

Catalytic Decomposition of the Fire Extinguishant Bromochlorodifluoromethane

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ABSTRACT

A study was made of the action of hopcalite catalyst on bromochlorodifluoromethane (CBrClF_2) at 600°F , both as a single air contaminant and in the presence of carbon monoxide (CO) as a second contaminant. A bench-scale catalytic burner, which simulated the operating conditions used in the catalytic burners of nuclear submarines, was employed to expose the contaminants to the catalyst at concentrations of about 100 ppm.

The results showed that less than 5% of the influent CBrClF_2 was decomposed in passing through the burner. Free chlorine (Cl_2) and/or bromine (Br_2), hydrogen chloride (HCl) and/or hydrogen bromide (HBr), and hydrogen fluoride (HF) were detected in the burner exhaust gas to confirm that a small amount of CBrClF_2 did decompose. A trace (<0.1 ppm) of phosgene (COCl_2) was detected in one instance of CBrClF_2 decomposition. The activity of the catalyst for CBrClF_2 was not affected by the presence of CO .

The activity of the catalyst for CO was only slightly reduced by the presence of CBrClF_2 . In a mixture of the two gases, all of the influent CO was oxidized within the first 40% of the 5-in. catalyst bed depth.

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CATALYTIC DECOMPOSITION OF THE FIRE EXTINGUISHANT BROMOCHLORODIFLUOROMETHANE

INTRODUCTION

Bromochlorodifluoromethane (CBrClF₂) is known also as Halon 1211 and Fluorocarbon 12Bl. Under normal conditions it is a colorless gas with a faintly sweet smell and a density about five times that of air. It can be readily liquefied by compression for storage in closed vessels, and at room temperature (72° F) its vapor pressure is about 25 psig.

As a fire extinguishing agent, Halon 1211 is effective against flammable liquid fires. It is electrically nonconductive and suitable for use against fires involving electrical equipment. It should not be used on fires of active metals and metal hydrides, nor against burning materials that contain their own oxidizer (1).

The extinguishing action of most common agents is through the physical processes of cooling and diluting, whereas the chemical extinguishants are able to interfere with the combustion process itself and thus are much more effective agents. Halon 1211 is of the latter type. It extinguishes fires by removing active species that are involved in the chain reactions, a process known as chain breaking. All the halogens are active in this way, but bromine is very much more effective than either chlorine or fluorine, and it is probable that Halon 1211 owes its high efficiency mainly to the presence of a bromine atom in the molecule (1).

Experimental data have shown that Halon 1211 can be tolerated by humans in concentrations up to 4% by volume for exposures of approximately 1 min. This indicates that its toxicity is of the same order as that of carbon dioxide, and Underwriter's Laboratories, Inc., in classifying chemicals according to toxicity, has placed Halon 1211 and carbon dioxide in the same group (1). However, exposure to Halon 1211 is generally of less concern than is exposure to its decomposition products.

Halon 1211 has characteristics which show promise for its application as a fire extinguishing agent for use in closed atmospheres such as in nuclear submarines. To qualify for submarine use, it is necessary not only that the characteristics of the Halon 1211 itself, but also the nature and quantity of any deleterious vapor which could result from its interaction with the shipboard air purification equipment be known.

A unit of submarine air purification equipment which might readily decompose a contaminant such as Halon 1211 is the CO/H₂ catalytic burner. In this device, the submarine air is continuously exposed to hopcalite catalyst at about 600° F to oxidize carbon monoxide (CO) to carbon dioxide (CO₂) and hydrogen (H₂) to water (H₂O). The CO₂ and water formed are removed by other types of air purification equipment. The CO/H₂ burner is an efficient instrument for controlling the concentration of CO and H₂ in submarine air, but its use presents certain problems which would not exist otherwise. For example, other contaminants in addition to CO and H₂ are decomposed by exposure to the hopcalite at an elevated temperature, and in some cases the decomposition products are more undesirable than the original contaminants.

A continuing series of studies of problems associated with the CO/H₂ burner has been carried on at NRL since before the time of the launching of the first nuclear submarine (2). The stabilities over hopcalite catalyst of a number of types of chemical compounds found or anticipated to be in nuclear submarine atmospheres have been investigated using a bench-scale catalytic burner. Included in this work were detailed studies of a number of halogenated hydrocarbons (3, 4, 5, 6, 7, 8). These compounds are used as refrigerants, fire extinguishing agents, aerosol propellants, degreasers, general solvents, and as a component of "non-flammable" adhesives. The action of hopcalite catalyst at 600° F to decompose low concentrations of these compounds in air has been found to range from very slight in the case of CCl₂F₂ (R-12), to virtually complete for CH₃CCl₃ (methyl chloroform).

As a result of recent interest in Halon 1211 for possible use as a fire extinguishing agent in nuclear submarines, the catalytic action of hopcalite on Halon 1211 was studied and is discussed in this report.

EXPERIMENTAL METHOD

Apparatus

The bench-scale burner used in this study was a reactor with an integral preheater, both of which were contained in a 28-in. length of 1-1/8-in. I. D. stainless steel tubing mounted vertically and surrounded by two separate electrical heaters. Figure 1 is a schematic of the burner. The heaters were independently powered by two variable voltage transformers, and the tube and heater assembly was heavily insulated with an aluminasilicate fiber blanket and wrapped with asbestos tape. The upper section of the tube was packed with stainless-steel turnings and served as a preheater for the inlet air. The lower section (reactor) of the tube contained the catalyst bed. One thermocouple was located in the burner tube between the preheater and catalyst and a second one at the exhaust end of the catalyst bed. The thermocouples ordinarily were connected to indicating pyrometers for convenient indication of the inlet and exhaust catalyst bed temperatures, but occasionally they were connected to a Rubicon potentiometer for more accurate measurement of these temperatures.

The burner was equipped with eight sampling lines of 1/8-in. O. D. stainless steel tubing to permit sampling of the air at the burner inlet, the preheater exhaust (between preheater and catalyst bed), at 1-in. increments of catalyst bed depth, and at the burner exhaust. The bed-sampling tubes were welded to the burner tube and had 0.072-in. O. D. extensions with square-cut ends which projected 3/8-in. into the catalyst bed. The inlet and exhaust sampling tubes were attached to the burner inlet and exhaust lines with tube fittings and did not project into the airstream. The sampling tubes were made about 4 ft long to provide cooling for the gas samples and were connected through stainless steel toggle valves with a gas chromatograph.

This burner is of a new design incorporating features that offer three major advantages over the burners used in earlier work. First, the use of two independently controlled heaters instead of the previous single heater permits a closer control of the catalyst bed temperature. Second, the use of additional catalyst bed sampling ports makes it possible to measure changes taking place within the bed that were not detectable by using only inlet and exhaust sampling, as was done previously. Third, the location of the preheater above the catalyst bed prevents catalyst dust from falling into the preheater and promoting some catalytic decomposition there.

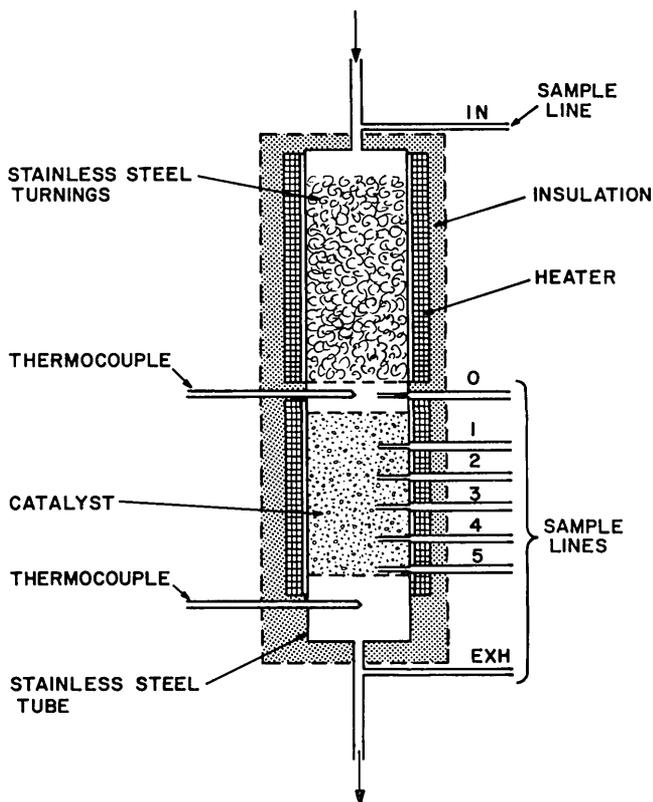


Fig. 1 — Bench-scale catalytic burner

In addition to the burner and analytical equipment, the major apparatus used in this study was the equipment required to prepare and deliver the contaminated gas stream to the burner inlet. Dry compressed air from the station line was supplied through a pressure regulator to a flowmeter for measurement of flow rate, after which the stream was divided into two parts. One portion of the air was passed through a water bubbler at room temperature and then remixed with the other portion to provide the desired humidity for the burner inlet air. The Halon 1211 was bled from a pressurized cylinder into the humid airstream with the rate of flow being controlled by a small needle valve. The CO was also bled from a pressurized cylinder into the air, but in this case the flow of gas was controlled by a small nozzle. The humid contaminated air then passed through a mixing loop and to the burner inlet. A gate valve in the burner exhaust line was used to maintain a slight pressure on the burner to permit easy leak-free sampling of the air for analysis.

Procedure

The apparatus used was scaled to bench size for convenience and economy in operation. Other operating parameters, except contaminant concentrations, were selected to simulate those of the shipboard burner. An airflow of 1 cfm was passed through the catalyst bed which had a 1-in.² cross-sectional area and a 5-in. bed depth. This provided a space velocity of 21,000 hr⁻¹. The catalyst temperature was 600 ± 10°F and the relative humidity of the inlet air was 50-60% at 25°C. The two contaminants, CO and Halon 1211, in the burner influent air were exposed to the hopcalite catalyst both separately and in

combination. Concentration levels of the contaminants were controllable in a range of ± 5 ppm during a day's run. During the series of runs, contamination levels used were 75-130 ppm for CO and 100-110 ppm for Halon 1211. These concentrations were based on estimates of the levels that could reasonably exist in submarine air and, to a certain extent, on analytical procedures.

The airstream was analyzed for CO and Halon 1211 at all sampling ports where detectable quantities of the contaminants remained unoxidized. When CO was used as a single contaminant, it was completely oxidized within the first 2 in. of catalyst bed and consequently the air was not analyzed for CO at points further downstream. Analyses of the gas at the burner inlet and preheater exhaust ports gave a measure of the thermal breakdown of the contaminants at 600°F, since between those two points the gas was heated to this temperature while being in contact with stainless steel only. Analyses made at succeeding sample ports gave a measure of the concentration of contaminants remaining at successive 1-in. increments of the catalyst bed. The decomposition of Halon 1211 was confirmed by the detection of breakdown products in the burner exhaust as well as by disappearance of the Halon 1211 as it passed through the burner.

According to Reference 1, the main decomposition products of CBrClF_2 upon exposure to a flame or hot surface at temperatures higher than 900°C are hydrogen bromide (HBr), hydrogen chloride (HCl), hydrogen fluoride (HF), bromine (Br_2), chlorine (Cl_2), and fluorine (F_2) with small amounts of carbonyl bromide (COBr_2), carbonyl chloride (phosgene or COCl_2), and carbonyl fluoride (COF_2). It was expected that any decomposition which might occur over hopcalite catalyst at 600°F would yield these same decomposition products, and the burner exhaust was analyzed for all of these potential breakdown products except COBr_2 , COF_2 , and F_2 . These three compounds were excluded because of analytical complexities. Blank analyses were made of the burner inlet air and of the burner effluent when CO was the sole influent contaminant.

ANALYTICAL TECHNIQUES

Halon 1211 was analyzed with a GC-2A gas chromatograph coupled with a Hewlett-Packard integrator and computer which converted the resulting peak areas to ppm concentrations of Halon 1211. A 12-ft by 1/4-in. column of 15% PEG 400 on Gas-chrom RZ at 70°C was used with a hydrogen-flame ionization detector. The 1/8-in. stainless steel sample lines from the burner were connected directly to the chromatograph sample loop.

Carbon monoxide was also analyzed with a GC-2A gas chromatograph, but in this instance the CO was converted to methane (CH_4) to permit its detection by the highly sensitive hydrogen-flame ionization detector. The sample to be analyzed was first passed through a chromatographic separation column containing equal weights of Poropak Q and T to separate CO_2 which would interfere with the analysis. The CO_2 was removed by backflushing and the sample then passed through a column of type 5A molecular sieve which separated the CO from any CH_4 which might have been in the air. Both the Poropak and molecular sieve columns were maintained at 65°C. From the molecular sieve column, the sample passed with a stream of hydrogen to a reactor (a bed of 10% Ni on Chromosorb W at 300°C) which converted the CO to CH_4 . Next, the sample passed to the hydrogen-flame detector where the original CH_4 and the converted CH_4 were detected as separate peaks. Readout was by integrator and computer as for the Halon 1211. Samples analyzed for CO were transferred from the burner to the gas chromatograph by means of a glass syringe.

Analyses of the burner exhaust gas for HCl and HBr were made with the NRL strong-acid test paper (9). This paper was calibrated to detect strong-acid vapors at concentrations ranging from 0.2 to 40 ppm. It is unresponsive to vapors of weak acids.

Dräger detector tube CH 303 was used to analyze the burner effluent for HF. It was calibrated to detect 1–15 ppm HF, but this range could be reduced to 0.5–7.5 ppm by proportionately increasing the volume of gas passing through the tube. The reaction of this tube is specific for HF.

The burner exhaust gas was analyzed for Cl₂ and Br₂ with Dräger detector tube CH 243. This tube was calibrated to detect concentrations of 0.2–3 ppm, but this range could be extended to approximately 30 ppm by passing a proportionately smaller volume of gas through the tube. In addition to Cl₂ and Br₂, the tube is also sensitive to nitrous fumes and chlorine dioxide.

Dräger detector tube CH 283, calibrated to detect concentrations of 0.25–15 ppm, was used to analyze the burner effluent for COCl₂. This detector tube is specifically sensitive to COCl₂.

Catalyst

The hopcalite catalyst was manufactured by the Mine Safety Appliances Corporation and was purchased through Navy supply channels on the military specification, Mil-C-21665 (Ships), 12 December 1958, amended 11 May 1959. It is a coprecipitate of copper and manganese oxides which has been analyzed as 78.3 wt-% MnO₂ and 13.1 wt-% CuO with a 7.9 wt-% ignition loss (presumably water or chemisorbed gases). The catalyst is about 6-mesh size and has an apparent density of 1 g/cm³.

Contaminants

The carbon monoxide used was purchased from Air Products and Chemicals, Inc. It was C.P. Grade, 99.3% purity.

No specification for the Halon 1211 used was available, but the chromatographic analyses indicated that it was of high purity. A chromatogram is shown in Fig. 2, and it can be seen that no impurities in the Halon 1211 were detected by the chromatograph. Further, no decomposition products were detected when the burner influent was contaminated with Halon 1211 and tested with the same detector tubes and detector paper used to analyze the burner effluent.

Results

The results of the experiments in which CBrClF₂ and CO were exposed to hopcalite catalyst at 600°F are summarized in Table 1. The data presented are averages of the measurements made and show the degree to which each compound was decomposed, both when exposed to hopcalite alone and with the other contaminant also present. The average

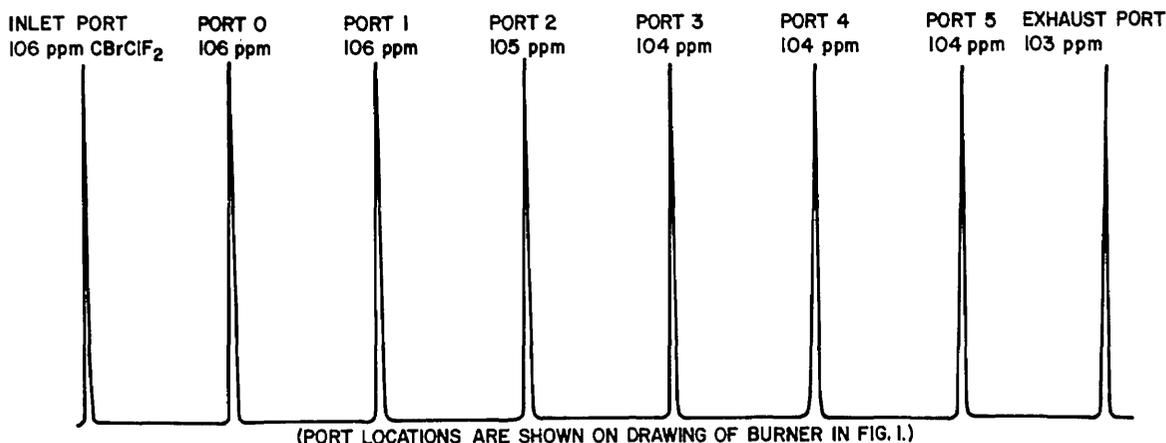


Fig. 2 — Chromatogram of CBrClF_2 analyses at different sampling ports of the bench-scale catalytic burner. Port locations are shown on Fig. 1.

of the chromatographic analyses shows that about 2% of the influent CBrClF_2 , when exposed as a single contaminant, was decomposed by the full catalyst bed. The simultaneous presence of CO did not measurably affect this degree of decomposition. The limit of reproducibility of the chromatograph is about 3%, so the fact that the average of all the measurements showed a 2% loss of CBrClF_2 in the burner indicates that there was probably a small amount (5% or less) of decomposition. A typical chromatogram of the CBrClF_2 analyses is shown in Fig. 2. This chromatogram, with successive samples having been taken at 3-min intervals, shows the concentration of CBrClF_2 at each sampling port.

The occurrence of a small amount of decomposition was confirmed by the detection of low concentrations of CBrClF_2 breakdown products in the burner effluent by means of detector tubes and acid detector paper. As may be seen in Table 1, the decomposition products detected were HCl plus HBr , Cl_2 plus Br_2 , and HF . Also, a trace (<0.1 ppm) of phosgene was detected in one instance.

When CO was exposed to hopcalite at 600°F as a single contaminant, it was completely oxidized within 20 to 40% of the 5-in. catalyst bed depth. At 20% of the bed depth, 3 to 6% of the inlet CO remained unoxidized while at 40% of the bed depth no CO was detected. The simultaneous presence of CBrClF_2 had only a slight effect on this degree of oxidation.

The action of hopcalite in decomposing CBrClF_2 and CO can be described in a more informative manner than that of Table 1. As discussed in a previous report (10), the conditions existing in the bench-scale reactor (and also in the shipboard reactor) including the high temperature, turbulent gas flow, and the large excess of oxygen make it reasonable to assume that the reaction in the catalyst bed is of first order. The following expression describes a first order reaction:

$$-\ln C_z = KZ, \quad (1)$$

Table 1
Catalytic Decomposition of CBrClF₂ and CO over Hopcalite at 600° F

Contaminants in Burner Influent			Decomposition Efficiency			Concentration of Decomposition Products in Burner Effluent* (ppm)			
Compound	Concentration (ppm)	Accompanying Compound	Concentration (ppm)	Percent Decomposed	Percent Bed Depth	HCl+HBr	HF	Cl ₂ +Br ₂	COCl ₂
CO	75-130	None	-	100	20-40	-	-	-	-
CBrClF ₂	100-110	None	-	<5†	100	5	3-8	10-12	N.D.‡
CO	110-120	CBrClF ₂	100-110	100	20-40	-	-	-	-
CBrClF ₂	100-110	CO	110-120	<5	100	2-3	10-15	10-15	Trace¶

*Values were obtained from Dräger tube and acid paper tests and are approximate.
 †Average of analyses was 2% loss of CBrClF₂ in burner. Reproducibility of measurements was about ±3%.
 ‡Not detected. Dräger tube was calibrated from 0.25 to 15 ppm and can detect <0.1 ppm.
 ¶One measurement detected <0.1 ppm. No COCl₂ was detected in other measurements.

where C_z is the concentration of reactant, as a fraction of the input concentration, remaining at bed depth Z . Bed depth Z is expressed as a fraction of the full bed depth, and K , is the effective reaction rate constant. The rate of decomposition in the reactor may be only partly determined by the chemical activity with heat and mass-transport effects also being significant. However, under the fixed operating conditions used in this work, it appears to be sufficient and practical to combine these variables into the one effective activity denoted as K in Eq. (1).

Equation (1) is plotted in terms of $1-C_z$ (the fraction of input reactant removed by the catalyst) vs bed depth Z for three different values of K in Fig. 3. The values of K chosen for these curves were the averages of the experimental values of K determined for CO and CBrClF_2 when they were exposed to hopcalite both singly and simultaneously. The ranges of measured concentrations of the contaminants used to calculate these experimental values of K are marked on Fig. 3, so that they may be compared with the curves to show the agreement of the experimental data with the theoretical equation. The curve drawn at a K value of 17.5 shows the activity of hopcalite for CO as a single contaminant, and that drawn at a K value of 14.6 shows the activity of hopcalite for CO in the presence of CBrClF_2 . The difference between the two curves is slight. The other curve at an average K value of 0.02 represents the activity of hopcalite for CBrClF_2 both as a single contaminant and in the presence of CO. In this instance, the data were grouped so closely that a single range is indicated for the experimental points and a single curve is drawn to represent both cases.

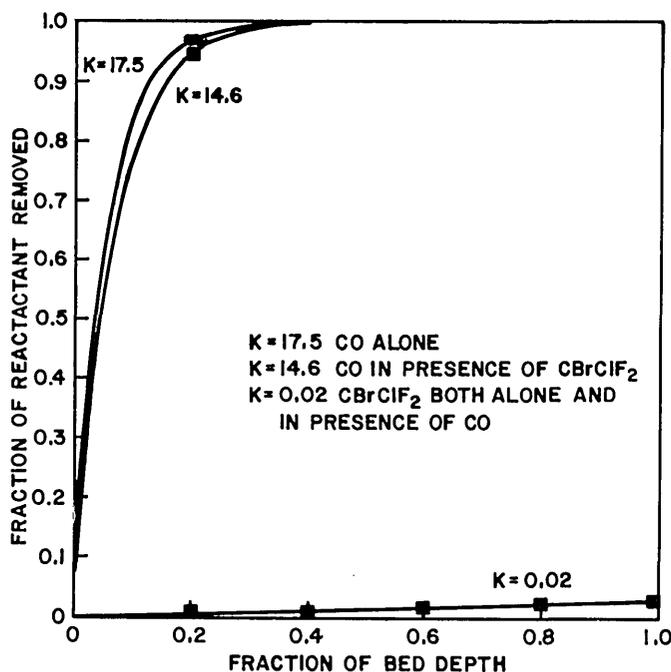


Fig. 3 — Catalytic decomposition of CBrClF_2 and CO over hopcalite at 600°F as a function of hopcalite bed depth. The range of the experimental points is shown by the length of the bars.

DISCUSSION

For a compound which only slightly decomposes, detector tubes and detector paper detect small concentrations of decomposition products directly and often disclose a slight degree of decomposition of a compound in the burner which is not shown by the chromatograph. The gas chromatograph measures the amount of decomposition as a small difference between the relatively large inlet and exhaust concentrations. Although the detector tubes and detector paper are very sensitive, they do not provide quantitative measurements which are reproducible within a small percentage of their range as does the chromatograph. They are valuable as a supplement to the gas chromatograph for qualitatively proving the presence or absence of low concentrations above their threshold limit.

It is typical that the amount of a compound decomposed in the CO/H₂ burner as determined by the gas chromatograph does not quantitatively agree with the amount of decomposition products detected in the burner effluent as measured by detector tubes and paper. One reason for this is that some of the reaction products are retained by the catalyst. In the common case, the concentrations of the breakdown products detected in the burner exhaust gas account for only part of the compound decomposed in the burner. The results of the CBrClF₂ measurements are different in that the decomposition products detected in the burner effluent fully accounted for the halogens in the decomposed CBrClF₂. On the basis of the maximum concentrations measured, the amount of breakdown products detected actually indicated a greater degree of oxidation (about 7%) than did the chromatographic measurements. This is not considered to be significant in view of the lesser precision obtainable from detector tube measurements. The gas chromatographic measurements of less than 5% decomposition of the influent CBrClF₂ are believed to be accurate.

SUMMARY AND CONCLUSIONS

The action of hopcalite catalyst at 600°F on bromochlorodifluoromethane (CBrClF₂) has been studied using the bench-scale burner which simulates the CO/H₂ burner of nuclear submarines. Concentrations of about 100 ppm each of CBrClF₂ and carbon monoxide (CO) were exposed to hopcalite both as single and as simultaneous contaminants. The following observations and conclusions were made.

1. The hopcalite catalyst decomposed less than 5% of the influent CBrClF₂ at 600°F.
2. Chlorine (Cl₂) and/or bromine (Br₂), hydrogen chloride (HCl) and/or hydrogen bromide (HBr), and hydrogen fluoride (HF) were detected in the burner effluent to confirm that a slight decomposition of CBrClF₂ did occur.
3. A trace (<0.1 ppm) of phosgene (COCl₂) was detected in one instance as a result of the CBrClF₂ decomposition.
4. The activity of hopcalite for the decomposition of CBrClF₂ was not affected by the presence of CO.
5. The hopcalite catalyst decomposed all of the influent CO within the first 40% of the 5-in. catalyst bed depth.
6. The activity of hopcalite for the decomposition of CO was only slightly affected by the presence of CBrClF₂.

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Carbon monoxide Catalytic combustion Halon 1211 Fluorocarbon 12B1 Bromochlorodifluoromethane Hopcalite Fire extinguishant Air purification						