MONS 088255

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Underwriters' Laboratories, Inc.
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207 E. OHIO ST., CHICAGO 11, ILLINOIS

INERTEEN 7336-9
60% AROCLOR 1260
40% TRICHLOROBENZENE
0.2% PPO

Miscellaneous Hazard 2689
Application No. 54C627

March 23, 1954

REPORT

on

LIQUID DIELECTRIC AND COOLING MEDIUM

Westinghouse Electric Corporation
Sharon, Pennsylvania

MONS 088256

TABLE OF CONTENTS

	Page No.
INTRODUCTION	1
DESCRIPTION	1
Product Covered by this Report	1
General Character and Use	2
Marking	2
THE INVESTIGATION	2
EXAMINATION AND TEST RECORD	3
Identification Tests	3
Flash Point Test	4
Fire Test	4
Ignition Temperature Test	5
Tests for Explosiveness of Vapors in Air	7
Explosive Range Tests	8
Decomposition Temperature Tests	9
Analytical Tests of Decomposition Products	9
Corrosion Tests	14
RECORD IN SERVICE	14
THE SUBMITTOR	15
Supervision of Product by the Laboratories	15
CONCLUSIONS	15
General Nature	15
Fire and Toxic Hazard	15
Corrosive Action	18
Stability	18
Uniformity	19
Conformity	19
RECOMMENDATION	

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INTRODUCTION

Since issuance of a listing of the dielectric medium Inerteen No 7336-8 covered by our report MH2689, dated May 17, 1948, this manufacturer has made a slight change in the composition of the dielectric medium, and has applied the designation Inerteen 7336-9 to the product.

DESCRIPTION

PRODUCT COVERED BY THIS REPORT:

Liquid dielectric and cooling medium "Inerteen No. 7336-9".

GENERAL CHARACTER AND USE:

This dielectric and cooling medium is a pale yellow liquid consisting essentially of a mixture of chlorinated hydrocarbons intended for use in electrical apparatus. The product is designed to render innocuous the free hydrogen chloride dissolved in the liquid after arcing has occurred.

MARKING:

Listee's name and "Inerteen No. 7336-9" on containers.

THE INVESTIGATION

The object of this investigation was to determine the fire and toxic hazard of the product.

The manufacturer gave confidential information as to the composition of the product, which was confirmed by Identification Tests.

Consideration of the composition of this product in comparison with that of Inerteen 7336-8 listed during manufacture, indicates that provided the flash point, fire point, and ignition temperature of the current product is approximately the same as that of Inerteen 7336-8, no difference in fire and explosion hazard is to be anticipated. The foregoing tests were therefore conducted on the current product, Inerteen 7336-9.

Considerations of composition indicate further that Inerteen 7336-9 would perform the same as Inerteen 7336-8 in all other tests previously conducted on Inerteen 7336-8. These tests are therefore recorded in this report as a matter of information, and completeness.

EXAMINATION AND TEST RECORD

DESCRIPTION OF SAMPLES

The manufacturer furnished a sample of the dielectric medium "Inerteen No. 7336-9".

IDENTIFICATION TESTS:

METHOD

Specific Gravity - The specific gravity was determined by means of a pycnometer.

Test for Chlorine - A few drops of the product together with a small piece of metallic sodium are heated in a test tube over a Bunsen burner. After the reaction is completed, the test tube is broken in water; the resulting solution is filtered; and nitric acid and silver nitrate solutions are added. The formation of a white precipitate indicates the presence of chemically combined chlorine.

Distillation Test - A 100-gram sample was distilled using a method (D20-30) of the American Society for Testing Materials.

RESULTS

The results of the identification tests are tabulated below.

Specific gravity at $\frac{15.6\ C\ (60\ F)}{15.6\ C\ (60\ F)}$	1.561
Test for chlorine	present
Distillation test	
Initial boiling point	209.0 C (408 F)
Per cent distilled below 235 C (455 F)	29.0 by weight
Per cent distilled between 235 C (455 F) and 270 C (518 F)	10.4 by weight
Per cent distilled between 270 C (518 F) and 365.5 C (690 F)	0.7 by weight
Per cent distilled above 365.5 C (690 F)	56.6 by weight
End point	390.0 C (734 F)
Residue, per cent	2.9 by weight
Distillation loss, per cent	0.4 by weight

FLASH POINT TEST:

METHOD

The flash point was determined with the Pensky-Martens closed tester using the method (D93-46) of the American Society for Testing Materials.

RESULTS

The flash point was 112.8 C (235 F). (1)

FIRE TEST:

METHOD

The fire test was made with the Cleveland open cup tester, using the method (D92-46) of the American Society for Testing Materials.

RESULTS

The sample gave negative (2) results in the Fire Test (3).

(1) -- In this connection see Note 3.

(2) -- Intermittent flashes were obtained but the product did not continue to burn. The sample was heated to the boiling point; the test was then discontinued.

(3) -- In view of the negative results obtained in the Fire Test, it is clear that the Flash Point Test cannot be depended upon to give a true measure of the fire hazard of the product. It was therefore necessary to conduct Tests for Explosiveness of Vapors in Air. See Page 7.

IGNITION TEMPERATURE TEST:

METHOD

The apparatus consists essentially of a combustion chamber surrounded by a solder bath which is heated at a constant temperature during the test by a gas burner. The temperature of the solder bath is measured by means of a calibrated thermocouple provided with a quartz tube to protect the hot junction.

The combustion chamber consists of a quartz flask of conical form with flat bottom, 4-1/2 in. (11.4 cm) in height, 2-3/8 in. (6.0 cm) in diameter at bottom, and 1-1/8 in. (2.8 cm) in diameter at top. It is of about 150 ml capacity (rated capacity 125 ml) having a ratio of surface area to volume of about 1.1.

Measured test samples in the liquid phase are introduced into the heated combustion chamber by means of a micro pipette. Different amounts of the sample are admitted to the chamber in successive tests in order to determine the minimum temperature at which the vapor of the liquid in any proportion with air will ignite. The residual vapors or gases in the combustion chamber are completely displaced by a stream of air in the interval between tests.

Ignition temperature tests are also conducted using a horizontal iron plate 10 in. square, and 1/4 in. in thickness, heated by a gas burner. Flames and hot gases from the burner are deflected from the upper surface of the iron plate by a strip of sheet iron extending 10 in. beyond each side of the plate. The deflector is bolted to the edges of the plate, asbestos gaskets being provided at the joint. A calibrated chromel-alumel thermocouple (No. 14 Awg) connected to a potentiometer is used to measure the temperature of the plate, the hot junction of the thermocouple being brazed to the center of the upper surface of the plate. Check measurements of the temperatures of the plate are also made employing an optical pyrometer of the disappearing filament type.

In these tests measured samples (up to 10 ml) were dropped on the center of the heated plate. A slow stream of air was applied to the evaporating sample in some of the tests. The minimum ignition temperature was determined by successive trial tests, during which the iron plate was maintained at progressively lower temperatures.

RESULTS

The results of the ignition temperature tests are tabulated below.

Ignition Temperature (4)	
Quartz Flask	Iron Plate (6)
563 C (1045.0 F) Lag 7 seconds (5)	716 C (1320.8 F)

In the ignition tests, using the heated iron plate, the combustion was very weak and did not show any tendency to propagate beyond the vicinity of the heated iron surface. It will be noted that there was no confinement of the vapors in the tests with the heated iron plate.

Note: (4) The main value of the ignition test is to determine the minimum temperature required to produce ignition under the most favorable conditions in the absence of a flame or spark, including ratio of vapor to air and ratio of heated surface to volume of vapor-air mixture. Under less favorable conditions, as when the liquid is applied to a hot plate, a higher temperature for ignition is required. A limitation of the ignition test in a small vessel is that it does not show whether flame propagation for any material distance will occur. After determining the ignition temperature there, it is necessary to obtain additional data having a bearing on flame propagation. These data are given by tests with hot plate and by tests for explosiveness of vapors in air.

(5) The term "lag" is used to denote the time interval between the introduction of the sample and the appearance of flame.

(6) Within experimental error, the optical pyrometer and the thermocouple gave the same values for the temperature of the iron plate.

TESTS FOR EXPLOSIVENESS OF VAPORS IN AIR:

METHOD

The apparatus consists of a cylindrical steel vessel, 6 in. in diameter (internal) and 26 in. long. It is provided with a small mica window and an outlet to the atmosphere. The apparatus is heated externally by gas burners. Openings are provided half-way between the ends of the vessel to admit electrode terminals connected to an induction coil. The electrode terminals are spaced to give a spark gap of 1/4 in.

An iron-constantan thermocouple connected to a potentiometer is used for rough measurements of the temperature of the vapor-air mixture inside of the vessel.

Samples ranging in volume from 20 to 80 ml were introduced into the cylinder which had previously been heated to a predetermined temperature. The outlet of the cylinder was closed with a loose asbestos plug. Sparks were passed between the electrode terminals at intervals of one minute.

The liquid was drained from the cylinder, and residual gases and vapors were displaced by a stream of air in the interval between tests.

RESULTS

No propagation of flame occurred at temperatures up to 110 C (230.0 F). Flame propagation with weak pressure effects occurred at 113 C (235.4 F). Moderate pressure effects were obtained at temperatures somewhat above 113 C (235.4 F).

EXPLOSIVE RANGE TESTS:

METHOD

The apparatus consists essentially of a glass chamber equipped with a spark gap and a stirrer. The chamber is cylindrical with the lower end hemispherical and is approximately 3 in. in diameter by 8 in. in height; it has a net volume of 905 ml. The apparatus is provided with a glass lid supporting the stirrer and the leads to the spark gap near the bottom of the chamber. An opening in the lid is provided for introduction of the sample. The glass chamber is immersed in a molten solder bath to a depth of 6 in.

In the tests the solder bath was maintained at a constant temperature of 400 C (752 F), a measured quantity of the liquid to be tested was placed in the apparatus and the vapors stirred. Sparks were then passed at the spark gap while observations were made for flame travel and pressure effects. Following the tests, the vapors were displaced by a slow stream of compressed air in preparation for the next test.

RESULTS

The results of the tests are tabulated below.

	Lower Limit	Upper Limit
Volume used in test, ml	0.042	0.105
Lb vaporized liquid per 1000 cu ft of air (calculated).	4.09	10.18
Per cent by volume (calculated)	1.6	3.9

Weak pressure effects were obtained in these tests as would be expected for mixtures near the limits of flammability.

DECOMPOSITION TEMPERATURE TESTS:

METHOD

The apparatus described in connection with Ignition Temperature Tests was used in these tests. The vapors evolved on introducing samples into the heated chamber were tested with moist blue litmus paper, a change in the color of the paper from blue to red indicating decomposition of the product. The minimum temperature at which litmus paper showed an appreciable change in color within a short time (three minutes) was considered to be the decomposition temperature.

RESULTS

The decomposition temperature was approximately 340 C (644 F).

ANALYTICAL TESTS OF DECOMPOSITION PRODUCTS:

METHOD

Hot Iron Surfaces - The cylinder previously described under Tests for Explosiveness of Vapors in Air was also used for exposing vapor-air mixtures to hot iron surfaces. Connections for withdrawing samples of the decomposition products for analysis were inserted in the outlet pipe. The cylinder was externally heated to a temperature of 600 C (1112 F) by gas burners.

Samples of the product in the liquid phase were introduced into the heated cylinder at a uniform rate. Samples of the decomposition products were withdrawn from the outlet pipe and hydrochloric acid, free chlorine, phosgene, carbon dioxide, oxygen, carbon monoxide, gases absorbed by bromine, and methane and other paraffin hydrocarbon gases were determined using the following methods.

Hydrochloric acid was determined by absorption with water and subsequent titration with standard alkali solution using methyl orange as an indicator. A 7-liter sample was drawn by a calibrated aspirator bottle through an absorption train consisting of three spiral form bubbling towers. Free chlorine interferes with the determination and is removed by mercury in contact with the water in the bubbling towers.

Tests for free chlorine were made by liberation of iodine from aqueous potassium iodide solution and subsequent titration with sodium thiosulphate solution using starch solution as an indicator. A 7-liter sample was drawn by a calibrated aspirator bottle through a train consisting of two spiral form bubbling towers containing potassium iodide solution.

Phosgene was determined by absorption in aqueous aniline solution. Phosgene reacts quantitatively with aniline to form diphenylurea, one molecule of phosgene being equivalent to one molecule of diphenylurea.

A 15-liter sample was drawn through a train of three bubbling towers containing freshly prepared aqueous aniline solution saturated with diphenylurea. Free chlorine interferes with the determination and was removed by granulated antimony contained in a tube through which the sample passed before it entered the bubbling towers. The precipitate of diphenylurea was separated from the aniline solution by filtration, washed with cold water, dissolved in ethyl alcohol, recrystallized, dried, and weighed.

Carbon dioxide, oxygen, carbon monoxide, gases absorbed by bromine and methane were determined in the Moorehead form of gas analytical apparatus after treating the sample with silver nitrate solution to remove hydrochloric acid.

Electric Arcs - The apparatus consists of a cylindrical steel vessel 10 in. in diameter (internal) and 18 in. high. It is provided with a small mica window and an outlet to the atmosphere. Openings are provided in the vessel to admit two electrode holders, one holder being fixed and the other movable. The electrodes are copper rods 1/2 in. in diameter; their arcing terminals are horizontal. A connection for withdrawing samples of decomposition products for analysis is inserted in the wall of the vessel. A small cylindrical settling chamber for the removal of solid products of decomposition is attached to the sampling connection.

The electrodes are connected to a 230-volt, d-c test circuit, the current being controlled by resistors. An ammeter is connected in the circuit and a voltmeter is connected across the electrode terminals.

A sample of the product, about 15 lb, was placed in the vessel, the arcing terminals of the electrodes being partially immersed in the sample. The arcing terminals were located on the axis of the cylinder 3-1/2 in. above the bottom. A continuous arc (170 amperes) was maintained between the electrodes until destruction of the arcing terminals (five minutes).

Samples of the decomposition products were withdrawn continuously during this period and tested for phosgene and chlorine using the methods previously described. Samples of the decomposition products were also collected in evacuated glass sampling tubes at the conclusion of the test and the chief constituents determined, employing the following methods.

Hydrochloric acid was determined by absorption in water and titration with standard alkali solution. Carbon dioxide, carbon monoxide, oxygen, gases absorbed by bromine, and paraffin hydrocarbon gases were tested for, using the Moorehead apparatus after treating the sample with water to remove hydrochloric acid.

Tests were also conducted with the arcing terminals of the electrodes 2-3/4 in. below the level of the sample in the vessel. A 21-lb sample of the product was used in these tests. The arcing terminals were located on the axis of the cylinder, 2 in. from the bottom. A continuous arc (125 to 190 amperes) was maintained between the electrodes until destruction of the arcing terminals. Samples of the decomposition products were withdrawn at intervals during the test (see Table I) and the chief constituents determined using the methods previously described.

RESULTS

The results of analytical tests of the decomposition products are shown in Table I.

TABLE I

RESULTS OF ANALYTICAL TESTS OF DECOMPOSITION PRODUCTS

Gaseous Component Determined	Per Cent by Volume of Gaseous Component: Decomposition by Hot Iron Surfaces — 5 Min After Start of Test	Decomposition by Hot Iron Surfaces — 13 Min After Start of Test	Decomposition by Electric Arcs, Terminals Partially Immersed in Sample — 5 Min After Start of Test	Decomposition by Electric Arcs, Terminals Immersed in Sample — 5 Min After Start of Test	Terminals Immersed in Sample — 7.5 Min After Start of Test	Terminals Immersed in Sample — 10 Min After Start of Test	Terminals Immersed in Sample — 11 Min After Start of Test
Carbon dioxide	15.2	15.6	0.7	0.0	0.0	0.0	0.0
Carbon monoxide	17.0	11.4	4.3	0.5	0.2	0.2	0.2
Oxygen	2.0	0.4	10.0	16.0	7.6	7.3	5.1
Gases absorbed by bromine	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Methane and other gaseous paraffin hydrocarbons	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Nitrogen (by difference)	58.3	65.1	51.0	65.8	31.7	30.1	21.6
		5 to 13 Min After Start of Test					
Hydrochloric acid		7.5	34.0	17.7	60.5	62.4	73.1

		Start of Test to 5 Min. After Start of Test	Start of Test to 11 Min After Start of Test
Chlorine	0.000	0.000	0.000
Phosgene	0.000	Trace (7)	Trace (7)

(7) Less than 0.001 per cent by volume.

MDNS 088270

CORROSION TESTS:

METHOD

The metals used in the corrosion tests were copper, yellow brass, iron, tin-lead solder, copper and tin-lead solder, and copper and iron coupled. Test specimens 3/4 in. wide and 6 in. long were cut from sheets of the metals. The specimens were buffed, measured, cleansed with soap and water, washed with ethyl alcohol and ethyl ether, dried, and weighed.

Each specimen was immersed for half its length in a sample of the product contained in a glass tube provided with a cork stopper. The tubes were heated to a temperature of 90 C in an electrically-heated oven controlled by a thermostat.

The samples were examined visually at weekly intervals during the test for evidences of corrosion. At the conclusion of the test, the specimens were cleansed, dried, and weighed and the change in weight per square centimeter of surface area calculated.

RESULTS

When heated in glass tubes containing the liquid at 90 C for 1608 hours, specimens of copper, yellow brass, copper and tin-lead solder, and copper coupled with iron showed no visible evidence of corrosion except for a slight discoloration of the surfaces above the liquid. Specimens of iron, iron coupled with copper, and tin-lead solder showed no change in appearance. The change in weight of the specimens was very small (less than 0.1 milligram per square centimeter).

R E C O R D I N S E R V I C E

Such materials have been in commercial service in increasing amounts during the past several years. The value of such liquids as dielectric and cooling media has been demonstrated.

THE SUBMITTOR

The submittor, Westinghouse Electric Corporation, is experienced in the manufacture of electrical apparatus and related products, many of which are listed by Underwriters' Laboratories, Inc.

SUPERVISION OF PRODUCT BY THE LABORATORIES:

This material will be placed under Reexamination Service.

CONCLUSIONS

GENERAL NATURE:

This product consists essentially of a mixture of chlorinated aromatic hydrocarbons.

The results of the Identification Tests indicate that the product is a mixture containing combined chlorine.

FIRE AND TOXIC HAZARD:

The product is nonflammable at ordinary temperatures. It is capable of forming moderately combustible and explosive mixtures with air under laboratory test conditions at higher temperatures, beginning at a temperature level of 113 C (235.4 F), but under practical conditions formation of combustible or explosive mixtures is regarded as extremely unlikely. The fire hazard is very small.

It will be noted that the flash point of the product is comparatively high, and that a so-called "fire point" was not obtained. While intermittent flashes occurred in the latter test, the product did not continue to burn. The flash point and so-called "fire point" tests, particularly the latter, in the case of chlorinated hydrocarbons are of limited value as a measure of fire hazard but are useful for purpose of identification. The practical significance of the above tests in the case of the product in question is that its fire hazard is of a low order.

As shown by the results of Tests for Explosiveness of Vapors in Air, the vapors failed to ignite or explode in tests at 110 C (230.0 F), but weak combustion occurred in tests at 113 C (235.4 F). Moderate explosions were obtained in tests at temperatures somewhat above this minimum value. At a temperature of 400 C (752 F) the lower limit of flammability (upward propagation) of the vapor was found to be 1.6 per cent by volume in air and the corresponding upper limit of flammability (upward propagation) was found to be 3.9 per cent by volume. It will be noted that this is a comparatively narrow explosive range.

The minimum ignition temperature was found to be 563 C (1045 F) as recorded in the results of Ignition Temperature Tests.

In the ignition tests using the heated iron no ignition occurred at plate temperatures below 714 C (1317.2 F). At higher temperatures the combustion was very weak and did not propagate beyond the vicinity of the heated iron surface.

In the tests with electric arcs the gases liberated, carbon monoxide and hydrochloric acid, were in such proportions as to be nonflammable alone or mixed with air. It will be noted that in the case of mineral oil under similar conditions, highly explosive gases would be formed.

The gases or fumes produced by burning or decomposition by hot metal surfaces or electric arcs include hydrochloric acid, carbon monoxide, and a small amount of phosgene. This will ordinarily constitute a toxic hazard only where the conditions are such as to cause confinement of the fumes as in a closed room in which combustion or decomposition is maintained by fire, highly heated surfaces, or electric arcs.

It is to be noted in this connection that oils in common use at present as the dielectric medium for electrical apparatus are readily combustible and when burning under conditions of restricted air supply form carbon monoxide in dangerous concentrations.

While phosgene is poisonous, our data indicate that the concentrations likely to result in the failure of a transformer or other electrical apparatus containing the product are not liable to endanger life.

The resulting concentration of hydrochloric acid in air when failure of a transformer or other electrical apparatus containing the product occurs will depend largely upon the conditions. It will constitute a toxic hazard only where the conditions are such as to cause a high concentration of the fumes as in a closed room. In ordinary use the small quantities of gases evolved will be vented to some safe location.

The exceedingly unpleasant and irritating fumes from the decomposition of the product even in concentrations of a very low order act not only to give warning of their presence but to prevent dangerous exposure of persons. While carbon monoxide is odorless and toxic, its formation from the product is always accompanied by the simultaneous formation of hydrochloric acid gas which gives adequate warning of its presence as brought out above.

It will be noted that combustion and decomposition of the product was maintained by heated surfaces (600 C, 1112 F) or by an electric arc in carrying out the Analytical Tests of the Decomposition Products. As shown by the results of the Decomposition Temperature Tests, the product was not appreciably decomposed at temperatures below 340 C (644.0 F).

CORROSIVE ACTION:

The product does not corrode the metals commonly used in electrical apparatus.

No corrosion occurred in the tests conducted on representative metals and alloys as recorded under Corrosion Tests.

It will be noted from the results of Analytical Tests of Decomposition Products that appreciable amounts of hydrochloric acid are formed on combustion or decomposition of the products in the presence of hot metal surfaces or electric arc.

STABILITY:

The product is reasonably stable and in use is unlikely to undergo decomposition resulting in an increase in fire hazard.

In the Decomposition Temperature Tests the product did not undergo appreciable decomposition at temperatures below 340 C (644.0 F). Consideration of the chemical structure and properties of the product also indicates that it is reasonably stable.

UNIFORMITY:

Consideration of the general process of manufacture indicates that it is practicable to maintain an acceptable degree of uniformity on a commerical scale.

CONFORMITY:

The product conforms to the definition of an askarel as given in Article 100, 1953 Edition of the National Electrical Code.

MONS 088276

R E C O M M E N D A T I O N

TO THE ELECTRICAL AND FIRE COUNCILS OF UNDERWRITERS' LABORATORIES, INC.:

We recommend promulgation of the following notice to subscribers and the action indicated thereby:

Guide No. 80 IO. March 23, 1954 File MH2689.

Westinghouse Electric Corp.,
469 Sharpsville Ave., Sharon, Pa.

Dielectric Medium.

Synthetic liquid intended for use as a dielectric and cooling medium in electrical apparatus, particularly transformers.
Nonflammable and nonexplosive at ordinary temperatures.
Marking: Listee's name and "Inerteen No. 7336-9".

Listed by report -- The description of this dielectric medium, an account of the examination and tests made or witnessed by Underwriters' Laboratories, Inc., and the conclusions derived therefrom are contained in a report filed MH2689 dated March 23, 1954, copies of which can be obtained upon application to the submittor or to Underwriters' Laboratories, Inc. (This product is not intended to replace oil as a dielectric and cooling medium unless equipment is designed for either oil or this product).

Listed -- Reexamination Service.

See description of Reexamination Service on guide card.

Tests by:
R. E. Dufour
C. C. Clogston
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Report by:
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Reviewed by:
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SUBMITTED BY:

A. F. Matson
A. F. MATSON
Chemical Engineer

The foregoing Recommendation has been accepted 4-26-54.

UNDERWRITERS' LABORATORIES, INC.

JW:CCW

(Signed) W. S. Austin
Secretary

MONS 088277