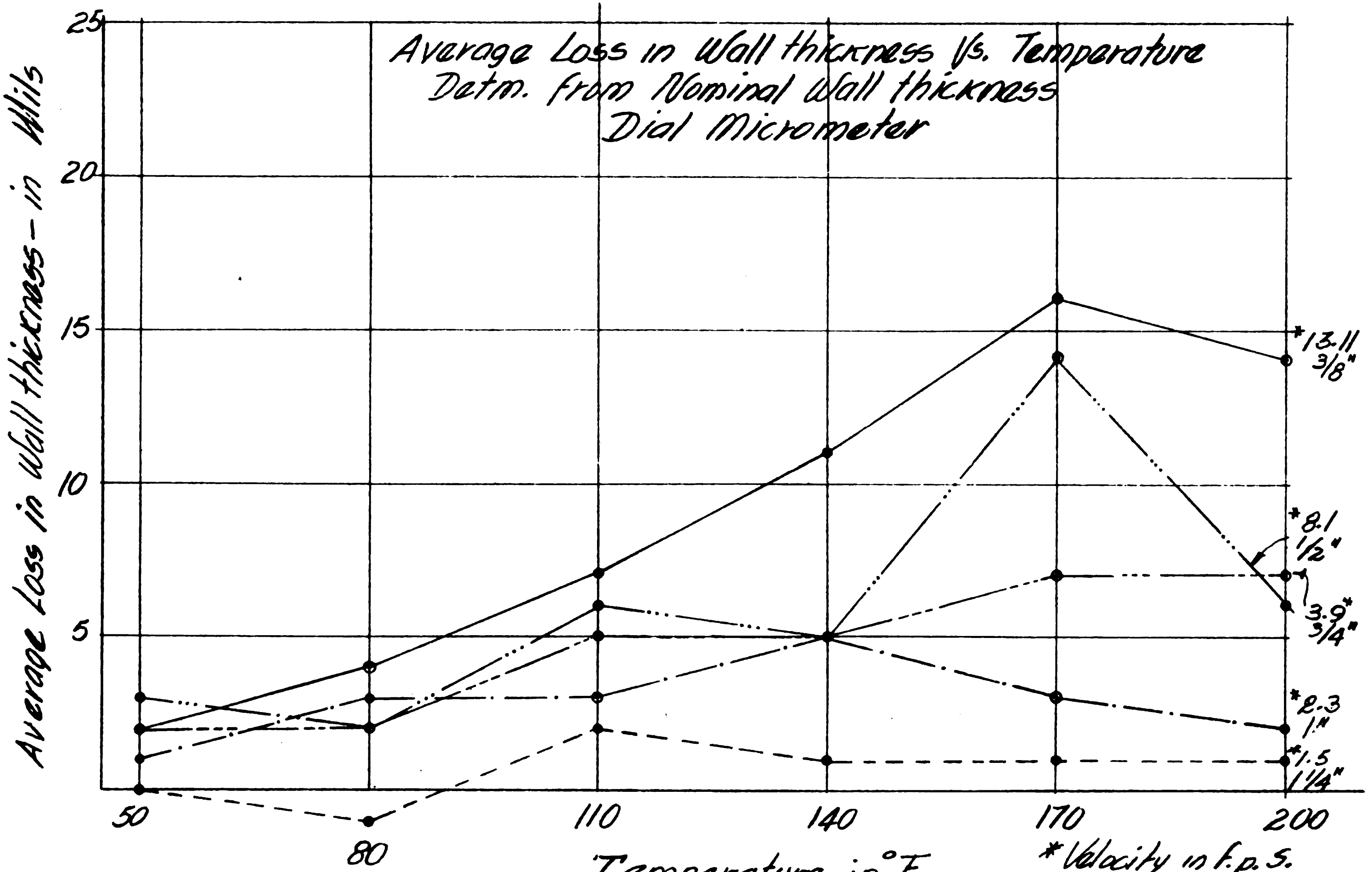

Average Loss in Wall thickness Vs. Temperature
Detm. from Nominal Wall thickness
Dial Micrometer
Average Loss in Wall thickness - in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F
*13.11
3/8"
*8.1
1/2"
3.9*
3/4"
*2.3
1"
*1.5
1 1/4"
* Velocity in f.p.s.

Figure # 12

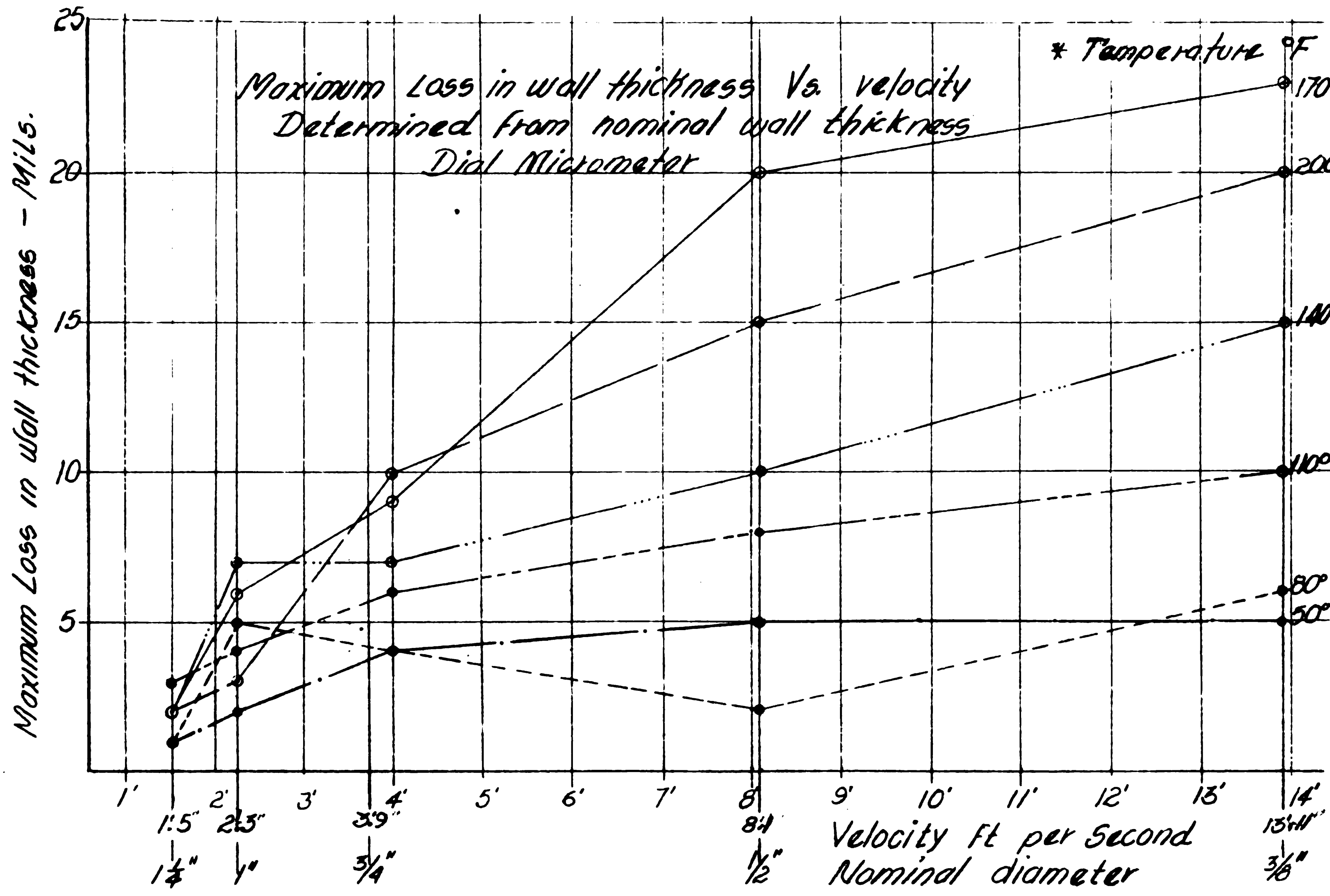
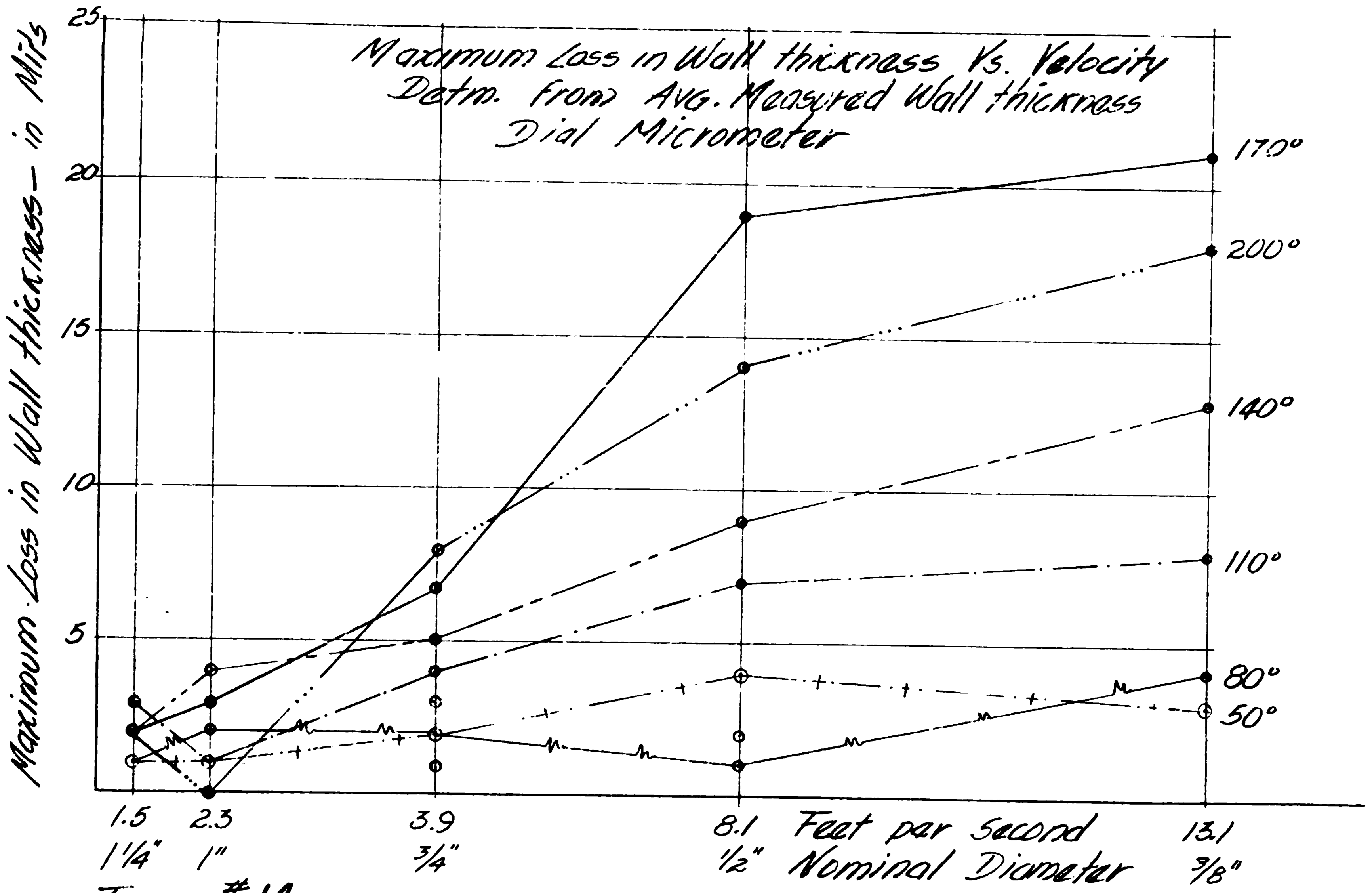
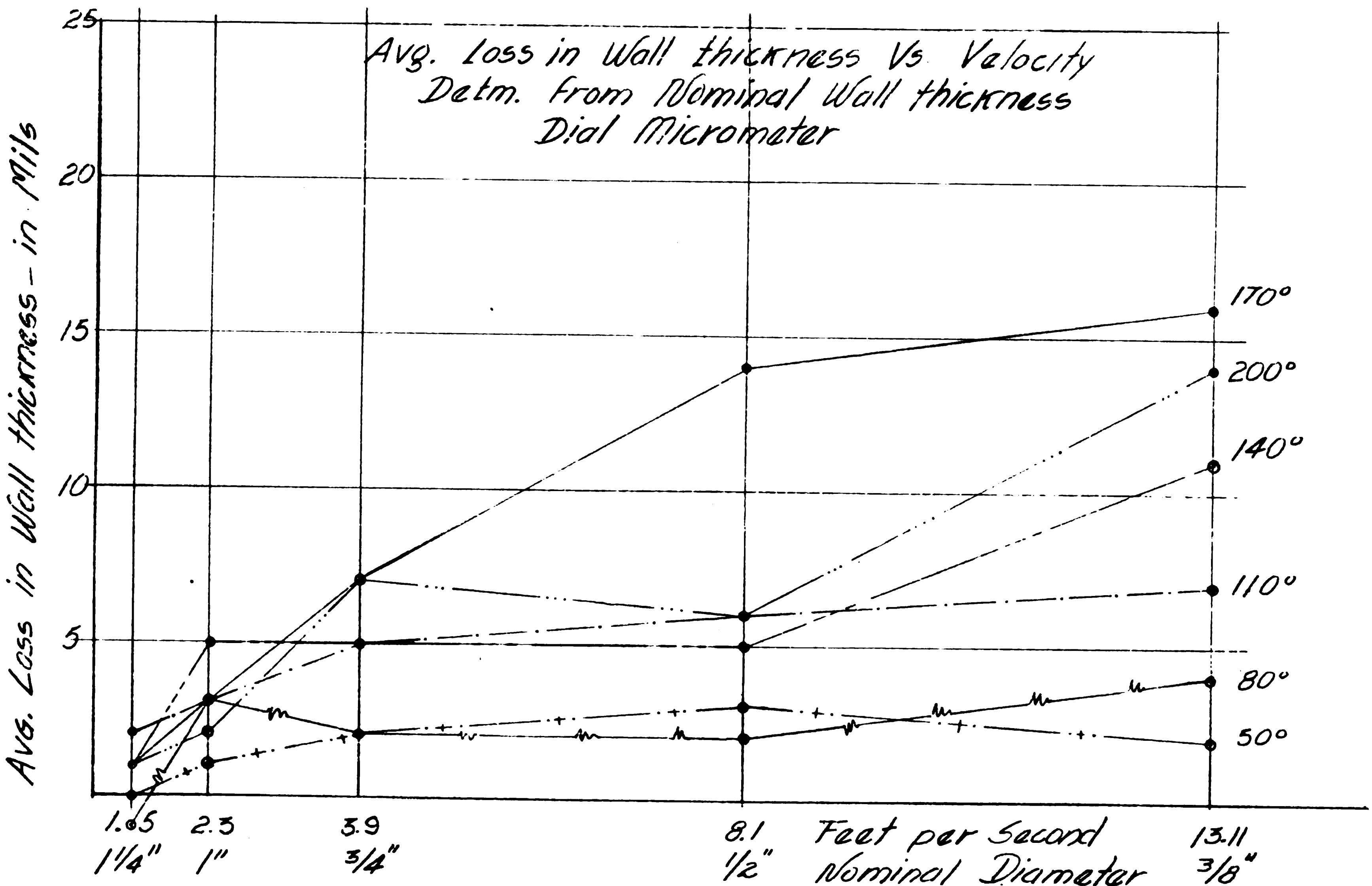

Maximum Loss in wall thickness Vs. velocity
Determined from nominal wall thickness
Dial Micrometer
* Temperature °F
170°
200°
140°
110°
80°
50°
25
20
15
10
5
Maximum Loss in Wall thickness - Mils.
1'
2'
3'
4'
5'
6'
7'
8'
9'
10'
11'
12'
13'
14'
1.5"
2.3"
3.9"
8.1
13.11"
Velocity Ft per Second
1¼"
1"
¾"
½"
⅜"
Nominal diameter

FIGURE # 13


Maximum Loss in Wall thickness Vs. Velocity
Detm. from Avg. Measured Wall thickness
Dial Micrometer
25
20
15
10
5
Maximum Loss in Wall thickness - in Mils
170°
200°
140°
110°
80°
50°
1.5
1¼"
2.3
1"
3.9
¾"
8.1
½"
13.1
⅜"
Feet per Second
Nominal Diameter
Figure #14



Avg. Loss in Wall thickness Vs. Velocity
Detm. from Nominal Wall thickness
Dial Micrometer
Avg. Loss in Wall thickness - in Mils
25
20
15
10
5
170°
200°
140°
110°
80°
50°
1.65
1 1/4"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Feet per Second
Nominal Diameter

Figure #15

Pointed Micrometer Measurements

For the second set of wall thickness measurements CAMA provided Brown and Sharpe No. 13 one inch micrometers with special 30° points. (Figure 6) The samples were held rigid in a clamp and readings taken carefully, as the points tended to scratch the soft copper.

The data were obtained as follows: ten random readings were made longitudinally; then deliberate attempts were made to find the ten deepest pits and the ten greatest islands. The average values of each of these sets of readings were calculated. (Tables VII)

The maximum (or pit), minimum (or island) and average thicknesses of metal lost by corrosion, as determined by comparison of these measured values (Table VII) with theoretical and average measured values of initial tubing wall thicknesses, are listed in Table VIII.

Table VII

Pointed Micrometer Readings

Key

MLP = minute localised pitting - smaller than probe

NSI = no specific islands

NSP = no specific pits

NLP = no localised pitting

	50°F	80°F	110°F	140°F	170°F	200°F
	.031	.031	.029	.023	.027	.025
	.033	.033	.029	.025	.022	.026
3/8"	.033	.034	.032	.022	.025	.016
	.035	.032	.029	.026	.020	.018
	.034	.032	.032	.022	.019	.031
	.032	.037	.033	.023	.020	.017
Random	.035	.033	.027	.028	.024	.023
	.033	.033	.029	.025	.016	.016
	.033	.033	.035	.021	.019	.026
	.034	.032	.027	.024	.021	.030
Average	.034	.033	.030	.024	.021	.023
	+.001	+.004	+.005	+.003	+.006	+.007
	-.002	-.002	-.003	-.003	-.005	-.007
	.034	.031	.027	.020	.014	.014
	.034	.031	.026	.021	.016	.016
	.033	.033	.027	.019	.013	.015
	.033	.031	.028	.021	.015	.018
Fit	.034	.032	.028	.023	.016	.018
	.033	.034	.029	.020	.014	.015
	.033	.034	.028	.024	.010	.016
	.032	.028	.023	.019	.015	.015
	.033	.028	.026	.020	.016	.014
	.032	.031	.026	.020	.014	.014
Average	.033	.031	.027	.021	.014	.016
	+.001	+.002	+.002	+.003	+.002	+.002
	-.001	-.003	-.001	-.002	-.004	-.002
	.035	.033	.033	.036	.027	.023
	.035	.034	.033	.034	.029	.027
	.036	.033	.036	.031	.030	.027
	.034	.035	.034	.031	.026	.030
Island	.035	.034	.032	.029	.026	.031
	.035	.033	.032	.032	.030	.028
	.034	.034	.033	.033	.031	.029
	.036	.033	.035	.033	.033	.030
	.035	.035	.033	.032	.029	.029
	.035	.033	.033	.034	.029	.031
Average	.035	.034	.033	.033	.029	.029
	+.001	+.001	+.003	+.003	+.002	+.002
	-.001	-.001	-.001	-.004	-.003	-.001

	50°F	80°F	110°F	140°F	170°F	200°F
	.037	.038	.034	.037	.025	.035
	.036	.038	.035	.037	.021	.037
1/2"	.036	.039	.033	.031	.028	.036
	.037	.037	.033	.036	.027	.038
	.035	.038	.036	.033	.030	.035
Random	.037	.038	.035	.034	.026	.028
	.035	.039	.036	.032	.027	.031
	.038	.038	.034	.037	.029	.032
	.036	.038	.034	.031	.025	.025
	.036	.038	.037	.036	.026	.029
Average	.036	.038	.035	.034	.026	.034
	+.002	+.001	+.002	+.003	+.004	+.004
	-.001	-.001	-.002	-.003	-.005	-.006
	.035	.035	.034	.031	.020	.025
	.034	.036	.034	.032	.022	.027
	.035	.038	.033	.030	.020	.029
	.035	.037	.034	.029	.024	.030
	.035	.038	.033	.031	.020	.029
Pit	.034	.038	.033	.030	.021	.023
	.036	.038	.033	.029	.019	.024
	.035	.038	.034	.029	.021	.025
	.035	.038	.033	.028	.019	.027
	.036	.037	.032	.028	.020	.025
Average	.035	.037	.033	.030	.021	.026
	+.001	+.001	+.001	+.002	+.003	+.004
	-.001	-.002	-.001	-.002	-.002	-.003
	.039	.039	.036	.038	.030	.038
	.039	.039	.037	.038	.034	.036
	.037	.038	.038	.037	.031	.036
	.037	.039	.038	.039	.032	.037
	.038	.040	.036	.040	.030	.037
Island	.037	.038	.037	.037	.033	.035
	.038	.039	.038	.039	.031	.038
	.038	.038	.037	.041	.032	.037
	.039	.038	.037	.038	.032	.037
	.038	.039	.037	.039	.031	.038
Average	.038	.039	.037	.039	.032	.037
	+.001	+.001	+.001	+.002	+.002	+.001
	-.001	-.001	-.001	-.002	-.002	-.002

	50°F	80°F	110°F	140°F	170°F	200°F
	.042	.044	.040	.040	.038	.040
	.042	.044	.040	.040	.038	.040
3/4"	.042	.044	.040	.041	.038	.040
	.042	.044	.040	.041	.039	.041
	.042	.045	.042	.041	.037	.040
Random	.043	.043	.041	.041	.040	.040
	.041	.044	.041	.039	.038	.041
	.041	.042	.040	.041	.038	.038
	.042	.043	.040	.043	.038	.040
	.042	.042	.040	.039	.036	.036
Average	.042	.044	.040	.041	.038	.040
	+.001	+.001	+.002	+.002	+.002	+.001
	-.001	-.002	-.000	-.002	-.002	-.004

	50°F	80°F	110°F	140°F	170°F	200°F
	N.L.P.	N.L.P.				
	.042	.041	.039	.038	.037	.037
	.042	.041	.040	.040	.037	.037
	.042	.042	.040	.039	.036	.038
	.042	.043	.040	.039	.037	.038
Fit	.042	.042	.041	.038	.036	.038
	.041	.043	.040	.038	.035	.037
	.042	.042	.039	.039	.036	.035
	.042	.042	.040	.039	.035	.038
	.042	.042	.040	.038	.035	.036
	.042	.042	.039	.038	.036	.034
Average	.042	.042	.040	.039	.037	.037
	+.000	+.001	+.001	+.001	+.000	+.001
	-.001	-.001	-.001	-.001	-.002	-.003

	50°F	80°F	110°F	140°F	170°F	200°F
		N.S.I.				
	.043	.045	.044	.041	.038	.043
	.043	.044	.043	.042	.038	.043
	.044	.044	.043	.043	.041	.044
	.043	.043	.043	.041	.040	.042
Island	.045	.046	.042	.040	.041	.044
	.043	.044	.043	.041	.041	.043
	.043	.043	.044	.043	.039	.044
	.045	.044	.044	.044	.040	.043
	.044	.043	.045	.041	.040	.042
	.043	.044	.043	.043	.040	.043
Average	.044	.044	.043	.042	.040	.043
	+.001	+.002	+.002	+.002	+.001	+.001
	-.001	-.001	-.001	-.002	-.002	-.001

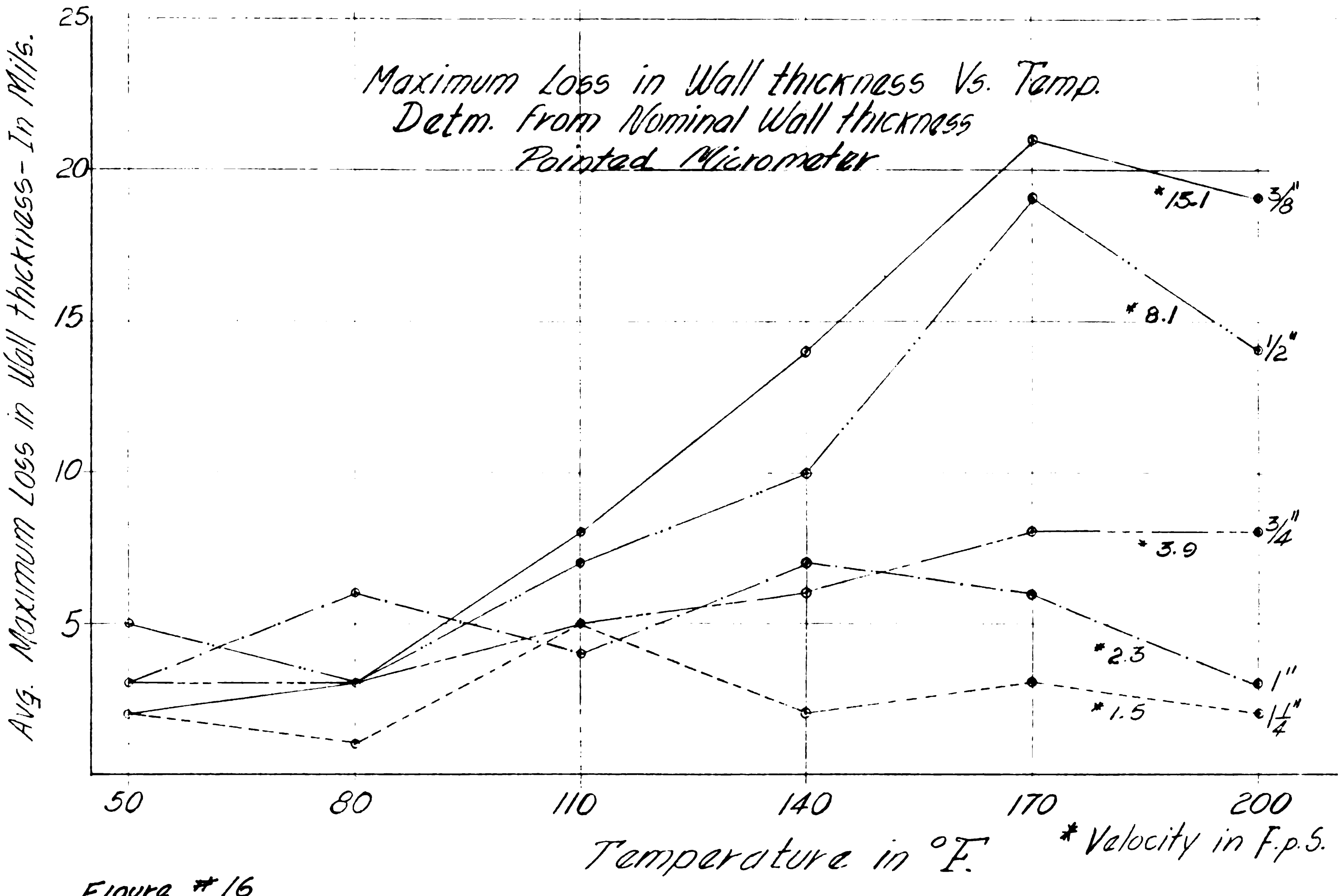
	50°F	80°F	110°F	140°F	170°F	200°F
	.048	.046	.046	.045	.044	.048
	.048	.048	.049	.043	.045	.047
1"	.048	.048	.046	.043	.045	.048
	.047	.046	.048	.044	.044	.048
	.048	.045	.046	.044	.046	.048
Random	.049	.044	.045	.043	.047	.047
	.048	.044	.047	.043	.045	.050
	.048	.046	.046	.044	.045	.047
	.048	.047	.046	.046	.045	.049
	.050	.046	.048	.045	.047	.048
Average	.048	.045	.047	.044	.046	.048
	+.002	+.003	+.002	+.002	+.002	+.002
	-.001	-.001	-.002	-.001	-.002	-.001
	N.S.P.					N.L.P.
	.047	.044	.046	.043	.044	.046
	.047	.045	.046	.044	.044	.047
	.048	.044	.047	.044	.045	.047
	.047	.044	.046	.042	.043	.046
Pit	.047	.044	.046	.043	.044	.047
	.047	.045	.045	.043	.043	.046
	.048	.044	.046	.043	.044	.047
	.047	.043	.046	.043	.043	.045
	.047	.044	.046	.043	.043	.047
	.048	.044	.047	.042	.043	.047
Average	.047	.044	.046	.043	.044	.047
	+.001	+.001	+.001	+.001	+.001	+.000
	-.000	-.001	-.001	-.001	-.001	-.002
	.050	.047	.048	.044	.046	.048
	.049	.048	.048	.045	.046	.048
	.050	.047	.048	.044	.047	.050
	.049	.048	.050	.047	.048	.048
Island	.049	.047	.048	.047	.047	.048
	.050	.048	.049	.045	.046	.048
	.049	.048	.050	.047	.046	.050
	.050	.047	.049	.047	.048	.048
	.050	.047	.048	.046	.047	.049
	.049	.048	.049	.047	.046	.048
Average	.050	.048	.049	.046	.047	.049
	+.001	+.000	+.001	+.001	+.001	+.001
	-.001	-.001	-.001	-.002	-.001	-.001

	50°F	80°F	110°F	140°F	170°F	200°F
	.053	.054	.050	.055	.052	.053
	.053	.054	.052	.055	.052	.053
1 1/4"	.053	.055	.051	.054	.053	.053
	.053	.054	.052	.054	.054	.052
	.053	.055	.050	.055	.055	.053
Random	.053	.055	.052	.055	.052	.053
	.053	.054	.051	.054	.053	.053
	.053	.055	.052	.054	.052	.052
	.053	.055	.053	.054	.052	.053
	.053	.054	.052	.055	.053	.054
Average	.053	.055	.052	.055	.053	.053
	+.000	+.000	+.001	+.000	+.002	+.001
	-.000	-.001	-.002	-.001	-.001	-.001
						M.L.P.
	.053	.054	.051	.053	.051	.052
	.052	.054	.051	.053	.052	.053
	.053	.054	.050	.052	.052	.053
	.053	.055	.050	.054	.051	.053
Pit	.053	.055	.049	.053	.053	.052
	.053	.054	.050	.053	.052	.052
	.052	.055	.050	.053	.052	.053
	.052	.055	.051	.052	.051	.052
	.053	.054	.050	.053	.052	.052
	.052	.053	.051	.053	.053	.053
Average	.053	.054	.050	.053	.052	.053
	+.000	+.001	+.001	+.001	+.001	+.000
	-.001	-.001	-.001	-.001	-.001	-.001
	.054	.056	.053	.055	.056	.054
	.054	.055	.052	.055	.055	.054
	.053	.056	.053	.056	.058	.053
	.054	.056	.054	.057	.057	.054
Island	.053	.057	.052	.055	.055	.053
	.053	.055	.053	.055	.054	.053
	.054	.057	.052	.057	.054	.054
	.053	.059	.053	.056	.055	.055
	.055	.056	.053	.055	.057	.054
	.054	.058	.052	.056	.055	.055
Average	.054	.057	.053	.056	.056	.054
	+.001	+.002	+.001	+.001	+.002	+.001
	-.001	-.002	-.001	-.001	-.002	-.001

Table VIII

Depth of Metal Lost by Corrosion (inches)

Nom. Tub. Dia.	Initial Wall Thickness		50°F			80°			110°			140°			170°			200°		
			Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)
3/8	+ Tol	.039	.005	.004	.006	.006	.005	.007	.009	.006	.012	.015	.006	.018	.018	.010	.025	.016	.010	.023
	Theor	.035	.001	.000	.002	.002	.001	.003	.005	.002	.008	.011	.002	.014	.014	.006	.021	.012	.006	.019
	Meas	.033	-.001	-.002	.000	.000	-.001	.001	.003	.000	.006	.009	.000	.012	.012	.004	.019	.010	.004	.017
1/2	+ Tol	.044	.008	.006	.009	.006	.005	.007	.009	.006	.011	.010	.005	.014	.018	.012	.023	.010	.007	.018
	Theor	.040	.004	.002	.005	.002	.001	.003	.005	.002	.007	.006	.001	.010	.014	.008	.019	.006	.003	.014
	Meas	.039	.003	.001	.004	.001	.000	.002	.004	.001	.006	.005	.000	.009	.013	.007	.018	.005	.002	.013
3/4	+ Tol	.049	.007	.005	.007	.005	.005	.007	.009	.006	.009	.008	.007	.010	.011	.009	.012	.009	.006	.012
	Theor	.045	.003	.001	.003	.001	.001	.003	.005	.002	.005	.004	.003	.006	.007	.005	.008	.005	.002	.008
	Meas	.043	.001	-.001	.001	-.001	-.001	.001	.003	.000	.003	.002	.001	.004	.005	.003	.006	.003	.000	.006
1	+ Tol	.054	.006	.004	.007	.009	.006	.010	.007	.005	.008	.010	.008	.011	.008	.007	.010	.006	.005	.007
	Theor	.050	.002	.000	.003	.005	.002	.006	.003	.001	.004	.006	.004	.007	.004	.003	.006	.002	.001	.003
	Meas	.047	-.001	-.003	.000	.002	-.001	.003	.000	-.002	.001	.003	.001	.004	.001	.000	.003	-.001	-.002	.000
1 1/4	+ Tol	.059	.006	.005	.006	.004	.002	.005	.007	.006	.009	.004	.003	.006	.006	.003	.007	.006	.005	.006
	Theor	.055	.002	.001	.002	.000	-.002	.001	.003	.002	.005	.000	-.001	.002	.002	-.001	.003	.002	.001	.002
	Meas	.055	.002	.001	.002	.000	-.002	.001	.003	.002	.005	.000	-.001	.002	.002	-.001	.003	.002	.001	.002



Maximum Loss in Wall thickness Vs. Temp.
Detm. from Nominal Wall thickness
Pointed Micrometer
Avg. Maximum Loss in Wall thickness - In Mils.
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F.
* Velocity in F.p.S.
*15.1
*8.1
*3.9
*2.3
*1.5
3/8"
1/2"
3/4"
1"
1 1/4"

Figure #16

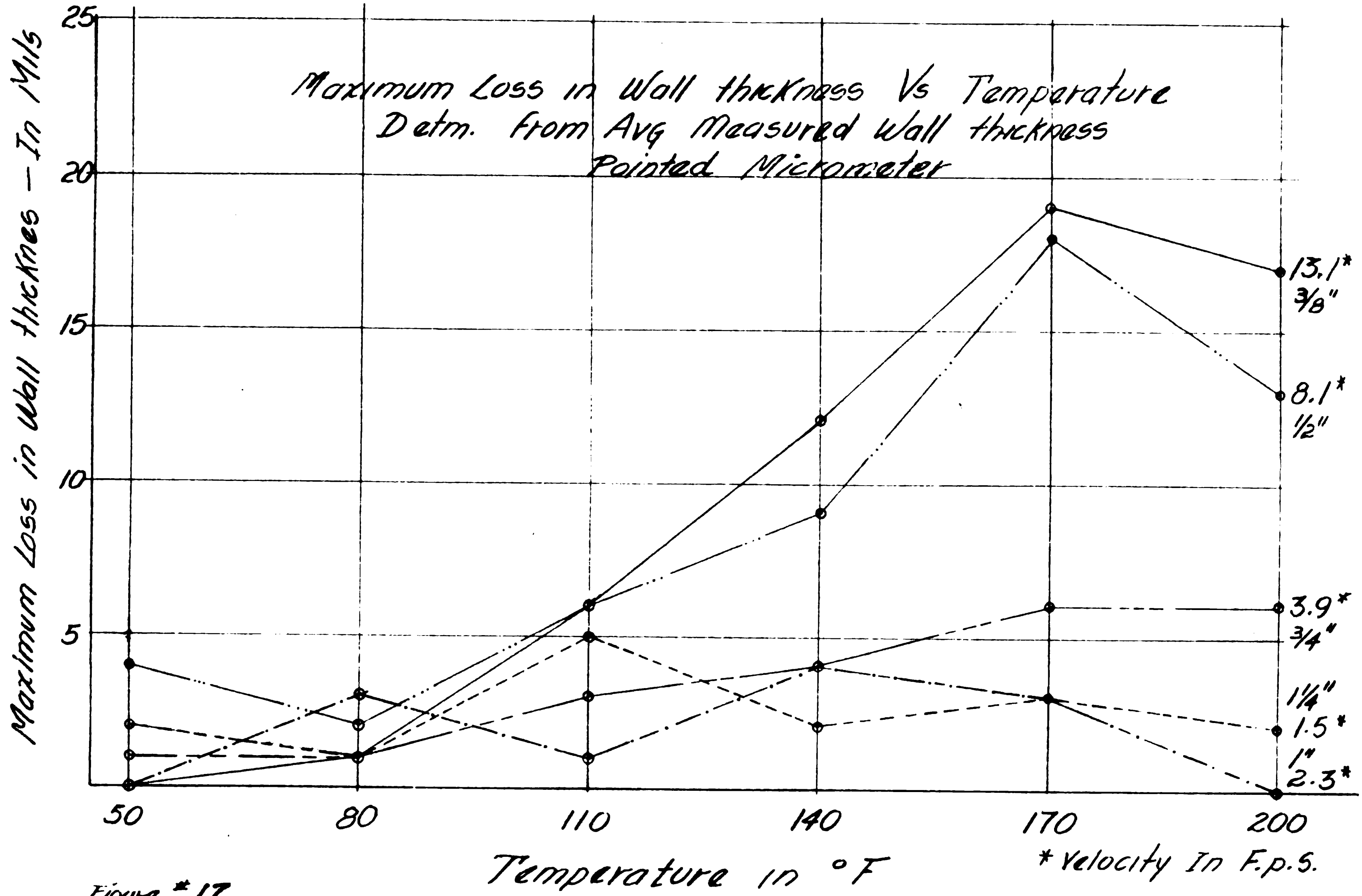
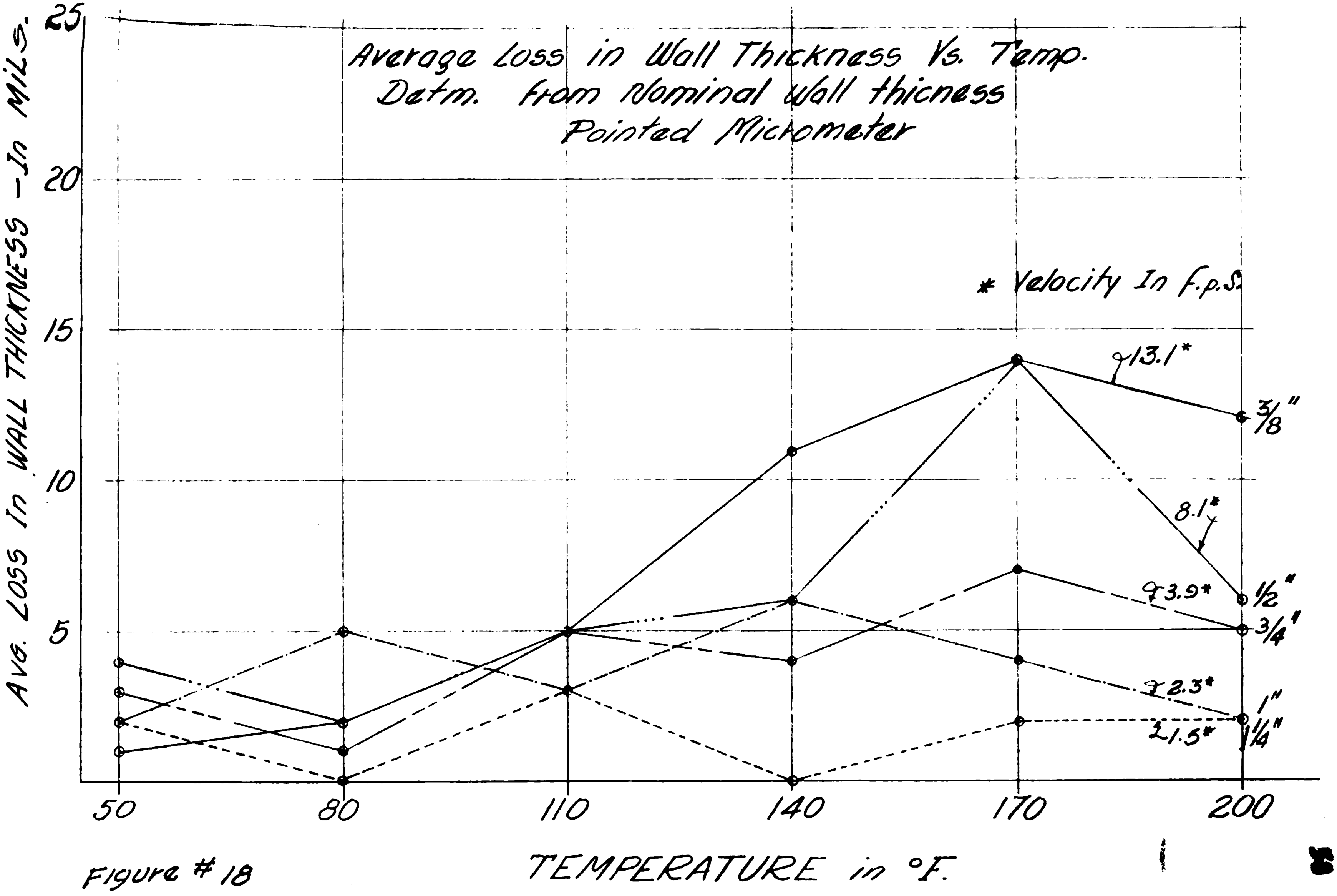

Maximum Loss in Wall thickness Vs Temperature
Detm. from Avg Measured Wall thickness
Pointed Micrometer
25
20
15
10
5
Maximum Loss in Wall thicknes — In Mils
50
80
110
140
170
200
Temperature in °F
13.1*
3/8"
8.1*
1/2"
3.9*
3/4"
1 1/4"
1.5*
1"
2.3*
* Velocity In F.p.S.

Figure * 17



Average Loss in Wall Thickness Vs. Temp.
Detm. from Nominal Wall thicness
Pointed Micrometer
* Velocity In F.p.S.
13.1*
8.1*
3.9*
2.3*
1.5*
3/8"
1/2"
3/4"
1"
1/4"
Avg. Loss In Wall Thickness – In Mils.
25
20
15
10
5
50
80
110
140
170
200
TEMPERATURE in °F.

Figure # 18

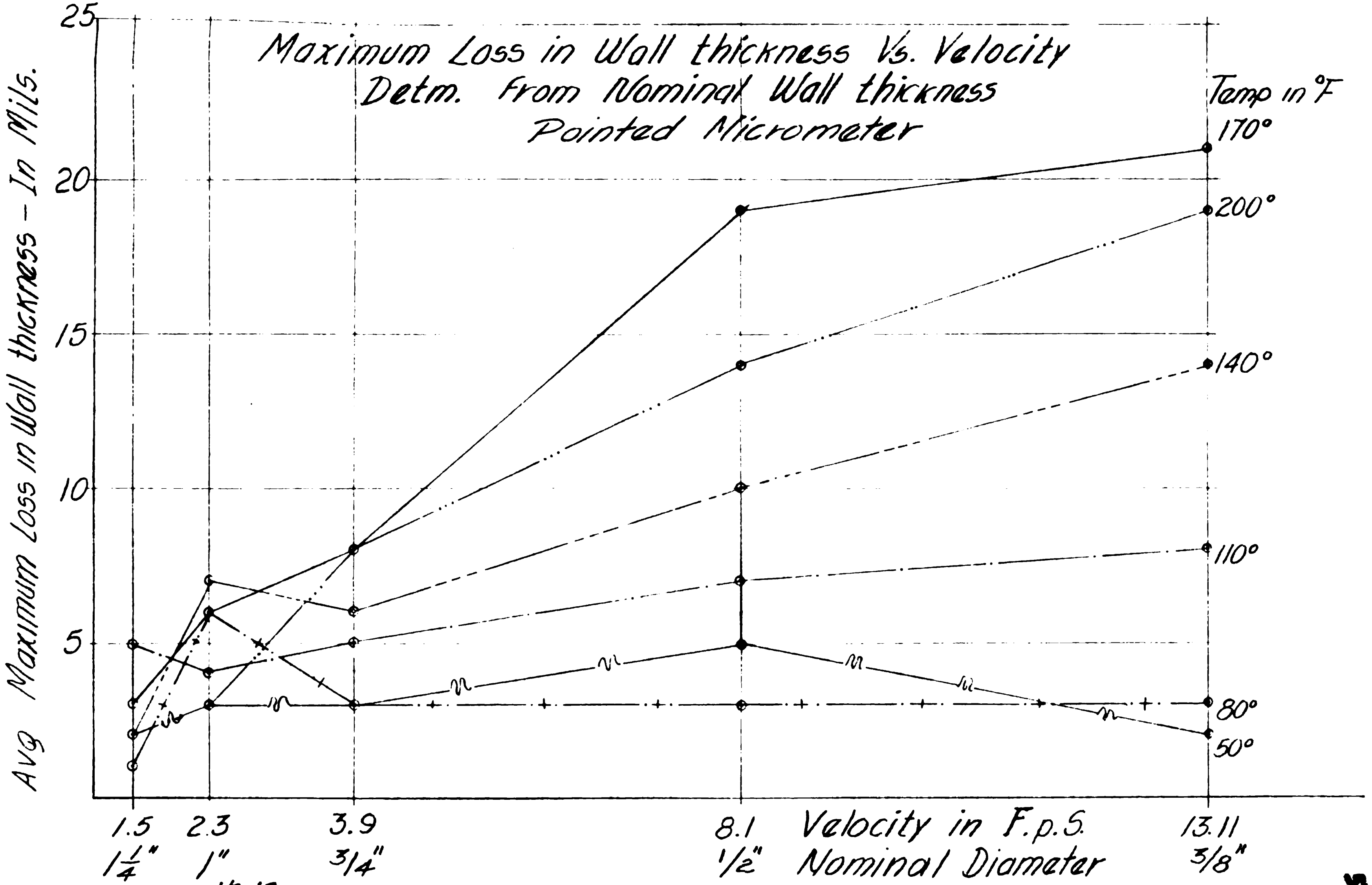

Maximum Loss in Wall thickness Vs. Velocity
Detm. From Nominal Wall thickness
Pointed Micrometer
Avg Maximum Loss in Wall thickness - In Mils.
25
20
15
10
5
Temp in °F
170°
200°
140°
110°
80°
50°
1.5
1 1/4"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Velocity in F.p.S.
Nominal Diameter

Figure # 19

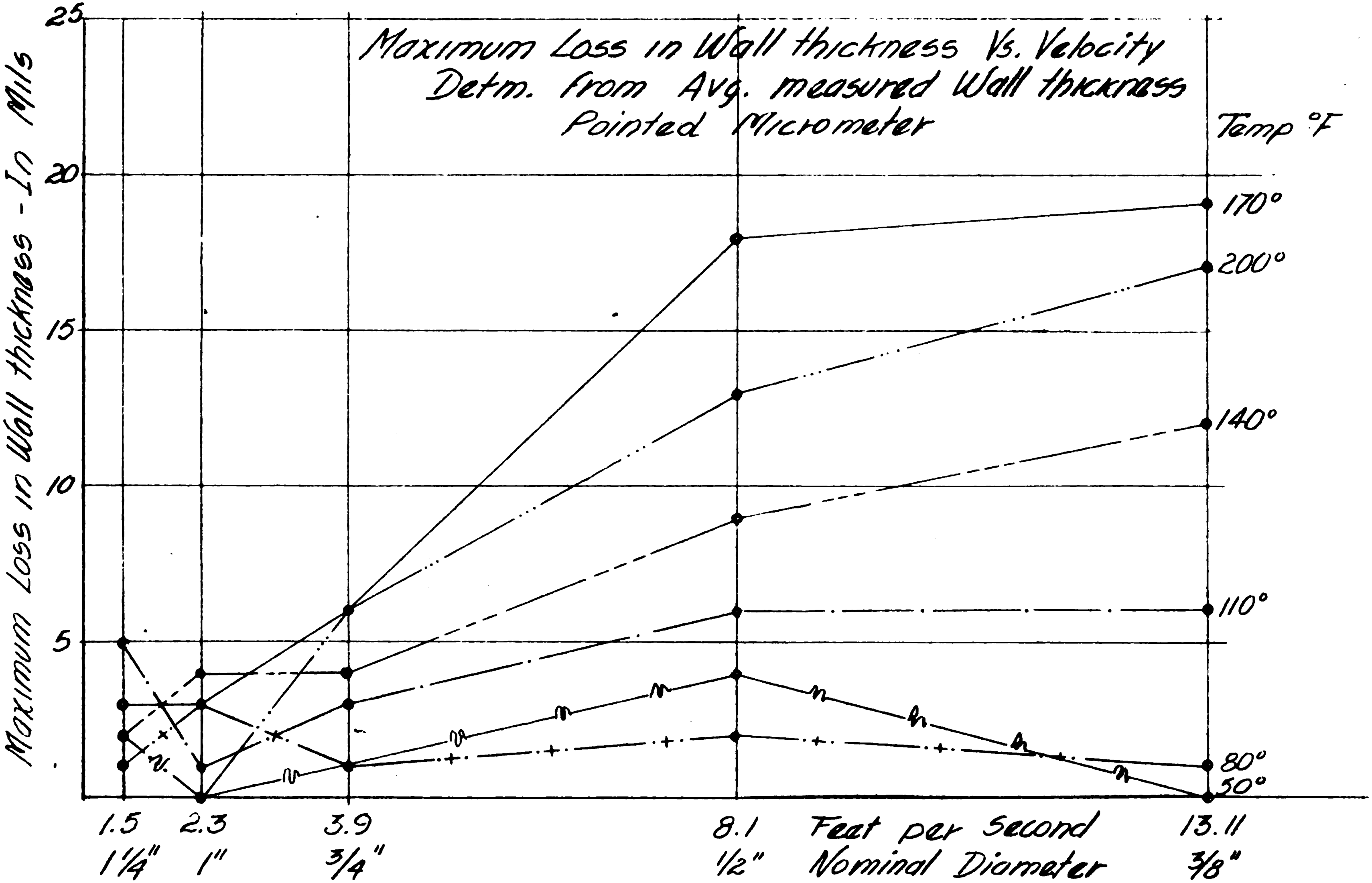

Maximum Loss in Wall thickness Vs. Velocity
Detm. from Avg. measured Wall thickness
Pointed Micrometer
Temp °F
170°
200°
140°
110°
80°
50°
25
20
15
10
5
Maximum Loss in Wall thickness - In Mils
1.5
1 1/4"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Feet per Second
Nominal Diameter

Figure # 20

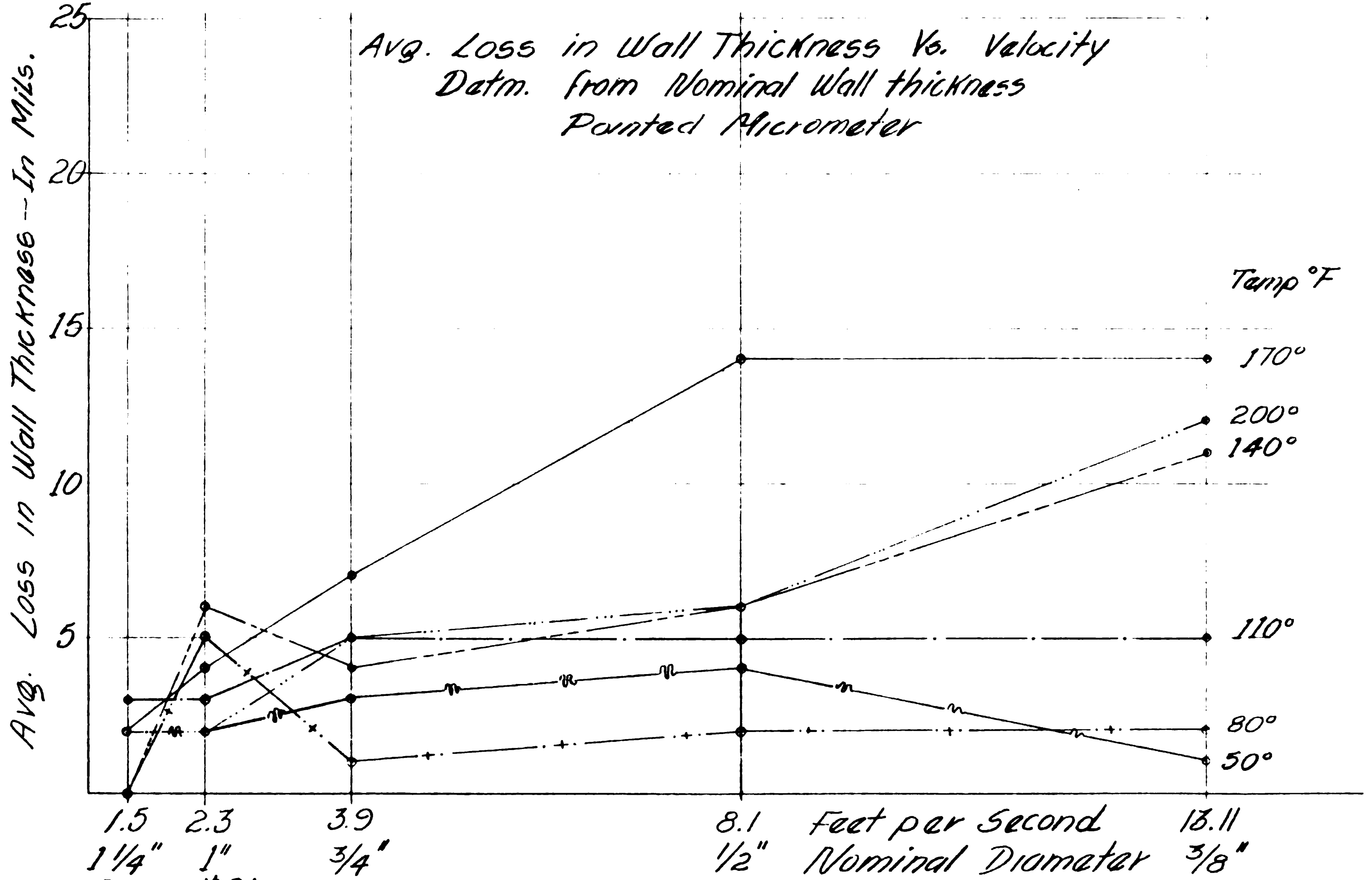

Avg. Loss in Wall Thickness Vs. Velocity
Detm. from Nominal Wall thickness
Pointed Micrometer
Avg. Loss in Wall Thickness -- In Mils.
25
20
15
10
5
Temp °F
170°
200°
140°
110°
80°
50°
1.5
2.3
3.9
8.1
Feet per Second
18.11
1 1/4"
1"
3/4"
1/2"
Nominal Diameter
3/8"

Figure #21

A correlating type of data was obtained by making a series of readings in each of several longitudinally marked segments of the tubing. These data have been recorded elsewhere.[11] No consistent trend in slope was indicated and the data are in good agreement with that reported here.

Radiographic Procedure

For the radiographic observations, the tubing specimens were placed, corroded side up, on the surface of an x-ray film holder under the x-ray tube. The initial radiographic work was done in the hospital x-ray room, using Kodak Blue Brand medical x-ray film.

The first plates were made with an intensifying screen, since it was not known how much of the x-radiation was being absorbed by the copper sample. Upon development, it was found that the exposure of the plate was more than adequate as indicated by the heavy blackening. Subsequent reduction in power on the x-ray tube did not completely eliminate the blackening. As a result the screen was eliminated. The succeeding attempts resulted in quite satisfactory plates. In order not to interfere with the regular operations of the hospital, this procedure was continued using an instrument in the Electrical Engineering Department. The 150 KV Westinghouse industrial x-ray unit was put in operating condition and employed for the remainder of the radiographic procedure. Following preliminary test exposures, plates of reasonably good visual intensity were obtained.

Microphotometer measurements were made by placing the film between two sheets of cleaned spectrographic glass in the appropriate bracket of a Jaco comparator-microphotometer, located in the Agricultural Chemistry Department. The output of this instrument was fed to a Bristol recording galvanometer. The instrument was set so the 100 o/o transmission corresponded to the lightest portion of the film. It was impossible to obtain full scale deflection upon checking the darkest portion of the film.

Changes in exposure were made in an effort to remedy the situation, but films of suitable contrast could not be obtained. The use of a copper sulfate bath to minimize background[9] did not appreciably change the results. Finally a different film, Kodak Industrial Type M, was ordered on the basis of the work of Liebhafsky[5] and Champion.[4]

This film gave much clearer negatives and some microphotometer readings were successfully completed. There was, however, an indication of blackening around the edges and at the ends of the samples, due to reflected radiation. In an effort to eliminate this difficulty, the samples were placed close together, face down, with small lead pieces at the ends to block reflected x-rays. (Figure 22)

The first step wedge used for comparative purposes was made from eight small leaves of 0.008" copper, soldered together on the edges to give a wedge with a range of 0.064 to 0.008". It was noted that the depth of steps was too great and the edges of the wedge allowed reflected x-rays to interfere, thus giving greater blackening.

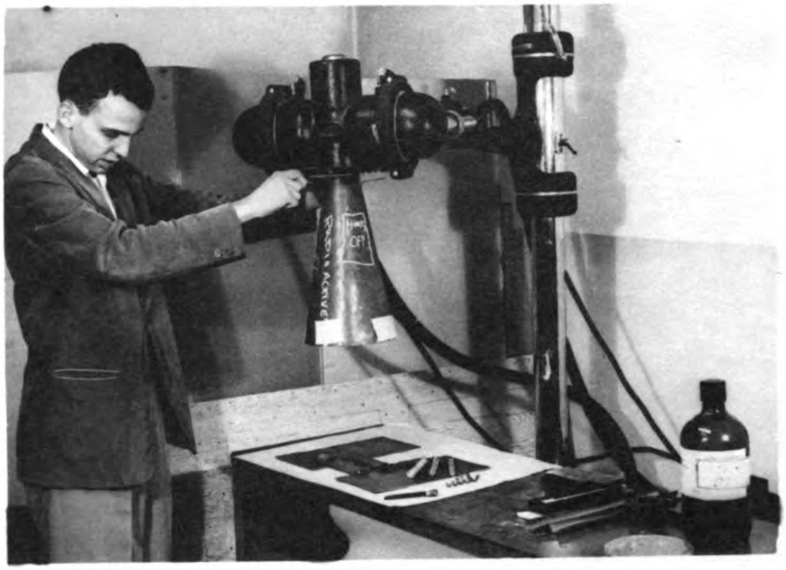
A new wedge was made from a one inch wide, 1/4 inch copper strip by milling the center in 0.003" steps from 0.010 to 0.060". The steps were in the center of a wide strip to minimize reflected radiation.

Contact prints of the radiographs are shown in Figures 23-27.

Figure 22.

Preparation for

Radiography



Exposure information for the radiographs is indicated in Figure 28.

Table IX'

Step Wedge Thicknesses (inches)

1	0.059	10	0.035
2	.057	11	.032
3	.054	12	.029
4	.052	13	.026
5	.049	14	.023
6	.046	15	.020
7	.044	16	.017
8	.041	17	.013
9	.0385	18	.010

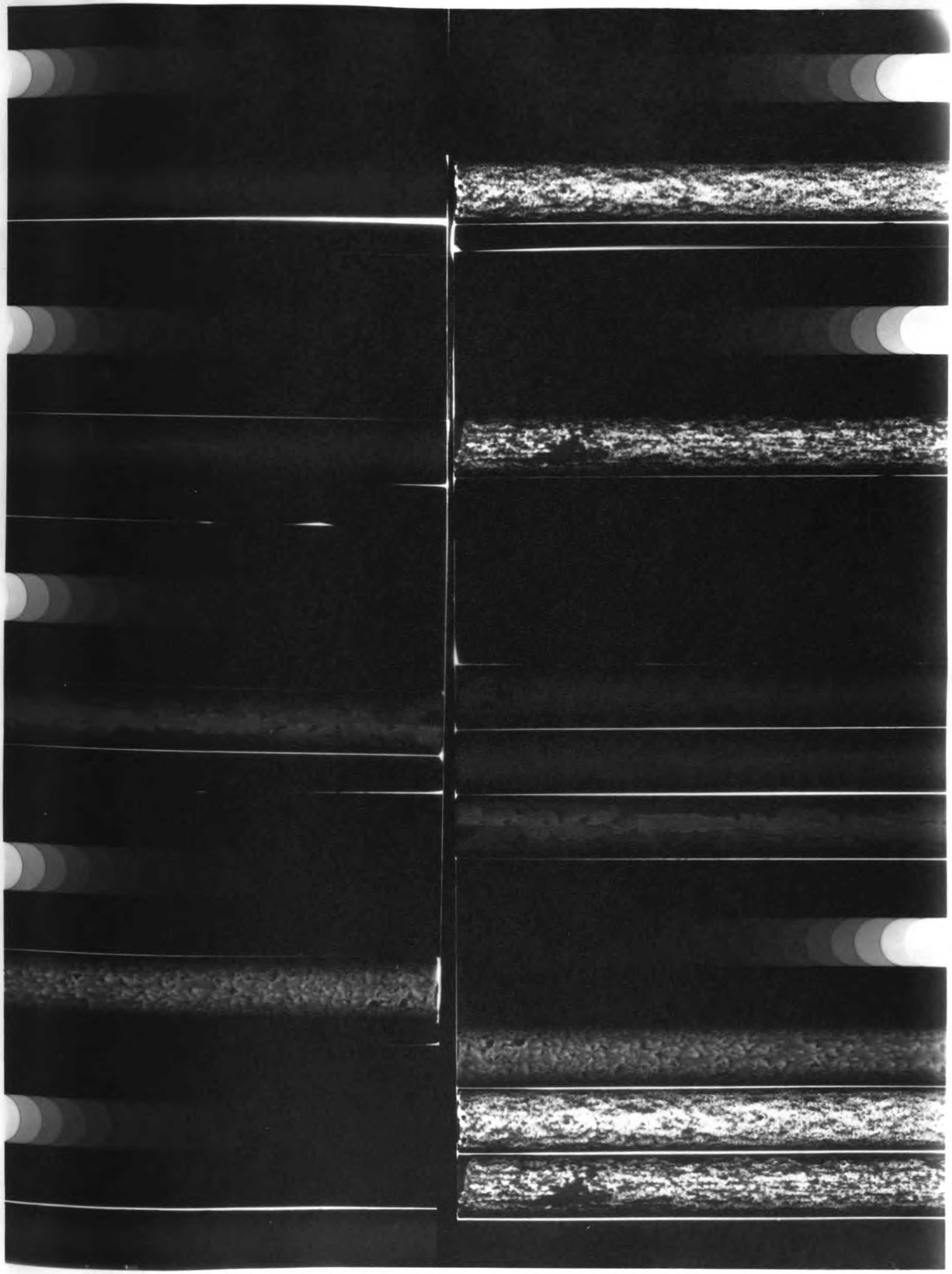


Figure 23. Radiograph of 3/8 inch Samples and Step Wedge (Plate U, page 70)

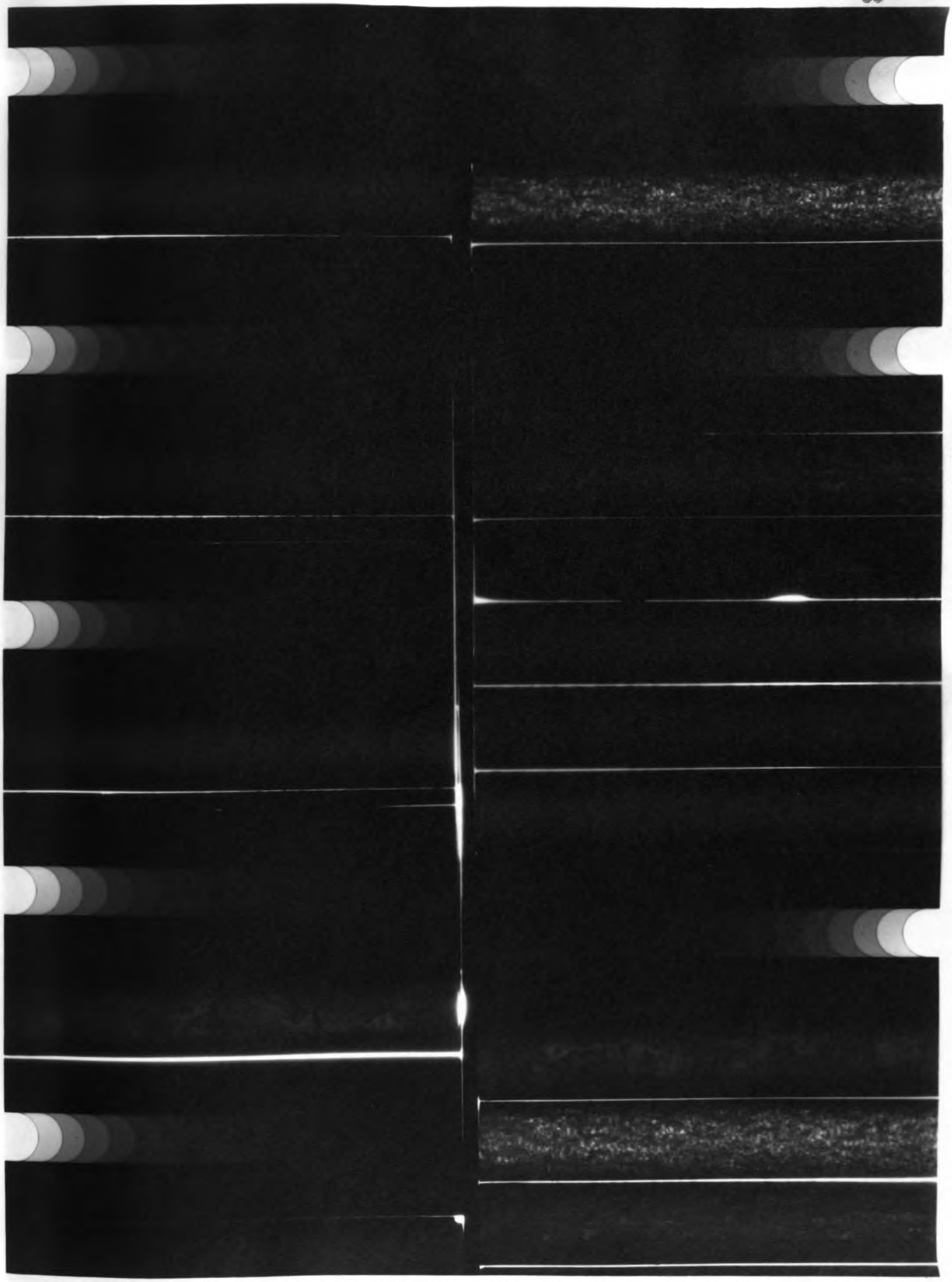


Figure 24. Radiograph of 1/2 inch Samples with Step Wedge (Plate V, page 70)

Figure 25. Radiograph of 3/4 inch Samples with Step Wedge (Plate W, page 70)

Figure 26. Radiograph of 1 inch Samples and Step Wedge (Plate X, page 70)

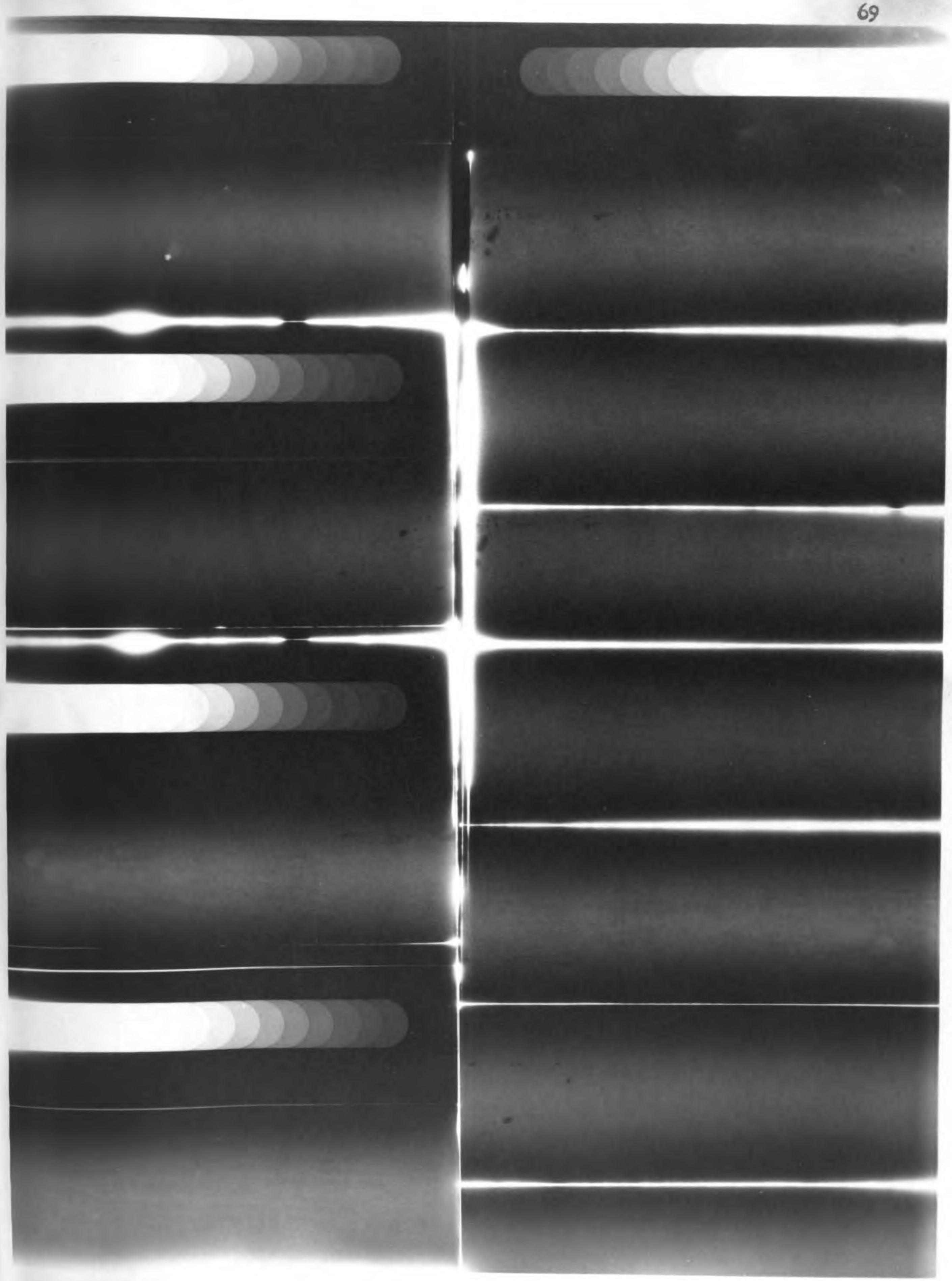


Figure 27. Radiograph of 1 1/4 inch Samples with Step Wedge (Plate Y, page 70)

50° Sample and Wedge	170° Sample and Wedge
80° Sample and Wedge	200° Sample and Wedge
110° Sample and Wedge	ALL SIX 3/8" SAMPLES AND WEDGE
140° Sample and Wedge	
New Tubing and Wedge	

PLATE "U" 3/8" TUBING
Exposure Time 30 Secds.

50° Sample and Wedge	140° Sample and Wedge
80° Sample and Wedge	200° Sample
110° Sample and Wedge	ALL SIX 1" SAMPLES <u>NO</u> WEDGE
170° Sample and Wedge	
New Tubing	

PLATE "X" 1" TUBING
Exposure Time 135 Secds.

50° Sample and Wedge	170° Sample and Wedge
80° Sample and Wedge	200° Sample and Wedge
110° Sample and Wedge	ALL SIX 1/2" SAMPLES AND WEDGE
140° Sample and Wedge	
New Tubing and Wedge	

PLATE "V" 1/2" TUBING
Exposure Time 45 Secds.

50° Sample and Wedge	170° Sample and Wedge
80° Sample and Wedge	200° Sample and Wedge
110° Sample and Wedge	ALL 1¼" SAMPLES EXCEPT 200°
140° Sample and Wedge	

PLATE "Y" 1¼" TUBING
Exposure Time 165 Secds.

50° Sample and Wedge	170° Sample and Wedge
80° Sample and Wedge	200° Sample and Wedge
110° Sample and Wedge	ALL SIX 3/4" SAMPLES <u>NO</u> WEDGE
140° Sample and Wedge	
New Tubing	

KEY

TO EXPOSURES OF RADIOGRAPHS

NOTE: All exposures at 50 KV and 15 ma with 24" plate to target distance

← PLATE "W" 3/4" TUBING
Exposure Time 75 Secds.

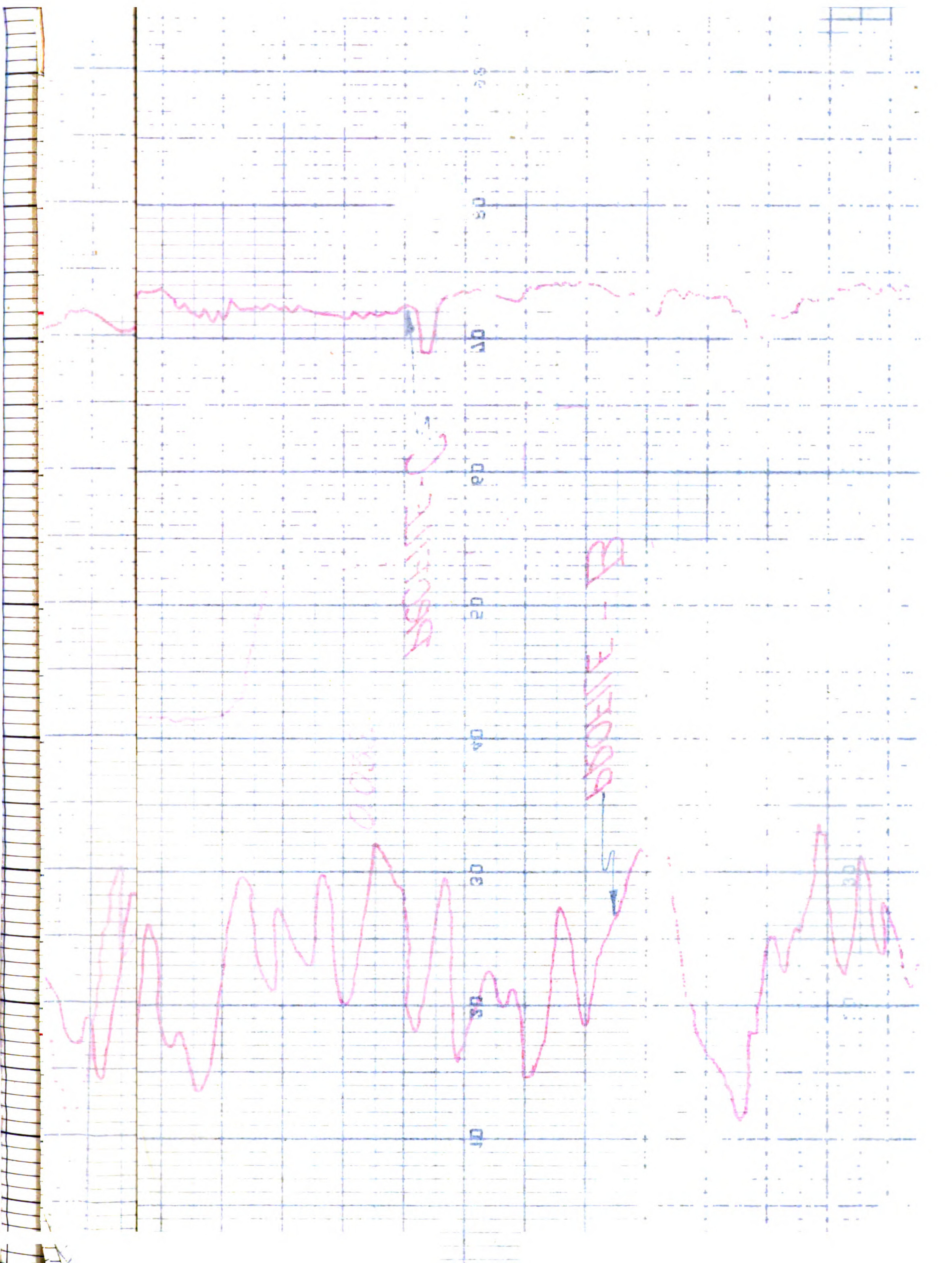
Figure #28

Microphotometer Charts

The radiograph corresponding to each sample was placed in the microphotometer as indicated above. The film sample moved in the instrument at 5 millimeters per minute and the slit light source was 80 microns by 1 millimeter. Tracings were made showing a cross-sectional profile of each corroded specimen, and of the step wedge radiographed with it. The thickness, in inches, of each step of the wedge had been measured previously (Table IX') and was recorded on the chart. From this standard, the maximum, minimum and average wall thickness values of the tubing were determined. (Table IX)

A microphotometer tracing (Figure 29) showing a 1/2 inch tube at 170°F, the corresponding step wedge and a new tubing section, illustrates the method.

The maximum (or pit), minimum (or island) and average thicknesses of metal lost by corrosion, as determined by comparison of these measured values with theoretical and average measured values of initial tubing wall thicknesses, are listed in Table X.


PROFILE - C
PROFILE - B


30
20
10

Radiographic-Microphotometer Measurements

Table IX includes the island, pit and average wall thickness measurements of the corroded tubing as observed from the microphotometer tracings obtained from corresponding radiographs. The form of each tracing is noted by a single descriptive word for a) the average corrosion and b) the degree of fluctuation representing the roughness of corrosion.

Table IX

3/8	Average	Average Island	Maximum Island	Average Pit	Minimum Pit	Notes a)	b)
50°F	0.033	0.034	0.044	0.031	0.030	even	little
80°	.031	.032	.033	.030	.028	even	little
110°	.028	.031	.036	.027	.025	even	some eff. end
140°	.025	.028	.030	.022	.020	even	wide
170°	.021	.025	.027	.015	.013	uneven	wide
200°	.025	.028	.031	.013	.016	uneven	some
1/2							
50°F	.036	.038	.039	.034	.033	even	little
80°	.039	.040	.042	.037	.036	even slope	very little
110°	.034	.036	.033	.032	.030	even	little
140°	.033	.037	.039	.030	.029	uneven	wide
170°	.024	.026	.030	.020	.018	even	wide
200°	.033	.034	.035	.031	.028	uneven	some

NOTE. -- Above "average, island or pit" values are estimated.

3/4	Average	Average Island	Maximum Island	Average Pit	Minimum Pit	Notes a)	b)
50°F	0.043	0.044	0.044	0.042	0.041	even	little
80°	.045	.045	.045	.043	.042	even slope	very little
110°	.040	.041	.042	.039	.035	even	little
140°	.041	.042	.043	.040	.033	uneven	some
170°	.039	.040	.041	.038	.036	even	some
200°	.042	.043	.044	.041	.039	even	some
1							
50°F	.043	.049	.050	.046	.045	even	very little
80°	.045	.046	.046	.045	.044	even	very little
110°	.048	.049	.049	.047	.046	even	some
140°	.046	.047	.047	.046	.045	even	some
170°	.047	.047	.048	.046	.046	even	some
200°	.050	.050	.051	.049	.049	uneven	some
1 1/4							
50°F	.055	.055	.056	.054	.053	uneven	little
80°	.056	.056	.057	.053	.052	even	some
110°	.051	.052	.053	.050	.049	uneven	some
140°	.053	.053	.054	.052	.052	even	little
170°	.054	.054	.055	.053	.053	even	some
200°	.053	.053	.054	.053	.052	even	little

Table X

Depth of Metal Lost by Corrosion (inches)

Nom. Tub. Dia.	Initial Wall Thickness		50°F			80°			110°			140°			170°			200°		
			Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)	Avg.	Isl. (Min)	Pit (Max)
3/8	+ Tol	.039	.006	.005	.008	.008	.007	.009	.011	.008	.012	.014	.011	.017	.018	.012	.024	.014	.011	.021
	Theor	.035	.002	.001	.004	.004	.003	.005	.007	.004	.008	.010	.007	.013	.014	.008	.020	.010	.007	.017
	Meas	.034	.001	.000	.003	.003	.002	.004	.006	.003	.007	.009	.006	.012	.013	.007	.019	.009	.006	.016
1/2	+ Tol	.044	.008	.006	.010	.005	.004	.007	.010	.008	.012	.011	.007	.014	.020	.018	.024	.011	.010	.013
	Theor	.040	.004	.002	.006	.001	.000	.003	.006	.002	.008	.007	.003	.010	.016	.014	.020	.007	.006	.009
	Meas	.039	.003	.001	.005	.000	-.001	.002	.005	.001	.007	.006	.002	.009	.015	.013	.019	.006	.005	.008
3/4	+ Tol	.049	.006	.005	.007	.004	.004	.006	.009	.008	.010	.008	.007	.009	.010	.009	.011	.007	.006	.008
	Theor	.045	.002	.001	.003	.000	.000	.002	.005	.004	.006	.004	.003	.005	.006	.005	.007	.003	.002	.004
	Meas	.044	.001	.000	.002	-.001	-.001	.001	.004	.003	.005	.003	.002	.004	.005	.004	.006	.002	.001	.003
1	+ Tol	.054	.006	.005	.008	.009	.008	.009	.006	.005	.007	.007	.007	.008	.008	.007	.008	.004	.004	.005
	Theor	.050	.002	.001	.004	.005	.004	.005	.002	.001	.003	.003	.003	.004	.004	.003	.004	.000	.000	.001
	Meas	.049	.001	.000	.003	.004	.003	.004	.001	.000	.002	.002	.002	.003	.003	.002	.003	-.001	-.001	.000
1 1/4	+ Tol	.059	.004	.004	.005	.003	.003	.006	.008	.007	.009	.006	.006	.007	.005	.005	.006	.006	.006	.006
	Theor	.055	.000	.000	.001	-.001	-.001	.002	.004	.003	.005	.002	.002	.003	.001	.001	.002	.002	.002	.002
	Meas	.055	.000	.000	.001	-.001	-.001	.002	.004	.003	.005	.002	.002	.003	.001	.001	.002	.002	.002	.002

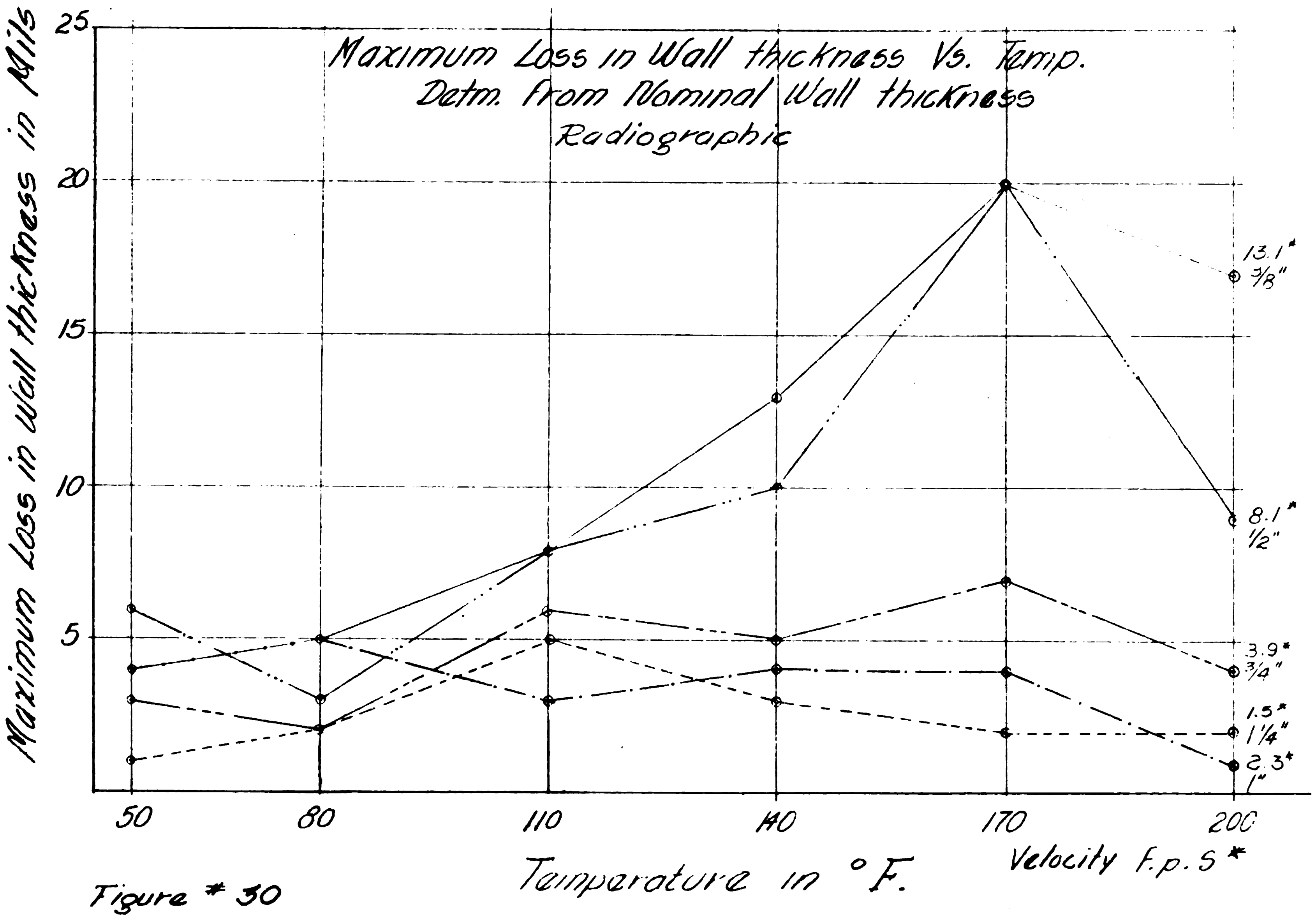

Maximum Loss in Wall thickness Vs. Temp.
Detm. from Nominal Wall thickness
Radiographic
Maximum Loss in Wall thickness in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F.
Velocity F.p.S *
13.1*
3/8"
8.1*
1/2"
3.9*
3/4"
1.5*
1 1/4"
2.3*
1"

Figure * 30

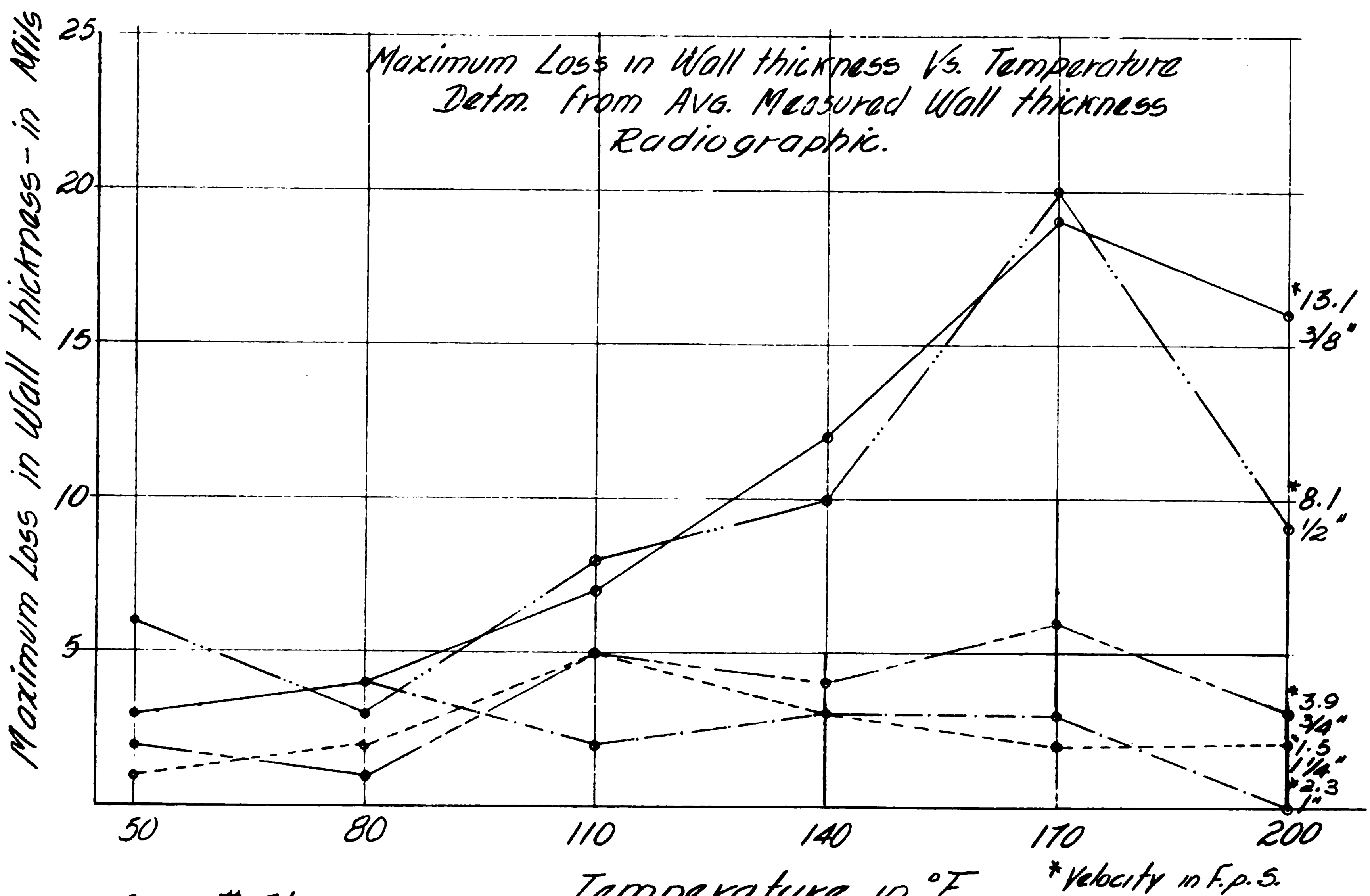
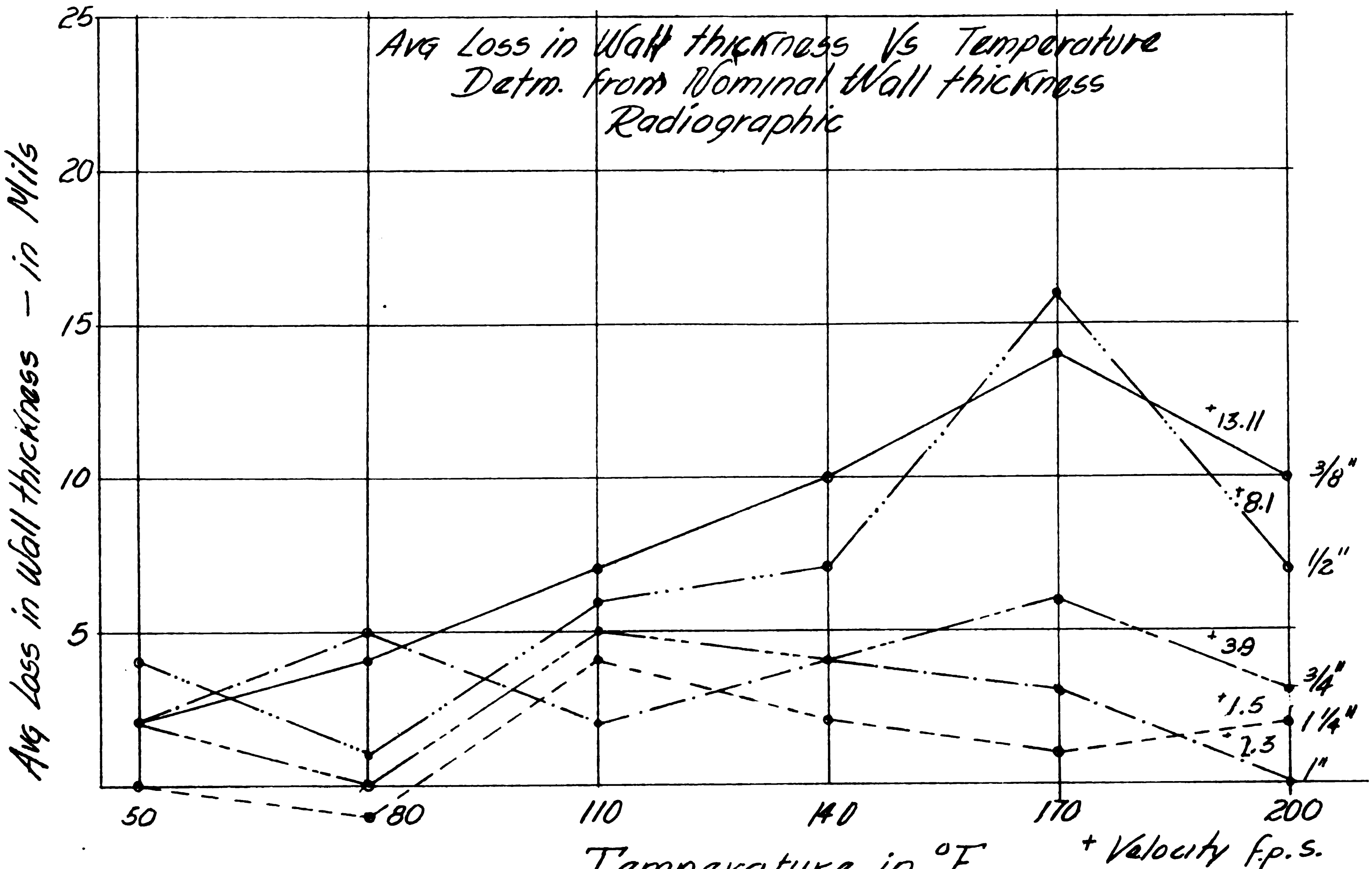

Maximum Loss in Wall thickness Vs. Temperature
Detm. from Avg. Measured Wall thickness
Radiographic.
Maximum Loss in Wall thickness - in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F
*13.1
3/8"
*8.1
1/2"
*3.9
3/4"
*1.5
1 1/4"
*2.3
1"
*Velocity in F.p.S.

Figure # 31



Avg Loss in Wall thickness Vs Temperature
Detm. from Nominal Wall thickness
Radiographic
Avg Loss in Wall thickness — in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F.
+ Velocity f.p.s.
+13.11
+8.1
+3.9
+1.5
+2.3
3/8"
1/2"
3/4"
1 1/4"
1"

Figure # 32

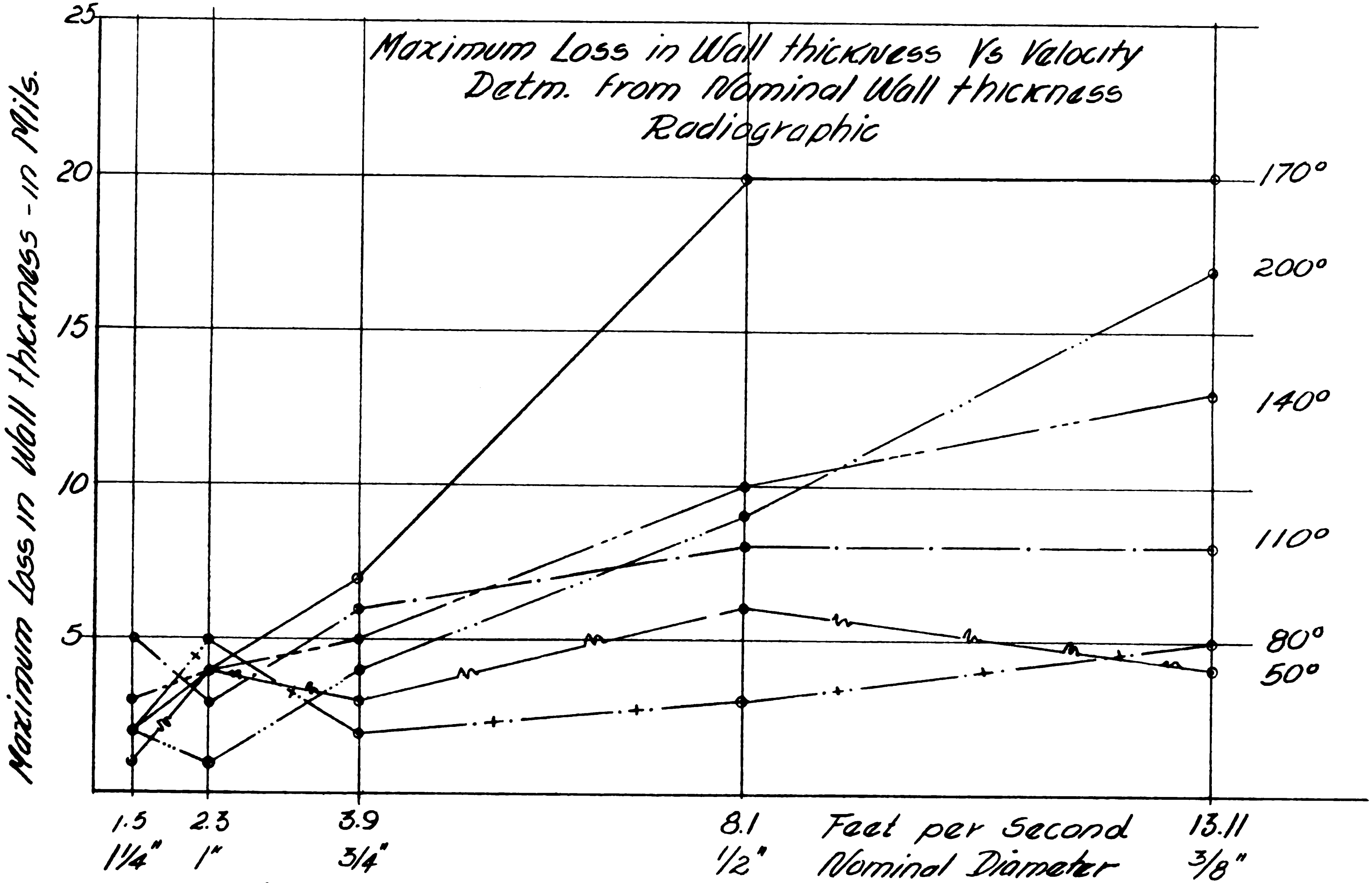
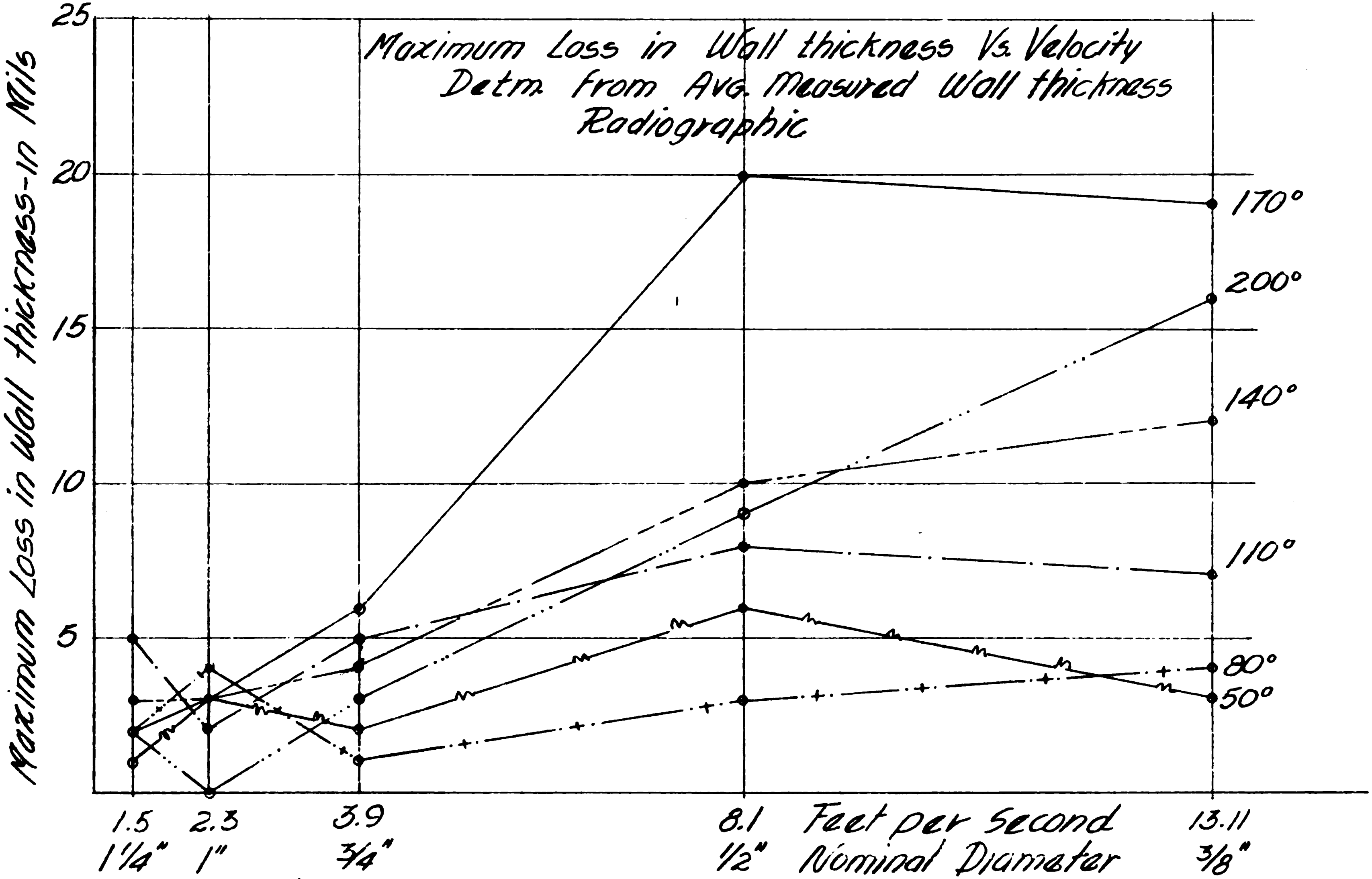

Maximum Loss in Wall thickness Vs Velocity
Detm. from Nominal Wall thickness
Radiographic
Maximum Loss in Wall thickness - in Mils.
25
20
15
10
5
1.5
1 1/4"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Feet per Second
Nominal Diameter
170°
200°
140°
110°
80°
50°

Figure # 33


Maximum Loss in Wall thickness Vs. Velocity
Detm. from Avg. Measured Wall thickness
Radiographic
Maximum Loss in Wall thickness-in Mils
25
20
15
10
5
170°
200°
140°
110°
80°
50°
1.5
1¼"
2.3
1"
3.9
¾"
8.1
½"
13.11
⅜"
Feet per Second
Nominal Diameter
Figure # 34

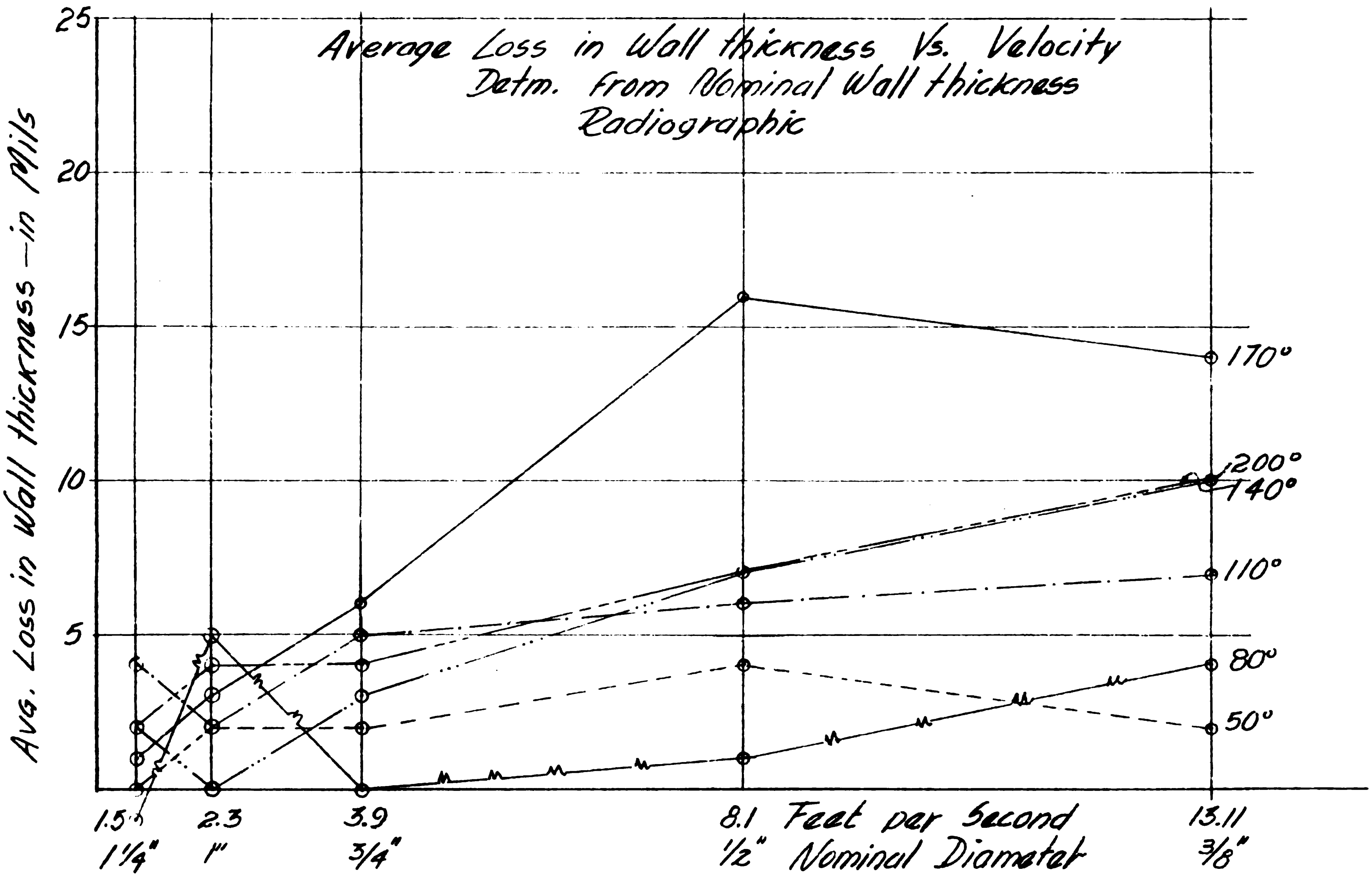

Average Loss in Wall thickness Vs. Velocity
Detm. from Nominal Wall thickness
Radiographic
Avg. Loss in Wall thickness —in Mils
25
20
15
10
5
170°
200°
140°
110°
80°
50°
1.5
1¼"
2.3
1"
3.9
¾"
8.1 Feet per Second
½" Nominal Diameter
13.11
⅜"

Figure # 35

Evaluation of Corrosion by Weight Loss

To evaluate the corrosion by loss of weight, unsectioned tubing was cut into three-inch lengths with a tubing cutter. New tubing of the same nominal diameter and make as the corroded specimens was used to obtain samples for "original" weight. This procedure was undertaken because the tubing was made to the same specifications as that originally used.

Two specimens were cut from each of the corroded sections and three from the new tubing sections. These were weighed on an analytical balance before and after cleaning. Cleaning operations involved immersing for twenty minutes in a 5 o/o potassium dichromate-sulfuric acid bath, rinsing with distilled water and finally rinsing with analytical grade ethyl acetate.

The lengths of these samples were determined by averaging four vernier caliper measurements. The average weight loss in grams per centimeter was determined as the difference between the average weight per centimeter of the new and the corroded tubing samples. A similar comparison was also made between the corroded tubing and the theoretical weight of new tubing according to specifications. Allowance was made for the slight loss due to the cleaning process. Results are included in Table XI.

The average wall thicknesses remaining (Table XII') were calculated from the average weights, using the relationship of the theoretical wall thickness to the theoretical weight. As an example, for the 3/8 inch sample at 50°F

$$\frac{.035}{2.947} = \frac{\text{average wall thickness}}{2.635}$$

then average wall thickness is 0.031 inches.

In Table XII' is listed the loss of copper by corrosion expressed as inches of wall thickness for the "Theoretical Loss", "Measured Loss", and "Inches per Year Loss". These data were obtained by comparing the "Remaining" wall thickness values with those for new and unused tubing expressed as theoretical and average measured wall thicknesses.

Table XI a

Gravimetric Procedure Summary

50°F	1/3" A_1	1/3" A_2	1/2" A_1	1/2" A_2	3/4" A_1	3/4" A_2	1" A_1	1" A_2	1 1/4" A_1	1 1/4" A_2
Total weight (grams)										
Before Cleaning	20.4393	20.7953	29.6313	29.5520	49.6683	49.6035	71.0323	70.9258	99.3049	98.4347
After Cleaning	20.3033	20.6265	29.3493	29.3978	49.4755	49.3319	70.6027	70.4743	98.7527	97.9525
Cleaning Loss	0.1305	0.1688	0.2315	0.1542	0.1923	0.2266	0.4301	0.4515	0.5522	0.5720
Total Length (inches)	3.035 3.032 3.040 3.024	3.090 3.075 3.036 3.041	3.050 3.040 3.054 3.032	3.055 3.057 3.056 3.049	3.059 3.054 3.052 3.045	3.078 3.075 3.042 3.045	3.077 3.074 3.055 3.043	3.055 3.055 3.061 3.034	3.075 3.079 3.033 3.063	3.053 3.035 3.033 3.025
Average (inches)	3.033	3.033	3.044	3.054	3.053	3.065	3.064	3.051	3.075	3.038
(centimeters)	7.703	7.331	7.732	7.758	7.753	7.735	7.781	7.750	7.811	7.716
Weight (g/cm)	2.636	2.634	3.796	3.789	6.331	6.343	9.073	9.093	12.643	12.694
Average	2.635		3.793		6.362		9.083		12.669	

Table XI b

30°F	3/8" A_s	3/8" A_a	1/2" A_s	1/2" A_a	3/4" A_s	3/4" A_a	1" A_s	1" A_a	1 1/4" A_s	1 1/4" A_a
Total Weight (grams)										
Before Cleaning	22.0307	21.9334	31.5597	31.8559	52.0495	51.6041	71.4477	72.5333	101.6309	101.1842
After Cleaning	21.9234	21.8214	31.3846	31.7089	51.8237	51.3939	71.1265	72.2195	101.1657	100.7145
Cleaning Loss	0.1073	0.1120	0.1751	0.1470	0.2208	0.2102	0.3212	0.3133	0.4652	0.4697
Total Length (inches)	3.050 3.073 3.079 3.059	3.061 3.055 3.043 3.029	3.080 3.077 3.050 3.045	3.075 3.075 3.082 3.062	3.075 3.075 3.053 3.069	3.056 3.070 3.050 3.025	3.000 3.014 3.016 3.006	3.075 3.063 3.066 3.041	3.081 3.082 3.069 3.060	3.075 3.073 3.064 3.052
Average (inches)	3.065	3.047	3.063	3.074	3.076	3.050	3.009	3.063	3.073	3.066
(centimeters)	7.786	7.739	7.780	7.807	7.812	7.748	7.643	7.779	7.813	7.733
Weight (g/cm)	2.815	2.819	4.034	4.061	6.634	6.633	9.306	9.284	12.940	12.932
Average	2.817		4.048		6.634		9.295		12.936	

Table XI c

110°F	3/8"		1/2"		3/4"		1"		1 1/4"	
	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2
Total Weight (grams)										
Before Cleaning	18.8658	19.1359	28.0291	27.9435	46.4395	46.7223	70.1546	69.3536	98.7940	97.5495
After Cleaning	18.7468	19.0260	27.8942	27.7962	46.2097	46.5099	69.8746	69.0696	98.4419	97.1815
Cleaning Loss	0.1190	0.1099	0.1349	0.1473	0.2298	0.2124	0.2800	0.2840	0.3521	0.3680
Total Length (inches)	3.044 3.045 3.045 3.039	3.064 3.070 3.075 3.053	3.044 3.032 3.033 3.013	3.036 3.032 3.023 3.012	3.050 3.054 3.051 3.007	3.070 3.061 3.048 3.040	3.086 3.060 3.060 3.047	3.023 3.016 3.044 3.038	3.094 3.078 3.075 3.040	3.031 3.032 3.039 3.034
Average (inches)	3.043	3.066	3.031	3.026	3.041	3.055	3.063	3.030	3.072	3.034
(centimeters)	7.730	7.786	7.698	7.686	7.723	7.759	7.781	7.697	7.802	7.706
Weight (g/cm)	2.425	2.443	3.623	3.616	5.983	5.994	8.980	8.973	12.616	12.610
Average	2.434		3.620		5.989		8.977		12.608	

Table XI d

1407	3/8"		1/2"		3/4"		1"		1 1/4"	
	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2
Total Weight (grams)										
Before Cleaning	14.7693	14.7500	27.6704	27.6546	44.7464	44.5546	67.6917	66.5659	93.1832	94.9342
After Cleaning	14.7047	14.6392	27.5916	27.5644	44.5863	44.3933	67.5036	66.3756	93.9455	94.7680
Cleaning Loss	0.0646	0.0603	0.0343	0.0332	0.1601	0.1613	0.1881	0.1903	0.2377	0.2162
Total Length (inches)	3.042 3.051 3.052 3.029	3.047 3.045 3.045 3.024	3.057 3.056 3.052 3.023	3.036 3.032 3.050 3.032	3.060 3.055 3.040 3.028	3.059 3.051 3.050 3.016	3.059 3.072 3.034 3.043	3.004 3.001 3.020 3.020	3.064 3.065 3.061 3.030	2.960 2.950 2.956 2.938
Average (inches)	3.044	3.040	3.047	3.038	3.043	3.044	3.066	3.011	3.055	2.951
(centimeters)	7.731	7.722	7.739	7.715	7.741	7.318	7.787	7.649	7.760	7.496
Weight (g/cm)	1.902	1.902	3.565	3.572	5.759	5.741	8.668	8.673	12.751	12.643
Average	1.902		3.564		5.750		8.669		12.687	

Table II e

170°F	3/8"		1/2"		3/4"		1"		1 1/4"	
	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2
Total Weight (grams)										
Before Cleaning	12.7146	12.8452	21.8208	22.0785	44.6076	45.3644	67.2268	67.1445	96.5546	97.3483
After Cleaning	12.6269	12.7596	21.7137	21.9652	44.4035	45.1704	66.9789	66.8793	96.2507	97.0128
Cleaning Loss	0.0377	0.0856	0.1011	0.1133	0.1991	0.1940	0.2479	0.2652	0.3039	0.3360
Total Length (inches)	3.036 3.025 3.033 3.024	3.040 3.050 3.048 3.038	3.018 2.999 2.987 2.990	3.031 3.041 3.040 3.020	3.022 3.034 3.032 3.015	3.090 3.077 3.076 3.051	3.064 3.065 3.063 3.033	3.069 3.040 3.040 3.020	3.045 3.055 3.053 3.043	3.069 3.066 3.072 3.044
Average (inches)	3.030	3.044	2.999	3.033	3.026	3.074	3.056	3.042	3.049	3.063
(centimeters)	7.695	7.732	7.616	7.704	7.686	7.807	7.763	7.727	7.745	7.780
Weight (g/cm)	1.640	1.650	2.851	2.851	5.778	5.786	8.627	8.654	12.428	12.470
Average	1.645		2.851		5.782		8.641		12.449	

Table II f

200°F	3/8"		1/2"		3/4"		1"		1 1/4"	
	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2	A_1	A_2
Total weight (grams)										
Before Cleaning	14.7541	14.7236	23.2375	23.5643	47.0457	46.7373	71.4633	71.9536	97.3173	98.6155
After Cleaning	14.6077	14.5743	23.0762	23.3969	46.7313	46.4614	71.1612	71.6504	96.3793	98.1731
Cleaning Loss	0.1464	0.1543	0.1613	0.1674	0.2639	0.2759	0.3021	0.3032	0.4390	0.4424
Total Length (inches)	3.040 3.030 3.039 3.040	3.015 3.024 3.026 3.016	3.043 3.053 3.051 3.024	3.057 3.040 3.044 3.034	3.034 3.039 3.038 3.012	2.993 3.008 3.013 3.006	3.059 3.030 3.032 3.020	3.076 3.065 3.076 3.050	3.033 3.044 3.034 3.011	3.012 2.990 2.980 2.973
Average (inches)	3.037	3.020	3.044	3.044	3.031	3.003	3.035	3.067	3.032	2.989
(centimeters)	7.715	7.672	7.732	7.731	7.698	7.639	7.710	7.790	7.701	7.592
Weight (g/cm)	1.393	1.399	3.631	3.672	6.077	6.081	9.230	9.193	12.530	12.931
Average	1.396		3.652		6.079		9.214		12.756	

Table XI g

Gravimetric Procedure Summary

New Tubing

	Weight Average (Three Samples) grams	Length Average (Four Measurements on Three Samples) inches	centimeters	Weight per Length grams/centimeter	Wall Thickness inches
3/8	21.519	3.015	7.658	2.810	0.034
1/2	31.039	3.040	7.722	4.026	0.038
3/4	34.034	2.028	5.151	6.617	0.044
1	71.126	3.006	7.634	9.317	0.048
1 1/4	99.866	3.057	7.765	12.861	0.054

Table XII

Weight of Metal Lost by Corrosion

Tubing Diameter	Initial Weight g/cm		Weight Loss g/cm 50°	80°	110°	140°	170°	200°
3/8	+ Tolerance	3.094	0.459	0.277	0.650	1.192	1.449	1.198
	Theoretical	2.947	0.312	0.130	0.513	1.045	1.302	1.051
	− Tolerance	2.800	0.165	-0.017	0.366	0.898	1.155	0.904
	Measured	2.810	0.175	-0.007	0.376	0.908	1.165	0.914
1/2	+ Tolerance	4.453	0.660	0.405	0.833	0.889	1.602	0.801
	Theoretical	4.241	0.448	0.193	0.621	0.677	1.390	0.589
	− Tolerance	4.029	0.236	-0.019	0.409	0.465	1.177	0.377
	Measured	4.026	0.233	-0.022	0.406	0.462	1.175	0.374
3/4	+ Tolerance	7.110	0.749	0.476	1.121	1.360	1.328	1.131
	Theoretical	6.771	0.410	0.137	0.782	1.021	0.989	0.792
	− Tolerance	6.432	0.071	-0.202	0.443	0.682	0.650	0.353
	Measured	6.617	0.255	-0.017	0.628	0.867	0.835	0.538
1	+ Tolerance	10.235	1.152	0.940	1.252	1.566	1.694	1.021
	Theoretical	9.748	0.665	0.453	0.771	1.079	1.107	0.534
	− Tolerance	9.261	0.178	-0.034	0.284	0.592	0.620	0.047
	Measured	9.317	0.234	0.022	0.340	0.648	0.676	0.103
1 1/4	+ Tolerance	13.814	1.145	0.878	1.206	1.127	1.365	1.058
	Theoretical	13.156	0.487	0.220	0.548	0.469	0.707	0.400
	− Tolerance	12.498	-0.171	-0.438	-0.210	-0.189	0.049	-0.258
	Measured	12.361	0.192	-0.075	0.253	0.174	0.412	0.105

Table XII'

Depth of Metal Lost by Corrosion

Tubing Diameter	Wall Thickness	50°	80°	110°	140°	170°	200°
3/8	Remaining	0.031	0.033	0.030	0.023	0.019	0.022
	Theoretical Loss	.004	.002	.0[illegible]5	.012	.016	.013
	Measured Loss	.003	.001	.004	.011	.015	.012
	Inches per Year Loss	.0029	.0015	.0036	.0033	.0117	.0095
1/2	Remaining	.036	.038	.034	.034	.027	.034
	Theoretical Loss	.004	.002	.006	.006	.013	.006
	Measured Loss	.002	.000	.004	.004	.011	.004
	Inches per Year Loss	.0029	.0015	.0044	.0044	.0095	.0044
3/4	Remaining	.042	.044	.040	.038	.031	.040
	Theoretical Loss	.003	.001	.005	.007	.007	.005
	Measured Loss	.002	.000	.004	.006	.006	.004
	Inches per Year Loss	.0022	.0007	.0036	.0051	.0051	.0036
1	Remaining	.047	.048	.046	.044	.044	.047
	Theoretical Loss	.003	.002	.004	.006	.006	.003
	Measured Loss	.001	.000	.002	.004	.004	.001
	Inches per Year Loss	.0022	.0015	.0029	.0044	.0044	.0022
1 1/4	Remaining	.053	.054	.053	.053	.052	.053
	Theoretical Loss	.002	.001	.002	.002	.003	.002
	Measured Loss	.001	.000	.001	.001	.002	.001
	Inches per Year Loss	.0015	.0007	.0015	.0015	.0022	.0015

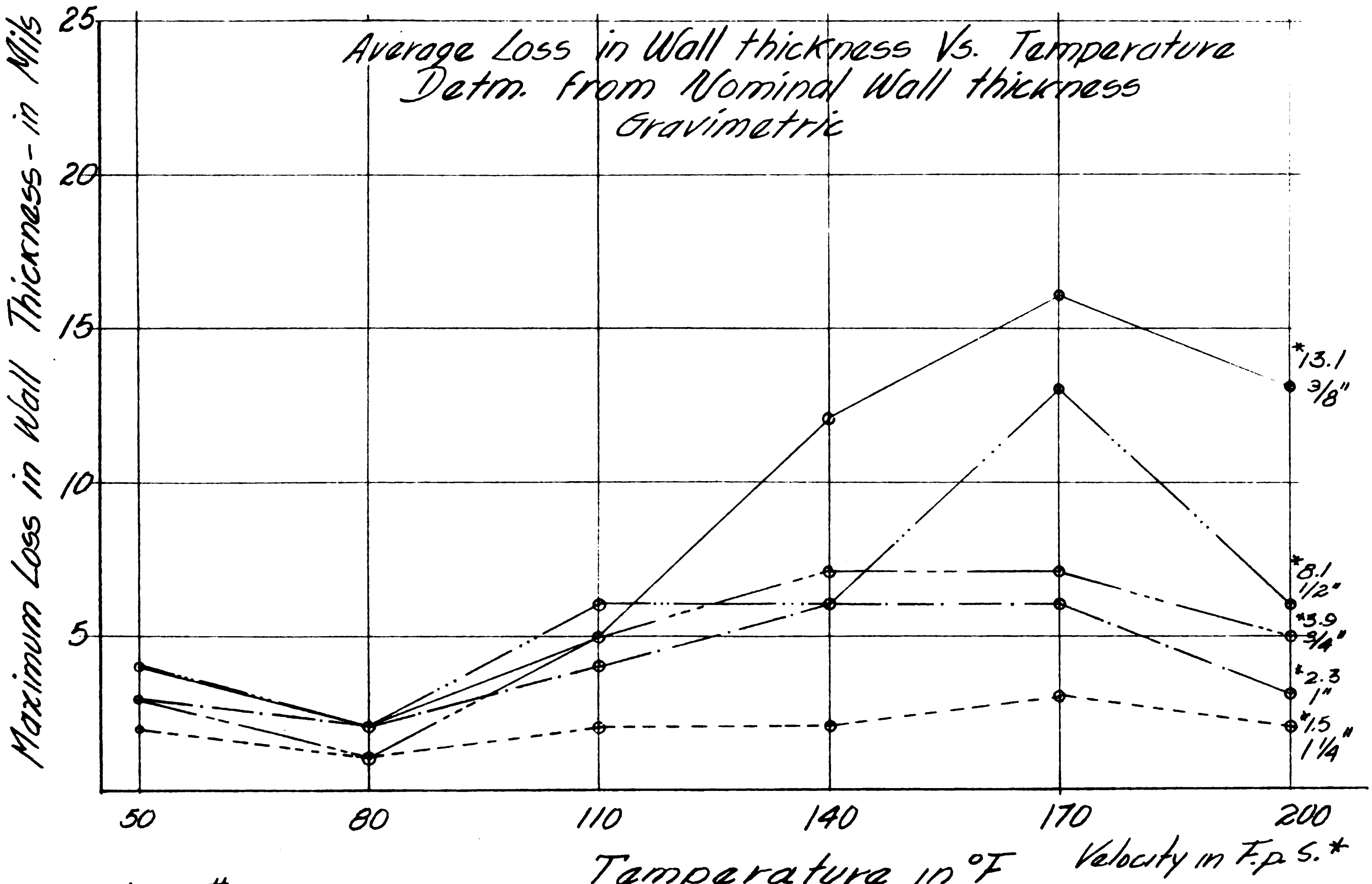

Average Loss in Wall thickness Vs. Temperature
Detm. from Nominal Wall thickness
Gravimetric
Maximum Loss in Wall Thickness - in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F
Velocity in F.p.S.*
*13.1
3/8"
*8.1
1/2"
*3.9
3/4"
*2.3
1"
*1.5
1 1/4"

Figure # 36

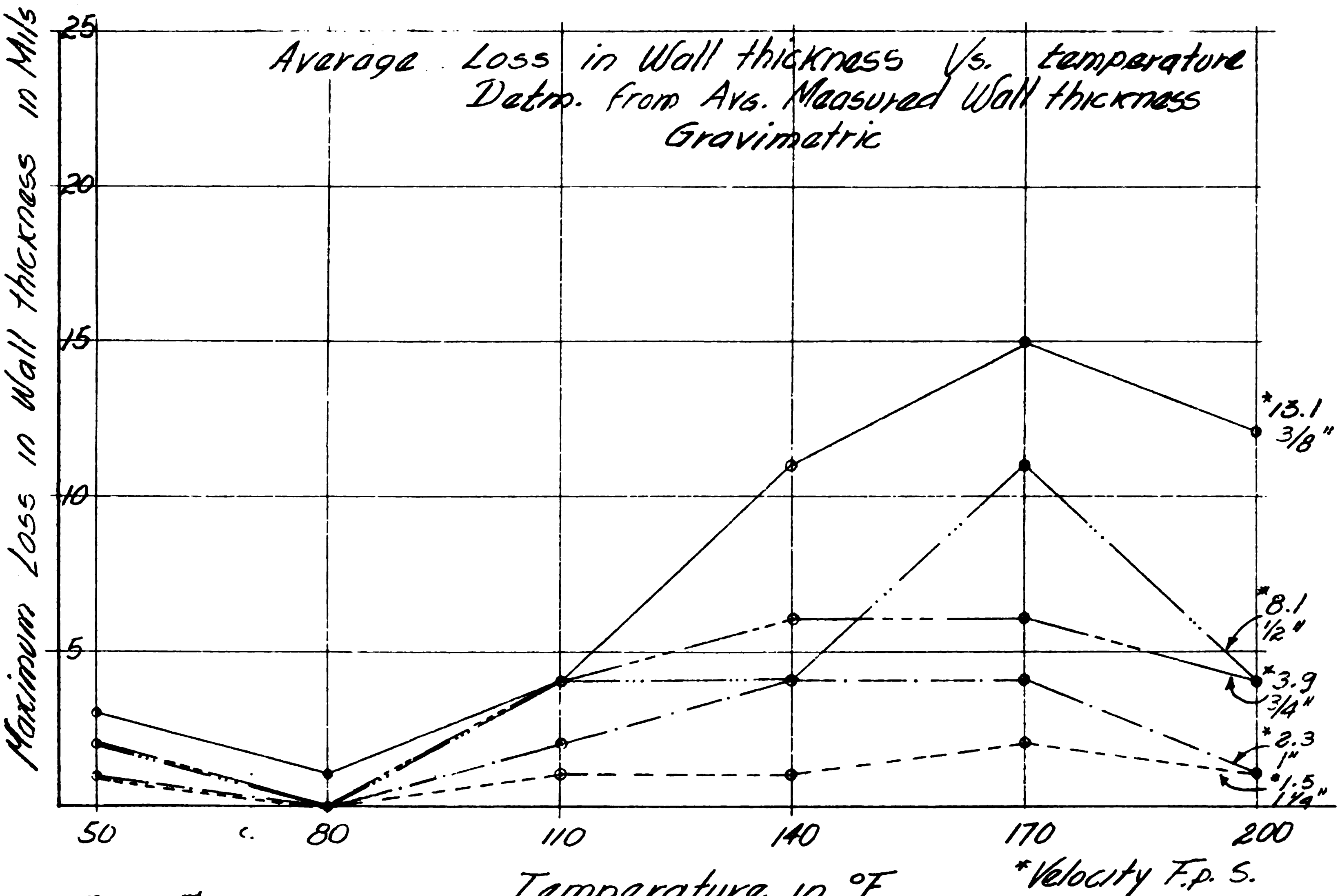
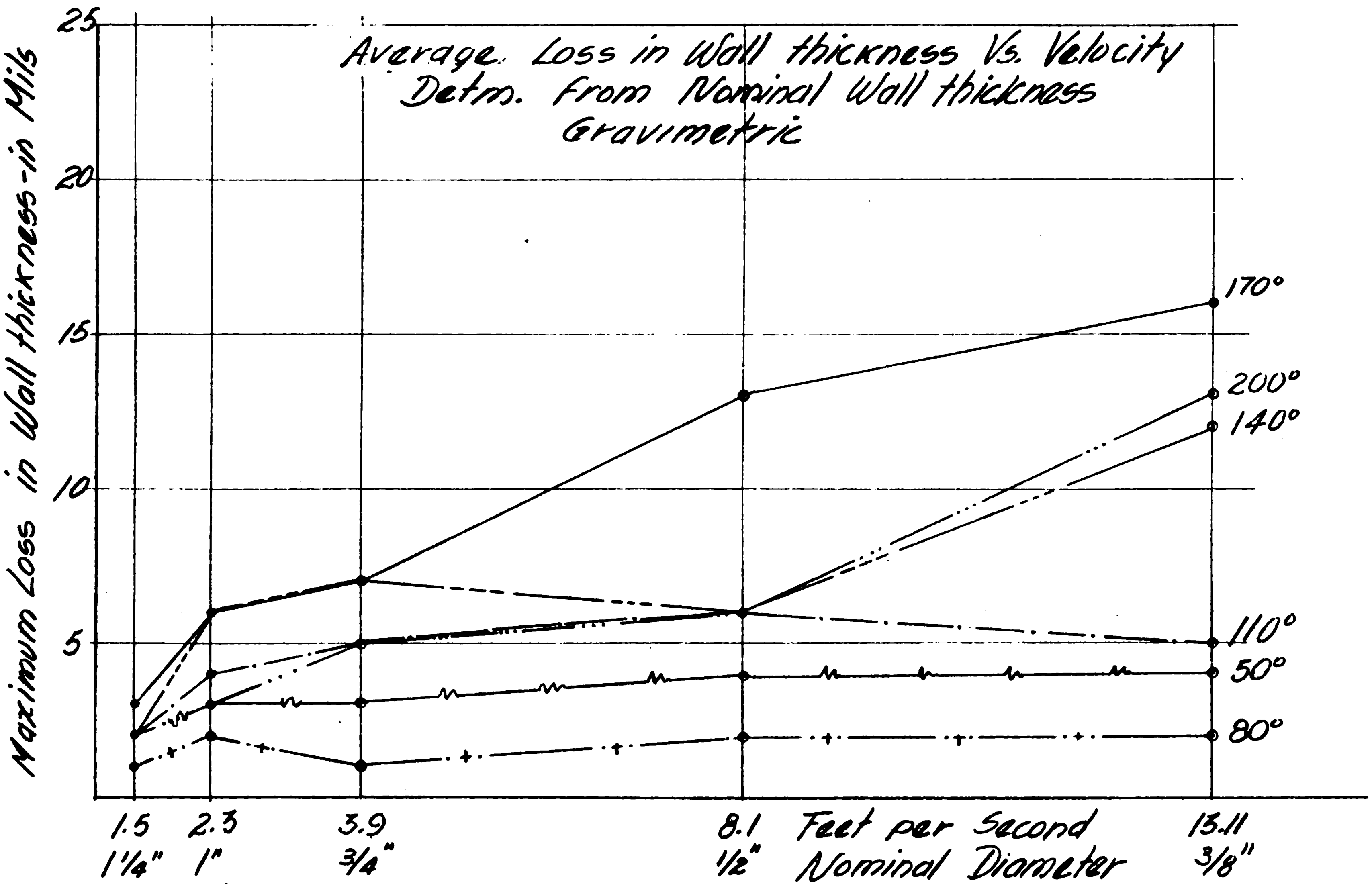
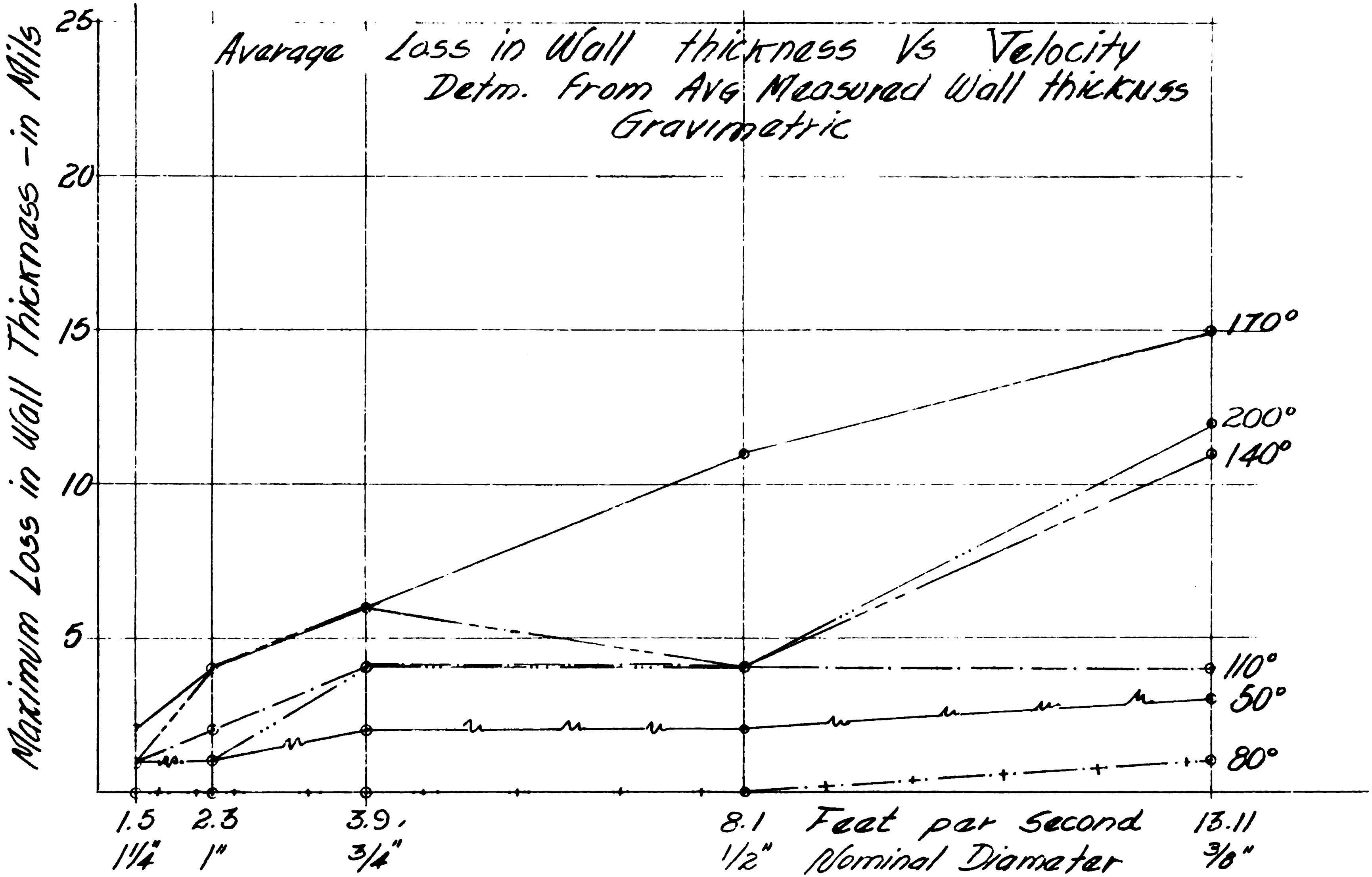

Average Loss in Wall thickness Vs. temperature
Detm. from Avg. Measured Wall thickness
Gravimetric
Maximum Loss in Wall thickness in Mils
25
20
15
10
5
50
80
110
140
170
200
Temperature in °F
*Velocity F.p.S.
*13.1
3/8"
*8.1
1/2"
*3.9
3/4"
*2.3
1"
*1.5
1 1/4"

Figure # 37



Average Loss in Wall thickness Vs. Velocity
Detm. From Nominal Wall thickness
Gravimetric
Maximum Loss in Wall thickness-in Mils
25
20
15
10
5
170°
200°
140°
110°
50°
80°
1.5
1¼"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Feet per Second
Nominal Diameter

Figure # 38



Avarage Loss in Wall thickness Vs Velocity
Detm. From Avg Measured Wall thickness
Gravimetric
Maximum Loss in Wall Thickness -in Mils
25
20
15
10
5
1.5
1¼"
2.3
1"
3.9
3/4"
8.1
1/2"
13.11
3/8"
Feet per Second
Nominal Diameter
170°
200°
140°
110°
50°
80°

Figure # 39

CONCLUSIONS
(EXPERIMENTAL I)

There is rather close agreement in results obtained by the four different methods of corrosion evaluation. (Table XIII) The effects on corrosion by changes in water temperature and velocity are indicated in the graphs. To give an indication of the imminence of failure, a set of graphs was drawn with the maximum loss in wall thickness plotted against velocity and against temperature. In a second set of graphs, the average loss in wall thickness was also plotted against velocity and against temperature. This second set of graphs, using average loss in wall thickness, was made in order that all four evaluation methods might be compared, as the gravimetric procedure yields only average values.

General Comments

Temperature Versus Loss in Wall Thickness Graphs
(Figures 10,11,12,16,17,18,30,31,32,36,37)

There is apparently a slight drop in the loss in wall thickness due to corrosion at 80°F, followed by a gradual increase as the temperature increases, with the maximum usually occurring prior to 200°F. This maximum is at 170°F in the 3/8, 1/2 and 3/4 inch tubing and at somewhat lower temperatures in the 1 and 1 1/4 inch tubing. It may be conjectured that the higher water velocities in the small diameter tubing may be preventing or hindering deposition of a protective coating, which would otherwise form at these temperatures, and that the coating probably formed more completely in the larger tubing.

Table XLII

Minimum Wall Thickness Remaining (Inches)

Nom. Diameter Tubing	Method of Measurement	50°	80°	110°	140°	170°	200°
3/8	Dial Mic.	.030	.029	.025	.020	.012	.015
	Point Mic.	.033	.031	.027	.021	.014	.016
	Radiographic	.031	.030	.027	.022	.015	.018
	Gravimetric	.031	.033	.030	.023	.019	.022
1/2	Dial Mic.	.035	.038	.032	.030	.020	.025
	Point Mic.	.035	.037	.033	.030	.021	.026
	Radiographic	.034	.037	.032	.030	.020	.031
	Gravimetric	.036	.038	.032	.032	.027	.032
3/4	Dial Mic.	.041	.041	.039	.038	.036	.035
	Point Mic.	.042	.042	.040	.039	.037	.037
	Radiographic	.042	.043	.039	.040	.033	.041
	Gravimetric	.042	.044	.040	.038	.033	.040
1	Dial Mic.	.048	.045	.046	.043	.044	.047
	Point Mic.	.047	.044	.046	.043	.044	.047
	Radiographic	.046	.045	.047	.046	.046	.049
	Gravimetric	.047	.043	.046	.044	.044	.047
1 1/4	Dial Mic.	.054	.054	.052	.053	.053	.053
	Point Mic.	.053	.054	.050	.053	.052	.053
	Radiographic	.054	.053	.050	.052	.053	.053
	Gravimetric	.053	.054	.053	.053	.052	.053

Velocity Versus Loss in Wall Thickness
(Figures 13,14,15,19,20,21,33,34,35,38,39)

As expected, the loss in wall thickness generally increases with velocity. This effect is much less pronounced in the low temperature range. It is only above 110°F that the velocity increase tends to influence the extent of corrosion. It is interesting to note that this is also the decomposition temperature range of several copper compounds assumed to be formed as the initial corrosion products.

Comparison of the Four Evaluation Methods

Comparison of the results of four methods of evaluation (Table XIII) shows that the average wall thickness remaining as determined gravimetrically differs considerably from the minimum remaining wall thickness in the 3/8 and 1/2 inch tubing from 140° through 200°F. This would indicate formation of several deep pits rather than extensive overall corrosion. This is borne out by examination of the radiographs of these samples.

Special Advantages of Evaluation Methods

The disadvantages of the various methods have been indicated in their respective discussions. It must be pointed out, however, that each method has certain advantages over the others. The micrometer procedures allow direct measurement of individual pits and probably consume less time than the other methods requiring conversions and calibrations of various instruments. The dial micrometer is much easier to read and contacts with consistant pressure, while the pointed micrometers can probably be handled more rapidly.

The radiographic procedure gives a clear, overall picture not obtainable by the other methods and is a permanent record for comparison. In addition, of course, the microphotometer tracings allow immediate determination of the extent of corrosion and present more of a physical picture of the severity and character of the corrosion. It is probable that this is the most comprehensive of the various evaluation methods. The number of operations involved necessitate use of considerable time and may allow a certain number of errors in addition to those inherent in measurements of any sort.

The gravimetric method, as employed in this evaluation, suffered in that the true original weights were not available. It is, however, a good method for general corrosion evaluation in that local pits and islands are averaged.

It must be noted that the agreement of results serves to indicate that the most rapid method, probably that with pointed micrometers, is sufficiently valid to be used alone in most instances. However, it is felt that the employment of several methods in conjunction better serves to evaluate the extent of the corrosion of tubing.

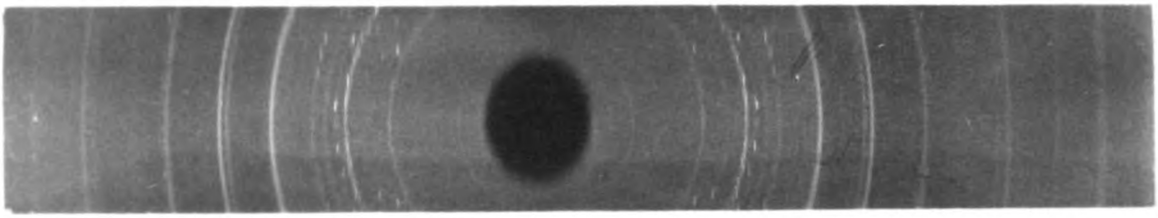
EXPERIMENTAL II — IDENTIFICATION OF DEPOSITS

Samples for powder pattern determinations in the early portion of this work were prepared by grinding the material with a boron carbide mortar and pestle and packing the resulting powder carefully into a 0.03 cm glass capillary. The capillary was then sealed with a match flame to give a sample tube of about one-half inch length. This tube was then mounted in a Phillips, Debye-Scherrer type, 11.4 cm camera. After alignment, the camera was loaded with Kodak No-Screen type film which had been cut and punched to fit the camera. The camera was then placed on the Norelco x-ray unit for exposure. All exposures were for 6 hours at 35 KV and 16 ma., using copper K-α radiation.

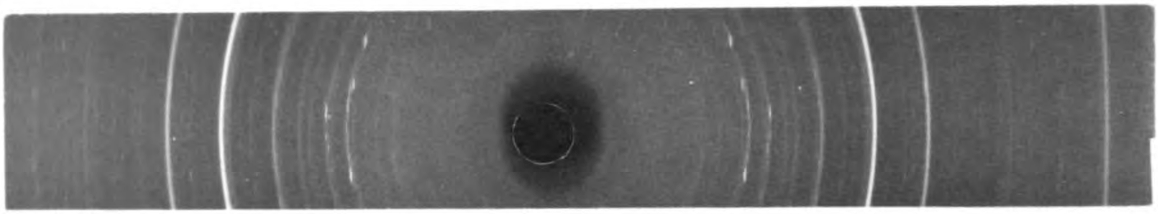
Exposures were made for the following compounds, both for the purpose of obtaining standards for comparison and to become familiar with the operation of the apparatus: NaCl, CuCl, $CuCl_2$, CuC_2, CuO, several copper carbonates and copper shavings from some of the tubing used in the panels. The first few attempts to obtain patterns from some of the deposits resulted in very densely fogged films with few discernable lines. Certain measures were taken to alleviate this situation. These included dessication over calcium chloride for three days and screening the powder through a 250 mesh silkscreen. In addition, new film was obtained and subsequent exposures resulted in measureable lines, although the background fog persisted. Separate runs, using an x-ray tube with an iron target, were made in an effort to determine whether the fogging could be due to secondary radiation of iron compounds in the deposit. Results would indicate some improvement, but at the cost of tripling exposure times to obtain even faint lines.

The patterns obtained from the various deposit samples are included (Figure 40) along with a list of the "d" spacings and the relative intensities of the major lines of the x-ray patterns. (Table XIV)

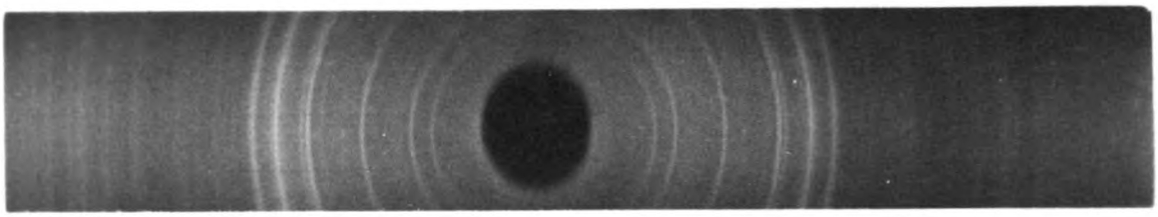
Identification of the lines was made by comparison of experimental "d" values to those in the Hanawalt, Rinn, Frevel Tables.[10] The line intensities are relative for each film and are designated 1-5, strongest to weakest. Only a few 2S distances were measured in order to determine a midpoint, the remainder of the S values were then obtained by subtraction of the midpoint value from the measured value of the lines. The corresponding "d" values were then determined from a graph made especially for the camera used. (Table XIV)



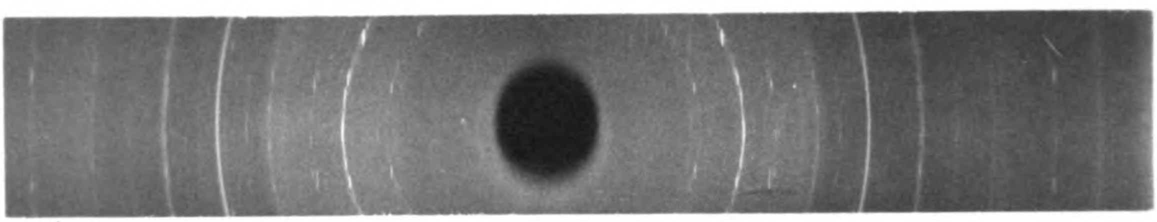
50°F



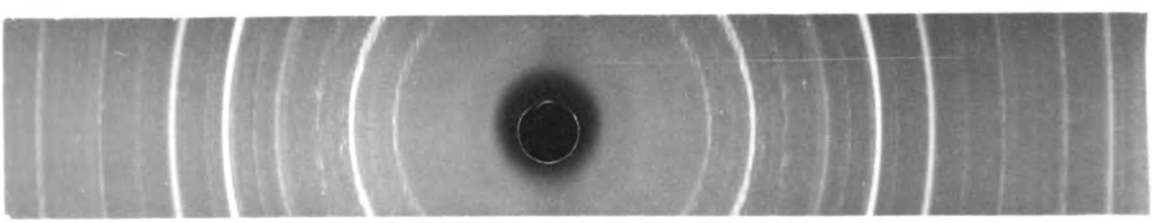
80°



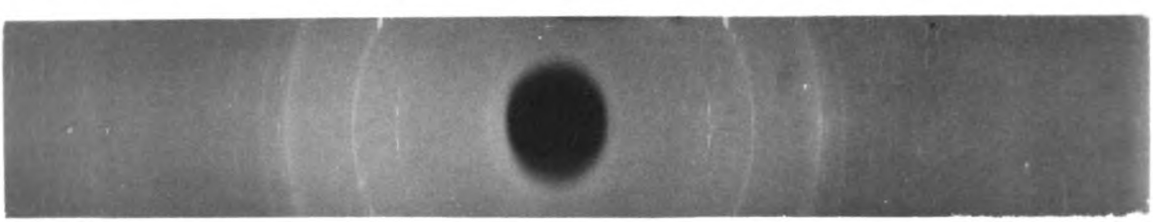
110°



140°



170°



200°

Figure 40. X-ray Powder Patterns of Deposit Material

Table XIV

Powder Pattern Identification

Line Intensity	2S (cm) Measured Distance	S (cm) Distance	d (Å) From Graph	Probable Identity
Deposit from 1 1/4" tubing at 50°F				
4	4.200	2.100	4.18	SiO_2 (4.25)
3		2.667	3.34	SiO_2 (3.35)
5		2.767	3.22	
5		2.932	3.04	$CaCO_3$ (3.04)
5		3.087	2.91	$CuCO_3$ (2.86)
1	7.305	3.653	2.45	FeO (2.47); Cu_2O (2.45); $CaCO_3$ (2.49)
5	8.455	4.228	2.14	FeO (2.14); Cu_2O (2.12)
2	8.685	4.343	2.08	Cu (2.08)
3		5.052	1.80	Cu (1.81); SiO_2 (1.82)
5		5.977	1.64	
4		6.142	1.51	FeO (1.51); Cu_2O (1.51)
5	13.630	6.865	1.37	SiO_2 (1.375)
5		7.412	1.29	Cu (1.277); Cu_2O (1.283)
5		7.712	1.25	
Deposit from 1 1/4" tubing at 80°F				
5		1.290	6.95	$Cu_3(PO_4)_2$ (6.9)
5		2.095	4.19	SiO_2 (4.25)
5	4.580	2.290	3.85	
5	5.185	2.593	3.42	$CuSO_4 \cdot H_2O$ (3.40)
4		2.668	3.34	SiO_2 (3.35)
5		2.875	3.10	CuCl (3.12)
4		2.933	3.04	$CaCO_3$ (3.04); $Cu_3(PO_4)_2$ (3.02)
5		3.165	2.83	$CuCO_3$ (2.86)
5		3.313	2.69	Fe_2O_3 (2.69); $FeCl_2$ (2.68)
3	7.295	3.648	2.46	Cu_2O (2.45); FeO (2.47); $CuSO_4 \cdot H_2O$ (2.46)
5		3.940	2.29	$CaCO_3$ (2.28)
1	8.695	4.348	2.07	Cu (2.08); $FeCl_2$ (2.08)
5		4.760	1.91	CuCl (1.91); $CaCO_3$ (1.92)
2	10.110	5.055	1.80	Cu (1.81)
5		5.400	1.70	$Cu_3(PO_4)_2$ (1.71); Fe_2O_3 (1.69)
5		5.735	1.60	CuCl (1.63)
5		6.125	1.51	Cu_2O (1.51); FeO (1.51)
5		6.815	1.38	SiO_2 (1.375)
3		7.418	1.29	Cu (1.277); Cu_2O (1.283)

Table XIV

Line Intensity	2S (cm) Measured Distance	S (cm) Distance	d (Å) From Graph	Probable Identity
Deposit from 1 1/4" tubing at 110°F				
2	2.965	1.483	5.98	$CuCO_3$ (6.0)
1	3.540	1.770	4.96	$CuCO_3$ (5.1)
1	4.850	2.425	3.65	$CuCO_3$ (3.68)
5		2.675	3.34	SiO_2 (3.35)
5		3.090	2.89	$CuCl_2$ (2.90)
1		3.137	2.85	$CuCO_3$ (2.86)
1		3.552	2.52	FeO (2.47); CuO (2.51)
1		3.857	2.34	$CuCl_2$ (2.36); CuO (2.31)
5	8.265	4.133	2.19	
5		4.235	2.13	FeO (2.14); Cu_2O (2.12)
5	8.780	4.390	2.05	Cu (2.08)
5		5.475	1.67	
5		5.630	1.62	CuCl (1.63)
5		5.803	1.59	
5		6.170	1.50	Cu_2O (1.51); FeO (1.51)
5		6.775	1.39	SiO_2 (1.375)
Deposit from 1 1/4" and 1" tubing at 140°F				
4		2.105	4.18	SiO_2 (4.25)
2	5.345	2.673	3.33	SiO_2 (3.35)
5		3.100	2.89	$CuCl_2$ (2.90)
5		3.640	2.47	Cu_2O (2.45)
5	7.890	3.945	2.28	
1	8.655	4.328	2.08	Cu (2.08)
5		4.575	1.98	
2	10.095	5.048	1.80	Cu (1.81)
5		5.485	1.67	
5	11.950	5.975	1.55	Cu_2O (1.51)
5		6.900	1.36	SiO_2 (1.375)
4		7.405	1.29	Cu (1.277); Cu_2O (1.283)
5		8.090	1.20	
Deposit from 1 1/4" and 1" tubing at 170°F				
4		2.105	4.18	SiO_2 (4.25)
5		2.425	3.65	$CuCO_3$ (3.63)
2	5.370	2.685	3.31	SiO_2 (3.35)
5		3.115	2.87	$CuCO_3$ (2.86); $CuCl_2$ (2.90)
4		3.660	2.45	Cu_2O (2.45)
5	7.905	3.953	2.28	
1	8.695	4.348	2.08	Cu (2.08)
5		4.595	1.97	
2	10.105	5.053	1.80	Cu (1.81); SiO_2 (1.82)
5		5.505	1.66	

Table XIV

Line Intensity	2S (cm) Measured Distance	S (cm) Distance	d (Å) From Graph	Probable Identity
5		5.970	1.54	Cu_2O (1.51)
4		6.820	1.38	SiO_2 (1.375)
3		7.410	1.29	Cu (1.277); Cu_2O (1.233)
5	15.960	7.980	1.22	

Deposit from 1 1/4" tubing at 200°F

Line Intensity	2S (cm) Measured Distance	S (cm) Distance	d (Å) From Graph	Probable Identity
4	4.160	2.080	4.24	SiO_2 (4.25)
1	5.320	2.660	3.34	SiO_2 (3.35)
2	7.115	3.558	2.52	CuO (2.51)
5	7.295	3.648	2.46	FeO (2.47)
4		3.865	2.33	CuO (2.31)

CONCLUSIONS
(EXPERIMENTAL II)

From Table XIV, it may be observed that certain lines appear in all the samples. The substances thus indicated as present in all the samples are silica, copper and an oxide of copper. The presence of metallic copper is due to the sampling technique. Because of the repetition of certain lines in the x-ray diagrams, which are known to be due to cuprous oxide, it may be assumed that cuprous oxide is present in all deposits except the 200°F sample. Similarly, cupric oxide may be assumed to be in this sample. Basic copper carbonate lines appear in the pictures of the deposits below 110°F; while lines attributable to several other copper salts appear randomly throughout the series. Traces of iron oxides are also indicated; they are most evident in the 80°F sample. A few faint lines remain unidentified due to time limitations and the lack, in this department, of an adequate system of cross references to the line indexes.

These results are in reasonable agreement with those expected on the basis of the composition of previously analyzed corrosion products of copper. The change in composition of the oxides and the lack of carbonate above 110°F are anticipated on the basis of the observed changes in color and structure of these deposits with increasing temperature. (Table III)

This presentation was not intended as an exhaustive study of deposit composition, but rather as a sort of general survey to verify expectations. More extensive analysis would, of course, involve investigation of the deposits of each diameter tubing as well as repeated runs on the above samples.

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