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FINAL REPORT

CHLORIDE AND WATER PERMEABILITY OF AUTOMOTIVE BRAKE HOSE

BY

JULES B. COUNTS

AND

ROBERT G. JAMISON

JULY 1972

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13. ABSTRACT

The object of this study was to determine the chloride and water permeability of hydraulic brake hoses. Permeability parameters measured were time interval, temperature, type of exposure, type of brake hose composition, type of brake fluid and the cation linked with the chloride ion.

It was found that sufficient chloride and water will permeate brake hoses to initiate corrosion of metal parts in the conventional brake system. The extent of permeability is greatly reduced in systems containing silicone fluids.

Details of illustrations in
this document may be better
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ABSTRACT

The object of this study was to determine the chloride and water permeability of hydraulic brake hoses. Permeability parameters measured were time interval, temperature, type of exposure, type of brake hose composition, type of brake fluid and the cation linked with the chloride ion.

It was found that sufficient chloride and water will permeate brake hoses to initiate corrosion of metal parts in the conventional brake system. The extent of permeability is greatly reduced in systems containing silicone fluids.

TABLE OF CONTENTS

	Page No.
TITLE PAGE	i
ABSTRACT	ii
INTRODUCTION	1
DETAILS OF TEST	1 - 2
RESULTS AND DISCUSSION	2 - 4
CONCLUSIONS	4
RECOMMENDATIONS	4
ACKNOWLEDGEMENT	5
REFERENCES	5
APPENDIX A	6
Tables I - XII	7 - 14
APPENDIX B	15
Photographs 1 - 3	16 - 18
APPENDIX C	19
APPENDIX D	21
Figures I - VI	22 - 26
DISTRIBUTION LIST	27 - 28
DD FORM 1473	29

I. INTRODUCTION

Considerable investigative work is being carried on at the present time on water pick up in brake systems. Water pick up in brake systems is a matter of grave concern, since it increases vapor locking tendencies and affects low temperature viscosity (11). Permeability of brake hoses is an important factor in the entry of water into a brake system, so it is desirable to know the extent of permeability under various conditions. Further, since large concentrations of salt are used on the roads during winter months in the northern states, chloride permeability may be a concomittant factor in water pick up by brake fluids and should be investigated.

Accordingly, work was initiated to investigate the parameters of time, temperature, type of exposure and type of rubber affecting permeability of automotive brake hoses. It was also considered advisable to evaluate the deleterious effect of chloride content on the stroking and storage properties of the brake fluid and the type of brake fluids.

II. DETAILS OF TEST

1. Brake Hose Assembly.

Two brands of brake hoses used by the two leading auto manufacturers were selected for the test and designated as A and B. Each hose was filled with the SAE Compatibility Fluid (5), and closed with a brass couple. The couples were sealed with solder and both ends of the hose were triple dipped in paraffin wax.

2. Types of Exposure.

The packed brake hose assemblies were exposed to the following types of exposure:

a) Water bath consisting of 3 gallon battery jar maintained at 36°F.

b) Water bath consisting of 3 gallon battery jar maintained at 100°F.

c) Five percent sodium chloride bath, 3 gallon battery jar, at 36°F.

d) Five percent sodium chloride bath, 3 gallon battery jar, maintained at 100°F.

e) Humidity cabinet conforming to Specifications JAN-H-31 and JAN-H-192 maintained at 100°F.

f) Salt spray cabinet (5%) - manufactured by GS Equipment Co., Model No. 22, maintained at 95°F.

g) Salt spray cabinet (20%) - manufactured by GS Equipment Co., Model No. 22, maintained at 95°F.

The hoses in tests f) and g) were suspended as shown in Photo 1, Appendix B. The hoses in all other tests were completely immersed.

In order to determine if the cation affected the degree of chloride permeability, test c) was repeated using sodium chloride, calcium chloride and ammonium chloride. Three brake fluids were evaluated in these tests; one SAE Compatibility Fluid and two silicone fluids designated as Codes C and D.

3. Test Procedure.

One brake hose of each type was removed from each of the above exposures at weekly intervals, the ends scrupulously cleaned, dried, and then opened. The fluid was drained and the chloride and water contents were determined. The water content was determined by Karl Fischer analysis (ASTM Method D-1123). The chloride content was determined by a method based on that of Barney and Bertolacini using mercuric chloranilate. The modified procedure is contained in Appendix C (9 and 10).

4. Stroking Test.

Chloride was added to the SAE compatibility brake fluid at the rate of 100 ppm. The stroking properties of the resultant fluid were determined according to Method 361.3 of Federal Test Method Standard No. 791-13. The stroking properties of the SAE compatibility brake fluid without chloride added were also determined for purposes of reference and comparison.

5. Brake Wheel Cylinder Storage.

A standard chloride solution containing 1 mg per ml. was prepared by diluting 1.51 grams of ammonium chloride to one liter with distilled water. The SAE compatibility brake fluid was then contaminated with this chloride solution so that the final chloride content of the brake fluid was zero, 20 ppm, 100 ppm, 200 ppm and 400 ppm. SAE standard 1-1/8 inch brake wheel cylinders (Bendix 2227391-L) were disassembled, thoroughly washed and then packed in quadruplicate with each of the contaminated fluids. The twenty packed cylinders were stored in the open laboratory at 70-80°F. for 30, 90 and 180 days storage period.

III. RESULTS AND DISCUSSION

The results of the chloride and water content analysis of brake fluids taken from brake hoses after various types of exposure are shown in Appendix A, Tables I-VII. At the outset, it should be pointed out that the brake hoses tested were not uniform in fabrication. This fact should be considered in the analysis of statistical data. It was found in the

initial screening test that maximum contamination was obtained at about 35 days, and accordingly, this was the time chosen for the duration of the tests. In each test there was sufficient water permeability to seriously change the boiling point and viscosity of the brake fluid, and sufficient chloride permeability to initiate and accelerate the rate of corrosion of metal parts in the brake systems. The average test results of each exposure are listed in Table VIII.

1. Comparisons of Types of Hoses.

Analysis of the brake hoses designated as A and B showed that both types had an inner layer of neoprene and an outer layer of styrene butadiene. The hoses differed from each other in that the B hose had a thinner fiber layer which was milled with a polyester type compound as contrasted with the A hose that was milled with styrene butadiene rubber. From the averages listed in Table VIII, it will be seen that the average water permeability of the A hose was 9.65% compared with 8.10% for the B hose. The average chloride permeability of the two hoses was comparable: 68 ppm for the A hose, 66 ppm for the B hose.

2. Effect of Salt Concentration.

Examination of the values listed in Table VIII shows that the increased amount of salt concentration in the salt spray had little effect on the chloride permeability.

3. Effect of Cation on Magnitude of Chloride Permeability.

From Tables IX, X and XI it can be seen that within experimental error and the uncontrolled factor of hose uniformity, the chloride permeability is independent of the cation with which it was linked.

4. Effect of type of Brake Fluid on Water and Chloride Permeability.

From Tables IX, X and XI, it can be seen that the use of silicone fluids drastically reduce the chloride and water permeability.

5. Results of Stroking Evaluation.

After 90,000 strokes at 1000 psi. and 250°F. and 210,000 strokes at 500 psi. and 158°F. (total 300,000 strokes) the brake system containing the salt/water contaminated fluid was in the following condition:

a) Master cylinder: The brass piston was extremely darkened; interior of the cylinder was in satisfactory condition; the spring and check valve were in satisfactory condition; and the primary cup exhibited excessive chipping, while the secondary cup exhibited light to heavy chipping.

b) Wheel cylinders: Tthe interior of the cylinder showed the normal wear while the four aluminum pistons showed excessive corrosive attack on the sides of the pistons (see Photo 2, Appendix B). The four wheel cylinders springs had areas of plating worn away. The interior of the cylinder was satisfactory. The wheel cylinder cups all showed excessive chipping.

The above test was repeated without chloride contamination. There was no excessive corrosion of metal parts and no excessive chipping of the rubber cups.

6. Brake Wheel Cylinder Stroage.

At the end of six months storage at room temperature, the brake wheel cylinders were opened and the fluid collected. The condition of the cylinders is listed in Table IX. All cylinders containing chloride were considered inoperable and failing. The cylinders were cut in half and the corrosive effect of the chloride can be seen in Photo 3. The spectrograms of the drained fluids are shown in Appendix D. Except for the water band at 6.1 microns, no perceptible change in composition is noted.

IV. CONCLUSIONS

The chloride and water premeability in all the tests except those containing silicone fluids was sufficient to initiate and increase the rate of corrosion of metal parts in the brake system. The chloride and water permeability was drastically reduced when the system contained silicone fluids. It increased at elevated temperatures.

It appeared that the thicker the fiber layer of the brake hose the greater the permeability. Chloride permeability was essentially independent of the cation to which the chloride was originally linked.

V. RECOMMENDATIONS

1. On the basis of findings in this report, it is recommended that a permeability test requirement be considered in the next revision of Military Specification MIL-H-13719C, Hose Assembly, Rubber, Hydraulic Brake.

2. That this investigation be extended to include the chloride analysis of brake fluids from random vehicles which have been exposed to winter driving conditions involving "salted" roads.

3. That the brake wheel cylinder storage program be continued.

4. That the results of this study be given wide publicity in the various committees, both industrial and governmental, which are studying various aspects of brake system safety.

VI. ACKNOWLEDGEMENT

The assistance of Mr. B. Caudill in the stroking evaluation tests and Mr. H. R. Sheets in the preparation and packing of the brake wheel cylinders for the storage program is gratefully acknowledged.

VII. REFERENCES

1. Specification JAN-H-792, Humidity Cabinet.

2. Specification JAN-H-31, Humidity Cabinet.

3. Specification MIL-H-13719C, Hose Assembly, Rubber, Hydraulic Brake.

4. Federal Test Method Standard No. 791-13, Method 361.3 dated January 1969, Stroking Properties of Hydraulic Brake Fluids.

5. SAE Standard, J 1703, Hydraulic Brake Fluid.

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9. J. Bertolacini, and J. Barney, "Colorimetric Determination of Chloride with Mercuric Chlorianilate", Analytical Chemistry, 29:1787 (Aug 1957).

10. R. Bertolacini and J. Barney, "Ultraviolet Spectrophotometric Determination of Sulfate, Chloride and Fluoride with Chloranilic Acid", Analytical Chemistry, 30:202 (Feb 1958).

11. CCL Report No. 198, "Effect of Water on Hydraulic Brake Fluid", Coating and Chemical Laboratory, Aberdeen Proving Ground, Maryland, dated May 1966.

APPENDIX A

TABLE I

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to 5% Salt Spray at 95°F.

			% Water			
			Hose A		Hose B	
	Chloride, ppm		Total		Total	
Days	Hose A	Hose B	Water	Permeability	Water	Permeability
0	Nil	Nil	0.46	--	0.46	--
7	5	5	7.11	6.65	8.67	8.21
14	18	25	12.66	12.14	8.10	7.64
21	200	170	10.90	10.44	8.60	8.14
28	50	116	7.90	7.44	7.30	6.84
35	12	12	8.04	7.58	8.50	8.04
Average	57	66		8.85		7.77

TABLE II

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to 20% Salt Spray at 95°F.

			% Water			
			Hose A		Hose B	
	Chloride, ppm		Total		Total	
Days	Hose A	Hose B	Water	Permeability	Water	Permeability
0	Nil	Nil	0.44	--	0.44	--
7	9	5	14.15	13.71	11.55	11.11
14	46	58	12.20	11.76	9.10	8.66
21	90	--	11.87	11.43	6.88	6.44
28	80	98	8.05	7.61	7.50	7.06
35	12	12	8.70	8.26	--	--
Average	47	43		10.55		8.32

TABLE III

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to 5% Salt Bath at 36°F.

	Chloride, ppm		% Water			
			Hose A		Hose B	
Days	Hose A	Hose B	Total Water	Permeability	Total Water	Permeability
0	Nil	Nil	0.46	--	0.46	--
7	5	5	3.95	3.49	3.35	2.89
14	5	5	9.22	8.76	10.40	9.94
21	23	60	8.40	7.94	9.30	8.84
28	25	50	5.30	4.84	7.00	6.54
35	275	400	7.80	7.34	15.95	15.49
Average	67	104		6.45		8.74

TABLE IV

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to 5% Salt Bath at 100°F.

	Chloride, ppm		% Water			
			Hose A		Hose B	
Days	Hose A	Hose B	Total Water	Permeability	Total Water	Permeability
0	Nil	Nil	0.44	--	0.44	--
7	5	5	9.95	9.51	9.45	9.01
14	50	60	7.60	7.16	9.59	9.19
21	110	50	11.50	11.06	10.50	10.06
28	680*	50	11.30	10.86	10.50	10.06
35	240	43	6.74	6.30	4.44	4.00
Average	101	42		8.98		8.46

*Not included in average.

TABLE V

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to Water Bath at 36°F.

			% Water			
			Hose A		Hose B	
	Chloride, ppm		Total		Total	
Days	Hose A	Hose B	Water	Permeability	Water	Permeability
0	Nil	Nil	0.46	--	0.46	--
7	Trace	Trace	11.50	11.04	10.60	10.14
14	Trace	Trace	7.04	6.58	5.94	5.48
21	Trace	Trace	7.89	7.43	7.53	7.07
28	Trace	Trace	10.49	10.03	9.49	9.03
35	Trace	Trace	9.84	9.38	5.58	5.12
Average	Trace	Trace		8.89		7.37

Table VI

Chloride and Water Permeability of Brake Hoses

Brake Fluids Sampled After Exposure to Water Bath at 100°F.

			% Water			
			Hose A		Hose B	
	Chloride, ppm		Total		Total	
Days	Hose A	Hose B	Water	Permeability	Water	Permeability
0	Nil	Nil	0.44	--	0.44	--
7	Trace	Trace	11.56	11.12	11.43	10.99
14	Trace	Trace	10.45	10.01	7.98	7.54
21	Trace	Trace	9.49	9.05	6.76	6.32
28	Trace	Trace	19.89	19.45	9.14	8.70
35	Trace	Trace	7.78	7.34	6.96	6.52
Average	Trace	Trace		11.39		8.01

TABLE VII

Chloride and Water Permeability of Brake Hoses

Brake Fluid Sampled After Exposure to Humidity Cabinet

	Chloride, ppm		% Water			
			Hose A		Hose B	
Days	Hose A	Hose B	Total Water	Permeability	Total Water	Permeability
0	Nil	Nil	0.46	--	0.46	--
7	Trace	Trace	0.78	0.32	0.76	0.30
14	Trace	Trace	8.17	7.71	6.04	5.58
21	Trace	Trace	8.89	8.43	8.46	8.00
28	Trace	Trace	24.80	24.34	14.60	14.14
35	Trace	Trace	24.00	23.54	12.74	12.28
Average	Trace	Trace		12.47		8.06

TABLE VIII

Summary of Average Chloride and Water Permeability
Of Brake Hoses After Various Exposures

	Chloride, ppm		% Water	
Exposure	Hose A	Hose B	Hose A	Hose B
5% Salt Bath (36°F.)	67	104	6.45	8.74
5% Salt Bath (100°F.)	101	42	8.98	8.46
5% Salt Spray	57	66	8.85	7.77
20% Salt Spray	47	43	10.55	8.32
Water Bath (36°F.)	--	--	8.89	7.37
Water Bath (100°F.)	--	--	11.39	8.01
Humidity Cabinet	--	--	12.47	8.06
Average	68	64	9.65	8.10

TABLE IX

Chloride and Water Content of SAE Brake Fluid from Brake Hose After Exposure to Various Salt Baths for 21 Days at 36°F.

		Cl (ppm)	H_2O (%)
a)	5% Na Cl (Hose A)	349	5.94
b)	5% Na Cl (Hose A)	349	6.11
a)	5% Na Cl (Hose B)	214	0.93
b)	5% Na Cl (Hose B)	377	1.34
	Average	330	3.58
a)	5% $CaCl_2$ (Hose A)	368	399
b)	5% $CaCl_2$ (Hose A)	428	580
a)	5% $CaCl_2$ (Hose B)	133	0.88
b)	5% $CaCl_2$ (Hose B)	139	5.19
	Average	254	3.57
a)	5% NH_4Cl (Hose A)	158	3.30
b)	5% NH_4Cl (Hose A)	270	6.96
a)	5% NH_4Cl (Hose B)	158	8.49
b)	5% NH_4Cl (Hose B)	256	3.29
	Average	211	541

TABLE X

Chloride and Water Content of Water Tolerant Silicone Brake Fluid from Brake Hose After Exposure To Various Salt Baths for 21 Days at 36°F.

	Cl (ppm)	H_2O (%)
a) 5% Na Cl (Hose A)	0	0.89
b) 5% Na Cl (Hose A)	0	1.20
a) 5% Na Cl (Hose B)	0	0.90
b) 5% Na Cl (Hose B)	0	0.63
Average	0	0.90
a) 5% $CaCl_2$ (Hose A)	0	0.54
b) 5% $CaCl_2$ (Hose A)	18	0.60
a) 5% $CaCl_2$ (Hose B)	18	0.84
b) 5% $CaCl_2$ (Hose B)	0	0.46
Average	9	0.61
a) 5% NH_4Cl (Hose A)	0	4.24
b) 5% NH_4Cl (Hose A)	0	1.47
a) 5% NH_4Cl (Hose B)	0	0.88
b) 5% NH_4Cl (Hose B)	0	0
Average	0	1.65

TABLE XI

Chloride and Water Content of Non-Hygroscopic Silicone Brake Fluid From Brake Hose After Exposure to Various Salt Baths for 21 Days at 36°F.

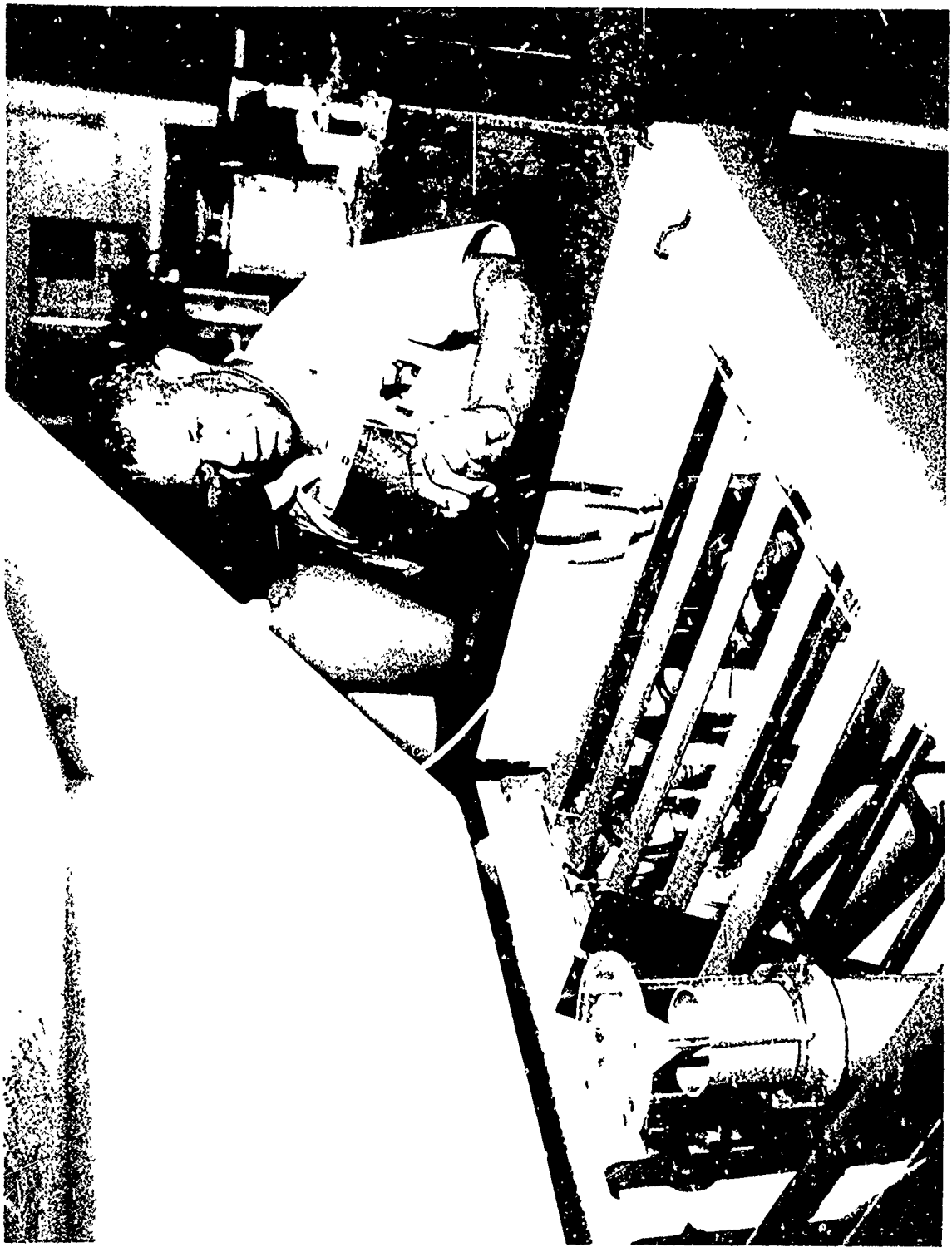
	Cl (ppm)	H_2O (%)
a) 5% Na Cl (Hose A)	Nil	N Nil
b) 5% Na Cl (Hose A)	Nil	Nil
a) 5% Na Cl (Hose B)	Nil	Nil
b) 5% Na Cl (Hose B)	Nil	Nil
Average		
a) 5% $Ca\ Cl_2$ (Hose A)	*Contaminated	Nil
b) 5% $Ca\ Cl_2$ (hose A)	Nil	Nil
a) 5% $Ca\ Cl_2$ (Hose B)	Nil	Nil
b) 5% $Ca\ Cl_2$ (Hose B)	Nil	Nil
Average		
a) 5% NH_4Cl (Hose A)	Nil	Nil
b) 5% NH_4Cl (Hose A)	Nil	Nil
a) 5% NH_4Cl (Hose B)	Nil	Nil
b) 5% NH_4Cl (Hose B)	Nil	Nil
Average		

TABLE XII

Condition of Wheel Cylinders after 6 Months Storage
With Brake Fluids Containing Chloride

Chloride Content	Appearance
0 ppm	Spring - OK. Right piston - Slight gum. Left piston - Slight gum and slight stain. Cylinder walls - Slight gum and slight stain.
20 ppm	Spring - OK. Right piston - Moderate gum and stain. Left piston - Moderate gum and stain; piston frozen. Cylinder walls - Moderate gum and stain.
100 ppm	Spring - OK. Right piston - Moderate gum and stain; piston frozen. Left piston - Moderate gum and stain; piston frozen. Cylinder walls - Moderage gum and stain; slight pitting.
200 ppm	Spring - OK. Right piston - Heavy gum and stain; piston frozen. Left piston - Heavy gum and stain; piston frozen. Cylinder walls - Heavy gum, stain and pitting.
400 ppm	Spring - OK. Right piston - Heavy gum buildup outside of piston, piston frozen. Left piston - Heavy gum buildup outside of piston, piston frozen. Cylinder walls - Heavy stain and pitting.

APPENDIX B



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APPENDIX C

Procedure for Ultraviolet Spectrophotometric Determination of Chloride in Brake Fluids with Mercuric Chloranilate

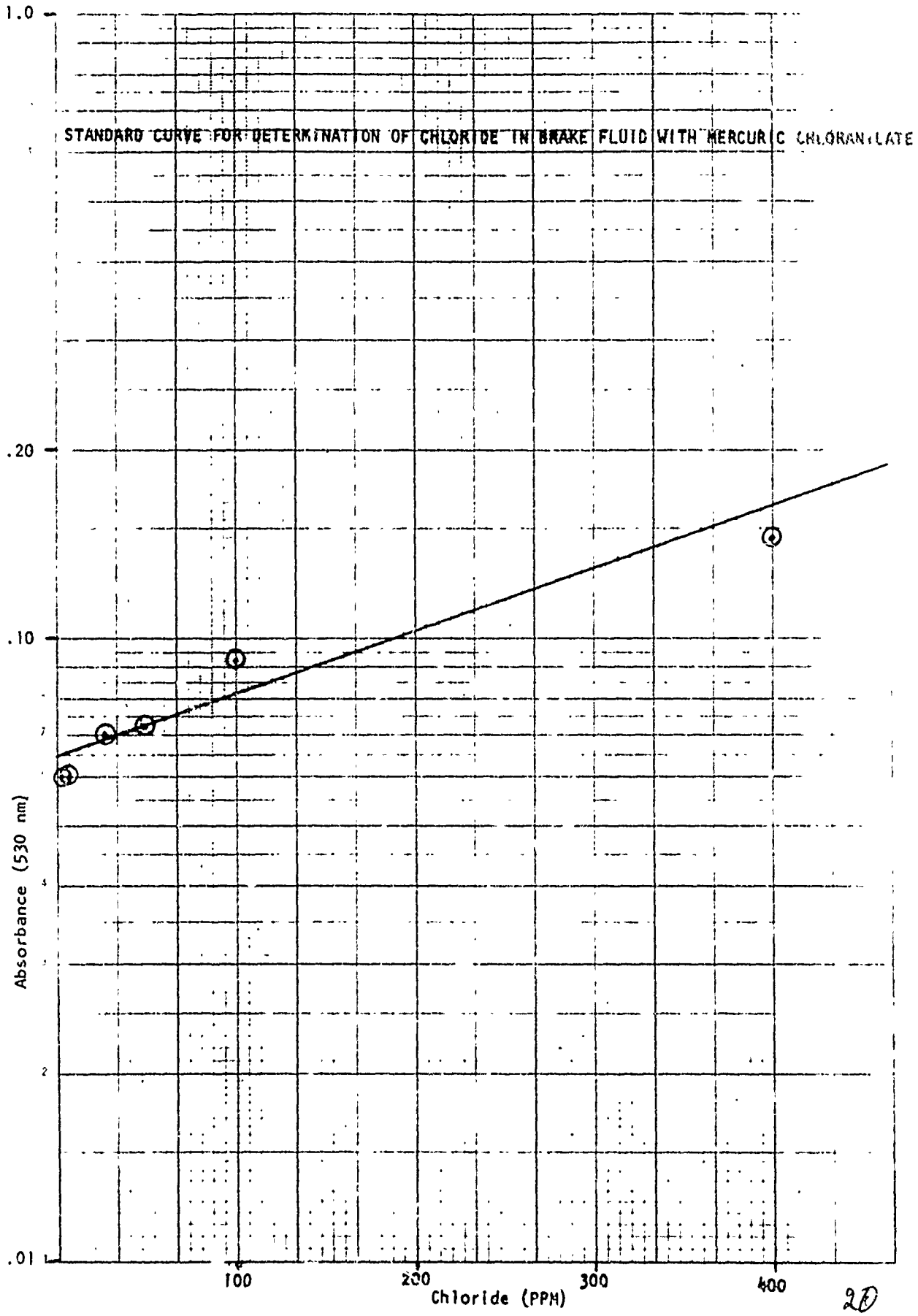
Reagents:

1N Nitric acid
Methyl cellosolve
Ammonium chloride (ACS)
Mercuric chloranilate (certified reagent grade)

Place 0.5 ml. of brake fluid in a 100 ml. volumetric flask. Add 5 ml. of 1N-nitric acid and 50 ml. of methyl cellosolve (ethylene glycol monoethyl ether). Add 0.2 gram of mercuric chloranilate. Dilute to the 100 ml. mark with distilled water and stopper the flask shake at least once a minute for fifteen minutes. Filter through Whatman #42 filter paper.

Read the absorbance of the filtrate on a Bechman "B" Spectrophotometer at 530 um. Compare to a standard curve constructed using a sample prepared by diluting 1.5105 grams of ammonium chloride to one liter with distilled water (see page 20). This gives a chloride standard of 1 mg per ml.

If the absorbance of the sample being tested exceeds the absorbance of the standard, the sample may be reduced accordingly. A 99% accuracy is obtained in the chloride range of 3 ppm to 400 ppm.


STANDARD CURVE FOR DETERMINATION OF CHLORIDE IN BRAKE FLUID WITH MERCURIC CHLORANILATE
1.0
.20
.10
.01
Absorbance (530 nm)
100
200
300
400
Chloride (PPM)

20

APPENDIX D

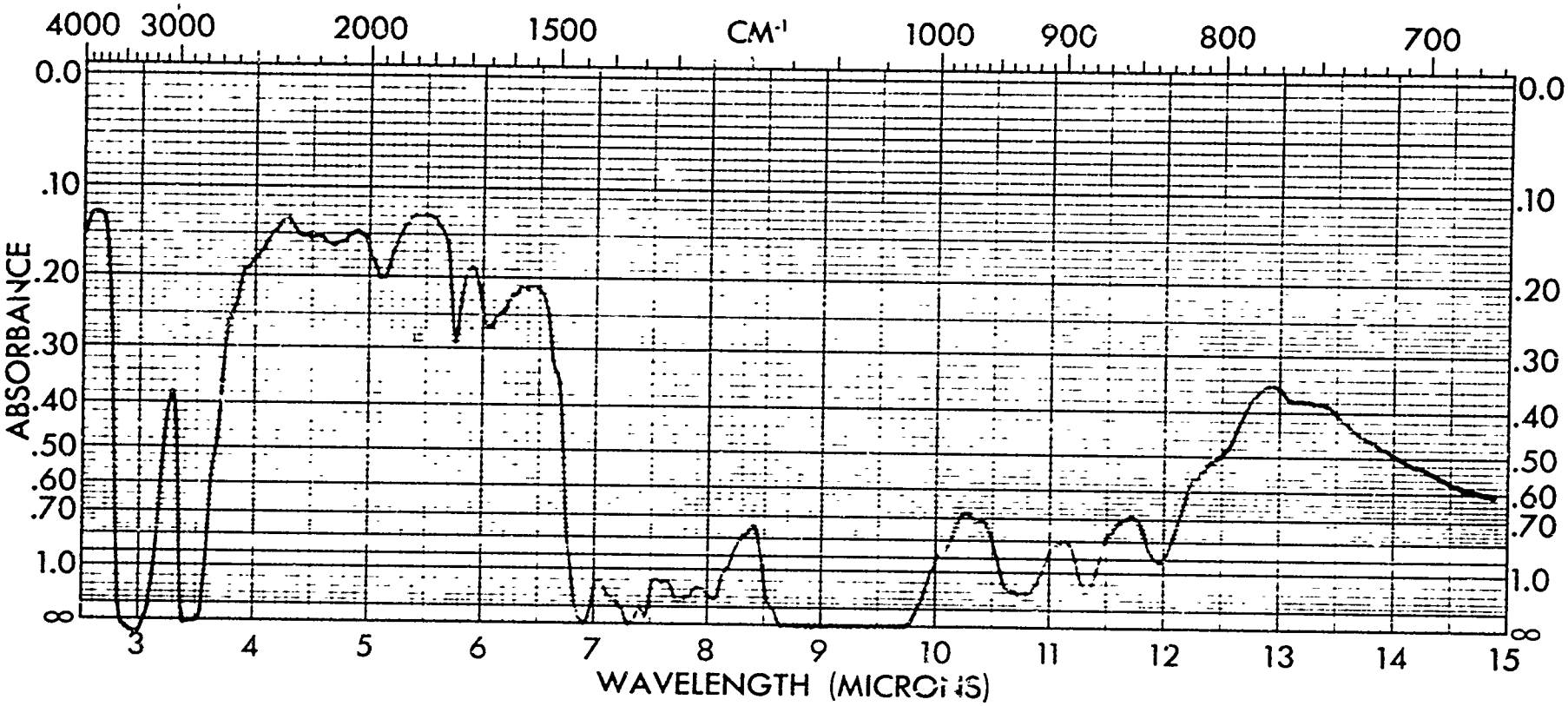


FIGURE 1 - SAE Compatibility Brake Fluid From Wheel Cylinder After Six Month's Storage. (No Chloride Added).

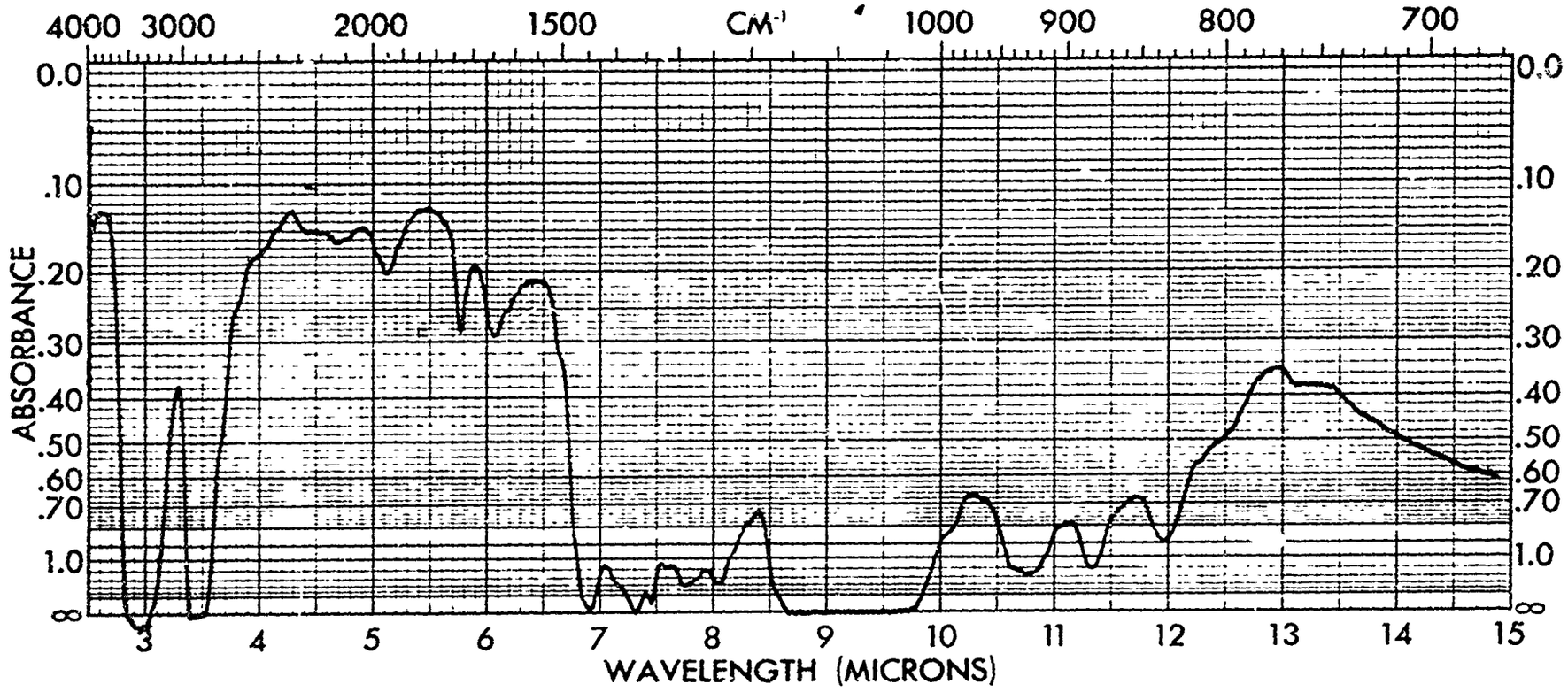


FIGURE 11 - SAE Compatibility Brake Fluid From Wheel Cylinder After Six Month's Storage. (20 ppm Chloride Added).

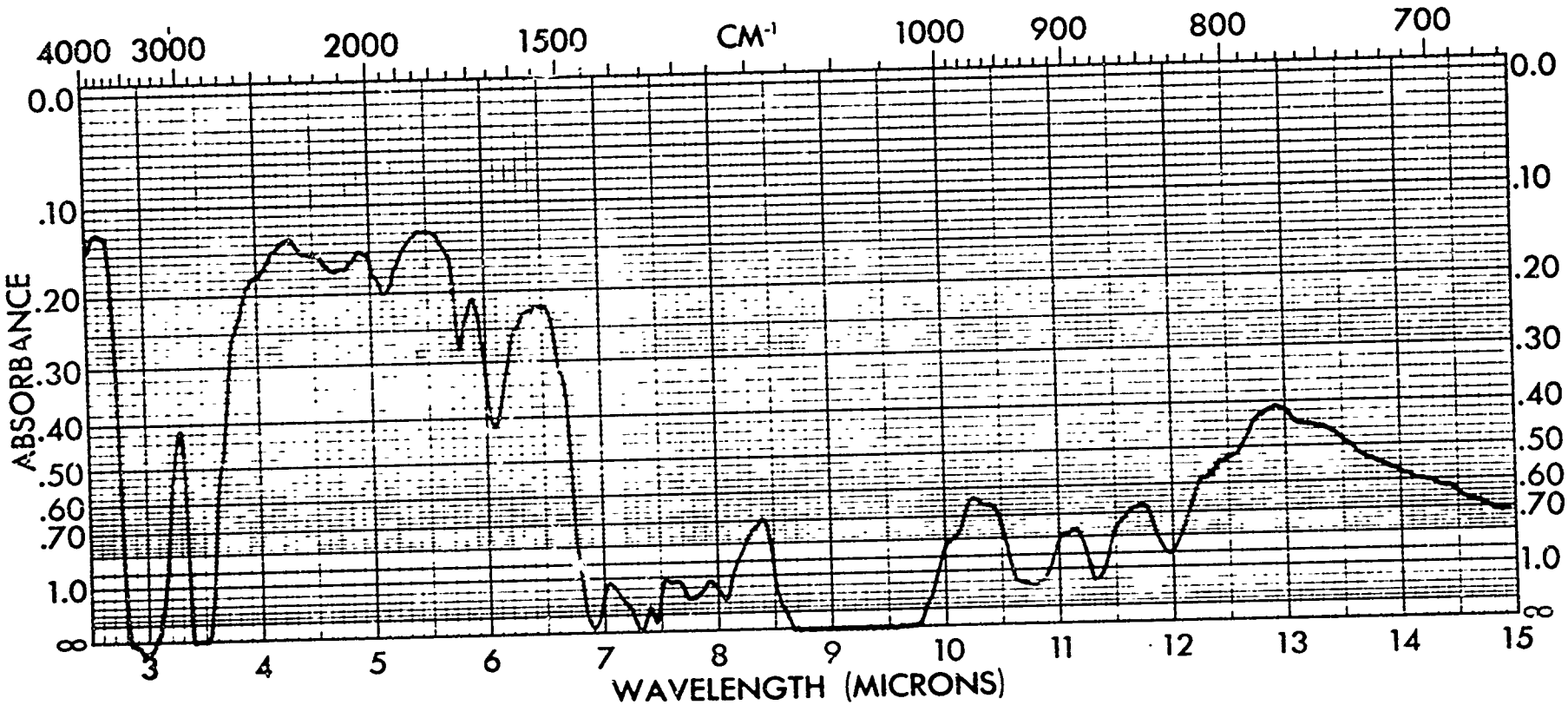


FIGURE III - SAE Compatibility Brake Fluid From Wheel Cylinder After Six Month's Storage. (100 ppm Chloride Added).

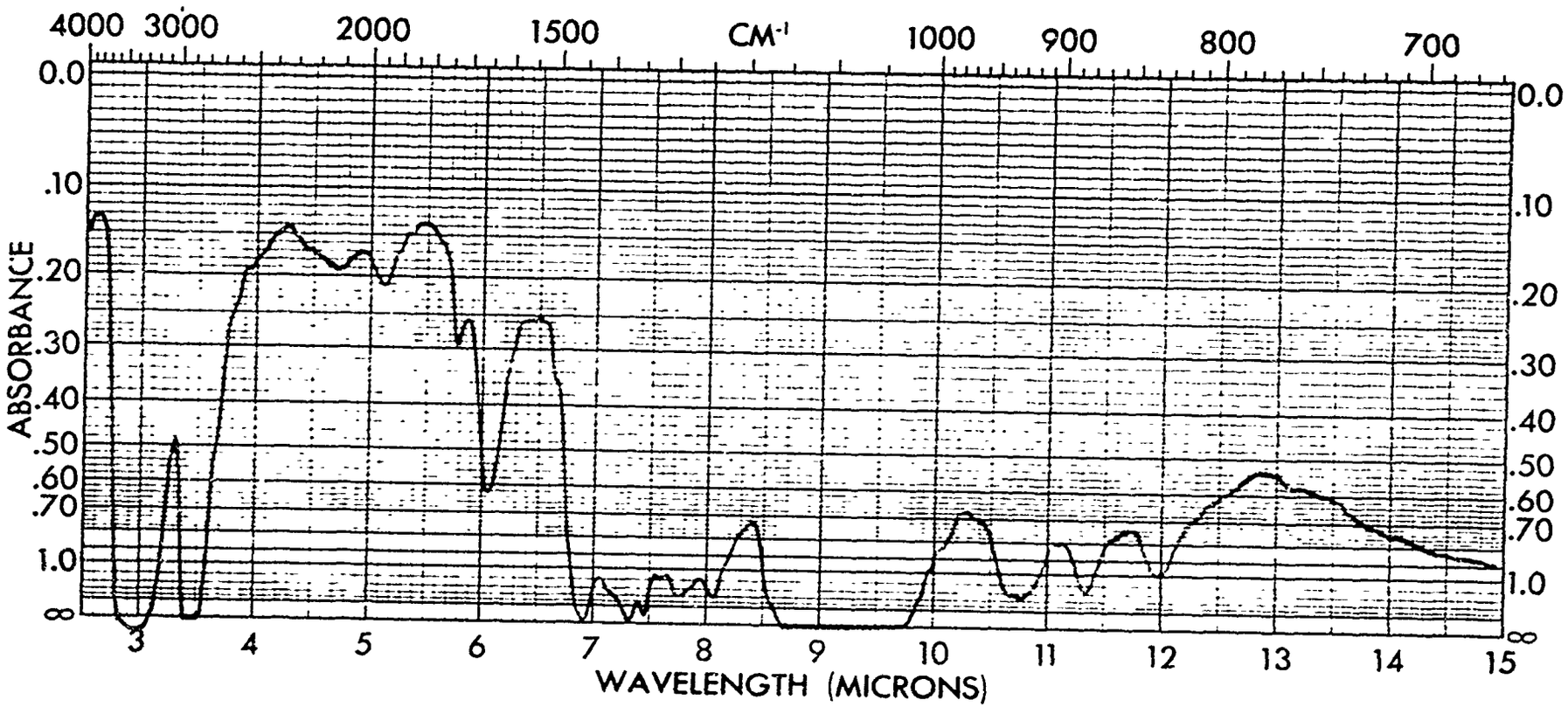


FIGURE IV - SAE Compatibility Brake Fluid From Wheel Cylinder After Six Month's Storage. (200 ppm Chloride Added).

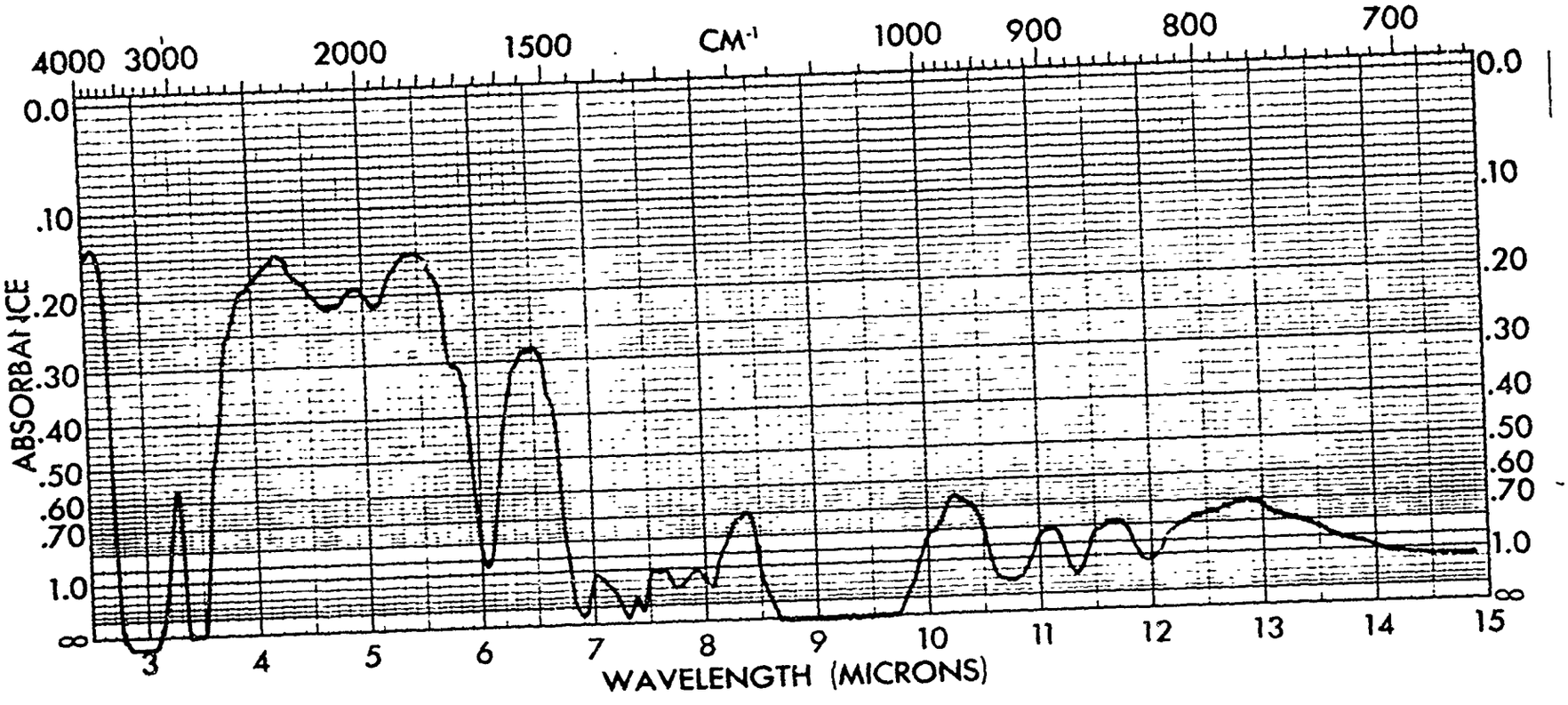


FIGURE V - SAE Compatibility Brake Fluid From Wheel Cylinder After Six Month's Storage. (400 ppm Chloride Added).