

DOBLE ENGINEERING COMPANY

ELECTRICAL INSULATION ENGINEERS AND CONSULTANTS
FIELD TESTING AND MAINTENANCE OF HIGH VOLTAGE INSULATION

June 4, 1971

Mr. J. C. Wygant
Monsanto Company
Organic Chemicals Division
800 North Lindbergh Boulevard
St. Louis, Missouri 63166

Dear Mr. Wygant:

Report No. 3
Electrical Insulating Fluid MCS 1112
Lot OR 180213

In accordance with your letter of March 11, we have performed tests on a sample of the subject fluid which we received in Belmont on March 24, 1971. The results of our tests are included in the attached copy of our Laboratory Report C-1236 (Sheets A-H). For comparison purposes, we have also listed the test limits specified in our Transformer Oil Purchase Specification (TOPS), and have included the results of tests performed on a sample of currently acceptable electrical insulating oil produced by sulfuric-acid refining of a predominantly naphthenic-base crude. Our tests consisted of:

- A. Screen tests, including
 - 1. Dielectric breakdown of samples before and after aging
 - 2. Neutralization Number of samples vs. hours of aging
 - 3. Interfacial Tension of samples vs. hours of aging
- B. Oxidation Tests
 - 1. Sludge-free life
 - 2. Power-factor valued oxidation (PFVO)
- C. Impulse Breakdown, before and after aging
- D. Gassing Tendency

DSW 201710

Addressee Monsanto Company

Date June 4, 1971

Page 2

We believe that our comparative screen-test data speak for themselves, noting that the Aniline Point and Viscosity constitute two areas of considerable difference between the two samples compared.

Our Sludge-Free Life and PFVO tests are commonly applied measurements of the chemical and electrical stability of insulating oils. As predicted, the Sludge-Free Life test does not appear to be applicable to dodecyl benzene, no second phase (end point) was observed during 140 hours of aging time. The acid-refined oil sample developed incipient sludge after 80 hours; therefore, its Sludge-Free Life is recorded as 72 hours (the last sludge-free sampling period). It would appear that better bases for comparison of the two fluids are offered by the PFVO Characteristic (power factor versus time), and the plots of Neutralization Number and Interfacial Tension versus aging time, all of which are included in the report.

The results of Impulse Breakdown tests before and after aging are tabulated for both fluids for comparison purposes.

Gassing Tendency tests indicate that the dodecyl benzene sample is an "absorber" based on our test. The latter is discussed in detail in the enclosed copy of a 1960 Doble Client Conference paper by Dr. G. H. von Fuchs.

We hope that the foregoing comments and our enclosures provide some of the information you require regarding the sample of dodecyl benzene which you submitted to us. Following your review of our data, please advise us of any comments or questions you may have.

Incidentally, as discussed in a telephone conversation between Mr. R. I. Lowe and Mr. P. G. Benignus on June 1,

DSW 201711



STLCOPCB4059778

Addressee Monsanto Company

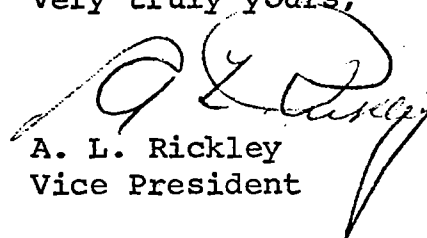
Date June 4, 1971

Page 3

we are interested in the possibility of your releasing any of the data we have recorded for possible discussion with our clients, particularly our Special Oil Committee.

We look forward to hearing further from you.

Very truly yours,



A. L. Rickley
Vice President

ALR:o
In duplicate

Enclosures

DSW 201712

3

dobe

STLCOPCB4059779

LABORATORY REPORT
TO
MONSANTO COMPANY

The following is a tabulation of the data obtained for comparative tests on a sample of a representative new oil and a sample of dodecylbenzene which we received in Belmont March 24, 1971. The samples were identified as indicated.

Tests	TOPS Limits	MCS 1112	Control
			Representative Mineral Oil
Acidity, mg. KOH/g	.025	<0.01	<0.01
Aniline Point, °C	78	44 ✓	75
Color, D-1500	0.5	1.0	<0.5
Corrosive Sulphur	Passes	Passes	Passes
Dielectric Strength, kV D-877	26 min.	52	46
Dielectric Strength, kV D-1816	20 min.	27	26
Flash Point, °C	130 min.	194	139
Fire Point, °C		214	160
Interfacial Tension, dynes/cm.	40 min.	48.3	50.2
Pour Point, °F	-40 max.	Passes	Passes
Power Factor, % at 100°C	0.3 max.	0.028	0.03
Power Factor, % at 25°C	0.05 max.	0.002	0.002
Specific Gravity 60/60	.865 -.910	0.897	0.885
Viscosity at 100°F, SUS	62 max.	144 ✓	61
Sludge-free Life, hours	64 min.	140+ ✓	72
Interfacial Tension after 140 hrs. oxidation dynes/cm		15.7	17.6

REPORT BY A. R. Bompane
A. R. Bompane

APPROVED BY A. M. Downey
A. M. Downey

(C-1236)

Sheet A

DSW 201713

May 24, 1971

Doble Engineering Company Belmont, Massachusetts



STLCOPCB4059780

SUPPLEMENTARY DIELECTRIC BREAKDOWN MEASUREMENTS

Oxidation Time (Hours)	MCS 1112		Representative Mineral Oil	
	D-877 (kV)	D-1816 (kV)	D-877 (kV)	D-1816 (kV)
	<u>D-877 (kV)</u>	<u>D-1816 (kV)</u>	<u>D-877 (kV)</u>	<u>D-1816 (kV)</u>
0	52	27	46	26
72	-	-	34	21
140	31	22	32	24

(C-1236)

Sheet B

DSW 201714

REPRESENTATIVE
MINERAL OIL

Sludge-free Life - 72 Hours

(C-1236)

Sheet C

% Power Factor @ 95°C

DSW 201715

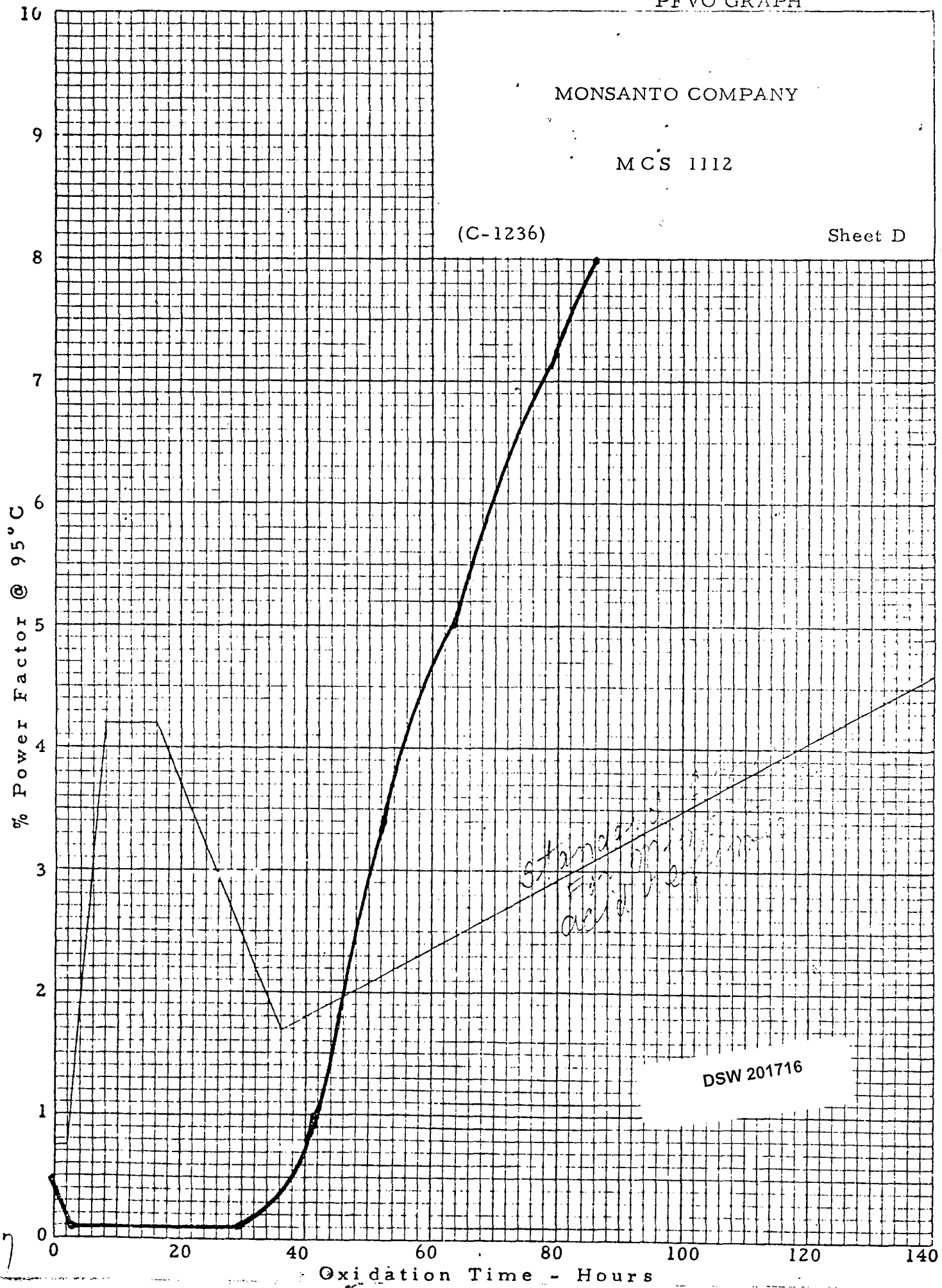
6
0 20 40 60 80 100 120 140
Oxidation Time - Hours

MONSANTO COMPANY

MCS 1112

(C-1236)

Sheet D



Neutralization Number

vs.

Hours of Oxidation

(C-1236)

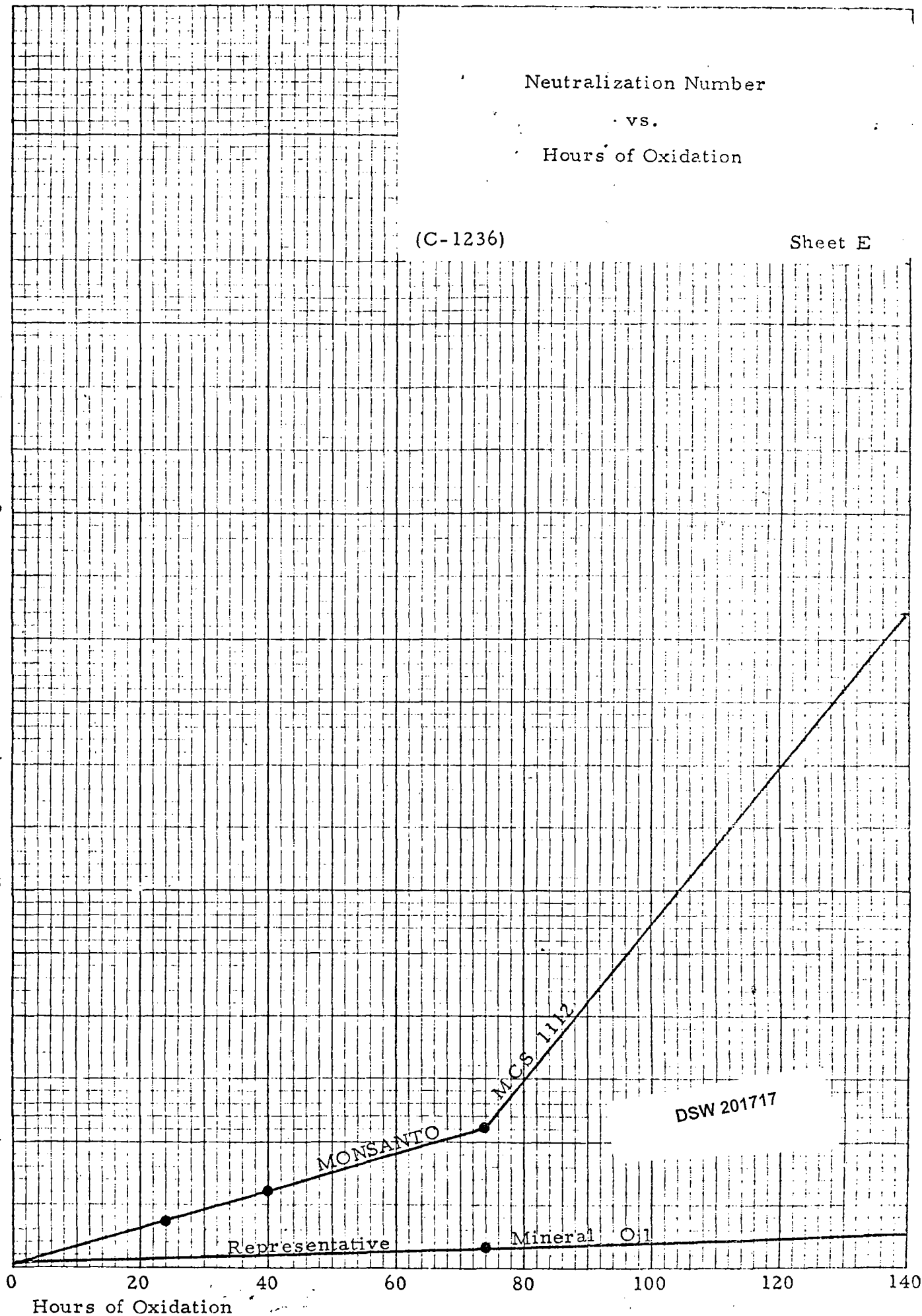
Sheet E

SPAULDING-MOSS INC.
BOSTON, MASS. 02210
MADE IN U.S.A.

NO. 4-10 SEMILINE GRAPH PAPER
10 X 10 PER INCH

Mg. KOH/gm.

Neutralization Number



DSW 201717

Interfacial Tension dynes/cm.

vs.

Hours of Oxidation

(C-1236)

Sheet F

SPAULDING-MOSS INC
BOSTON, MASS. 02210
MADE IN U.S.A.

NO. 4-10 SEMILINE GRAPH PAPER
10 X 10 PER INCH

Interfacial Tension dynes/cm.

Hours of Oxidation

Representative Mineral Oil

Monsanto MCS 1112

DSW 201718

STLCOPCB4059785

IMPULSE TESTS ON MONSANTO MCS 1112 AND A REPRESENTATIVE MINERAL OIL

1. MCS 1112 - As Received

<u>Electrode Configuration</u>	<u>Test Wave Description</u>	<u>Polarity</u>	<u>Breakdown</u>		<u>Avg.</u>	<u>Temperature</u>		<u>Barometric Pressure</u>	<u>Date</u>
			<u>Crest</u>	<u>Voltage</u>		<u>Amb.</u>	<u>Sample</u>		
Point to Sphere 1.0-Inch Gap Space	1.2x50 μ secs	Negative		145-kV	146-kV	75°F	75°F	29.97	5-11-71
				155-kV					
				145-kV					
				150-kV					
				135-kV					

2. MCS 1112 - After 140 hrs. Oxidation Time

Point to Sphere 1.0-Inch Gap Space	1.2x50 μ secs	Negative		120-kV	120-kV	75°F	75°F	29.97	5-11-71
				115-kV					
				115-kV					
				130-kV					
				120-kV					

3. Representative Mineral Oil As Received

Point to Sphere 1.0-Inch Gap Space	1.2x50 μ secs	Negative		160-kV	156-kV	77°F	77°F	29.63	5-4-71
				145-kV					
				155-kV					
				155-kV					
				165-kV					

DSW 201719

4. Representative Mineral Oil - After 72 Hours Oxidation Time

<u>Electrode Configuration</u>	<u>Test Wave Description</u>	<u>Polarity</u>	<u>Breakdown Crest</u>		<u>Temperature</u>		<u>Barometric Pressure</u>	<u>Date</u>
			<u>Voltage</u>	<u>Avg.</u>	<u>Amb.</u>	<u>Sample</u>		
Point to Sphere 1.0-Inch Gap Space	1.2x50 μ secs	Negative	140-kV	122-kV	75°F	75°F	-30.14	5-19-71
			115-kV					
			105-kV					
			130-kV					
			120-kV					

5. Representative Mineral Oil - After 140 Hours Oxidation Time

Point to Sphere 1.0-Inch Gap Space	1.2x50 μ secs	Negative	110-kV	117-kV	73°F	73°F	29.75	5-14-71
			130-kV					
			115-kV					
			115-kV					
			115-kV					

DSW 201720

GASSING TENDENCY TEST DATA

ASTM, D-2300

Procedure B

This test was performed to measure the rate at which gas is evolved or absorbed by the insulating fluid when subjected to electrical stress of sufficient intensity to cause ionization.

Gassing Tendency

<u>Sample Designation</u>	<u>Cubic Millimeters/Minute*</u>
MCS1112	-38
Representative Mineral Oil	+2

*Conditions of Test

Test Temperature	65° Centigrade
Test Voltage	10-kV
Saturating Gas	Hydrogen
Test Period	100 Minutes (between 20 and 120 minutes elapsed from start of test -- i.e., voltage application)
Test Method:	Doble (Modified Thornton-Pirelli) ASTM D-2300

DSW 201721

(C-1236)
Sheet H

DOBLE GASSING TENDENCY TEST

G. H. von Fuchs
Doble Engineering Company

* * *

Resistance to gassing under high electrical stress has long been recognized as one of the essential properties of cable oils. Quite a number of tests have been proposed but so far none has found general acceptance, partly because of the complexity of the test apparatus and procedures involved and partly because of the lack of repeatability and reproducibility of the test and the length of time required.

During 1957 ASTM became interested in developing a gassing test and the Doble Company agreed to co-operate. The apparatus proposed by Okonite (1) was a modification of the Beaven-Cockburn-Thompson Cell (2) which in turn was evolved from the Pirelli cell (3). When an Okonite type apparatus was obtained, it turned out to be so complex in design that it was soon decided to make a fresh start.

One characteristic all the previous test procedures had in common was the requirement that the test oils were first to be thoroughly de-gassed and then re-saturated with the gas to be used. This required an elaborate, vacuum tight degassing and re-saturating system and resulted in expensive apparatus and cumbersome and time-consuming test procedures.

It occurred to us at the Doble Company, that if the oil to be tested could be freed of extraneous gases in the gassing cell by bubbling the test gas through it for a period of time, both the apparatus and the procedure would be simplified. Another advantage not immediately apparent, was that since there would be no requirement for creating or holding a vacuum, no stopcock grease need contaminate the apparatus, the oil under test being used as a lubricant. Among other improvements introduced by the Doble Company were a reduction in the dead gas space to the absolute minimum to speed up stripping and a specially-designed heating bath to increase precision of test results. The use of a bath also permitted testing at several temperatures.

The Doble gassing test apparatus shown in Figure 1 contains the following parts: (1) a transformer with control-box as a source of the high-voltage a-c current, which is normally 10,000 volts, but lower and higher voltages can also be used; (2) a thermostatically-controlled oil bath with stirrer and low-lag blade-type electric heaters, a thermometer reading to 0.1°C, and a mercury thermo-regulator connected to a super-sensitive electronic relay; (3) a gassing cell and manometer assembly suspended in the bath; and (4) a pressure-regulated source of test gas which may be hydrogen, nitrogen, helium or any other reactive or inert gas or gas mixture. The operator is protected by a transparent safety shield (not shown) which is equipped with an electrical interlock. The manometer outlet is connected to a plastic tube leading out-of-doors. This is an essential safety feature when the test gas is hydrogen. The gassing cell and manometer

DSW 201722

assembly shown in Figure 2 is composed of a Pyrex glass cell with the part under stress constructed of true-bore tubing. This cell has an outer electrode of painted-on silver with a vertical slit for observation of the oil level and a copper band for connecting to ground. The silver electrode is covered by a coating of oil-resistant enamel.

The principal novelty and distinguishing feature of the apparatus is the hollow high-voltage electrode which permits stripping the oil sample in the cell with the test-gas at atmospheric pressure while the cell is immersed in the thermostatically controlled oil bath maintained at test temperature. Thus the need for vacuum tight, complicated auxiliary equipment normally used for de-gassing the oil sample is eliminated; and this operation, as well as saturation of the oil sample with the test gas, is accomplished in a greatly simplified single step. Therefore considerable time is saved and the two operations can be completed in 20 minutes instead of the several hours which were required previously.

The manometer is connected to the side-arm of the gassing cell by a standard taper ground-glass joint and held in place by spring clamps. The manometer has an etched-on millimeter scale and is so constructed that the manometer-liquid (di-butyl-phthalate) can not contaminate the oil in the test cell.

A schematic drawing of the cell and manometer assembly is shown in Figure 3. During part of the stripping operation, the test gas, which enters the cell at the lower end of the inner electrode, passes through the test oil and through the manometer liquid. This is intended to remove all extraneous gas from bulb A and also explains the need for bulb C. Bulb A serves as a reservoir for the manometer liquid when hydrogen absorbing oils are tested, while bulb B permits the use of the full length of the graduated manometer arm when gas is evolved. The stopcock is used both to by-pass the manometer during the last ten minutes of the stripping period and to equalize the manometer liquid levels after stopping the gas flow.

Detailed dimensions of both the glass cell and the inner (high tension) electrode are shown in Figure 4. This electrode is made of 10 mm o. d. centerless ground stainless steel seamless tubing and contains a brazed in stainless steel capillary tube as gas passage. The electrode is supported and centered in the glass cell by a precision machined, recessed 24/40 tapered Teflon plug. A 1/8" needle valve with gas inlet is on top of the electrode.

The test procedure is as follows: 4.8 ml of the oil to be tested is added to the cell and the electrode inserted using a few drops of the test oil as lubricant and sealant. The cell and manometer assembly is suspended in the oil bath previously heated to the test temperature (95°C). The gas inlet and outlet connections are attached and the test gas passed through the oil and through the manometer liquid as a steady stream (about three bubbles/second) for ten minutes. Following this, the bypass stopcock of the manometer is opened and gas stripping and saturation of the oil sample is continued for another ten minutes. Then the gas supply and the electrode inlet valve are turned off and the bypass stopcock is closed, the high-voltage and ground leads are connected to the cell and the safety shield is lowered. Before turning on the current, the manometer level is read and the readings repeated at one minute intervals until two successive readings give the same value, which is recorded. Then the current is turned on by gradually increasing the voltage until the test voltage (10,000 volts) is reached, this voltage being maintained throughout the test. The ASTM testing time is normally 100 minutes and is preceded by a 20-minute run-in period. The alleged purpose of this run-in period is to arrive at a

DSW 201723

relatively steady reaction rate. Thus in our routine gassing test, manometer readings are taken 20 minutes and 120 minutes after the current is turned on and the difference is recorded in millimeters per 100 minutes.

If a decrease in gas volume is observed such as with oils absorbing hydrogen in the test, the results are marked with a negative sign.

The test results are expressed in cubic millimeters per minute which is arrived at by multiplying the millimeter change in liquid level by the manometer constant and dividing the result by the test time in minutes. In our case the manometer constant is 25 cubic millimeters per millimeter change in manometer level.

$$\text{Average gassing rate} = \frac{V_2 - V_1 \times 25}{100} = \frac{V_2 - V_1}{4} = \text{cubic mm/minute}$$

V_2 is the reading of the manometer at 120 minutes.

V_1 is the reading of the manometer at 20 minutes.

Gassing test values for fourteen commercial oils with hydrogen and with nitrogen as test gases are shown in Table I, which also contains the specific optical dispersion (4) values of these oils.

While hydrogen is the generally accepted test gas, we at the Doble Company believe that the use of nitrogen is more realistic. Hydrogen is hardly ever encountered in normal service, while nitrogen is the principal constituent of cavities believed to be the initiators of the gassing reaction. Nitrogen is also commonly used as an inert gas blanket in electrical equipment.

In the gassing reaction, hydrogen is split off from hydrocarbons under electrical stress. Saturated hydrocarbons which are the principal constituents of over-refined oils lose hydrogen much more readily than do the so-called "unsaturates" or aromatics. The chemical action of electrical stress is comparable to that of thermal stress which in the petroleum industry is referred to as "cracking". Due to their unsaturated nature, the thermally and electrically more stable aromatics are capable of chemically absorbing the hydrogen atoms split-off from the saturated hydrocarbons before these atoms have a chance to combine to molecular hydrogen. Since gassing is largely a vapor phase reaction, oils containing the right kind of (light) aromatics will resist gassing.

Specific dispersion is a measure of total aromaticity and thus does not always correlate with gassing tendency. Examples of such inversions are Samples 1 and 2 and Samples 13 and 14. In the case of Samples 1 and 13, it is assumed that the oils have less of the light aromatics than their counterparts, Samples 2 and 14. According to its specific dispersion value (100.4) Sample 2 is very low in aromatic content: it is practically a white oil. Thus it is interesting to note that Sample 1 with a substantially higher aromatic content gasses more, when either hydrogen, or nitrogen is used as a test gas.

While the right kind of aromatics are capable of absorbing activated hydrogen atoms and thus make an oil an absorber when using hydrogen as a test gas, certain highly reactive olefinic compounds absorb hydrogen even more readily. An experimental cable oil of low aromaticity but containing such a reactive, presumably olefinic, compound as an additive, was capable of absorbing more hydrogen than was a commercial cable oil of

DSW 201724

much higher aromatic content. However, when the same two oils were tested with nitrogen, as shown in Table II, the situation was reversed, Sample 1 formed more gas than Sample 2.

These results and similar confirmatory work with argon and helium as test gases convinced us at the Doble Company, that gassing tests with hydrogen are not always reliable because the test can be passed when additives are present which are capable of absorbing activated hydrogen when it is used as a test gas and is thus present in excess; but these same additives do not readily absorb the hydrogen atoms split off from saturated hydrocarbons under electrical stress.

Nitrogen activated in an electric field is not completely inert. In the gassing test with a highly aromatic oil, we were able to show that nitrogen is absorbed giving negative test values. When the same oil was tested with truly inert gases such as argon or helium, the manometer level remained constant even when the test was extended over 8 hours (500 minutes) showing that no hydrogen was liberated.

These findings further justify the Doble Company's preference for electrical oils which contain light aromatic hydrocarbons, since the proper type of aromatics will absorb nitrogen as well as hydrogen, and such oils will tend to decrease or will even eliminate voids in cables by absorption and thus further decrease the chances for gas formation under electrical stress.

In conclusion, the Doble Gassing Tendency test presented here shows considerable promise. The design of the gassing cell is simple and the test is easily repeatable and reproducible.

As evidenced at recent ASTM meetings, there is considerable interest in a practical gassing test for cable oils. While some of the transformer people are not as yet concerned with the need for such a test, improvements in transformer design will bring about this requirement for transformer oils as well. In the meanwhile, the use of non-gassing transformer oils should be regarded as additional insurance for trouble-free service performance. This is expressed by the Doble Company's inclusion of the gassing tendency test in their oil specifications.

REFERENCES

- (1) S. C. Bartlett and R. B. Blodgett; "A Correlation Between Gas Evolution in Electrically Stressed Oils and Other Estimates of Oil Aromatic Content" Paper presented at the Twenty-Eighth Annual Meeting, N. R. C. Conference on Electrical Insulation, October 26-28, 1959, Pocono Manor Inn, Pocono Manor, Pa.
- (2) G. H. Beaven, J. A. Cockburn and C. N. Thompson; "The Evaluation of Gassing Tendencies of Insulating Oils: Apparatus, Procedure and Effect of Experimental Variables", Journal of the Institute of Petroleum, Vol. 35, No. 311, p. 735-754, November 1949.
- (3) G. Palandri and A. Pelagatti; "Insulating Oils for Electric Cables" paper presented at Convention of the Associazione Elettrotecnica Italiana in Bellagio, Italy, October 1954.

DSW 201725

- (4) G. H. von Fuchs and A. P. Anderson; "Effect of Solvent Extraction on Aromaticity of Lubricating Oils" Industrial and Engineering Chemistry Vol. 29, pages 319-324. (March 1937)

TABLE I

Doble Gassing Tendency Test -- (120-20 min) at 95°C

Sample No.	Cubic mm/min.		Aromatics Spec. Disp. *	Sample No.	Cubic mm/min.		Aromatics Spec. Disp. *
	H ₂	N ₂			H ₂	N ₂	
1	+34.5	59.9	105.8	8	-7.8	18.4	114.4
2	+32.6	44.8	100.4	9	-8.6	21.0	115.4
3	+9.2	33.4	107.0	10	-11.2	23.6	114.4
4	+8.0	29.8	109.0	11	-11.2	20.0	114.9
5	+8.0	34.9	111.1	12	-11.5	18.8	114.4
6	-0.4	24.6	113.2	13	-18.6	21.1	119.7
7	-1.5	28.6	111.2	14	-27.0	14.8	117.0

* Specific Dispersion by Dispersometer

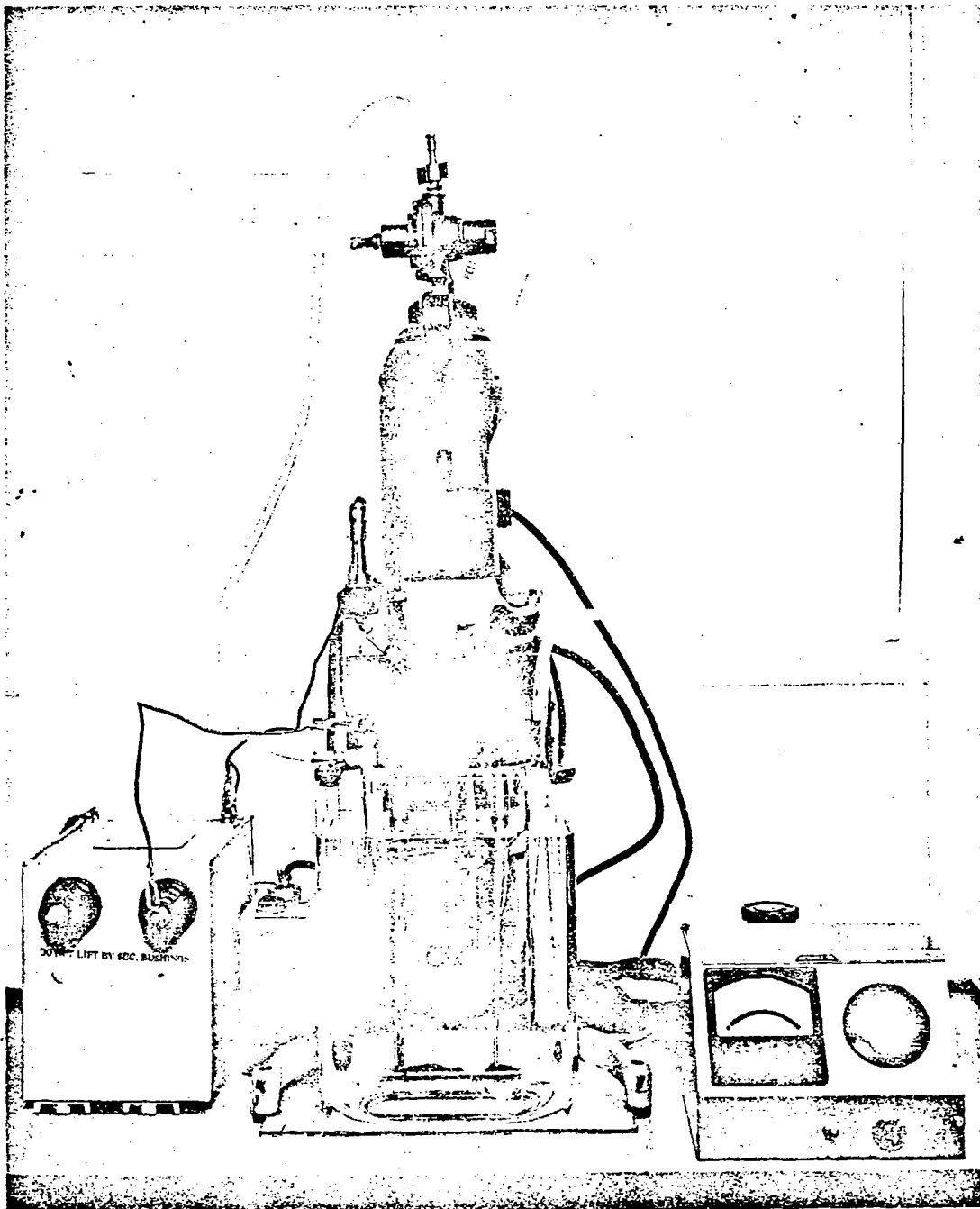
TABLE II

Sample No.	Doble Gassing Tendency		Aromatics Specific Dispersion*
	H ₂	N ₂	
1	-20.1	+27.3	100.5
2	-8.9	+17.3	115.5

* By Dispersometer

Note: Sample 1 contains a hydrogen absorbing additive

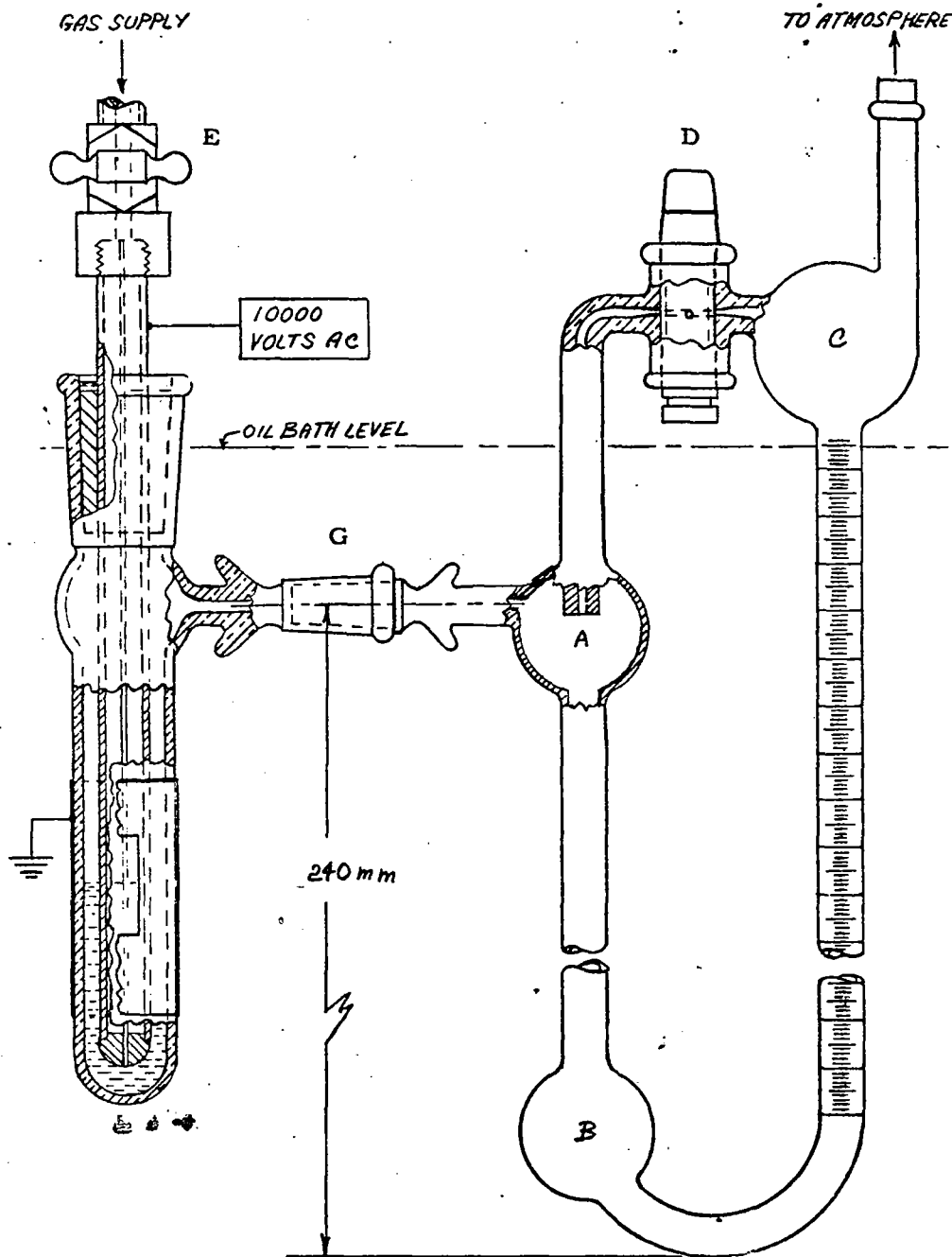
DSW 201726



Doble Gassing Test Apparatus

FIGURE 1

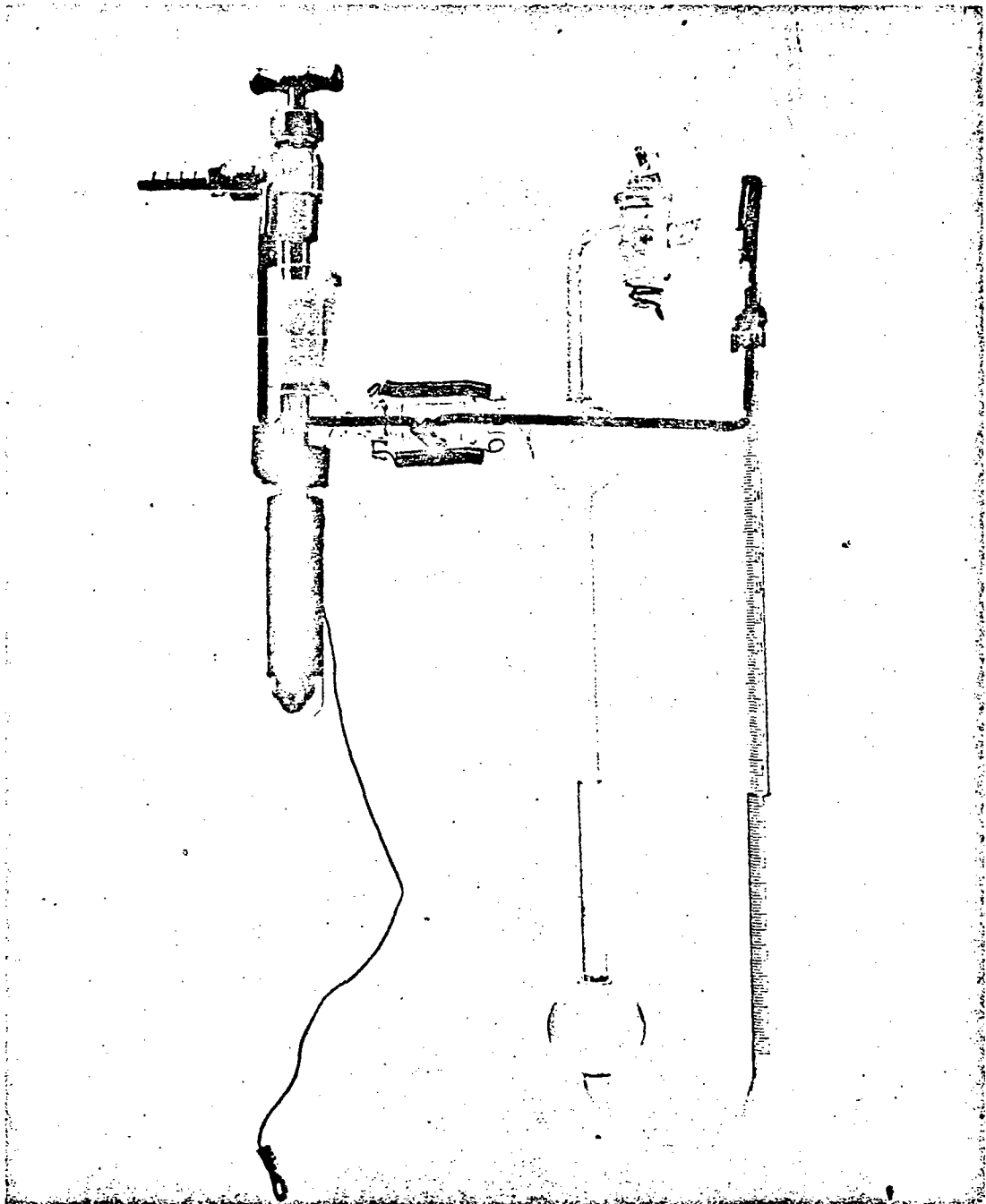
DSW 201727



Schematic Diagram of Cell and Manometer Assembly

FIGURE 3

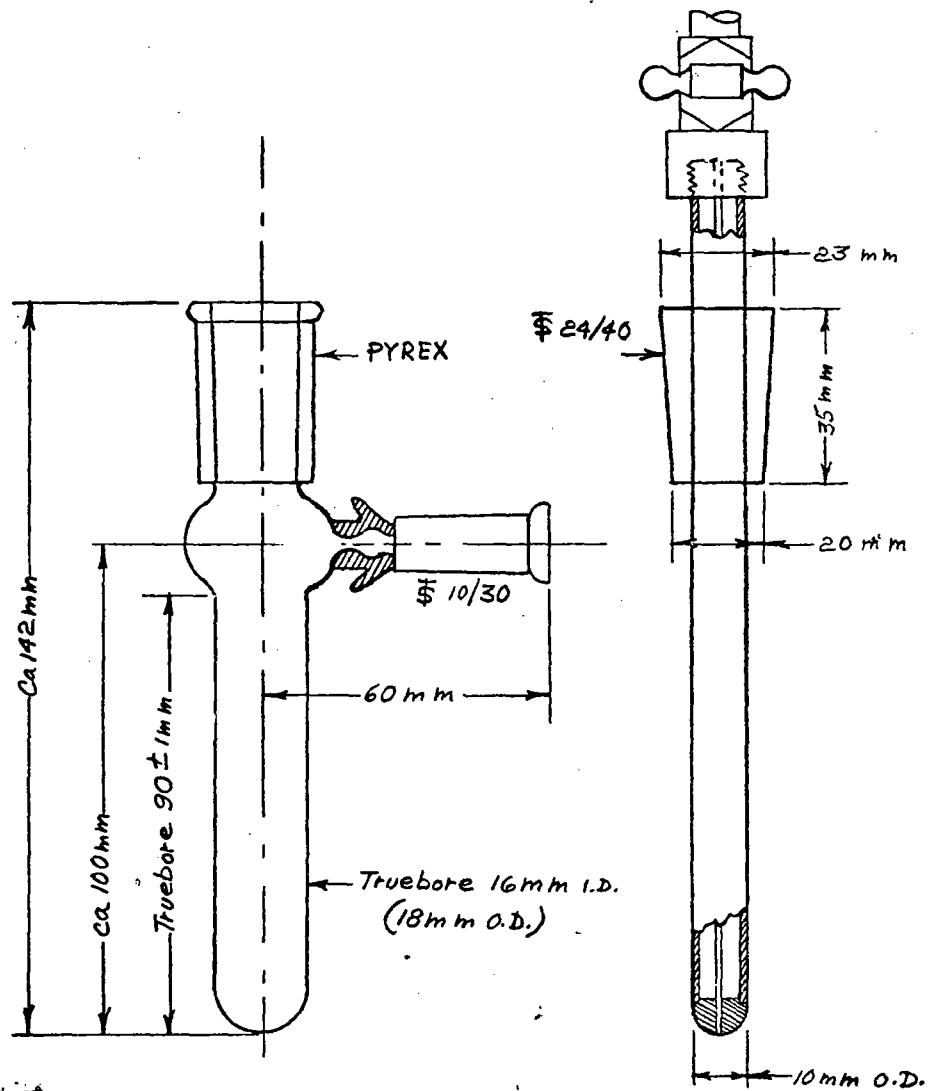
DSW 201728



Gassing Cell and Manometer Assembly

FIGURE 2

DSW 201729



Detailed Dimensions of the Glass Cell and the Inner
(High-Tension) Electrode

FIGURE 4

DSW 201730