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FINAL REPORT

ON

APPLICATION WORK ON PRESENT

PLASTICIZERS - 1953-54

FLAMMABILITY STUDIES WITH THE SPI TESTER

Job No. 2-02-750.01-2616 Part IV

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I. INTRODUCTION

The problem of accurately determining the flammability of plastic specimens has been a difficult test development program for the plastics industry. Development of the flammability tester by the technical committee under the sponsorship of the Society of the Plastics Industry was a definite step in the proper direction.

Because Monsanto had considerable interest in flame retardant plasticizers, such as phosphate esters and Aroclors, it was deemed advisable to organize a complete testing program in order to point up the advantages of these products. It was also desirable to promote trade acceptance of the SPI tester by the industry so that many laboratories could better appreciate the value of flame retarded products.

An extensive program of preparation of vinyl films and subsequent flammability testing yielded such potentially valuable information for the promotion of our products that a technical paper was delivered at the Third Annual Film and Sheeting Conference in New York. A reprint of this paper constitutes the detailed section of this report.

II. SUMMARY

Eighty-three plasticized polyvinyl chloride films were calendered at Springfield and extensive flammability tests run on the SPI tester at St. Louis. Over two thousand flammability tests were made. A technical paper was prepared covering these results.

III. CONCLUSIONS

1. The SPI tester gave the most definitive flammability results that we have been able to achieve. It has been promoted for ASTM acceptance as a standard test method and a tentative procedure has been developed by this organization.
2. A truly flame retarded film can be prepared by using as plasticizers tricresyl phosphate, Santicizers 140 and 141, and other plasticizers either in combination with the above ones, or with antimony oxide.

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3. Sovaloid C (an aromatic hydrocarbon) and tri-2-ethylhexyl phosphate gave flame retarded films alone but were relatively ineffective when used in combination with more flammable plasticizers.

4. Refer to technical paper for other conclusions.

IV. REFERENCE TO PREVIOUS WORK

Final Report 117-2422, Part II, by P. W. Spink entitled "Sales Service Work on Plasticizers and Resins" briefly mentions this flammability work.

Final Report 117-2414, Part II, by P. W. Spink entitled "Application of Present Plasticizers" (July 30, 1953) also mentions, but does not detail this flammability program.

Job number 117-2616 succeeded 2414 as the job number covering "Application Work on Present Plasticizers" and was the job number to which the cost of the reprints were charged. Parts I, II, and III on 2616 were reported previously but did not deal with flammability.

V. DETAILS

See reprint at end of report.

VI. ACKNOWLEDGMENTS

Mr. G. R. Buchanan suggested this overall program as a means of promoting S-141 sales. Mr. R. R. Lawrence was very cooperative with the calendering program. Mr. E. E. Cowell was co-author on the flammability paper but the printers inadvertently omitted his name from the reprint.

VII. APPENDIX

Notebook pages concerned with flammability studies with the SPI tester:

A-27417 to and including A-27437

J. R. Darcy

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FLAMMABILITY TESTS WITH THE SPI TESTER

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SUMMARY: The SPI tester provides an unexcelled method for determining flammability. Heretofore unsuspected factors can be measured. The development of this instrument will undoubtedly prove valuable to the vinyl industry.

Polyvinyl chloride type plastics possess an inherent advantage over many other classes of materials because they can be made flame retardant. Just how great this advantage was could not be accurately assessed until The Society of The Plastics Industry, Inc. through its Group VII Flammability Committee under the Chairmanship of Mr. J.R. Blegen, developed the flammability tester shown in the accompanying photograph. Results of round robin tests made with the first experimental instrument were so encouraging in accuracy and reproducibility that the manufacture of this tester was undertaken by Custom Scientific Instruments Company and a flammability specification, based on this instrument, has been written into the Proposed Commercial Standard for General Purpose Vinyl Film by the SPI. Many methods of determining the Flammability characteristics of plastics had been proposed and evaluated before. However, the SPI Flammability Committee found early that no existing method took into consideration all the significant factors involved in practical application nor foresaw everyday hazards. Results of many of the tests were misleading; invariably they defied reproduction; no single test was accepted as standard.

Reported herein are the results obtained on polyvinyl chloride film and sheeting when using an early model of the SPI tester. We followed our interpretation of the general procedures of testing outlined in the SPI publication:

"Proposed Commercial Standard - General Purpose Vinyl Film - A Recorded Voluntary Standard of the Trade".

Test Instrument.

A brief review of the instrument will aid explanation of results to be given later. Figure 1 shows a sample holder and some of the essential features of the instrument. The gas supplied from a butane cylinder is measured by means of the manometer; the flame burns near the bottom of the tester. The smooth plastic specimen is secured between the frames of the sample holder, and this holder is slid down the track of the specimen holder rack. Upon arriving at the bottom end of the track, a switch actuates the ignition timer. This electric time-clock continues to operate until a thread stretched across the specimen holder where the flame is held is burned through. At this exact time, the burner is extinguished and the specimen is removed with hand. The ignition timer, which stops the electric time-clock, is then reset.

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but the rate timer is automatically shut off when a high thread located on the specimen is burnt, thus limiting the time. Microburner temperature within the tester is measured by means of a thermocouple fairly centrally located; this temperature is registered in °F. on the pyrometer located atop the instrument.

The ignition timer registers the time required for the flame to travel from near the bottom of the specimen holder to the first thread; this reading then defines an important flammability parameter, that of ignition time. It is obvious that the burning rate timer measures the time required for the flame to travel six inches, thus providing information on this important factor in flammability studies (burning rate). Temperature developed in the instrument can be taken as a comparative measure of the heat evolved upon burning; this is a third important flammability factor.

Group Classifications:

One of the complicating features of using the SPI flammability tester is that various procedures of testing must be followed. This complication is well worthwhile because the grouping of materials into various categories of flame retardance is an important reason for the reliability of the tests.

Group I was established for materials which vary in burning characteristics from those which burn readily to those which are slightly flame retarded. Figure 2 shows additional details of the specimen holder. The test is made with the sample at a 45° angle with a 1/2" flame emitted from a #22 hypodermic needle burner. The tip of the burner is located 9/16" from the specimen. This group is further subdivided alphabetically from "a" through "d".

Group I-a specifies the cabinet door be closed.

I-b is an open-door test.

I-c required edge ignition with door closed.

I-d is the same as "b" but a measurement of inches burned is called for.

Group II tests are also made with the sample at 45° angle but the needle burner and the microburner are both used to edge-ignite the sample while the cabinet door is closed. Group III tests are made for materials which will not support combustion once the source of heat has been removed. A 2" flame from a microburner is used to ignite a 3" x 10" sample supported vertically above the flame. Figure 3 depicts the type of holder used. These tests are run inside the cabinet with the door closed.

Preparation of Samples

Only dry, uncured resins degraded by infrared at 2900 cm⁻¹ were used except where specifically noted. The stabilizer system generally used was 0.5 phr 301 (a) and 0.5 phr 302 (a) resin of 0.1 phr 301 (a) and 0.1 phr 302 (a).

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50 PHR modified calcium stearate (National Lead Co., Plumb-O-Sil) and 100 PHR modified calcium stearate (National Lead Co., Plumb-O-Sil) and then calendered on a laboratory calender. All samples were made as closely as possible to 4 mil thickness, except in the cases noted.

Film samples were cut to 3" x 9" size and conditioned 3 hrs. in an air circulation oven at 50°C., then cooled in a desiccator before testing. For Group 3 tests the sample length was increased to 10".

Effect of Plasticizers:

A series of 10 different plasticizers at 50 PHR concentration was tested for flammability in every classification. At least five samples were tested on each formulation in each classification, and whenever results varied by over 50% from the mean, additional tests were made until agreement within a series was obtained. Table I contains the average results for each formulation.

Diocetyl sebacate imparted the fastest burning rate and burned by every test. Diocetyl adipate and diocetyl phthalate were possibly the next most flammable. Santicizer 160 (butyl benzyl phthalate) was slightly less flammable than the aforementioned plasticizers. Paraplex G-50, which exhibited a slow burning rate, was completely consumed whenever ignited and developed excessively high temperatures. This was the only plasticizer which gave an afterflame in test #3, which means that the film continued to burn after removal of the ignition flame. (The other flammable plasticizers had been burned out during the 10 seconds flaming time.)

Tricresyl phosphate, Santicizer 140 (cresyl diphenyl phosphate) and Santicizer 141 (alkyl diaryl phosphate) were quite flame retardant. Sovaloid C, a petroleum hydrocarbon cut, showed surprising flame retardance. Trioctyl phosphate was the least flame resistant of those compositions which did not burn completely.

It is interesting to note that ignition time in seconds given under groups 1-a, b and d, and c was not significant in indicating whether the composition would burn completely. Thus, Santicizer 141 gave an ignition time of 2.1 seconds but was self-extinguishing, whereas, DOP gave an ignition time of 2.3 and burned all the way. The times denoted in the table under seconds burning have a different significance when "boxed in". All these compositions were self-extinguishing and the figures cited are the seconds burning before extinguishment. As previously explained, burning rate is defined as the time required for the flame to travel 6". In columns under groups b and d, the inches burned are recorded as well as the time required to burn this distance, so some rough assessment of burning rate for cases where 6" could be calculated.

Class 1-c gave no data at all with the exception of one composition. This was no the effect of edge-igniting the samples but rather the result of moving the flame to a non-flammable distance from the burning conditions under

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group II test. It is noted that at least half of the composition was melted and consumed in the test. The remaining portion was burned away.

Effect of Plasticizer Concentration:

Table II supplies data on the effect of increasing the concentration of 4 plasticizers from 40 to 70 PHR by increments of 10 PHR. We were unable to find significant differences in the DOP compositions. It was thought that the burning rate would become faster with increasing concentrations of plasticizer, but this relationship was not detected. The most noticeable trend developed was the elevation in temperature with an increasing concentration of DOP. It was surprising that the 40 PHR dioctyl phthalate film burned at the comparative fast rate of 4.6 seconds. This series of samples were re-tested later.

Increasing the concentration of Santicizers 140 and 141 had very little effect on any test. Trioctyl phosphate became measurably more flammable with increasing concentrations until all the specimens were consumed at 60 and 70 PHR concentration in the Group II test. It is noteworthy here that in Group II tests a temperature increase of 40°F. developed from the ignition flame alone.

Effect of Plasticizer Mixtures:

It is when testing a mixture of plasticizers that we bring out the practical flame-retarding efficiency of plasticizers. By holding constant the concentration of plasticizer at 50 PHR, using DOP as the building plasticizer, and then adding flame retarding plasticizers, we can assess this factor. Table III shows that 10 PHR of tricresyl phosphate or cresyl diphenyl phosphate (S-140) produces a self-extinguishing film. A proportion of 15 parts of Santicizer 141 with 35 parts DOP is self-extinguishing. As much as 20 parts of trioctyl phosphate failed to effect self-extinguishment. Neither chlorinated paraffin (Chlorafin 42-S) nor Sovaloid C, at 15 parts, prevented the film from burning.

Effect of Film Thickness:

When a 50 PHR DOP plasticized composition was increased in thickness from 4 mil through 8, 12 and 20 mils, definite trends were established. Both the time required for ignition and the time measured for burning 6 inches increased as the thickness increased. The temperature registered in the cabinet generally became higher as more fuel was charged in the form of thicker compositions.

Table IV shows that a 30 PHR DOP/15 PHR tricresyl phosphate formulation, although it was retarded in burning, did show signs of flame retardance at 8 mils and was significantly flame retarded in the form of a charred residue. It was also noted that the temperature developed in the DOP composition did not increase with increasing thickness.

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Effect of Antimony Oxide

The addition of 2 PHR of antimony oxide flame retarded 40 PHR DOP composition. Increasing concentrations up to 5 PHR had the effect of further enhancing flame retardance. This higher concentration of antimony oxide flame retarded all types and mixtures of plasticizers tested. Where the opacifying and pigment covering properties of antimony oxide can be tolerated, such as in white opaque film and sheeting, flame retarding with antimony oxide is another way of achieving this desirable property with vinyls.

Table V supplies the detailed data on these tests.

Effect of Fillers:

Table VI contains information of the flammability effects of calcium carbonate and titanium dioxide when added as fillers. Increasing the concentration of Atomite (calcium carbonate) filler accelerated the burning rate and diminished the ignition time when DOP was used as the plasticizer. With a 35 PHR DOP-15 PHR Santicizer 141 formulation, a similar increase in Atomite content had no definite effect and all stocks were self-extinguishing. The flammability effect of 5 PHR of Atomite or titanium dioxide was minimal with all plasticizers tested. It would be interesting to investigate the effect of carbon black on flammability.

Miscellaneous Factors:

The change of stabilizers from the lead type to the epoxy plus barium-cadmium type had very little effect except for prolonging the burning rate of the dioctyl adipate film.

Vinylite VYNW (95% PVC / 5% PVAC) appeared to be only very slightly more flammable than straight polyvinyl chloride. Shorter burning rates were observed for tests 1-b, c and d, and also for Group II when using DOP as the plasticizer. DOP in this resin could be readily flame retarded with phosphate plasticizers and with antimony oxide.

After having run about 2500 tests, during the course of which time several parts of the tester were repaired or replaced, the first series dealing with various concentrations of DOP was retested in Class I-a. Results show somewhat longer burning times than determined previously. The 40 PHR composition was, however, still the most rapid burning. As yet we have not determined whether an aging effect on the film can be detected, or whether the conditions of the tester have changed over the 3-month period of almost continuous usage. All films were calendared during a week and testing started the next week with this group of films.

Discussion of Work

The SP flammability tester is highly sensitive in detecting flame

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...and a properly selected, some amazingly small flammability
...in a desiccator in order to obviate the variable humidity factor on the burning
rate of the thread. This same sensitivity engendered consternation and des-
pair on occasions when necessary repairs and slight changes in the instru-
ment resulted in significant changes in the burning rate or ignition time of
a "standard" specimen.

One measurement which almost invariably proved reliable was that of
determining if the composition was self-extinguishing. By using as a criterion
the amount burned, rather than the rate of burning, very clear-cut and defin-
ite results were obtained. These results are illustrated in the photographs of
the plastics specimens after burning. Although variations could be detected
between the burning rates of several "flammable" compositions, the profound
difference between films was simply whether they continued to burn or not.

Samples of commercial film from six different manufacturers were
evaluated with the tester. Two were found to be effectively flame retarded,
three had burning characteristics resembling those imparted with phthalate
plasticizers, and one was almost completely consumed, thus suggesting the
presence of a proportion of a polymeric plasticizer. It so happened that the
most flammable film was described by accompanying literature as having
some outstanding property other than flame resistance.

All commercial films passed the flammability specification of a 5-second
minimum burning rate as proposed by the SPI. All formulations in this ex-
perimental series which registered a burning rate of less than 5 seconds were
reviewed. Upon re-examination after aging, no film burned in less than 5
seconds.

It therefore appears that the proposed SP' specification can be met by
almost any vinyl formulation.

Future of the SPI Tester:

The SPI tester provides an unexcelled method for determining flam-
mability. It will undoubtedly prove valuable to the trade. Probably, specifications
for flame retardant films by legislation in the future will increasingly call
upon this tester. The ASTM has already started machinery in motion to de-
vise a test method for this instrument.

Another advantage likely to accrue from the development of this tester
is that since a reliable test now exists, the vinyl trade can use it to point up
a possible cause of the problem. Only seven years ago the production
of flame-retarded transparent vinyl film was a troublesome problem. The
plasticizer's then a liability, lacking a suitable property for compara-

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very unstable to heat or light. On the other hand, the situation has changed. Now several good plasticizers are available to impart flame retardance and the significant advances made in stabilization raises no problems when they are used.

The materials indicated in this paper by trademarks are supplied by the following companies:

Vinylite	Bakelite Corp. (Unit of Union Carbide & Carbon Corp.)
Atomite	Thompson-Weinman and Co.
Paraplex	Rohm & Haas Co.
Sovaloid C	Socony-Vacuum Oil Co.
Clorafin 42-S	Hercules Powder Co.
Plumb-O-Sil	National Lead Co.
Mark(stabilizers)	Argus Chemical Lab.
Opalon 300	Monsanto Chemical Co.
Santicizer	Monsanto Chemical Co.

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TABLE I

0800P CLASSIFICATION

TABLE II

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TABLE III

GROUP CLASSIFICATION

1 band d 15° Angle 1/2" Flame Door Opened Inches burned measured				1 c 15° Angle Edge Ignition Door Closed				11 15° Angle Both Burners Edge Ignition				111 Vertical specimens 2" Burner flames for 10 seconds			
Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned
1.8	4.2	120	3-1/2					6	4.8	140	all				
3.2	4.0	140	2					.5	4.7	150	2-3/4				
1.0	3.0	140	all					.5	5.3	80	1-1/2				
1.9	6.9	110	2					.4	5.6	140	3-1/2				
1.9	7.3	180	2-1/2					.5	7.3	150	3-1/2				
2.7	10.0	40	2					.5	6.2	180	1-1/2				
2.0	10.0	200	all					.5	6.3	200	all				
2.7	8.2	140	all					.2	8.3	200	all				
2.7	15.0	80	2					.2	7.0	200	2				
2.7	5.0	280	all	1.2	6.2	120		.2	7.0	250	all				
2.7	5.7	210	all					.2	6.8	260	all				
2.9	5.7	200	all					.1	6.8	240	all				
2.9	7.0	240	all	1.5	5.0	200		.6	5.4	160	all				
2.9	7.2	240	all	1	5.0	200		.6	7.1	200	all				
2.9	7.2	140	all					.4	7.5	220	all				
2.9	7.5	180	all					.3	7.5	240	all				
2.9	7.5	180	all					.3	7.5	240	all				

TABLE IV

[illegible]

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TABLE V

Effect of Antimony Oxide on Flammability

GROUP CLASSIFICATION

1c 15° Angle Edge Ignition Door Closed	1d 15° Angle 1/2" Flame Door Opened Inches Burned Measured	1e 15° Angle Both Burners Edge Ignition	1f Vertical Specimens 2" Burner Flame for 10 seconds						
Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	After Ign. Sec.	Inches Burned
2	6.6	210	all	2	6.6	210	all	---	all
1	6.0	200	3	1	6.0	200	6-1/2	---	6-1/2
1	5.7	195	2-1/4	1	5.7	195	2-1/4	---	4
1	5.6	180	1-1/4	1	5.6	180	1-1/2	---	2-1/2
1	4.4	80	1-1/2	1	4.4	80	1-1/4	---	1-1/4
1	6.0	113	1-1/2	1	6.0	113	1-1/4	---	2-1/2
1	6.2	160	2-1/4	1	6.2	160	2-1/4	---	2-1/2
1	6.2	160	1-1/2	1	6.2	160	1-3/4	---	2-1/2
6	4.3	120	1-1/2	6	4.3	120	1-1/2	---	2
7	5.9	90	1-1/2	7	5.9	90	1-3/4	---	2-1/2
1	6.2	160	1-1/2	1	6.2	160	1-3/4	---	2-1/2

TABLE VI

Effect of Fillers on Flammability

1c 15° Angle Door Closed Inches Burned Measured	1b and 1d 15° Angle Door Opened Inches Burned Measured		1e 15° Angle Edge Ignition Door Closed		1f 15° Angle Both Burners Edge Ignition		1g 15° Angle Specimens 2" Burner Flame for 10 seconds		
Seconds Burning for Ignition	Max. Temp.	Ign. Sec.	Burn. Sec.	Max. Temp.	Ign. Sec.	Burn. Sec.	Max. Temp.	Inches Burned	After Inches Burned
2.5	160	2.7	5.9	140	all	5	4.6	all	all
2.5	170	3.3	4.1	160	all	4	5.8	all	all
2.2	200	3.1	8.0	160	all	4	4.1	all	all
2.2	220	3.2	5.2	170	all	4	5.2	all	all
3.5	SE	3.0	5.1	---	1-1/2	6	6.0	1	2
3.5	SE	3.2	4.6	---	1-1/2	6	6.0	1-1/2	2-1/2
3.5	SE	2.6	4.1	---	1-1/2	2	6.0	5	all
2.6	160	2.6	4.1	130	all	3	6.0	all	all
3.5	SE	3.4	5.6	---	2	4.9	320	all	all
3.5	SE	3.8	4.6	---	MB	1	5.0	140	4-1/2
3.7	SE	4.4	4.3	---	MB	1	5.0	170	2-1/2
4.0	SE	4.7	3.8	---	MB	6	5.7	130	5
2.3	SE	2.7	3.8	---	MB	1	5.4	220	5
2.3	SE	2.3	5.2	160	all	5	5.6	all	all
2.3	SE	2.3	5.2	---	1-1/2	5	5.6	all	2-1/2
2.3	SE	2.7	5.2	---	1-1/2	5	5.6	all	2-1/2
2.3	SE	2.7	5.2	---	1-1/2	5	5.6	all	5-1/2
2.3	SE	2.7	5.2	---	1-1/2	5	5.6	all	all

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TABLE VII

Effect of Changing Stabilizers

GROUP CLASSIFICATION									
Ia 15° Angle 1/2" Flame Door Closed	Ib and d 15° Angle 1/2" Flame Door Opened Inches Burned Measured	Ic 15° Angle Edge Ignition Door closed			Ie 15° Angle Both Burners Edge Ignition			Iii Vertical specimen 2" Burner flame for 15 seconds	
Seconds Burning for Time Ignition Seconds	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Ign. Sec. Burr. Sec. Max. Temp.	Inches Burned	After 15 sec. Flame Burned
1.7 6.5	240	2.2 7.6	140	all	.8 4.6	340	all	all	all
2.7 5.1 SE		3.5 5.6	---	1-1/4	.6 15.7	120	2-1/2	2-1/2	---
2.4 6.9 SE		2.2 6.8	60	1-1/4	.6 4.7	240	all	all	---
1.2 12.2	240	1.2 12.2	200	all	.6 4.5	340	all	all	---

TABLE VIII

Effect of Changing Resin

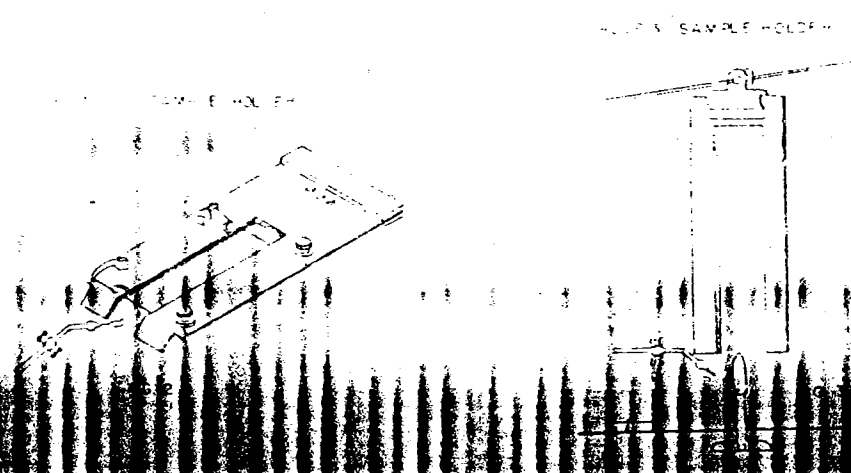
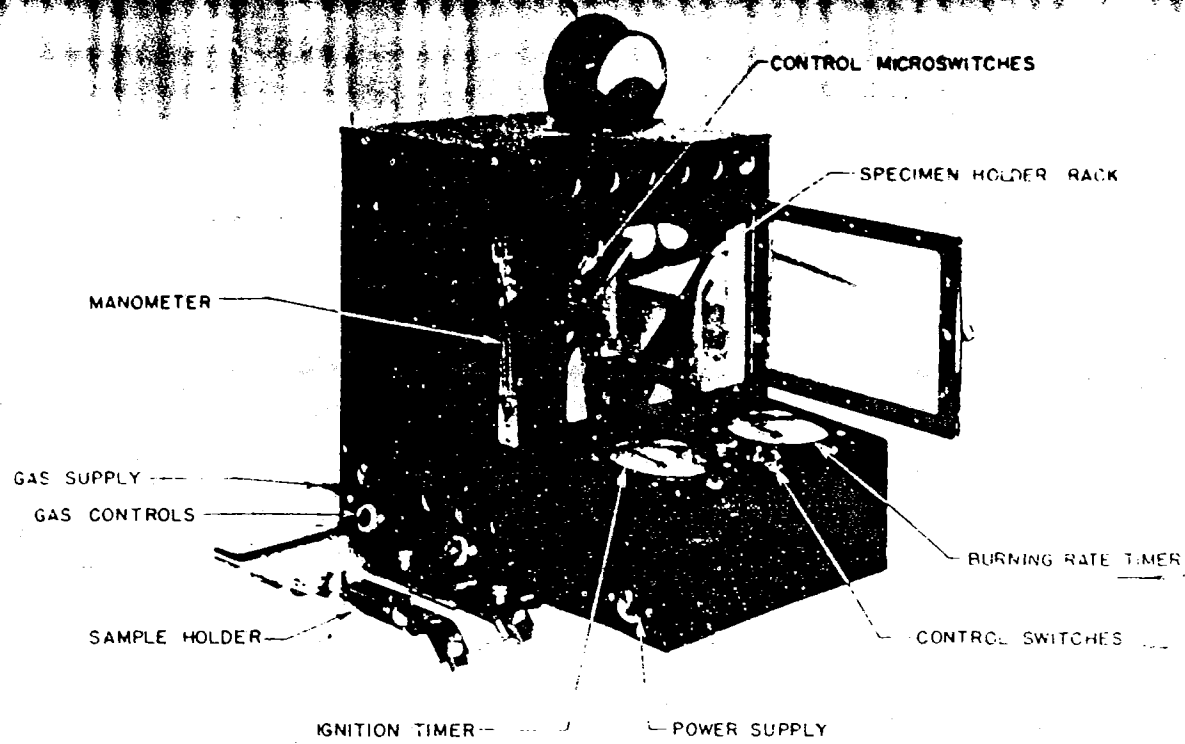
VINYL DOP-50	2.1 5.7	1.5 1.5	4.7 4.7	120 all	1.2 1.2	5.0 5.0	210 210	all	---
DOP-40-50	2.4 5.2 SE	2.2 2.2	5.0 5.0	---	2-1/4 2-1/4	NE NE	120 120	2-1/2 2-1/2	---
DOP-30-50	2.2 6.3 SE	2.2 2.2	6.2 6.2	40	2-1/2 2-1/2	NE NE	160 160	2-1/2 2-1/2	---
DOP-50-50	2.7 4.0 SE	2.5 2.5	7.1 7.1	---	1-1/2 1-1/2	NE NE	150 150	2 2	---

TABLE IX

Effect of Film Aging and/or Reproducibility

DOP-50	3.0 6.9	280 280	1.8 6.4	300 300	1.9 8.0	320 320	1.9 8.3
DOP-50	1.8 6.4	280 280	1.9 8.0	300 300	1.9 8.3	320 320	1.9 8.3

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DOP-45 TCP-5	DOP-40 TCP-10	DOP-35 TCP-15	DOP-40 SI40-5	DOP-40 SI40-10
DOP-40 SI41-10	DOP-35 SI41-15	DOP-30 TOF-20	DOP-35 C42-S-15	DOP-35 SOV.C-15

DOP	TCP	SI40	SI41	SI60
DOP	TCP	SI40	SI41	SI60

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