

UNITED STATES  
DEPARTMENT OF THE INTERIOR  
J. A. KRUG, SECRETARY

BUREAU OF MINES  
JAMES BOYD, DIRECTOR

REPORT OF INVESTIGATIONS

TOXICITY AND FLAME RESISTANCE OF THERMOSETTING PLASTICS

This report covers an investigation conducted jointly by the Bureau of Ships, the Material Laboratory, New York Naval Shipyard, U. S. Navy Department, and the Bureau of Mines, U. S. Department of the Interior



BY

LAWRENCE B. BERGER, H. H. SCHRENK, JAMES A. GALE,  
RALPH W. STEWART, AND LORENZ E. SIEFFERT

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

TOXICITY AND FLAME RESISTANCE OF THERMOSETTING PLASTICS<sup>1/</sup>

By Lawrence B. Berger,<sup>2/</sup> H. H. Schrenk,<sup>3/</sup> James A. Gale,<sup>4/</sup>  
Ralph W. Stewart,<sup>5/</sup> and Lorenz E. Sieffert<sup>6/</sup>

INTRODUCTION

The purpose of the investigation discussed in this article was to evaluate the toxic hazard from gases produced when thermosetting plastic materials used for electrical insulating purposes are burned or thermally decomposed in enclosed spaces. For brevity the term "toxicity" is used throughout the article to refer to the nature of gases produced under these conditions. A need for immediate study of the problem was created as a result of the battle damage reports received from various United States Fleet units. These indicated that the fumes emitted when these materials were burned or thermally decomposed had, in several instances, resulted in toxic atmospheres. In attempting to develop a suitable test method for evaluating toxicity, it was recognized that no single test procedure could be expected to duplicate the variety of conditions that might exist in an actual fire aboard ship. In view of the immediate need, however, it was felt that considerable information could be obtained, at least to classify comparatively the several types of materials as to the toxic effect caused by burning or thermal decomposition, through the development of a sound empirical method which could be applied to all of the materials. On this basis, it was decided to modify the Navy flame-resistance test for thermosetting plastics to render it applicable to the determination of the toxic gases evolved when

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The opinions or assertions contained herein are the private ones of the authors and are not to be construed as official or reflecting the views of the Navy Department, the Naval service at large, or the United States Department of the Interior.

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2/ Chemist, Bureau of Mines, Central Experiment Station, Pittsburgh, Pa.

3/ Chief, Health Division, Bureau of Mines.

4/ Chemical engineer, Material Laboratory, U. S. Naval Shipyard, New York, N. Y.

5/ Electrical chemical engineer, Bureau of Ships, U. S. Navy Department, Washington, D. C.

6/ Electrical engineer, Bureau of Ships, U. S. Navy Department, Washington, D. C.

this test was employed. A wide variety of materials was tested, both molded and laminated, to cover the types commonly used by the Navy in electrical applications. Toxicity determinations were conducted at the Bureau of Mines Central Experiment Station, Pittsburgh, Pa. Parallel tests, to determine the flame resistance of the materials, were conducted at the Material Laboratory, U. S. Navy Yard, New York, N. Y.

#### EQUIPMENT

The Navy test method and equipment for flame-resistance tests of thermo-setting plastics, as used at the Material Laboratory, have been previously described in the literature,<sup>7/</sup> <sup>8/</sup> <sup>9/</sup> Briefly, the equipment consists of a specimen support, heater coil, and spark plugs, arranged in an enclosure of sufficient size. The enclosure is provided with vent holes, distributed around the sides adjacent to the base, to admit fresh air and an exhaust fan at the top, operated at minimum suction just sufficient to carry off smoke and gases. A sliding door at the front of the enclosure, with shatterproof glass window, permits access to the equipment and a clear view of the interior. The specimen support is an ordinary four jawed lathe chuck suitably secured to the base of the enclosure. The heater coil consists of seven turns of No. 10 (0.102-inch diameter) Nichrome resistance wire, space wound to 0.25 inch per turn and 1-3/16 inches diameter. The coil ends are clamped into heavy copper lugs with the axis of the coil coincident with the axis through the opening in the specimen support and with the lower end of the coil 1/2 inch above the top of the support.

Two spark plugs, with extended electrodes, diametrically opposite, are placed with their longitudinal center lines in a horizontal plane 1/2 inch above the top of the heater coil, to ignite gases emitted from the heated specimen. The spark plugs are mounted in such a manner that they may be moved to within 1/8 inch of the surface of the specimen when in operation or away from the specimen after ignition occurs, to prevent their electrodes from becoming fouled by soot. A suitable electric circuit is provided to maintain continuous sparking at the electrodes.

Current is supplied to the heater coil through the heavy lugs which are, in turn, connected to the secondary of a transformer. Current is controlled by means of a variable auto-transformer in the primary.

Specimens 1/2 inch by 1/2 inch by 5 inches, of the type commonly used for flexural strength tests, were used. Samples of molded materials were fabricated to finished dimensions in a standard test mold and laminated samples were cut from 1/2-inch-thick sheet stock.

<sup>7/</sup> Gale, J. A., Stewart, R. W., and Alfors, J. B., Determining the Flammability of Thermosetting Materials: Plastics, June 1945, p. 56.

<sup>8/</sup> Gale, J. A., Stewart, R. W., and Alfors, J. B., Flame Resistance of Thermosetting Plastics: A.S.T.M. Bull., Dec. 1944, pp. 23-27.

<sup>9/</sup> Joint Army-Navy Specification JAN-P-14 dated Sept. 30, 1944.

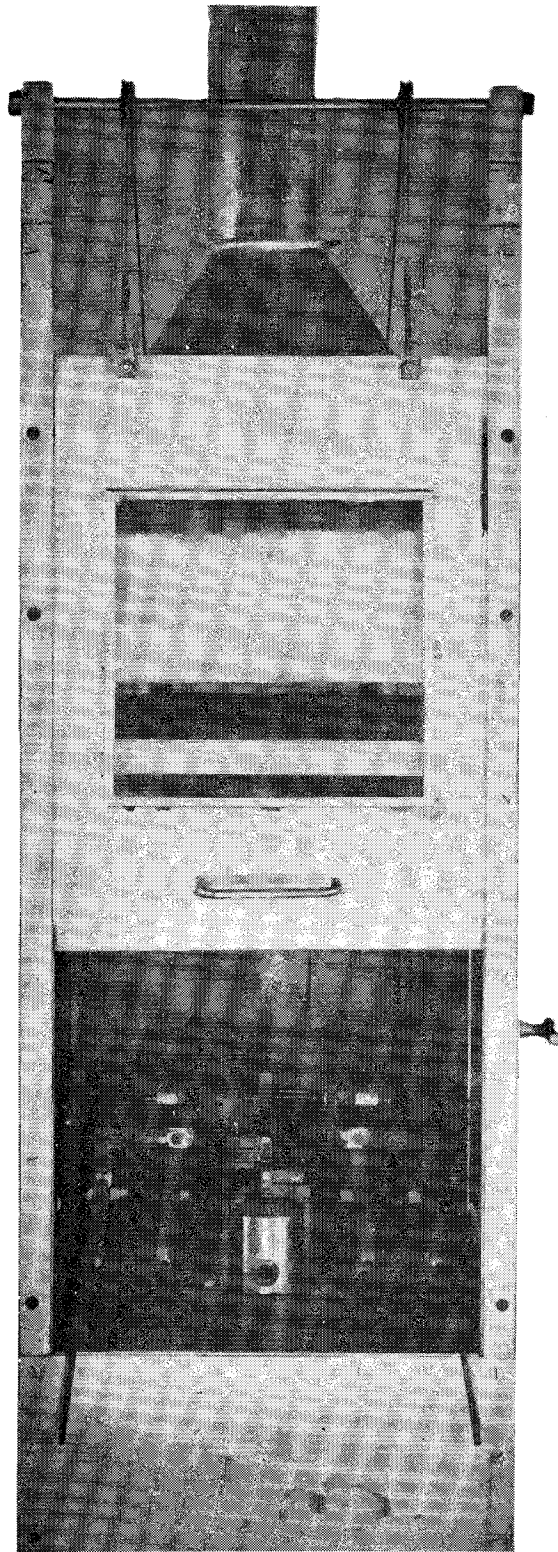


Figure 1. - Flame-test apparatus; general arrangement.

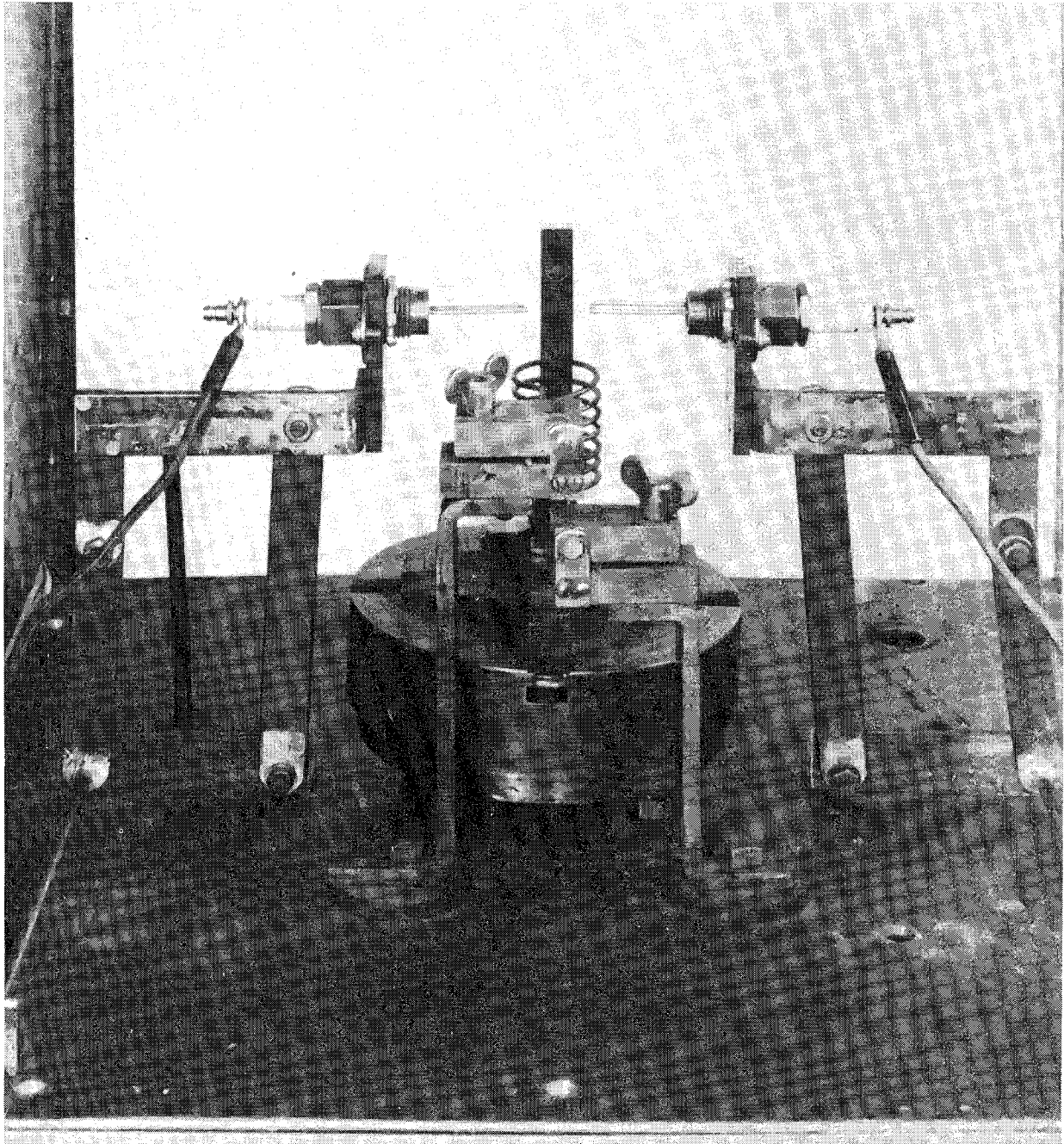


Figure 2. - Front view of accessories, spark plug, heater coil, and supporting legs and specimen.

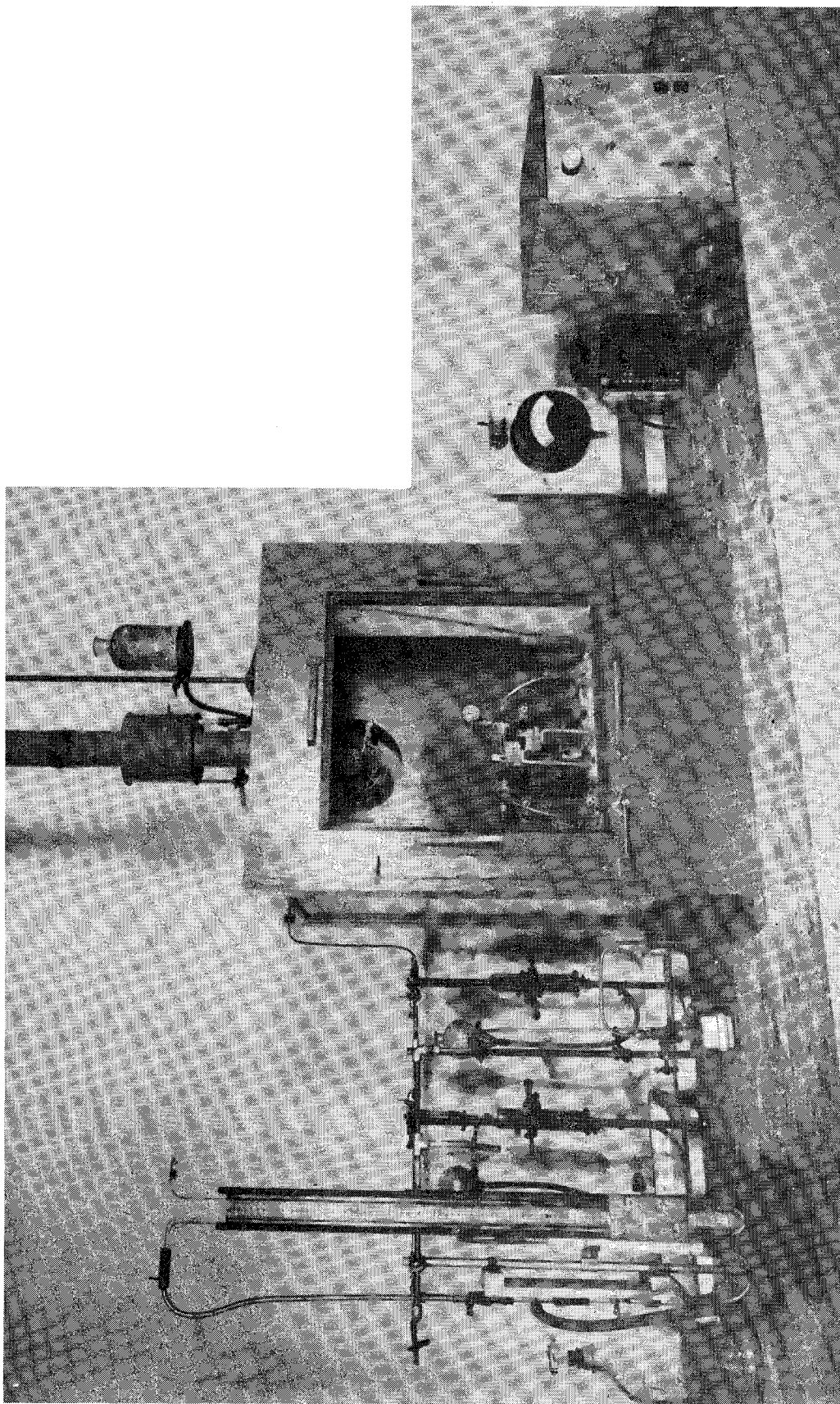


Figure 3. - Toxicity-test apparatus; general arrangement.

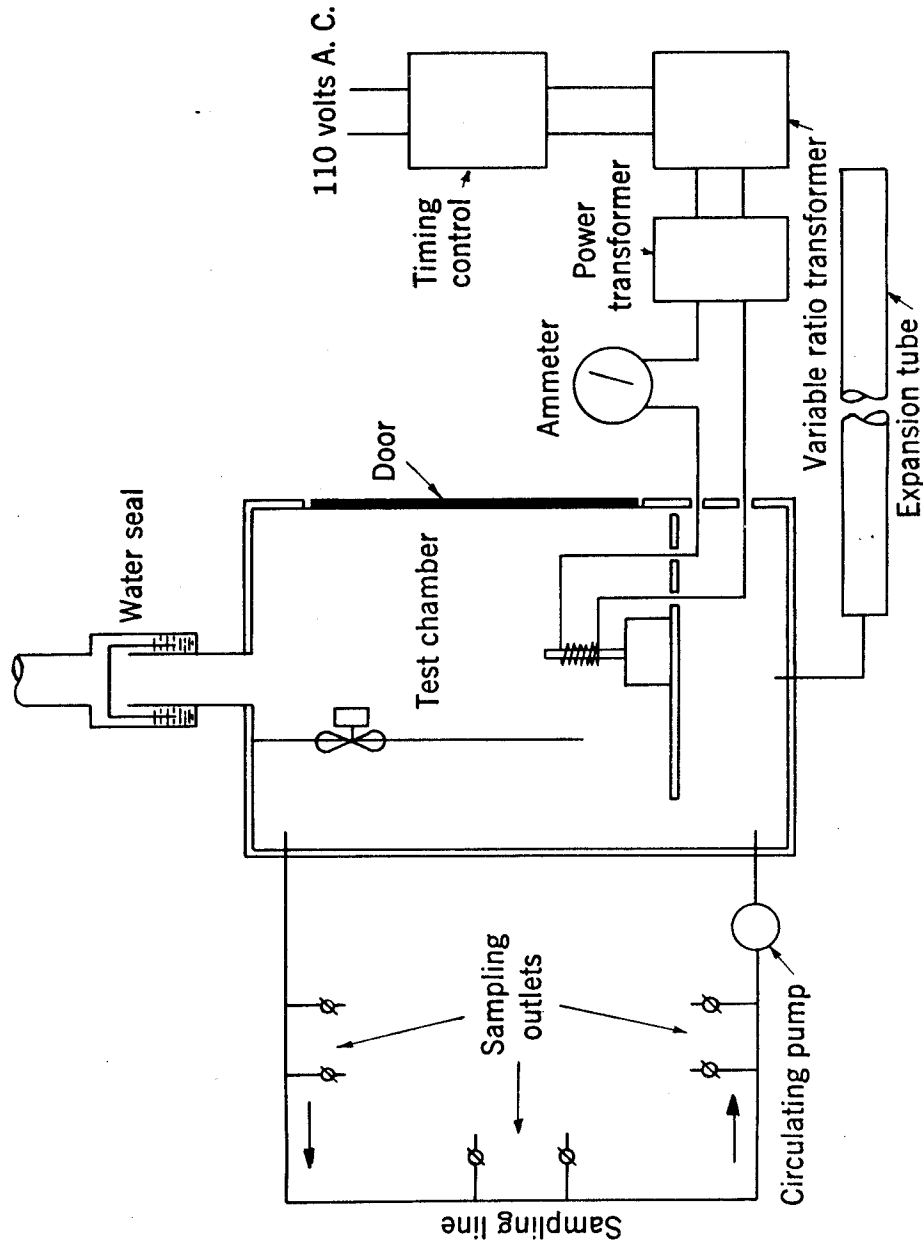


Figure 4. - Toxicity-test apparatus; diagrammatic sketch showing accessory apparatus and gas-sampling arrangement.

A photograph of the entire flame-resistance test equipment is shown in figure 1. Figure 2 shows a close-up of the specimen support, spark plugs, and heater coil, with the specimen in position for test.

The test apparatus used at the Bureau of Mines Central Experiment Station for determining toxicity of gases emitted by the specimens is similar to the flame-resistance test equipment, except that the specimen support, spark plugs, and heater coil are enclosed in a gas-tight chamber instead of a ventilated one.

The test chamber is of wood construction, with glass sides and with a small window set into the door at the front of the chamber to permit observation of the specimen during the test. The inside dimensions of the chamber are 24-3/4 inches by 21 inches by 32-3/4 inches, and its volume is 270 liters. The door of the chamber closes on a soft rubber gasket and is secured when closed by four refrigerator-door-type clamps. The chamber was found by test to be essentially gas-tight. A baffle plate, to facilitate mixing of the chamber atmosphere, is situated 6 inches from the rear wall and extends downward from the top of the chamber to within 5 inches of the base supporting the flammability-test equipment. A 4-inch fan is mounted in a circular opening near the top of the baffle. This fan is operated only after burning of the test specimen has ceased, circulation of the atmosphere of the chamber being obtained during the test by convection set up by the heating coil and by combustion of the specimen. The chamber may be ventilated at the conclusion of a test by connection to an exhaust ventilation duct. This duct is closed off from the chamber by a water seal during test. To prevent undue pressure changes in the chamber during test, as a result of heating and cooling, the chamber atmosphere is permitted to expand through a suitable connection into a 3-inch-diameter metal tube, open at one end, during operations that evolve heat. When burning of the test specimen has ceased and cooling of the chamber atmosphere ensues, the gases discharged by thermal expansion from the chamber into the tube are again drawn into the chamber from the tube by contraction due to cooling. With this arrangement, error from loss due to expansion on heating and dilution of the test chamber atmosphere during cooling is negligible.

The test apparatus for determination of toxicity of gases, including gas sampling equipment, is shown in figure 3. Figure 4 shows a diagrammatic sketch of the same apparatus.

#### METHOD OF TEST

The test procedure was essentially the same for both the flame-resistance and toxicity determinations. The heating coil was clamped in the copper lugs, and the specimen was inserted into the support so that it was within, but not touching, the heater coil and so that the specimen extended 2 inches above the top of the coil. The spark plugs were moved into position near the specimen, and the prescribed current (55 amperes) was passed through the heating coil. The time required for ignition was noted. Heating was continued for 30 seconds after the sample had ignited, and burning time was taken as the time required for the flame to extinguish itself after the heater coil was deenergized.



The complete procedure for determination of flame resistance is described in detail in the references previously cited.<sup>10/</sup> The flexural strength of the specimen was determined after test, where possible, and the ignition temperature was determined, using the ignition-temperature conversion curve. This curve was developed by measuring the temperature at the edge of a specimen under test by means of thermocouples and is reproduced here for convenience (fig. 5). It will be noted that the curve has been extended to include ignition time up to 450 seconds.

In determination of the toxicity of the gases, the chamber atmosphere was stirred for 5 minutes by the fan at the conclusion of the test, and samples of the atmosphere were collected for chemical analyses. The volumes of the various toxic gases indicated by these analyses served as an index of the toxic hazard presented by the specimen under test.

In the event of nonignition of the samples, the coil current was discontinued after 600 seconds in the flame-resistance test and after 480 seconds in the toxicity test.

#### RESULTS OF INVESTIGATION

All materials tested produced toxic gases in some amount, and therefore only the relative hazard was considered rather than attempting to interpret test data in a manner that might result in arbitrary classification of materials as "safe" or "unsafe."

Concentrations of toxic gases produced by burning or thermal decomposition of the materials depend, in addition to the individual characteristics of the materials, upon the following factors:

- (a) Quantity of material involved,
- (b) Whether material ignites and burns; or does not ignite and is thermally decomposed by heat from adjacent sources,
- (c) Size of enclosure,
- (d) Rate of ventilation in enclosure, and
- (e) Temperature and duration of heating of material.

As in an actual fire the possible variations in the foregoing conditions cannot be predicted, the first approach to the study of production of toxic gases was to subject the materials to essentially uniform test conditions and to determine the quantities of the significant toxic gases produced under such conditions.

Under the empirical test conditions, the order of difference in the indicated toxicity of the thermosetting plastic materials was not extremely wide. Toxicity should be considered in the choice of thermosetting plastic materials for specific applications only if the factors mentioned above are such as to represent an extreme condition, under which high concentrations of toxic gases may be expected. On the basis of the test results, it is

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<sup>10/</sup> See footnotes 7, 8, and 9.

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All materials tested produced toxic gases in some amount, and therefore only the relative hazard was considered rather than attempting to interpret test data in a manner that might result in arbitrary classification of materials as "safe" or "unsafe."

Concentrations of toxic gases produced by burning or thermal decomposition of the materials depend, in addition to the individual characteristics of the materials, upon the following factors:

- (a) Quantity of material involved,
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As in an actual fire the possible variations in the foregoing conditions cannot be predicted, the first approach to the study of production of toxic gases was to subject the materials to essentially uniform test conditions and to determine the quantities of the significant toxic gases produced under such conditions.

Under the empirical test conditions, the order of difference in the indicated toxicity of the thermosetting plastic materials was not extremely wide. Toxicity should be considered in the choice of thermosetting plastic materials for specific applications only if the factors mentioned above are such as to represent an extreme condition, under which high concentrations of toxic gases may be expected. On the basis of the test results, it is

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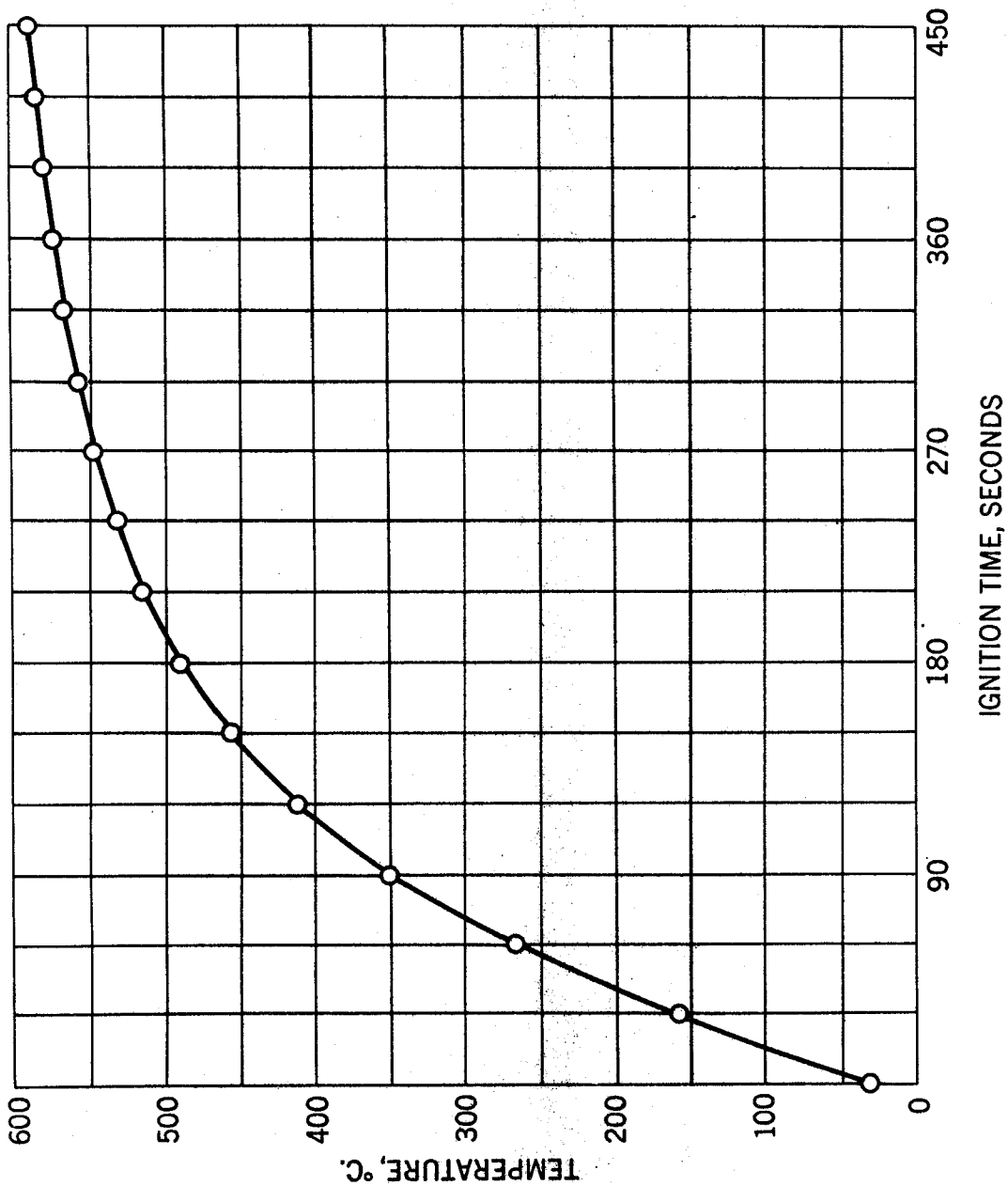


Figure 5. - Relationship between ignition time and temperature of plastic test-piece; coil current, 55 amperes.

not believed that, even under such conditions, toxicity should be the sole criterion of suitability of material.

As has been previously ascertained, the order of difference in flame resistance between various compositions of molded and various compositions of laminated thermosetting plastic materials is quite large, depending upon the type of resin and filler used. The factors considered in evaluating flame resistance were:

- (a) Ignition time (or ignition temperature),
- (b) Burning time,
- (c) Percentage of flexural strength retained after burning.

The toxic gases of chief significance produced under the described test conditions were carbon monoxide, cyanides, and ammonia. In addition to these gases, the test atmospheres contained aldehydes and smoke, and, in some instances, trace amounts of oxides of nitrogen, phenols, and amines. As in any combustion process, carbon dioxide was formed and the oxygen content of the test chamber atmosphere was decreased. The magnitude of the hazard created in an actual fire by production of carbon dioxide and depletion of oxygen would be dependent upon the quantity of material burned and the size of the enclosure in which the fire took place.

The volumes of carbon monoxide, cyanides, and ammonia produced in the tests are given in tables 1 and 2. Table 1 gives the ranges in volumes of these three toxic gases obtained for the various grades of molded thermosetting materials of each type as well as the number of grades of each type tested. Table 2 gives the ranges in volume of these three toxic gases obtained for the various grades of laminated thermosetting materials of each type as well as the number of grades of each type tested. It is emphasized that these volumes are the observed values obtained under the particular test conditions employed by the Bureau of Mines Central Experiment Station.

TABLE 1. - Volumes of toxic gases produced in Bureau of Mines tests on molded thermosetting materials

Navy type	Number of materials	Resin - filler	Range of volume of gases produced, milliliters <sup>1/</sup>		
			Carbon monoxide	Cyanides	Ammonia
MFI-20	7	Phenolic-asbestos	51-108	0-6.7	6.3-95
MFI-10	3	Phenolic-asbestos	65-88	1.6-3.9	27-267 <sup>2/</sup>
MFG	4	Phenolic-asbestos	69-88	2.2-4.4	137-307
MFH	4	Phenolic-asbestos	45-79	1.9-2.9	124-252
MFE	4	Phenolic-mica	51-92	3.0-4.6	6.4-164
	22	Phenolic-mineral	45-108	0-6.7	6.3-307
CFG	3	Phenolic-wood-flour	45-85	1.4-2.1	9.7-15
CFI-20	3	Phenolic-cotton	55-89	.6-4.8	7.4-55
CFI-40	2	Phenolic-cotton	68-125	1.8-2.1	9.3-20
	8	Phenolic-cellulose	45-125	.6-4.8	7.4-55
-	3	Melamine-glass (ignited)	50-132	9.7-17	20-97
-	1	Melamine-asbestos (ignited)	32	5.2	24
G-2	2	Melamine-asbestos (ignited)	80-96	6.5-11.3	22-29
	6	Melamine-mineral (ignited)	32-132	5.2-17	20-97
-	3	Melamine-glass (not ignited)	140-199	14-19	361-480
-	1	Melamine-asbestos (not ignited)	158	13	725
	4	Melamine-mineral (not ignited)	140-199	13-19	361-725
		Melamine-mineral (ignited and not ignited)	32-199	5.2-19	20-725
G-2	1	Modified melamine-cellulose	53	3.2	16
G-3	2	Melamine-cotton flock	108-162	1.5-13	27-73
Types G-2 and G-3 combined	3	Melamine-cellulose	53-162	1.5-13	16-73

1/ Gas volumes calculated from volume of test chamber and concentration of constituent. Volumes at 60°F. and 30 inches of mercury.

2/ 1 value high in 3 materials tested.

*11 Samples 1/2" x 1/2" x 5" - 14 ea. in*  
*1/ Test chamber 24" x 21" x 30"*  
*Volume - 270 liters*  
*100 milliliter = 1/2700 pounds in 400 pounds*  
*per milliliter*  
*100 milliliter = .04%*

TABLE 2. - Volumes of toxic gases produced in Bureau of Mines tests on laminated thermosetting materials

Navy type	Number of materials	Resin - filler	Range of volume of gases produced, milliliters <sup>1/</sup>		
			Carbon monoxide	Cyanides	Ammonia
GMG .....	4	Melamine-glass (ignited) .....	46-92	7.3-12	67-112
AMG .....	1	Melamine-asbestos (ignited) .....	78	16	111
	5	Melamine-mineral (ignited) .....	46-92	7.3-16	67-112
GMG .....	6	Melamine-glass (not ignited) ....	82-141	7.4-13	268-376
AMG .....	1	Melamine-asbestos (not ignited)..	178	15	471
	7	Melamine-mineral (not ignited) ..	82-178	7.4-15	268-471
		Melamine-mineral (ignited and not ignited) .....	46-178	7.3-16	67-471
FBG .....	2	Phenolic-cotton .....	74-163	2.5-3.1	6.6-8.8
GBE .....	1	Phenolic-aniline-formaldehyde-glass .....	69	5.5	7.1
PBE .....	1	Phenolic-paper .....	127	3.7	3.3

<sup>1/</sup> Gas volumes calculated from volume of test chamber and concentration of constituent. Volumes at 60°F. and 30 inches of mercury.

CONCLUSIONS

Molded Thermosetting Plastic Materials. - On the basis of table 1, the following conclusions are drawn as to the toxicity of various types of molded thermosetting plastic materials:

- (a) Comparison of phenolic-mineral-filled and cellulose-filled molded materials.
- (1) The volumes of carbon monoxide and cyanides, respectively, are approximately equal for both classes of material.
  - (2) The range of volumes of ammonia obtained for phenolic-mineral-filled materials is wider than that obtained for phenolic-cellulose-filled materials.
  - (3) Phenolic-asbestos-fabric-filled materials (MFI-20) and phenolic-cotton-fabric-filled materials (CFI-20) gave off approximately equal volumes of ammonia as well as of carbon monoxide and cyanides, and may be considered equivalent from the toxic standpoint.
  - (4) Phenolic-asbestos-fiber-filled materials (MFG and MFH) gave off appreciably more ammonia than any of the phenolic cellulose filled types.
- (b) Comparison of mineral-filled-phenolic and melamine-molded materials.
- (1) The volumes of all three toxic gases considered were higher for the melamine-mineral-filled materials that did not ignite than for the phenolic-mineral-filled materials.
  - (2) The melamine-mineral-filled materials which did not ignite gave off greater volumes of the gases considered than the melamine-mineral-filled materials which ignited.
- (c) Comparison of cellulose-filled-phenolic and melamine-molded materials.
- (1) The volumes of carbon monoxide, cyanides, and ammonia, respectively, are approximately equal for both classes of material.

Laminated Thermosetting Plastic Materials. - On the basis of table 2, the following conclusions are drawn as to the toxicity of various types of laminated thermosetting plastic materials:

- (a) Comparison of melamine-mineral and phenolic-cotton laminated materials.
- (1) The volumes of carbon monoxide given off are approximately equal for both classes of material.



- (2) Types GMG melamine-glass and AMG melamine-asbestos laminated materials gave off considerably greater volumes of cyanides and ammonia than did type FBG phenolic-cotton laminated material.
- (3) The melamine-mineral laminated materials which did not ignite gave off greater volumes of carbon monoxide and ammonia than the melamine-mineral laminated materials which ignited.

The results of the flame-resistance tests conducted at the Material Laboratory indicate that molded thermosetting plastic materials may be grouped into four general classifications, as follows:

- Class I - Low ignition time, long burning time, complete carbonization, no flexural strength remaining after burning - cellulose-filled phenolic materials.
- Class II - Low ignition time, medium burning time, complete carbonization, no flexural strength remaining after burning - low impact strength materials of the following compositions:
- (a) Mineral-filled phenolic (mica and asbestos)
  - (b) Mineral-filled melamine
  - (c) Cellulose-filled melamine
- Class III - Medium ignition time, medium burning time, some flexural strength remaining after burning - asbestos-filled phenolic materials of high impact strength.
- Class IV - High ignition time, short burning time, some flexural strength remaining after burning - mineral-filled melamine materials of high impact strength (glass and asbestos).

The range of values obtained for the various grades of molded thermosetting materials of each type and the number of grades of each type tested are given in table 3.

On the basis of the results shown in table 3, it appears that:

- (a) For the same type of resin, an inorganic filler will provide a more flame-resistant plastic than an organic filler.
- (b) For the same type of filler, a melamine resin will provide a more flame-resistant plastic than a phenolic resin.

It should be noted that an increase in flame resistance will result in a thermal decomposition of the material which will evolve more toxic gases than if the same material had ignited.

TABLE 3. - Flame resistance of molded thermosetting materials -  
Material Laboratory tests

Class	Navy type	Number of materials	Resin - filler	Ignition time range, seconds	Ignition temperature range, degrees C.	Burning time range, seconds	Percent flexural strength remaining after burning
I..	CFI-40	2	Phenolic-cotton .....	60-90	275-350	270-300	0
	CFI-20	3	Phenolic-cotton .....	90-120	350-405	240-330	0
	CFG	3	Phenolic-wood-flour .....	60-150	275-450	150-270	0
II..	MFE	4	Phenolic-mica .....	60-150	275-450	120-210	0
	G-2	1	Modified melamine-cellulose .....	60	275	120	0
	G-2	2	Melamine-asbestos .....	120-180	405-490	150-180	0
	MFH	4	Phenolic-asbestos .....	120-180	405-490	90-150	0
	G-3	2	Melamine-cotton flock .....	120-270	405-575	30-60	0
III..	MFI-20	7	Phenolic-asbestos .....	120-210	405-525	120-210	20-35
	MFI-10	3	Phenolic-asbestos .....	120-240	405-560	60-180	0-40
	MFG	4	Phenolic-asbestos .....	150-240	405-560	60-90	45-60
IV..		4	Melamine-glass .....	240 $\frac{+1}{-}$	560 $\frac{+1}{-}$	30-60 $\frac{+1}{-}$	30-50
		1	Melamine-asbestos .....	120 $\frac{+1}{-}$	405 $\frac{+1}{-}$	40 $\frac{+1}{-}$	6

1/ In most cases, materials did not ignite within 600 seconds and the current in the coil was turned off.

## GENERAL SUMMARY

Due to the practical limitations imposed by the empirical nature of the tests, a complete answer to the multitudinous problems connected with toxicity of the gaseous decomposition products of molded and laminated thermosetting materials, resulting from fires aboardship, cannot be given. It is considered, however, that the tests were sufficiently broad in scope to provide information of value as a guide to the relative toxic hazard of these materials in connection with the choice of materials for shipboard applications. It is emphasized that all materials tested produced toxic gases in some amount, and therefore none of the materials may be considered as presenting no toxic hazard if burned or thermally decomposed in an enclosed space.

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