April 28, 1964 E. A. DIEMAN 3,131,029

SYSTEM FOR ACCELERATED CONDENSATION CORROSION

Original Filed Dec. 18, 1957 2 Sheets-Sheet 1

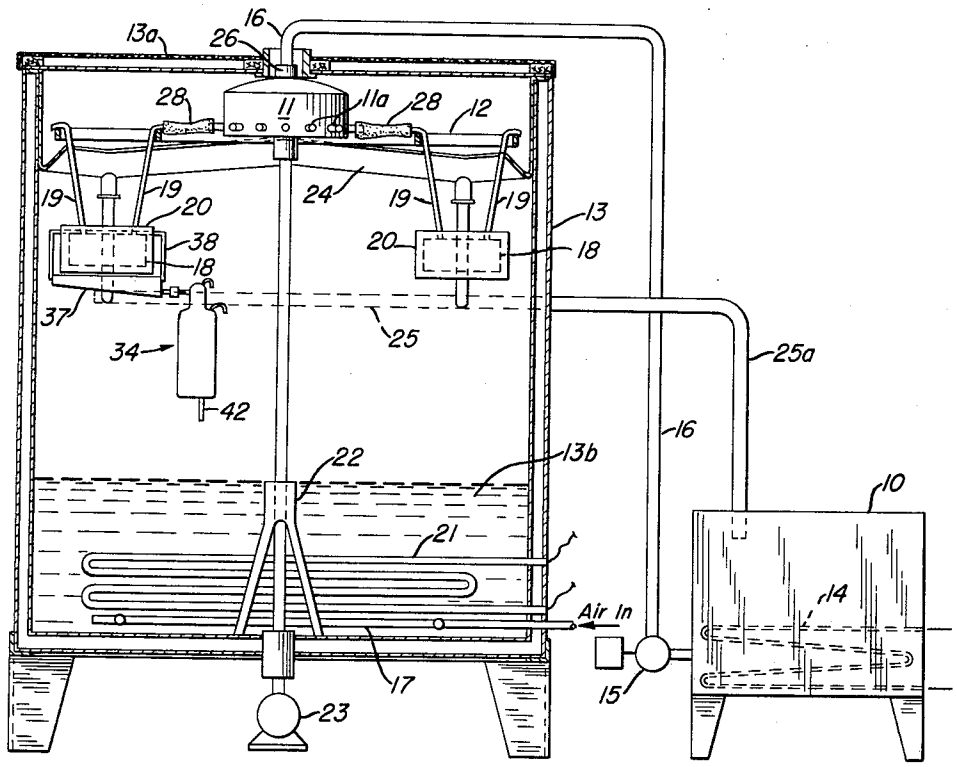


Fig. 1

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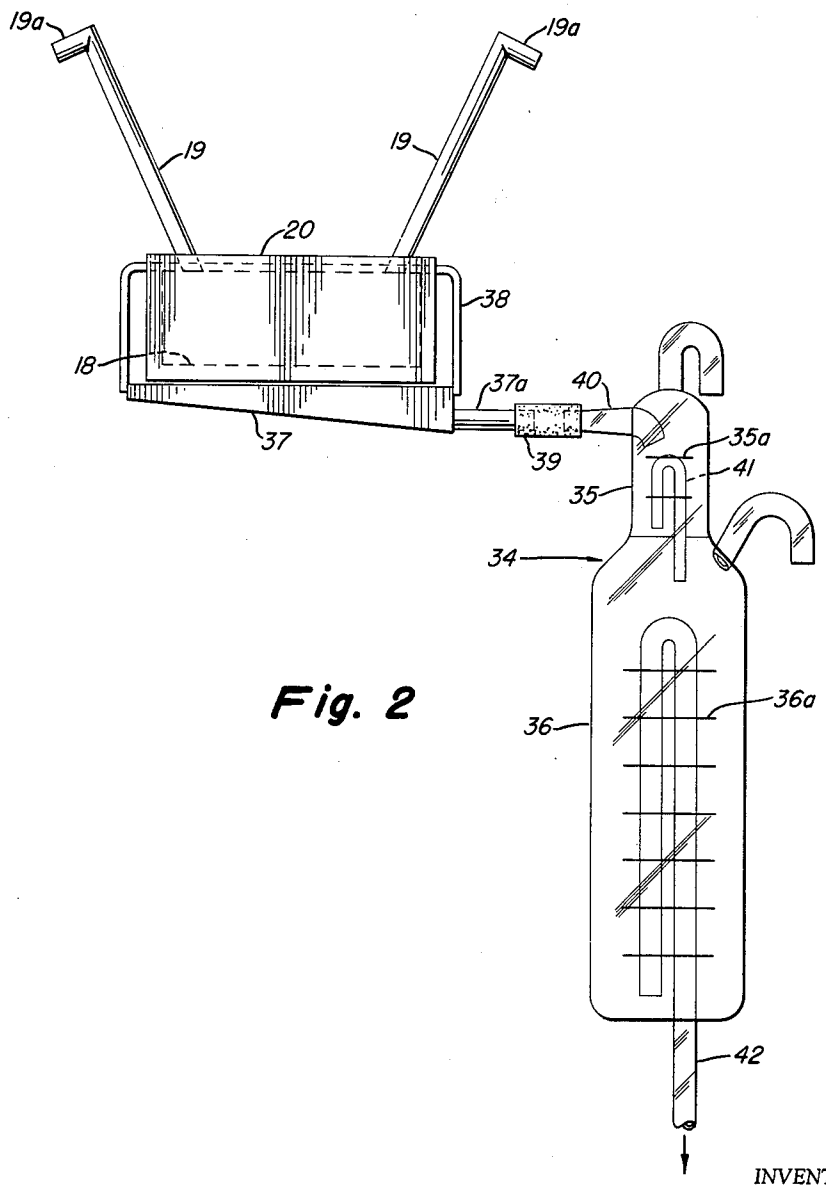


Fig. 2

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3,131,029

SYSTEM FOR ACCELERATED CONDENSATION CORROSION

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Original application Dec. 18, 1957, Ser. No. 703,530. Divided and this application Aug. 18, 1960, Ser. No. 50,436

1 Claim. (Cl. 23—253)

This is a division of my application, Serial Number 703,530, entitled "System for Accelerated Condensation Corrosion," filed December 18, 1957, and now abandoned.

This invention relates to a system for conducting corrosion tests on specimens for evaluating anti-corrosion properties of rust preventive compositions. More particularly, the invention relates to a test for quality control of rust preventives.

The humidity cabinet test procedure covered by JAN–H–792 specification is widely used for laboratory evaluation of rust preventives. In this test, steel specimens coated with rust preventive compositions are exposed to a humid atmosphere at 120° F. and are periodically examined for initial rusting. There are two objections to the test: (1) long testing time and (2) poor precision. The long testing time is objectionable to manufacturers of rust preventives because of holdup of shipments pending quality approval. The poor precision makes evaluations subject to question.

The JAN–H–792 specification is intended to provide continuous condensation on the test specimens and the long test time is due to the low condensation on the specimens. Recently, an apparatus has been developed whereby the rusting of the specimens is accelerated by increased condensation on the panels, obtained by keeping the specimens at a lower temperature than the surrounding atmosphere.

My co-pending application, Serial Number 581,486, filed April 30, 1956, issued as U.S. 2,897,060 and entitled, "Accelerated Condensation Corrosion Testing System," covers a suitable apparatus for my accelerated test. The humidity cabinet contains a plurality of test specimens maintained at a temperature differential with respect to the atmosphere within the cabinet. The metal specimens are maintained at a constant temperature by circulating a cooling fluid through the individual specimen structures which are suspended on a rotated stage. Water from an external coolant tank is pumped into a reservoir mounted onto the rotating stage. The coolant flows by gravity to the hollow or box-like structure supporting a panel specimen, and the coolant overflows into a trough from which it is returned by gravity to the coolant tank.

In some evaluations, it is desired to expose the specimens to a preselected extent of condensation without time limitation and in others it is desired to determine the condensation occurring on each specimen over a selected time interval in addition to obtaining uniformity of exposure and control in evaluating corrosion preventives. Heretofore apparatus for accomplishing these desired results has not been available. The techniques for specimen preparation have been unsatisfactory in that no significant improvement in precision has been obtainable with the techniques heretofore proposed.

It is therefore a primary object of my invention to provide a system which gives accelerated and reproducible results in evaluating the anti-corrosion properties of rust preventive compositions. It is a further object of the invention to provide an apparatus for conducting tests on individual compositions separately or on a plurality of compositions simultaneously. An additional object of the invention is to provide a system wherein the test can be made in a short time, have good precision,

and be suitable as a quality control test. Still another object is to provide a system for accelerated corrosion testing wherein the test specimens are prepared in accordance with a uniform preparation procedure to avoid variables which otherwise occur prior to the application of the preventive composition under test. A further object of the invention is to provide an apparatus associated with each of the selected specimens for determining the rate of condensation on an individual specimen. These and other objects of the invention will become apparent as the description thereof proceeds.

Briefly according to my invention, short testing time is obtained by accelerated condensation on the test specimens and good precision is obtained by close control of condensation and by the use of a specimen preparation procedure which minimizes the uncontrolled variables.

In one procedure the specimens are conditioned by sandblasting, water dipped, methyl alcohol dipped, and finally slushed in rust preventive. If it is desired to simulate conditions on highly machined metal parts the conditioning of the specimen can be conducted under water. Thus the conditioning may be conducted in two stages, the first stage being with an abrasive belt and the second stage being by hand polishing under water. This is followed by the alcohol dip before slushing in rust preventive.

To collect the condensate I provide a self-siphoning receptacle which receives condensate from a pan suspended under a specimen. The collector comprises two superposed sections, both sections including graduated portions. The upper graduated portion is of about 10 ml. capacity and receives condensate dropwise from the collecting pan. Upon becoming filled, the upper portion is emptied of fluid by a siphon arrangement into the lower graduated portion of about 200 ml. capacity. When the lower has become filled, the entire contents discharge into the humidity cabinet.

Further details and advantages of the invention will be described in conjunction with the preferred procedures and preferred embodiments of the apparatus, forms of which are illustrated in the accompanying drawing herein:

FIGURE 1 illustrates an apparatus of the type in which my invention may be used; and

FIGURE 2 is an enlarged elevation of a test specimen unit and condensate collector.

The basic features of the test apparatus are shown in FIGURE 1 wherein coolant water from an external coolant water from an external coolant tank **10** is pumped into a reservoir **11** mounted on a rotating stage **12** within the humidity cabinet **13**. The coolant tank **10**, typically having a capacity of about 5 gallons, is provided with temperature control means **14** to maintain the desired temperature. The pump **15**, with a minimum delivery capacity of about 1.5 gallons per minute is connected to the coolant tank **10** and delivers coolant to the reservoir **11** by line **16**. Air introduced by distributor **17** passes upwardly through the pool of water **13***b* in the bottom portion of the cabinet **13** and thence through the fabric cover **13***a*.

The coolant flows by gravity from the reservoir **11** through the hollow or box-like specimen structures **18** suspended from the rotating stage **12** by hooked ends **19***a* of the tubes **19**. The specimen structures **18** comprise the standard corrosion panel **20** forming one wall of the box-like specimen **18**. The panels **20** are thus maintained at a lower temperature than the surrounding humid atmosphere within the cabinet **13** and accelerated condensation is obtained. The atmosphere in the cabinet **13** is maintained humid and at the desired temperature level by means of the heater **21** within pool **13***b* of the water in the lower portion of the cabinet **13**. The stage **12** is

rotated by motor 23 driving the mounting base 22 which in turn supports and rotates the stage 12.

The coolant flows from the reservoir chamber 11 into the specimen unit 18 on the rotated stage 12. The reservoir 11 is stainless steel 2.5 inches deep and 6.0 inches in diameter. The tube 26 projects from the top of the reservoir 11 and through the cover 13*a* of the cabinet 13 to receive the coolant from line 16. The coolant overflows from the specimen units 18 into a trough 24 mounted on the inside wall of the cabinet 13 and is returned by gravity to the coolant tank 10 by the manifold 25 and the return tubing system 25*a*.

A plurality of stainless steel tubes 11*a* about 0.25 inch in diameter and about 1 inch long extend outwardly from the bottom edge of the reservoir 11 and are connected by flexible tubing 28 to the inlet tube 19 of the box-like specimen unit 18, details of which are shown in FIGURE 2. The test specimen 20 is a 2.0 by 4.0 by 0.125 inch plate of 1020 steel which is conditioned as described herein to provide the test surface.

The box-type specimen unit 18 is formed by soldering the 1020 steel plate 20 to the open side of a stainless steel box about 1.5 by 3.5 by 0.5 inches. Water inlet and outlet tubes 19, of 0.25 inch copper tubing, are attached near the ends of a 3.5 inch side of the box and project upwardly, terminating in hooks 19*a*, to support each specimen unit 18 on the rotating stage 12 within the cabinet 13. Coolant from tank 10 is pumped through the specimen units 18 at a rate of about 350 to 400 ml. per minute per specimen at a selected temperature for the required test operating conditions to attain the desired condensation rate.

The front wall of the cabinet 13 is fitted with a 6 by 15 inch window (not shown) which is positioned to permit observation of the specimens 18 as they are rotated on the stage 12. An internal windshield wiper (not shown) and an external spot light (not shown) are directed through the window to facilitate the viewing of the specimen surfaces without disturbing the cabinet 13.

Reverting to FIGURE 2, I have shown details of means for collecting the condensate from a specimen unit 18. Such collecting means may be applied to either a dummy test specimen, a control specimen, or a specimen actually under test depending upon the procedure being employed.

As described above, rust preventives may be rated in "time to failure" of a preventive film. However, since the amount of condensate formation on the coated steel specimens is the factor which promotes corrosion, the amount of condensate collected until rust appears on the specimens gives a true evaluation. Thus the collection of condensate compensates for any irregular or abnormal conditions in or about the humidity cabinet 13. The amount of condensate formed is a direct result of the operating and ambient conditions and not necessarily related only to time.

Changes in barometric pressure, temperature and humidity of the surrounding air, slight irregularity in air put through the cabinet 13 and undetected variations in temperature therein all contribute to the amount of condensate formed on the test panel. Since absolute control of these factors would involve extremely complicated and expensive equipment, the collection of the condensate compensates for any irregularity or combinations of conditions which may arise. Accordingly, the device of FIGURE 2 diminishes the necessity of strict temperature and air control in the apparatus of FIGURE 1.

The self-siphoning collector 34 comprises two sections, the small graduated upper receptacle 35 with its scale 35*a* and a larger graduated accumulation receptacle 36 with its scale 36*a*. A pan 37 is suspended by bale 38 below the specimen unit 18 and discharges through duct 37*a* into collector inlet 40. The graduated receptacle 36 is preferably transparent, is of about 10 ml. capacity, and receives condensate dropwise from the collecting pan 37.

Upon filling the upper graduated receptacle 35, the fluid flows by siphon action through siphon tube 41 into the lower accumulating receptacle 36. When this lower receptacle 36 has been filled, it discharges by siphon tube 42 into the pool 13*b* within the cabinet 13, thereby permitting continued and uninterrupted measurements to be taken. By providing the two stages 35 and 36, the collector 34 can operate on dropwise collection of condensate which is at a rate normally insufficient to activate the self-siphoning collector.

To prepare the specimen the 10–20 steel surface is sandblasted in two directions, horizontally and vertically, at a pressure of about 100 p.s.i. and with the surface about 1 inch from the nozzle of the sandblaster. After said blasting, the specimen is rapped sharply against the inside wall of the blasting equipment to remove adhering sand and immediately subjected to the following treatment inside the blasting chamber:

(1) Dipped in distilled water for 5 seconds,
(2) Immediately thereafter dipped with agitation in C.P. methanol for 5 seconds,
(3) Air dried for 15 seconds,
(4) Dipped in the rust preventive for 1 minute and,
(5) Allowed to drain for 16 to 18 hours at room temperature.

Three specimens 18 are also prepared for a single evaluation of the composition under test and two additional specimens are prepared and treated with a reference composition. The prepared specimens are mounted on the rotating stage 12 in the cabinet 13, the stage 12 being loaded to its full capacity to maintain repeatable operating conditions. The test is then operated under conditions shown in Table I, Specification JAN–H–792 operating conditions being included for comparison.

TABLE I

Operating Conditions

	Accelerated Test		Spec-JAN-H-792	
	Controlled	Resulting	Controlled	Resulting
Cabinet Water Bath, ° F.	130±1.0			130
Specimen Coolant Water, ° F	90±0.5			
Aeration, cu. ft./hr.	31.2±1.0		31.2±4.0	
Coolant Water Circulation, gal./min.	1.5±0.1			
Cabinet Atmosphere, ° F.		110	120±2.0	
Temperature Differential, ° F [1]		20		(0)
Condensation, ml./hr./specimen		12±0.7		(none to 0.2)

[1] Between cabinet atmosphere and specimen coolant water.

The specimens under test are viewed through the window of the cabinet at half-hour intervals and a specimen is rated "fail" at the time stain or rust is first observed provided that the amount of stain or rust is greater at the next inspection. The hours-to-fail values obtained in the three specimens of the test are averaged and the result a single value is reported as the test life of the oil under test.

In addititon to the method of specimen preparation involving the sandblasting of the surface I may prepare the specimen by polishing the surface in contact with water. This method offers advantages over the sandblasting technique and is useful where the inhibitor being tested is to be used on highly machined parts.

The steps in preparing a polished surface in contact with water according to the "under water" method is as follows:

(1) Machine polish using abrasive belts, the final belt being 240 grit aluminous oxide,
(2) Hand polish under water using 280–*c* grit, wet or dry, trimite Carborundum abrasive paper,

(3) Dip the wet specimen for 5 seconds in absolute methyl alcohol,
(4) Dry by shaking in air for about 15 to 20 seconds,
(5) Slush in rust preventive for 1 minute,
(6) Drain for 16 to 20 hours and expose in the humidity cabinet.

This preparation technique requires less specialized equipment, may be conducted in the laboratory and possible contamination by ambient air is avoided.

With the sandblast preparation method the time of submergence under water may be as short as one second or as long as about 30 seconds, or even longer.

The alcohol dip serves to dissolve, and thereby remove, excess water from the water-dipped specimen surface. Other solvents for water, e.g., acetone, may be used instead of alcohol.

A comparison of test time and precision using the conventional and the water-dip specimen procedures on sandblasted specimens is shown in Table II.

TABLE II

Effect of Specimen Preparation on Test Time and Precision

	Tests	Test Life, Hours		Standard Deviation, Percent
		Range	Avg.	
Conventional	10	2–11	6	45
Water-Treated	15	10–15	13	13

Repeatability refers to the variability of test life values and is expressed as standard deviation, percent, in accordance with ASTM recommendations. A higher but satisfactorily rapid testing time was obtained with the water-treated specimens. Repeatability using the water-dip treatment is about 3 times better than with conventionally prepared specimens. The significance of the data on water-treated specimens is strengthened by the fact that the results are from tests run over a substantial period.

A comparison of test time and precision of my accelerated test and the JAN–H–792 test is shown in Table III.

TABLE III

Comparison of Test Time and Precision

	Tests	Test Life, Hours		Standard Deviation, Percent
		Range	Avg.	
ACCT	15	10–15	13	13
JAN–H–792	6	98–344	224	40

The test life values reported for the JAN–H–792 tests were obtained by averaging the hours-to-failure values for each of the five specimens in a test. Testing time in the accelerated test is about 1/20 that required in the JAN–H–792 test.

Precision in the accelerated test is about 3 times better. Precision in the JAN–H–792 probably would be improved with water-treated specimens but the objectionably long testing time would still exist.

Other rust preventives have been tested in the accelerated test apparatus with test life values ranging from 4 to 40 hours. Testing times have ranged from 1/20 to 1/50 of those obtained in JAN–H–792 tests. Repeatability has been of the same order of magnitude as reported on the oil in Table III. Tests have also been run on oils with various levels of additive treatment. Test life versus concentration curves for different additives differ markedly.

The accelerated test, using a condensation rate of 12 ml./hour, satisfies the short-testing-time, good-precision requirements for a quality control test. Further test work is required to determine correlation of accelerated test evaluations with service performance of different rust preventives. Tests at a lower condensation rate may be desirable in such correlation studies.

Although the invention has been described by the reference to particular embodiments thereof, it is to be understood that these are by way of illustration only and that they are not intended to limit the invention thereto since alternative modes of operation and embodiments of the invention will become apparent to those skilled in this art without departing from the spirit of the invention as described.

I claim:

An apparatus for use in collecting and measuring condensate from a corrosion test specimen maintained within a test cabinet which comprises: a condensate collection pan positioned to directly collect condensate collecting on said test specimen unit, and a graduated self-siphoning receptacle means connected to said pan to receive and measure the condensate collected in said pan, said receptacle means comprising two sections, the upper section being of relatively small capacity and the lower section being of relatively large capacity, said receptacle means further comprising siphon means discharging from said upper section into said lower section and second siphon means discharging condensate from said lower section into said test cabinet.

References Cited in the file of this patent

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