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WADC TECHNICAL REPORT 54-451
PART 1

EVALUATION OF SURFACE TREATMENTS FOR LOW-ALLOY STEELS

Part 1. Test Method for Heat-Resistant Corrosion Protective Coatings on Steel

SAM TOUR

SAM TOUR & CO., INC.

NOVEMBER 1954

WRIGHT AIR DEVELOPMENT CENTER

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Part 1. Test Method for Heat-Resistant Corrosion Protective Coatings on Steel

SAM TOUR

SAM TOUR & CO., INC.

NOVEMBER 1954

MATERIALS LABORATORY
CONTRACT No. AF 33(616)-406
PROJECT No. 7351
TASK No. 73512

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
200 - 23 March 1955

FOREWORD

This report was prepared by Mr. Sam Tour of Sam Tour & Co., Inc. under Contract No. AF 33(616)-406. The contract was initiated under Project No. 7351, Metallic Materials, Task No. 73512, High Temperature Alloys, formerly RDO No. 619-11, and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt J. R. Miller acting as project engineer.

In the carrying out of the work covered by this report, the author gratefully acknowledges the cooperation extended by the suppliers of test panels and coating materials and the direct assistance of the staff of Sam Tour & Co., Inc.

ABSTRACT

The test procedure involves two types of artifically created environments. The first step is a cyclic or repeated exposure for one week at the desired temperature in an atmosphere of products of combustion. The second step is a cyclic exposure for one week in an alternate condensation corrosion test unit at room temperature or slightly above.

The atmosphere containing products of combustion is obtained by the use of a kerosene torch, kerosene plus suitable additives and an excess of air. The atmosphere produced in this manner is conducted into a full muffle in a temperature controlled furnace. The panels are hung on racks in the furnace muffle where they are heated to the desired temperature for 6 hours each day for five continuous days. During the remaining 18 hours of each day they remain in the closed muffle with no heat applied to the furnace and no artificial atmosphere being introduced into the muffle.

The alternate condensation test equipment has a large turntable revolving about 15 times per hour and on which the specimen panels are mounted. The turntable carries the panels successively through (a) a tunnel where they are cooled with dry air, (b)a tunnel where they encounter warm moist air so as to collect a layer of condensate and (c) an open space where the condensate may evaporate into the room atmosphere.

Sixteen different coatings have been tested. Some of the coatings were tested at temperatures of 600°, 800° and 1000°, others, at 1000°, 1200° and 1400°F in the atmosphere of combustion products of kerosene with additives. Eight of the sixteen coatings mentioned above have been tested at 1000°F in the atmosphere of the combustion products of kerosene without additives.

When the additives were not used, different corrosion effects were observed. These panels withstood the testing conditions in the furnace and in the alternate condensation test better than when the additives were used.

It should be pointed out that this test method is specialized and rather severe; the success or failure of a coating in this test does not imply similar results under other conditions of service or tests.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

TABLE OF CONTENTS

Section		Page Number
I	Introduction	1
II	Development of the Test Furnace	1
III	Development of the Kerosene Combustion System	7
IV	Kerosene Additives	8
V	The Alternate Condensation Corrosion Test	9
VI	Base Metals Used for Tests	10
VII	Types of Coatings Tested	10
VIII	Test Procedure	14
IX	Test Results	15
X	Discussion and Conclusions	22

PART I

Section I - Introduction

This final summary report on Part I of Phase II of Contract No. AF 33(616)-406 on "Research On Surface Treatment Of Low Alloy Steels" covers the work done during the period of May 1953 to June 1954.

A program for the development of an adequate, practical and applicable test procedure or procedures for the relative evaluation of various types of coatings on plain carbon and low alloy steels for use in aircraft at service temperatures from 400 to 1200°F was outlined in Progress Report #5 on this contract. The program was approved under date of July 7, 1953. Actual work began during the month of August 1954. Progress Reports #9 to #17 inclusive for each successive month from September 1953 to and including May 1954 contain much of the details.

In the development of a satisfactory test procedure it was necessary to carry out tests on a number of different types of coatings supposedly serviceable in the range of temperatures under consideration. No attempt was made to carry out evaluation tests of the many types of commercial coatings on the market. Actually the test procedure was used as a tool for Part II of Phase II of the contract which was devoted to a study of paint siliconizing and paint chromizing of plain carbon and titanium-boron low alloy steel.

Section II - Development of the Test Furnace

A gas fired full muffle gas curtain atmosphere controlled furnace was selected for the work. A resistance wound electric muffle furnace should be equally suitable. It was considered inadvisable to use an electric furnace with the electrical resistors exposed within the furnace to the atmosphere to be generated.

It was decided to generate the desired atmosphere by the combustion of kerosene plus additives with an excess of air so as to obtain a final product containing from 10 to 12% of oxygen. It was not found possible to do this by attaching a kerosene torch directly to the atmosphere combustion space in the curtain furnace. It was necessary to develop a separate kerosene combustion system and a series of Venturi couplings in order to obtain and maintain the desired oxygen content of the products of combustion for introduction into the furnace muffle.

Figure 1 (Plate 15862) is a photograph of the complete system. The units from left to right are the kerosene pressure vessel, the regulating valves, the torch, three Venturi couplings, a bleed off pipe, the combusted gas supply pipe and the gas fired full muffle furnace.

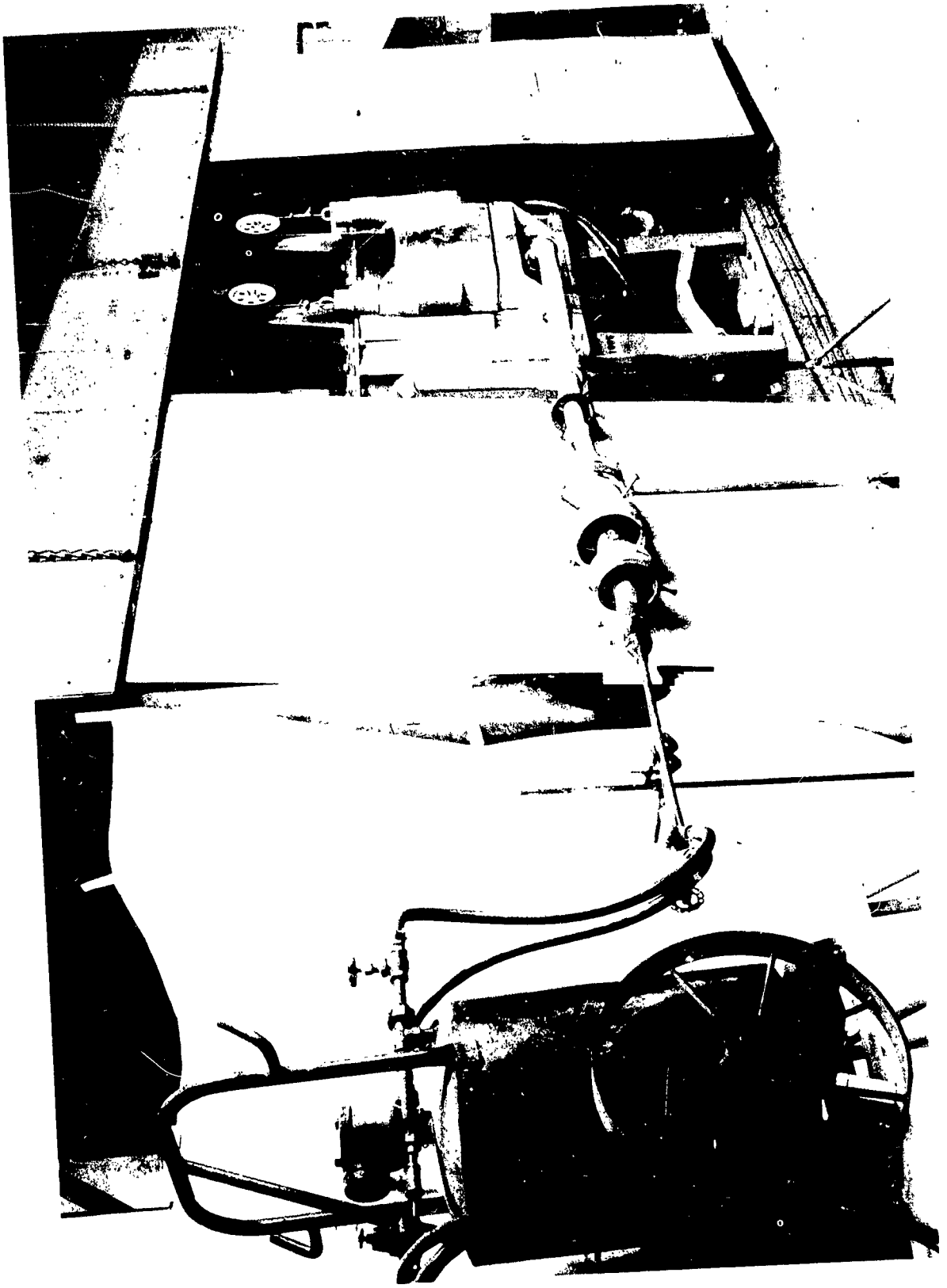

FIG. 1 (PLATE 15862)
TEST FURNACE SYSTEM

Figure 2 (Plate 15863) is a close-up of the kerosene burner regulating valves and the torch.

Figure 3 (Plate 15864) is a close-up of the three Venturi couplings in series.

Figure 4 (Plate 15865) is a close-up of the muffle furnace with a part of the piping system. A rack with panels can be seen in the open furnace.

The kerosene torch burner which is known as a Mahr Safety Torch is often used in foundries for the skin drying of sand molds. The burner has a 15 gallon tank and required compressed air at between 50 and 125 psi. The average kerosene consumption of the burner was approximately 5 gallons per 6 hour run. The burner nozzle was directed into the first of three Venturi couplings installed in series. The piping system, beyond the last Venturi, consisted of 2" pipe and pipe fittings as shown. The additional exhaust or bleed off which was partly open during operations was found necessary to avoid back leakage through the Venturis. Most of the combustion gases from the kerosene burner went through the piping system into the muffle of the furnace.

The furnace was of the eight burner two manifold type, heated by city gas. The furnace was equipped with a Premix gas-air proportioning control and its own air blower system. When all eight burners were used, the maximum operating temperature in the furnace was approximately 1900°F; the lowest, 1200°F. The operating temperature was brought down much lower by using fewer burners.

Necessary burner modifications were determined for operating the furnace at 600°F ± 5, 800°F ± 5, 1000°F ± 10, 1200°F ± 10, 1400°F ± 10. Temperature variations were determined for each of the above temperatures at nine points in the muffle. Using different combinations of burners, the temperature variations within the muffle could be held to within 50°F.

Ordinarily a combusted gas curtain furnace is heated by burners set underneath the muffle as well as by the gas curtain coming from the slot at the front of the muffle inside the furnace door. As the gas curtain was substituted with the flow of products of combustion of kerosene, it had the effect of cooling the front of the muffle. Necessary readjustments were made in the heating burners to effect the uniform temperature distribution throughout the muffle.

Holders, for about 40 panels each, were designed and constructed, one of carbon and one of stainless steel. Each holder had two bars, 5/16" in diameter, to accommodate the panels. A hole in the back wall of the muffle enabled the combustion products to pass through the muffle. During all runs, the holder was placed in the muffle in such a position that the panels were parallel to the flow of gases.

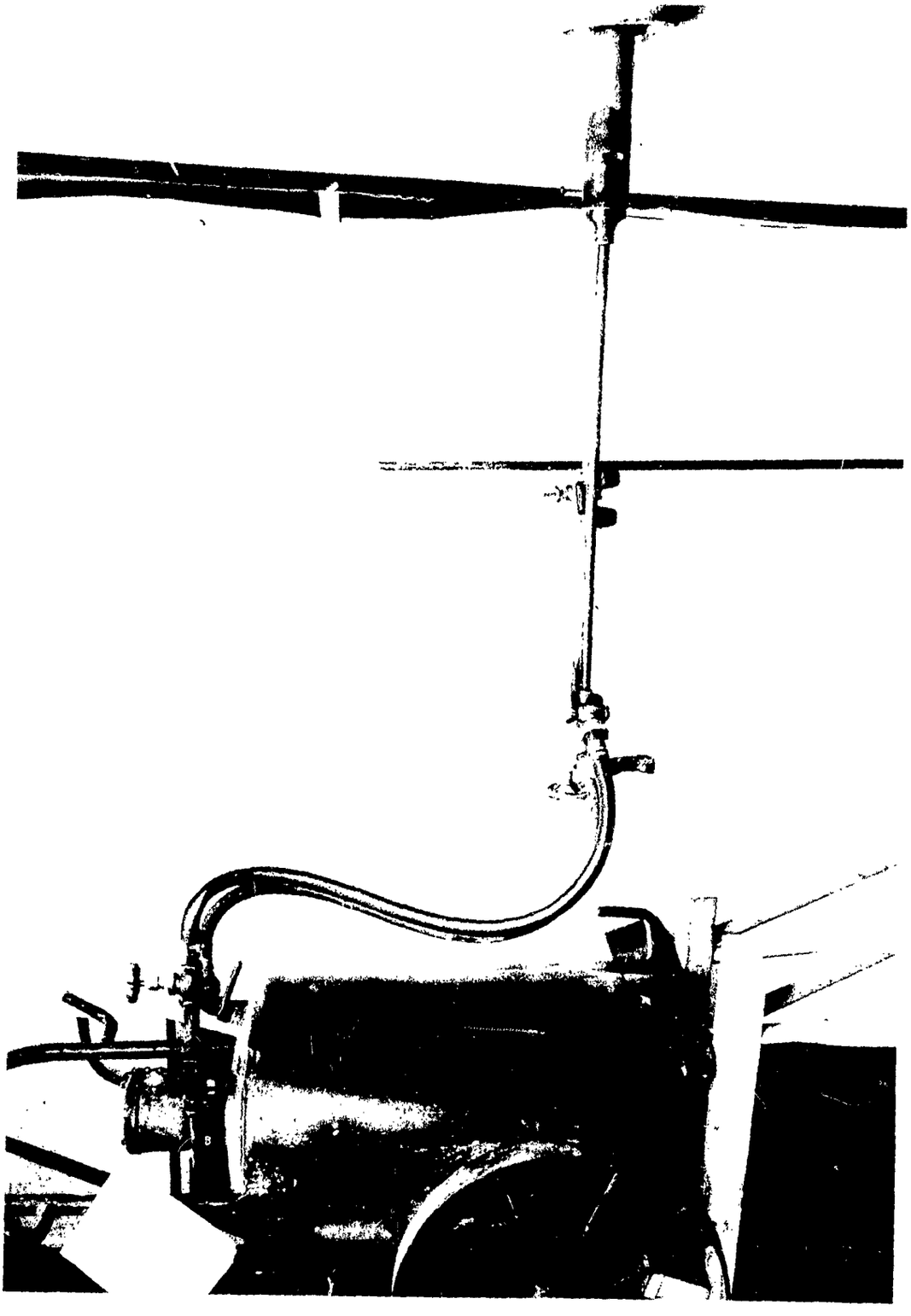

FIG. 2 (PLATE 15863)
KEROSENE BURNER

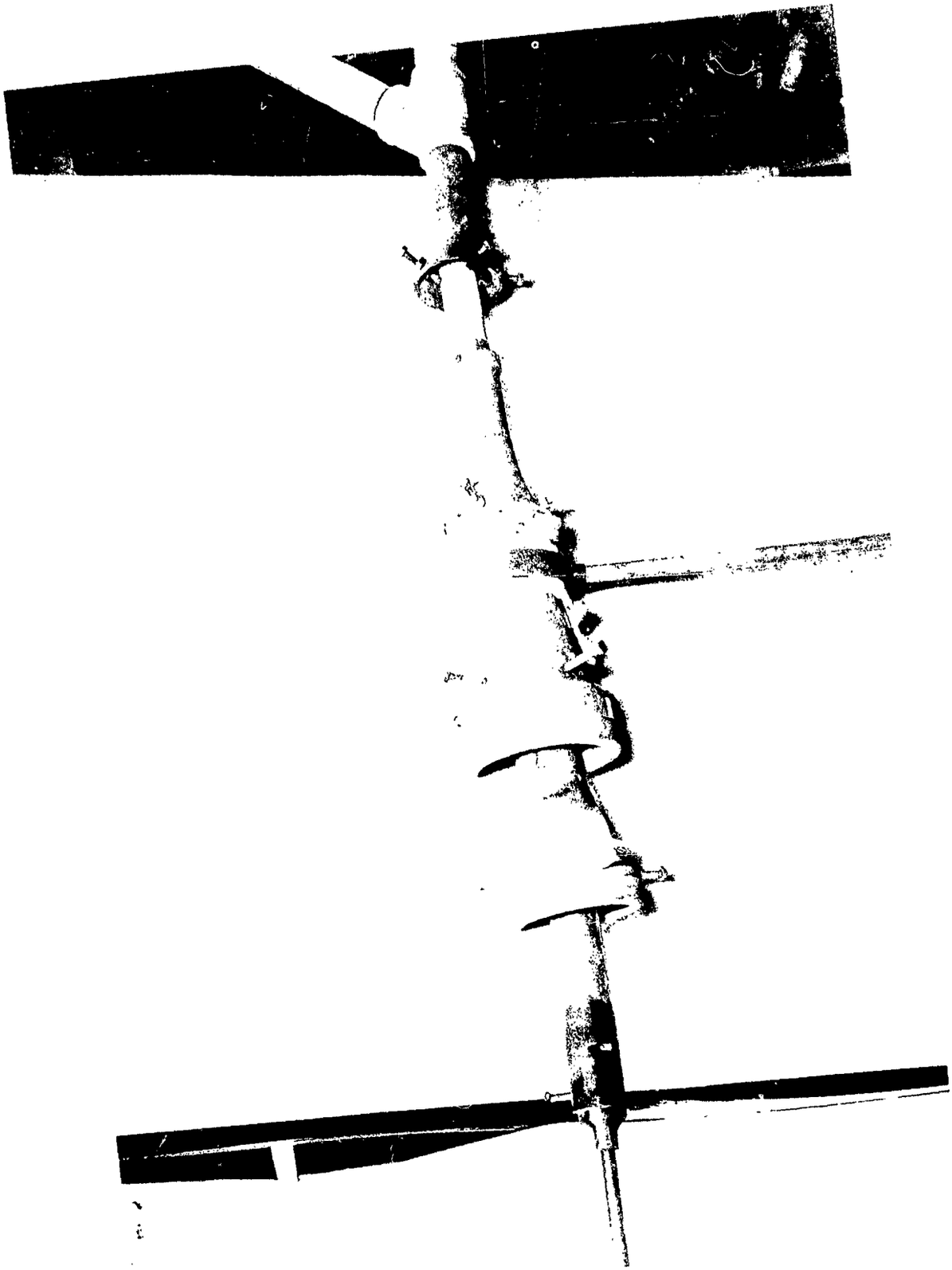

FIG. 3 (PLATE 15864)
VENTURI COUPLINGS

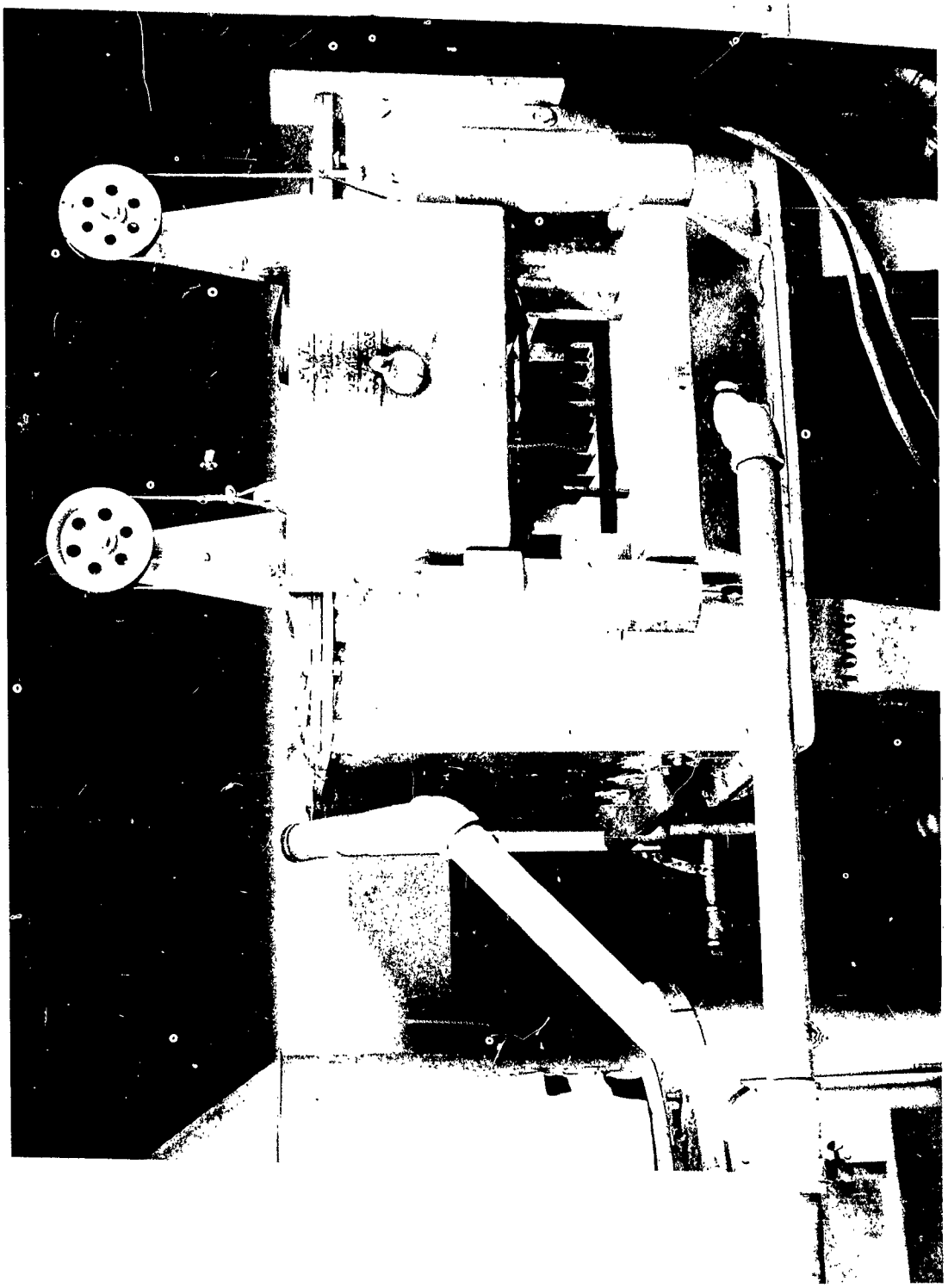

FIG. 4 (PLATE 15865)
MUFFLE FURNACE

Several preliminary test-runs were made without panels, with uncoated plain carbon steel panels, and with aluminum metallized panels in the muffle. During these runs, the temperatures of the muffle were 1000°F and 1200°F. Combusted gas atmospheres or room atmosphere were used in these preliminary runs.

Section III - Development of the Kerosene Combustion System

The kerosene combustion system was modified several times in order to (a) decrease the burning intensity, (b) increase the uniformity of combustion, (c) obtain a thinner flame and, (d) increase the oxygen content of the combustion gases.

To obtain the above ends, the following modifications were made. The air orifice, the kerosene orifice and the cap outlet on the kerosene burner were made smaller, resulting in a decrease in the intensity of the flame. The air hose and kerosene hose were shortened between the kerosene tank and the torch. The kerosene tank was raised three feet above the floor level. A regulating air valve was inserted into the air line to obtain a steady air flow into the system. The wide nozzle of the kerosene burner was replaced with a narrow one. Two such nozzles were made; one of low carbon steel and one of stainless steel. To increase the amount of oxygen present in the combustion products, the Venturi couplings were placed in series in the line to the furnace. Adjustment of the position of the entrance pipes to the Venturis made it possible to control the oxygen content of the products entering the furnace muffle.

To further improve the uniformity of combustion operation and to eliminate occasional back-firing in the kerosene torch system, it was found necessary to place an exhaust line in the pipe system through which the excess of combustion gases could escape. To increase the flow of the combustion products into the test furnace, the inlet into the muffle was enlarged from 1" diameter to 2½" diameter.

To avoid lead poisoning of operating personnel, the entire system, consisting of the kerosene torch, the by-pass lines and the furnace were enclosed under a large hood with suction draft ventilation. Complying with precautionary specifications of the Ethyl Corporation, an industrial gas mask from the Mine Safety Appliances Co. was used by the persons attending the kerosene burner. In order to determine the lead concentrations in the air, a colorimetric Hellige comparator was obtained from the Ethyl Corporation. The instrument had a detective range of 1-20 microns of lead per cubic foot and facilitated determinations of lead in the atmosphere. The instrument was calibrated with standard solutions made in the laboratory. The maximum safe lead concentration in air is considered

to be 3 micrograms per cubic foot. Using a Mine Safety Appliance Co. air sampler and the Hellige comparator the air in the laboratory space around the kerosene-furnace installation was sampled and analyzed for three locations daily. The average lead concentration in the air was held to about one microgram per cubic foot of air.

Analyses of the atmosphere in the muffle were made daily with a Hayes orsat gas analysis unit. Oxygen, carbon dioxide and carbon monoxide percentages were determined. The test atmosphere was maintained at 8 to 12% oxygen, 5 to 6.5% carbon dioxide and zero percent carbon monoxide.

No attempt was made to analyze the gases from within the muffle for lead, sulphur, or bromine content.

Section IV - Kerosene Additives

Kerosene was selected as the fuel to be burned rather than gasolene because of its higher flash point, less explosion danger and its higher solubility for such additives as might be desired to be used. It was desired to create an atmosphere within the test muffle that would be much more active than any that would occur in service burning either ethyl gasolene or fuel oils containing sulphur and vanadium compounds.

Tetraethyl lead and ethylene dibromide were obtained from the Ethyl Corporation as aviation type which was reported to contain 64.8% by volume of lead tetraethyl, 28.6% by volume of ethylene dibromide and 6.6% by volume of kerosene. Throughout the testing program, when tetraethyl was used, the quantity used was 13 mls added to each gallon of kerosene. This is several times the amount normally used in ethyl gasolene.

Originally it was planned to add sulphur to the kerosene in the form of thiophene. Some of this material was obtained and tried but was not found to have any advantages over the simple use of carbon disulphide. Throughout the testing program, when sulphur additions were used, the quantity used was 19 mls of carbon disulphide per gallon of kerosene.

Considerable time was spent in finding a vanadium compound that is sufficiently soluble in kerosene. A number of organic vanadium compounds were obtained such as stearates and palmitates, but they were not sufficiently soluble. Finally vanadium oleate was made in the laboratory from oleic acid and vanadium pentoxide. This soap was found to be soluble in kerosene up to a concentration of 0.05% of vanadium. Throughout the testing program, when vanadium was used, the quantity of vanadium oleate used was such as to give 0.05% of vanadium in the kerosene.

Section V - The Alternate Condensation Corrosion Test

It is often desired to know whether or not a coating still provides good corrosion protection after having been heated to service or test temperatures. Simple high temperature resistance of a coating is insufficient. A coating must continue to provide corrosion protection at room temperatures or below after it has been exposed at elevated temperatures encountered in service even though its surface has become contaminated with combustion products, etc., during such service.

Having in mind the above objectives, such tests as the salt spray test and an artificial rain test were considered. The salt spray is applicable only where sea or salt air is a factor. It is a standard method of measuring continuity of surface coating rather than corrosion protection. The artificial rain test has the limitation of bridging over pin holes and concealing imperfections, even though they may exist. Any test where excess moisture is applied might wash away the combustion products and change the corrosion rate.

The alternate condensation tester, developed by Sam Tour & Co., Inc., was considered the most suitable tester for panels which had been subjected previously to the high temperature tests. The tester made it possible to obtain reliable information as to the extent of corrosion attack and defects of coatings including pin-holes, cracks, etc; it did not have any of the above limitations.

The test is an accelerated test simulating service conditions more than any test yet devised. Its principle is alternate condensation of water vapor and its subsequent evaporation.

During the test, the panels, which are mounted on a round revolving table, enter chambers with different conditions of temperature and humidity. In the first chamber, the panels meet a stream of cool and dry air of approximately 70°F and 25% relative humidity. In the second chamber, the panels meet a stream of air which has been saturated with water vapor at a thermostatically controlled temperature of 160 to 170°F. Specimens, however, can be exposed to varying temperatures and varying humidity conditions, as well as to air carrying desired contaminants in controlled quantities. Exposing the specimens to different corrosive atmospheres can be made by making use of a third or fourth chamber where these atmospheres can be introduced.

As the dry and cool panels leave the first chamber and enter the second chamber, water vapor condenses to form a layer of dew or condensate on them. This water is of maximum corrosivity; it searches out pin-holes and does not bridge them. Evaporation of this condensate takes place on the way from the second to the first chamber. This evaporation lasts during approximately half a revolution of the table and can be accelerated by a fan blowing at the panels. The test simulates the actual weather conditions when

the dew precipitates at night and evaporates during the day. The duration of a full alternate condensation cycle which can be regulated at will is usually four minutes and repeats continually for a desired period of time.

Figure 5 is a photograph of the alternate condensation tester in operation in the test of coated steel drums.

Section VI - Base Metals Used for Tests

The steels used for the tests were (1) a 3 Cr-1 Mo-TiB steel supplied by the United States Air Force, Wright Air Development Center, Wright-Patterson AF Base, Ohio, and (2) SAE 1010 steel (hot rolled and pickled) obtained from a commercial producer. Panels, 4" x 8", made of these two steels were used. Such test panels were large enough to show tendencies towards pin-holes in coatings and to permit the taking of test specimens for tensile and bend test purposes after various amounts of exposure, had the latter been found necessary.

All panels were 14 gauge or 0.064" thick. Each panel had a 3/8" hole near the center of one 8" edge, for mounting in the test furnace. The hole was made before any coating was applied on a panel. Each panel was identified by a notch code system.

Section VII - Types of Coatings Tested

In order to test the test method it was desired to include a range of coatings. Some of the coatings chosen were expected to fail at a low temperature (cadmium), some to corrode slightly and some to withstand the conditions of the test. Simultaneously it was desired to use the test to evaluate certain new and promising coatings for plain carbon steel and for the titanium-boron low alloy steel.

For testing purposes, the panels were divided into three groups and coatings applied as indicated below.

<u>Group I</u>

1. Hot dip aluminum (13% silicon alloy) applied on 12 plain carbon steel panels by Arthur Tickle Engineering Works, Inc., Brooklyn 2, N. Y.

2. Hot dip aluminum (13% silicon alloy) applied on 6 TiB steel panels by Arthur Tickle Engineering Works, Inc.

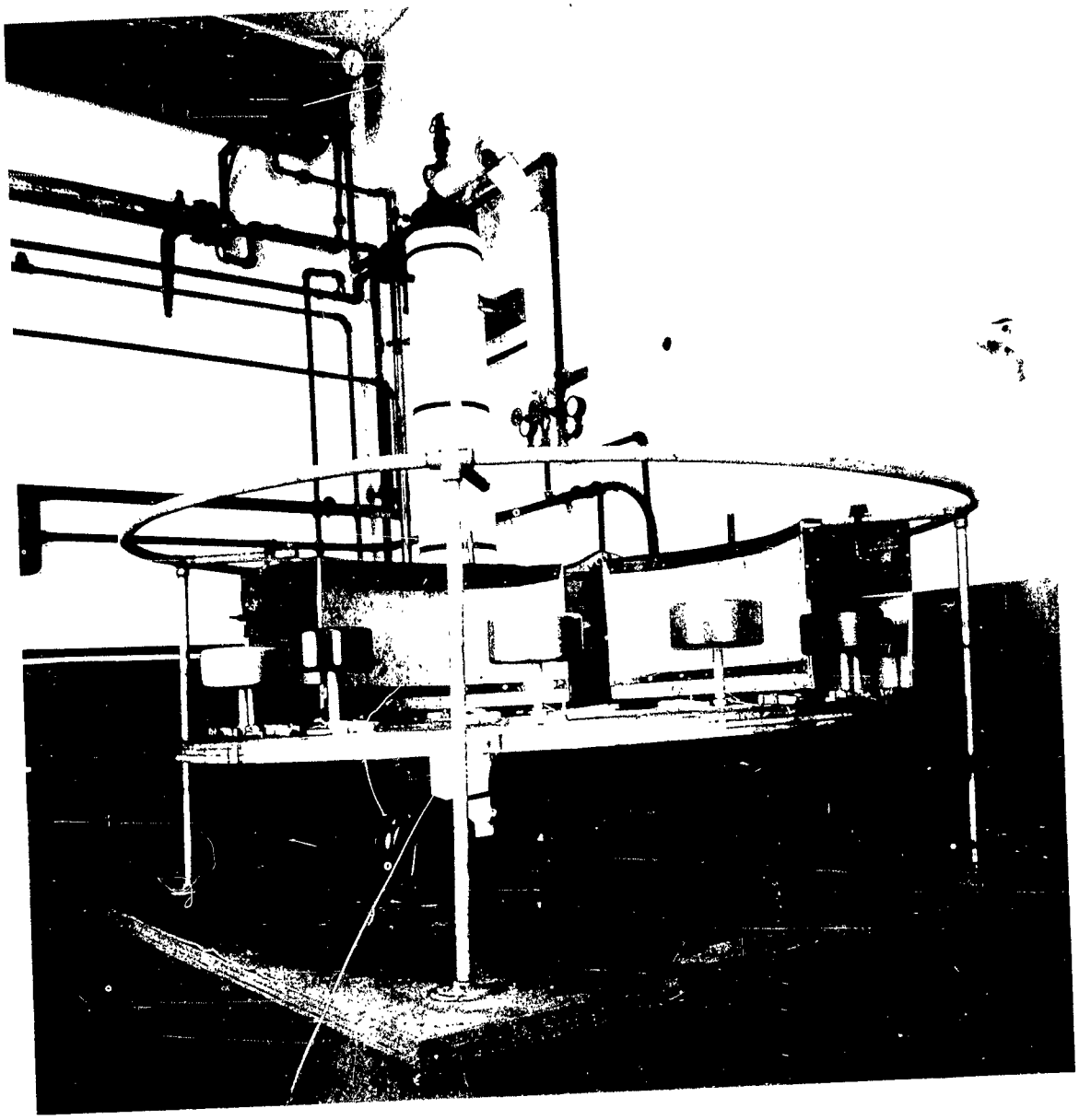

FIG. 5
ALTERNATE CONDENSATION CORROSION TESTER

3. Sprayed and fused aluminum (Metco Process 11) applied on 12 plain carbon steel panels by Metallizing Engineering Co., Inc., Long Island City, N. Y.

4. Sprayed and sealed aluminum (Metco System 120) applied on 12 plain carbon steel panels by Metallizing Engineering Co., Inc.

5. Paint siliconizing applied on 12 TiB steel panels by Sam Tour & Co., Inc.

6. Paint siliconizing applied on 12 plain carbon steel panels by Sam Tour & Co., Inc.

7. Paint chromizing applied on 12 TiB steel panels by Sam Tour & Co., Inc.

8. Paint chromizing applied on 12 plain carbon steel by Sam Tour & Co., Inc.

Group II

1. Electroless nickel applied on 12 plain carbon steel panels by Metal Processing Co., Cedar Grove, N. J.

2. Electroplated nickel applied on 12 plain carbon steel panels by Hanson-Van Winkle-Munning, Matawan, N. J.

3. Silicone aluminum paint applied on 12 plain carbon steel panels by Sam Tour & Co., Inc. The paint was obtained from General Electric Co., Schenectady, N. Y.

4. Corronizing (nickle-zinc) applied on 12 plain carbon steel panels by Standard Steel Spring Co., Coraopolis, Pa.

5. Sicon paint 3 x 222 - Silicone base aluminum pigmented paint applied on 12 plain carbon steel panels at Sam Tour & Co., Inc. The paint was obtained from Midland Industrial Finishes Co., Waukegan, Ill.

6. Sicon paint 7 x 273 - Silicone base aluminum pigmented paint applied on 12 plain carbon steel panels by Sam Tour & Co., Inc. The paint was obtained from Midland Industrial Finishes Co.

7. Sprayed aluminum applied on 12 plain carbon steel panels by Metallizing Engineering Co., Inc.

8. Cadmium electroplated applied on 12 plain carbon steel panels by a commercial electroplater.

9. Epoxy base, aluminum pigmented paint applied on 12 plain carbon steel panels by Sam Tour & Co., Inc. The paint was obtained from Midland Industrial Finishes Co.

Group III

All the coatings of Group III were applied on plain carbon steel panels as follows:

1. Paint chromizing,
2. Paint siliconizing,
3. Electroless nickel,
4. Electroplated nickel,
5. General Electric silicone aluminum,
6. Sicon paint 3 x 222,
7. Sicon paint 7 x 273,
8. Epoxy paint.

The thicknesses of the coatings were determined metallographically and found to average as follows:

1. Hot dip aluminum (13% Si alloy) on low carbon steel - 0.0039.

2. Hot dip aluminum (13% Si alloy) on TiB steel - 0.0046.

3. Aluminum sprayed and fused - 0.007.

4. Aluminum sprayed - 0.006.

5. Siliconizing on low carbon and titanium-boron steels (½ hour at 1850°F) - 0.013.

6. Chromizing on low carbon (½ hour at 1950°F) - 0.0015.

7. Chromizing on titanium-boron steel (15 min. at 1950°F and 3 min. at 2100°F) - 0.0012.

8. Corronizing (nickel-zinc) on low carbon steel - 0.0032.

9. Electroless nickel on low carbon steel - 0.0006.

10. Cadmium electroplate on low carbon steel - 0.0015.

11. Nickel electroplate on low carbon steel - 0.0009.

12. Silicone aluminum paint on low carbon steel - 0.0009.

13. Sicon paint 3 x 222 on low carbon steel - 0.0006.

14. Sicon paint 7 x 273 on low carbon steel - 0.0005.

15. Epoxy paint on low carbon steel - 0.0004.

Section VIII - Test Procedure

The coated panels were exposed in triplicate in the test furnace at the following temperatures: Group I at 1000°, 1200° and 1400°F; Group II at 600°, 800° and 1000°F. Both these groups were exposed to the atmosphere of combustion products of kerosene plus additives. The panels of Group III were exposed at 1000°F to an atmosphere of combustion products of kerosene without additives.

Each group was exposed in the furnace for 6 hours at the specified temperature and then allowed to cool and remain in the furnace for the balance of a 24 hour period. This procedure was repeated through five 24 hour cycles. The purpose of cooling after the heating in the furnace was to allow the condensation products to form which, in themselves, could have been corrosive. Each group of panels was started at a low furnace temperature. The panels were examined each day, when in the furnace.

After the furnace exposure at a given temperature, two exposed panels from each category were selected for testing in the alternate condensation tester. In addition to the two furnace exposed panels, one previously untested panel of each category was included to determine the effect of the alternate condensation test alone. In the alternate condensation tester the exposure was for 100 continuous hours with daily examination for evidence of failure.

Panels that failed after a given furnace test temperature alone or after the alternate condensation test, which followed the furnace test, were removed from further test. Panels that did not fail were returned to the furnace for testing at the next higher temperature.

The lowest temperature range considered for heat resisting coatings was 400 to 600°F. The test method under development used a 600°F test temperature to evaluate materials in this range. If a test temperature of only 400°F were used, it would not be possible to determine the margin of safety of coatings at 400°F.

Coatings that were fully satisfactory in the test at 1200°F might have been so near to failure that they would not stand long periods of time at 1200°F. The 1400°F test was included and was used on coatings that withstood the 1200°F test in order to determine the margin of safety of these coatings at the 1200°F limit.

The panels were mounted on a rack and placed in the furnace spaced about ½" apart and separated from one another by stainless steel rings. Thus the panels were distributed throughout the whole muffle of the furnace.

The schedule and sequence of the tests were as follows:

<u>1st Week</u>

Group II in test furnace at 600°F.

2nd Week

Group I in test furnace at 1000°F.
Group II in alternate condensation test.

3rd Week

Group I in the alternate condensation test.
Group II in test furnace at 800°F.

4th Week

Group I in test furnace at 1200°F.
Group II in alternate condensation test.

5th Week

Group I in the alternate condensation test.
Group II in test furnace at 1000°F.

6th Week

Group I in test furnace at 1400°F.
Group II in alternate condensation test.

7th Week

Group I in the alternate condensation test.

Throughout the above program of testing, panels which failed at a given temperature were replaced with fresh panels for the next higher temperature of test. Throughout the above program the atmosphere in the test furnace consisted of the products of combustion of kerosene plus the additives previously described.

Following the above program, on Groups I and II, Group III was tested at 1000°F with the atmosphere in the test furnace consisting of the products of combustion of kerosene alone, with no additives, followed by the alternate condensation test.

Section IX - Test Results

Results of Tests - Group I

The weekly results in brief are shown in Table I.

1. Hot Dip 13% Si-Al on Plain Carbon Steel

This coating had a glossy silver appearance before exposure. Panels with this coating withstood all exposures in the test furnace with no visible change except a dulling of the coat. Exposure to alternate condensation tests had no effect on previously unexposed panels or on panels which had been exposed at 1000°F or 1200°F in the test furnace. However, panels which had been subjected to 1400°F were corroded in the alternate condensation test. The corrosion product was red-brown and spread lightly over most of the panel. No blistering or scaling took place.

TABLE I

Results of Tests - Group I

Coating	After 1000°F	After Alternate Condensation	After 1200°F	After Alternate Condensation	After 1400°F	After Alternate Condensation
Hot Dip 13% Si-Al on plain carbon	Unaffected	Unaffected	Unaffected	Unaffected	Unaffected	Slightly Corroded
Hot Dip 13% Si-Al on TiB steel	1 panel corroded slightly. Others unaffected	Slightly Corroded	Slightly Corroded	Slightly Corroded (blank unaffected)	Slightly Corroded	Slightly Corroded
Sprayed Al Process 11	Unaffected	Unaffected	Unaffected	Unaffected	Corroded	Corroded
Paint Chromizing plain carbon	Corroded	Corroded	Corroded	Corroded	Corroded	Corroded
Paint Chromizing TiB steel	Corroded	Corroded	Corroded	Corroded	Corroded	Corroded
Paint Siliconizing plain carbon	Corroded	Corroded (blank unaffected)	Corroded	Corroded	Corroded (cast broke away from base)	Corroded (blank unaffected)
Paint Sili-conizing TiB steel	Corroded	Corroded	Corroded	Corroded	Corroded	Corroded
Sprayed Al Method 120	Unaffected	Unaffected	Unaffected	Unaffected	Unaffected	Corroded

2. Hot Dip 13% Si-Al on TiB Steel

Upon leaving the test furnace of the three panels tested at 1000°F, only one showed signs of slight corrosion, while the other two were tarnished to a dull silver, as contrasted to their original bright silvery appearance. Exposing the tested panels to alternate condensation caused slightly more corrosion, while previously untested panels showed no signs of attack. Exposure at 1200°F and 1400°F caused somewhat more corrosion, and subsequent testing in the alternate condensation tester again increased corrosion. The corrosion product was pinkish and was spread lightly over the panel. No blistering or scaling occurred.

3. Sprayed and Fused Aluminum on Low Carbon Steel - Process 11

These panels had a rough, dull gray coat. Exposure at 1000°F, 1200°F and to the following alternate condensation tests did not show any visible effects. Corrosion did occur, however, during exposure at 1400°F, and the alternate condensation test aggravated this condition. The corrosion was fairly uniform with the corrosion product being red-brown in color. No blistering or scaling occurred.

4. Sprayed Aluminum on Low Carbon Steel - Method 120

Included under Group II.

5. Paint Siliconizing on TiB Steel

The appearance of these panels were the same as for #6.

6. Paint Siliconizing on Plain Carbon Steel

These panels had a dark gritty coat. Thirty hours at 1000°F produced corrosion on all panels tested. Tests at 1200°F caused corrosion and scaling of the coating, while at 1400°F, the coat was severely blistered and broke away from the base metal. Testing in the alternate condensator increased corrosion. The condensation test, however, gave varying results on panels not exposed in the Stewart furnace. Several panels were not corroded at all, while others were corroded. The corrosion product was red-brown and in some cases, was localized rather than spread uniformly over the panel.

7. Paint Chromizing on TiB Steel

The results of the testing of these panels were the same as for the plain carbon steel. Before testing the panels were of a metallic lustre but with encrustations in some areas.

8. Paint Chromizing on Plain Carbon Steel

These panels had a metallic lustre but with some dark encrustations spread over portions of the surfaces. All exposures

in the test furnace caused corrosion as well as scaling. The alternate condensation test worsened this corrosion, and caused corrosion of previously untested panels. The corrosion product varied from red to red-brown in color and was spread uniformly over the entire panel.

Results of Tests - Group II

The weekly results in brief are shown in Table II.

1. Electroless Nickel

These panels withstood exposure at 600°F, however, the original silvery appearance was changed to dark gray. Exposures at 800°F and 1000°F caused light corrosion, and all exposures in the alternate condensator brought about corrosion. The corrosion product was brown. Its formation was not accompanied by blistering or scaling.

2. Electroplated Nickel

These panels were a shiny silvery gray before exposure and withstood all exposures in the Stewart furnace, the only visible effect being a darkening of the coat. Likewise, the alternate condensation test caused no visible signs of corrosion.

3. Silicone Aluminum Paint

The bright, silvery coat of these panels did not change in the test furnace at 600°F or 800°F, while slight attack did take place at 1000°F. All panels, after exposure in the test furnace at any temperature failed in the alternate condensation test. Previously untested panels were unaffected. The corrosion product was red-brown in color and spread over most of the panel. No blistering or scaling was observed.

4. Corronizing

These panels retained their dark gray appearance throughout all exposures in both the test furnace and in the alternate condensator. No corrosion was apparent at any time.

5. Sicon Paint 3 x 222

Panels in this group were corroded in all tests except that at 600°F. The coating was silvery before testing and had a red-brown corrosion product spread uniformly over it after testing. No blistering or scaling was observed.

6. Sicon Paint 7 x 273

This coating withstood only the exposure at 600°F. All other exposures, both in the test furnace and in the alternate condensator, caused the formation of a red-brown corrosion product on the bright coat of the panel. No blistering or scaling was apparent.

TABLE II

Results of Tests - Group II

Coating	After 600°F	After Alternate Condensation	After 800°F	After Alternate Condensation	After 1000°F	After Alternate Condensation
Electroless Nickel	Unaffected (discolored)	Corroded	Corroded	Corroded	Corroded	Corroded
Electroplated Nickel	Uncorroded (discolored)	Unaffected	Uncorroded (discolored)	Unaffected	Uncorroded (discolored)	Unaffected
Silicone Aluminum Paint	Unaffected	Corroded (blank unaffected)	Unaffected	Corroded (blank unaffected)	Corroded	Corroded (blank unaffected)
Corronizing	Unaffected	Unaffected	Unaffected	Unaffected	Unaffected	Unaffected
Sicon Paint 3 x 222	Unaffected	Corroded	Corroded	Corroded	Corroded	Corroded
Sicon Paint 7 x 273	Unaffected	Corroded	Corroded	Corroded	Corroded	Corroded
Sprayed Al Method 120	Unaffected	Unaffected	Uncorroded	Uncorroded	Uncorroded	Corroded
Cadmium Electroplate	Corroded	Corroded (blank unaffected)	Corroded	Corroded (blank unaffected)	Corroded	Corroded (blank unaffected)
Epoxy Paint	Unaffected	Corroded	Unaffected	Corroded	Corroded	Corroded

7. Sprayed Aluminum - Method 120

These panels had a rough silvery coat before testing. All Group II testing did not affect the panels. Panels in this category were also tested with Group I panels. At 1200°F and 1400°F the coating flaked off in some areas, but at no time was corrosion of the base metal apparent. Areas of one panel exposed at 1400°F took on the appearance of Process 11 panels. After 1400°F, alternate condensation caused corrosion of the base metal, the corrosion product being red-brown in color.

8. Cadmium Electroplate

The bright coat of these panels was covered with a red-brown corrosion product after each exposure in the test furnace. The alternate condensation test aggravated this condition, but did not affect previously untested panels. No blistering or scaling was observed.

9. Epoxy Paint

These panels had a silvery coating. Exposures at 600°F and 800°F produced no visible attack. Exposure at 1000°F caused attack. After test furnace exposure, all panels failed in the alternate condensation test. The corrosion product was red-brown; its formation was not accompanied by blistering or scaling.

Results of Tests - Group III

To determine the rigidity of the test method and also to provide a further evaluation of the coatings, it was decided to test a number of panels from both Groups I and II in the test furnace at 1000°F using kerosene with no additives. These coatings were called Group III. The results are shown in Table III.

1. Paint Chromizing on Plain Carbon Steel

Corrosion and some scaling took place for the first twelve hours of testing. After that, little or no change was apparent.

2. Paint Siliconizing on Plain Carbon Steel

These panels became corroded after 6 hours; this corrosion increased with the time of the test. The alternate condensation test which followed this exposure yielded results which did not differ from any previous alternate condensation exposures for these same panels.

3. Electroless Nickel

No apparent effect.

4. Electroplated Nickel

No apparent effect.

TABLE III

Comparison Tests
With and Without Additives

30 Hours at 1000°F

Coating	Kerosene With Additives	Kerosene Without Additives
Electroless Nickel	Corroded	Unaffected
Electroplated Nickel	Corroded	Unaffected
G. E. Silicone Al Paint	Corroded	Unaffected
Sicon 3 x 222	Corroded	Corroded
Sicon 7 x 273	Corroded	Corroded
Epoxy	Corroded	Unaffected
Paint Chromizing	Corroded	Corroded
Paint Siliconizing	Corroded	Corroded

5. Silicone Aluminum Paint

No change in appearance.

6. Sicon Paint - 3 x 222

Panels slightly corroded after 6 hours. However, this corrosion did not increase with time.

7. Sicon Paint - 7 x 273

These panels behaved like Sicon 3 x 222.

8. Epoxy

Not affected.

Section X - Discussion and Conclusions

The corrosive agents, sulphur, vanadium, bromine and lead, added to the kerosene in the amounts described, greatly increased the corrosiveness of the atmosphere of combustion products.

The alternate condensation test proved to be of great value in attacking the affected areas, pin-hole porosity and other defects of the coatings, as well as in determining the extent of failures. As an example, such paint coatings as Sicon Paint - 3 x 222, Sicon Paint - 7 x 273 and Epoxy paint appeared completely unaffected by the furnace exposure to 600°F. No difference in appearance was observed between them and blanks. However, when these panels and blanks were subjected to alternate condensation testing the furnace tested panels failed quickly while the blanks remained unaffected.

The test work has shown that there is a vast difference between exposure in an atmosphere containing the products of combustion of hydrocarbon fuels and exposure at the same or higher temperatures in air alone. In other tests run on siliconized and chromized panels, the panels have been heated in air at a temperature of 1350°F for 70 to 128 hours with no scaling or flaking. When exposed to a test atmosphere containing the products of combustion of kerosene these coatings did not withstand 30 hours at 1000°F.

The test, as developed, shows a high degree of selectivity. Poorly protective coatings are readily distinguished from fair coatings and fair coatings are distinguished from good coatings.

Incidental by-products of the development of this method of test are as follows:

1. Electroless nickel coatings may not be as satisfactory as electroplated coatings.

2. Silicone-aluminum paints do not provide corrosion protection after service exposures at 600°F.

3. Sprayed and sealed aluminum (metallizing) provides protection up to 800°F.

4. Corronizing provides protection up to 1000°F.

5. Sprayed and fused aluminum provides protection up to above 1200°F.

6. Hot dip 13% silicon-aluminum coatings provide protection up to above 1200°F.

7. Hot dip 13% silicon-aluminum coatings may be applied to the titanium-boron low alloy steels as a precoat prior to their conditioning heat treatment.

It is possible that the test with additives is entirely too drastic for general application. It may be that coatings that fail to withstand bromine and lead compounds are penalized unduly by the use of these additives.

Additional development work in connection with this test method should be planned so as to determine the relative effects on finishes of the individual constituents of the test furnace atmosphere. The products of complete combustion of a pure hydrocarbon, such as kerosene are carbon dioxide and water vapor. The products of combustion of the additives which were used in the kerosene include sulphur dioxide, lead oxide, lead, lead bromide, hydrobromic acid, bromine, vanadium pentoxide and probably some sulphates. The relative corrosivity of each of these products is unknown either at the test furnace temperature or as condensate deposits plus moisture on the surfaces tested.

Additional test runs should include a variety of coatings, as before, exposed to atmospheres produced by the combustion of

1. Kerosene alone
2. Kerosene plus tetraethyl lead
   (a) 4 ml per gallon
   (b) 8 ml per gallon
3. Kerosene plus ethylene bromide
   (a) 2 ml per gallon
   (b) 4 ml per gallon
4. Kerosene plus carbon disulphide
   (a) 10 ml per gallon
   (b) 20 ml per gallon
5. Kerosene plus 0.05% vanadium as vanadium oleate.

It may be that the vanadium addition has little effect at service temperatures below 1000°F. It may be that for a standard test for coatings to be used in a service where the fuel does not contain tetraethyl lead, these additives should not be specified.

The oxygen, carbon dioxide and water vapor content of the test furnace atmosphere can be varied, within limits, by control of the kerosene torch.

The flexibility of this test method is quite evident.

1. The fuel can be varied.

2. The type and amount of impurities in the fuel can be varied.

3. The temperature of the test furnace can be varied.

4. The time of exposure in the test furnace can be varied.

5. The alternate condensation test period can be varied.

6. The alternate condensation test can be changed to include exposure to other types of atmosphere rather than plain air.

In its present state, the test is capable of differentiating between poor and good coatings from the standpoint of porosity, adherence, heat resistance and protection against corrosion. It may be possible to extend the test so as to establish minimum thickness requirements for specific coatings.