

TECHNICAL NOTE**CRIMINALISTICS**

Katherine Hutches,¹ Ph.D. and James Lord,² M.S.

A New Kind of Molotov? Gasoline–Pool Chlorinator Mixtures*

ABSTRACT: This paper investigates the reaction between pool chlorinators and gasoline. In particular, the propensity for self-ignition and the resulting chemical products were studied. An organic pool chlorinator was combined with gasoline in varying proportions in an attempt to form a hypergolic mixture. None of the combinations resulted in self-ignition, but larger quantities of chlorinator produced vigorous light-colored smoke and a solid mass containing isocyanuric acid and copper chloride. Additionally, the chlorinating abilities of different commercially available pool chlorinators were explored. When Ca(ClO)₂ and sodium dichloro-s-triazinetrione-based chlorinators were used, the presence of gasoline was still visible after 10 days, despite limited chlorination. The trichloro-s-triazinetrione-based chlorinator, however, caused efficient chlorination of the C₂- and C₃-alkylbenzenes, making gasoline no longer identifiable.

KEYWORDS: fire debris, forensic science, gas chromatography–mass spectrometry, hypergolic mixture, ignitable liquid, pool chlorinator

It is widely known that certain mixtures of chemical reagents will self-ignite. For example, when potassium chlorate, sugar, and sulfuric acid are combined, the mixture will ignite nearby fuels such as gasoline (1). Another self-igniting mixture, also known as a hypergolic mixture, is the combination of inorganic pool chlorinator (generally calcium hypochlorite-based products) with brake fluid (2). Other reactive mixtures are used to create overpressure devices. It has been recently reported that the addition of organic pool tablets containing trichloro-s-triazinetrione, also known as trichloro or trichloroisocyanuric acid (TCCA), to isopropanol results in the production of toxic gases, but not actual ignition (3).

A new “self-igniting” mixture has been referred to by at least two online sources (<http://www.freewebs.com/lkjhgfdsafruit/anar666.htm> [accessed July 19, 2010], <http://www.skepticalfiles.org/new/034doc.htm> [accessed July 19, 2010]). According to the instructions, gasoline is added to a glass bottle and crushed pool tablets are placed into a cloth that is then carefully inserted into the top of the bottle. When the bottle is thrown at the intended victim, the glass bottle allegedly explodes (<http://www.bombshock.com/forum/explosives-pyrotechnics/9787-chlorine-fuel-tank.html> [accessed July 19, 2010]). Another online forum stated that an “instant fireball explosion” resulted from the combination of these two reagents (<http://www.tfproject.org/tfp/tilted-knowledge-how/3916-dry-ice-bombs.html> [accessed July 19, 2010]).

The work presented here was initiated as the result of a device submitted to the laboratory. This device consisted of a glass bottle, burned cloth, and a white substance. Laboratory analysis determined that the white substance, which had since turned brown, contained

calcium and chlorine, but the specific chemical composition could not be determined. The burned cloth and the unknown substance were each extracted using passive headspace concentration and the extracts analyzed by gas chromatography–mass spectrometry (GC–MS). Gasoline was identified in the sample with the unknown substance, but no ignitable liquid could be identified on the cloth. The total ion chromatogram (TIC) for the cloth had a poorly resolved hump in the region of C₁₀–C₂₀, with a mirroring hump in the aromatic extracted ion profile (EIP) (Fig. 1). Mass spectral library searches of the peaks within the poorly resolved envelope included a broad array of chlorinated aromatic compounds. Although toluene and the C₂- and C₃-alkylbenzenes were present, they were not present in the expected ratios (Fig. 1B), and there were no recognizable patterns consistent with a petroleum product.

The research presented in this paper sought to determine (a) whether the combination of pool chlorinator and gasoline is an effective hypergolic mixture and (b) whether this combination would yield the wide range of chlorinated aromatics observed in the case. There is some literature suggesting that the chlorination of aromatic compounds by trichlor is plausible (4–6). In these studies, it is noted that the chlorination of aromatic systems is possible under polar and free-radical conditions. Chlorination of benzene by trichlor, in particular, is possible in the presence of anhydrous ferric chloride or 50% aqueous sulfuric acid. Neat gasoline does not provide any of these conditions and presents a new environment in which to study the chlorination of aromatics by trichlor.

Three types of pool chlorinator, one inorganic and two organic, are typically used by consumers and therefore were the focus of this study. These are shown in Table 1. The inorganic chlorinator is a calcium hypochlorite-based product. One of the organic chlorinators uses trichlor in combination with copper sulfate pentahydrate. The other uses sodium dichloro-s-triazinetrione, or dichlor. The structures for trichlor and dichlor are shown in Fig. 2. Initially, only the inorganic and trichlor-based chlorinators were investigated. The dichlor-based chlorinator was added during the long-term chlorinator portion of the study.

¹Bureau of Alcohol, Tobacco, Firearms and Explosives, Forensic Science Laboratory, 355 North Wiget Lane, Walnut Creek, CA 94598.

²Bureau of Alcohol, Tobacco, Firearms and Explosives, Fire Research Laboratory, 6000 Ammendale Road, Ammendale, MD 20705.

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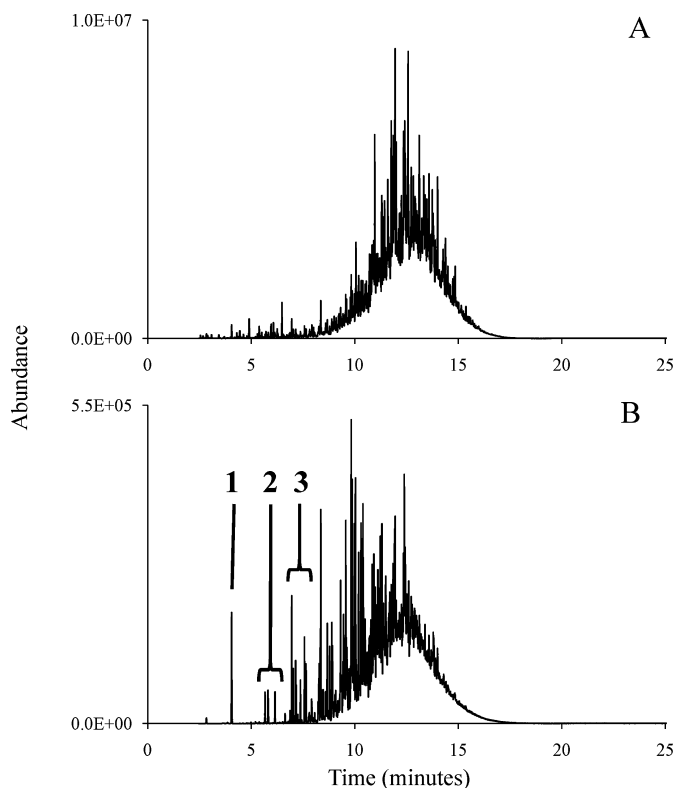


FIG. 1—Chromatogram of debris in the original case: (A) TIC and (B) corresponding aromatic EIP. (1) Toluene; (2) C_2 -alkylbenzenes; (3) C_3 -alkylbenzenes. NOTE: This sample was analyzed under a different temperature program from the test samples. The oven temperature was initially held at 40°C for 3 min, ramped at $15^\circ\text{C}/\text{min}$ to 300°C , where it was held for a final 5 min.

TABLE 1—Designations and ingredient listings for chlorinators used in this study.

Chlorine Source	Ingredients
hth pace [®] dual action 3" chlorinating tabs (Arch Chemicals, Norwalk, CT)	Trichloro-s-triazinetrione (trichlor) (93.5%) Copper sulfate pentahydrate (1.5%) Inert ingredients (5%)
Shock [®] Plus (Pool Time, Lawrenceville, GA)	Sodium dichloro-s-triazinetrione (58.2%) Other (41.8%)
hth Shock 'N Swim [®] (Arch Chemicals)	Calcium hypochlorite (47.6%) Other (52.4%)

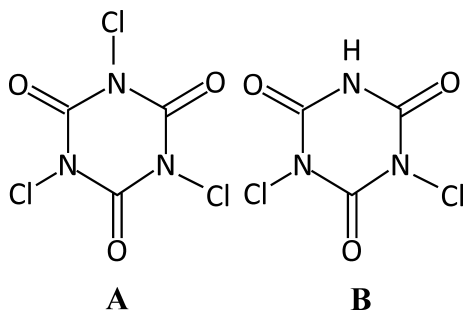


FIG. 2—Molecular structures of (A) trichlor and (B) dichlor.

During preliminary testing, it was noted that the inorganic chlorinator did not produce any visible signs of reaction while the trichlor-based chlorinator produced discernable heat when combined

with gasoline. As such, this was the main chlorinator of interest for the ignitability testing, although all three chlorinators were investigated in an attempt to recreate the chlorinated aromatic pattern observed in the case.

Materials and Methods

Chlorinators

Three chlorinators purchased at local hardware stores were investigated in this study and are listed in Table 1. In each of the experiments listed below, no cloth was used as it was believed by the authors that the cloth described in the online forums was likely used merely to prevent premature reaction and was not necessary for reaction.

Ignitability Testing—Small Scale

Increasing quantities of the shaved trichlor-based product were added to 20 mL of gasoline purchased from a local gas station. The quantities of this chlorinator were chosen for feasibility reasons. Each combination was tested once, with the exception of the 20 g/20 mL mixture, which was tested twice. Liquids were analyzed using GC–MS, and solids were analyzed using X-ray fluorescence (XRF) and X-ray diffraction (XRD). The temperature was monitored using an analog thermometer with an upper temperature limit of 250°C (Taylor USA, Oak Brook, IL).

Ignitability Testing—Large Scale

The large-scale ignitability testing was performed at the Fire Research Laboratory in Ammdendale, MD. A mass/volume ratio of 1 g chlorinator to 1 mL gasoline was found to be the most energetic during the small-scale testing, so this ratio was used during the large-scale testing. For safety reasons, the amounts of chlorinator were limited to 100 and 130 g of shaved trichlor-based chlorinator, which were combined with 100 and 130 mL of gasoline, respectively, inside 12-ounce clear glass beer bottles. In a third bottle, 100 g of the inorganic chlorinator was combined with 100 mL of gasoline. The temperature in each of these was monitored using a Type K glass-insulated thermocouple. The smoke produced was not analyzed.

Long-Term Chlorination

Approximately 10 g of each of the three chlorinators was combined with approximately 20 mL of gasoline in closed glass bottles. This amount of chlorinator was determined during the first phase of testing to be sufficient for visible reaction without causing the highly energetic reaction described later. These mixtures were allowed to sit at room temperature inside a fume hood for approximately 36 days. The mixtures were sampled and analyzed by GC–MS at 0.5, 1, 2, 3, 7, 10, 14, 21, 28, and 36 days after combination.

GC–MS Analysis

The GC–MS analyses were conducted using an Agilent 6890N gas chromatograph with 5973 mass spectrometer (Agilent Technologies, Santa Clara, CA). The column was a J&W DB-1MS dimethyl polysiloxane column, 30 m \times 250 μm \times 0.25 μm (Restek Chromatography Products, Bellefonte, PA). Sample volumes of 1.0 μL were injected with a split ratio of 30:1. Helium was used as a carrier gas. The mass spectrometer had a source temperature of

300°C and was set for a full scan over an m/z range of 33–300 amu in electron ionization mode. The oven temperature was initially held at 37°C for 2 min, ramped at 5°C/min to 120°C, and then ramped at 12°C/min to 280°C, where it was held for a final 4 min. Each sample was prepared by diluting 7 μ L of sample with approximately 1 mL of carbon disulfide (Fisher Scientific, Pittsburgh, PA). The presence of 2-chloro-1,4-dimethylbenzene and 2-chloro-1,3,5-trimethylbenzene was confirmed with standards (Sigma-Aldrich, St. Louis, MO).

XRF Analysis

The solid mass that remained after the reaction of trichlor product and gasoline was analyzed by XRF. This analysis was performed on an EDAX Eagle μ -Probe Spectrometer (PV9791/47 Model Supermax 160-5) with EDAM III Analyzer (PV9803/13), operated at 20 kV (EDAX Incorporated, Mahwah, NJ). All samples were ground and mounted on carbon stubs with carbon tape.

XRD Analysis

In addition to XRF, the ground solids were also analyzed by XRD. This analysis was performed on a Rigaku Miniflex X-ray diffractometer (Rigaku Americas, Woodlands, TX). The XRD was operated at 30 kV and 15 mA, scanning from 5 to 80° with a scan rate of 0.020°/sec.

Results and Discussion

Ignitability Testing—Small Scale

The first series of tests involved examining the reaction between small amounts of chlorinator and gasoline. During the first two trials, 0.9 and 3.4 g of the trichlor product were each added to 20 mL of gasoline. Only minor physical changes were noted. Within 30 min, each of the mixtures turned slightly green, but no chlorination was observed in the corresponding chromatograms. When the amount of chlorinator was increased to 11.2 g, more energetic physical effects were observed. Approximately 1 min after combination, the mixture began bubbling; at 2 min, the mixture turned opaque and orange, and the temperature increased to approximately 64°C. The mixture temperature peaked at 76°C after 3 min and then gradually decreased.

Thirty seconds after approximately 20 g of trichlor product was combined with gasoline, the mixture began bubbling vigorously. The temperature of the mixture climbed above 250°C within 3.5 min and was accompanied by the production of a great deal of light-colored smoke. After completion of the reaction, a solid mass remained and no liquid was apparent in the beaker. XRD of the solid mass identified cyanuric acid, a precursor to trichlor, in all portions of the solid mass sampled, and copper chloride was identified in the bottom of the mass. When the 20:20 combination was repeated, a solid mass with similar composition was obtained.

It should be noted that while the greater amounts of chlorinator produced a large amount of smoke, none of the combinations resulted in spontaneous ignition, much less the “instant fireball” suggested by one of the various online forums. It was thought that scaling-up the size of the mixture might increase the overall thermal output and give the desired reaction. Additionally, it was noted that the original online forums suggested confining the mixture to a bottle, the shape of which would potentially impede dissipation of the heated gaseous products. For this reason, large-scale ignitability testing was conducted.

Ignitability Testing—Large Scale

In an effort to determine the effects of larger quantities, the amounts of gasoline and chlorinator used in this phase of testing were increased from the 20:20 combination, and the mixtures were tested in open glass bottles. The first mixture of trichlor product and gasoline (100 g and 100 mL, respectively) did not initially react as expected. The chlorinator was added to the gasoline through a funnel, so the entire amount of chlorinator did not contact the gasoline at the same time, as with the small-scale testing. After combination, the mixture rapidly heated to 71°C over 1.5 min. For the next 10 min, the temperature only increased by approximately 4°C, and none of the bubbling, smoke production, or other energetic responses from previous tests were observed. This delay may have been due to inefficient mixing; thus, the bottle was agitated and shortly afterward the temperature rapidly increased to 330°C. This mixture finally produced a vigorous jet of white smoke, and a solid mass was left in the bottle.

Because of the initial difficulty in delivering the chlorinator to the gasoline in the first large-scale test, the remaining two tests were performed with the gasoline added through a funnel to the chlorinator. The second mixture of trichlor product and gasoline (130 g and 130 mL, respectively) reacted immediately on combination, reaching a peak temperature of 359°C within approximately 5.5 min. While the speed of the reaction was faster than the previous reaction, the rapid spike in temperature and the maximum temperature reached were similar. Similar to the previous tests, copious amounts of white vigorous smoke and a solid mass were produced.

The third mixture tested contained 100 g of the inorganic chlorinator and 100 mL of gasoline. This mixture exhibited markedly different behavior from the other two large-scale tests. The temperature slowly rose to a maximum of approximately 100°C over 25 min at a slow, steady rate. This is very different from both mixtures of the trichlor-based chlorinator and gasoline, which increased rapidly to 240°C over 1.7 min and 279°C over 1.2 min, respectively. The inorganic mixture eventually produced white smoke 16 min after combining the two ingredients.

Long-Term Chlorination

Figure 3 shows the chromatograms resulting from the combination of the inorganic chlorinator and gasoline as well as a chromatogram of neat (un-evaporated) gasoline for comparison. The inorganic chlorinator initially showed potential for chlorination of the aromatic components in gasoline, but this potential did not develop in any substantial manner. Within 1 day, the chlorination of some of the C₃-alkylbenzenes became evident in the TIC and in the aromatic EIP (Fig. 3B). The peak corresponding to 1,3,5-trimethylbenzene was no longer visible, and small peaks attributable to singly chlorinated derivatives of 1,3,5-trimethylbenzene, such as 2-chloro-1,3,5-trimethylbenzene, appeared. Peak ratios for the C₄-alkylbenzenes and polynuclear aromatics showed slight differences, but no change was observed in the alkane and indane EIPs. Only minor changes were observed beyond the first day of reaction. Despite the loss of one of the peaks in the C₃-alkylbenzenes, the overall pattern in Fig. 3B is readily identifiable as gasoline.

Figure 4 depicts the chromatograms from the mixture of the dichlor product and gasoline. This mixture required more time for chlorination to become apparent in the TIC than the previous mixture, but more extensive chlorination was observed with this chlorinator than with the inorganic chlorinator. Three days after the chlorinator was combined with the gasoline, a chromatogram similar to that observed for the inorganic chlorinator after 1 day of

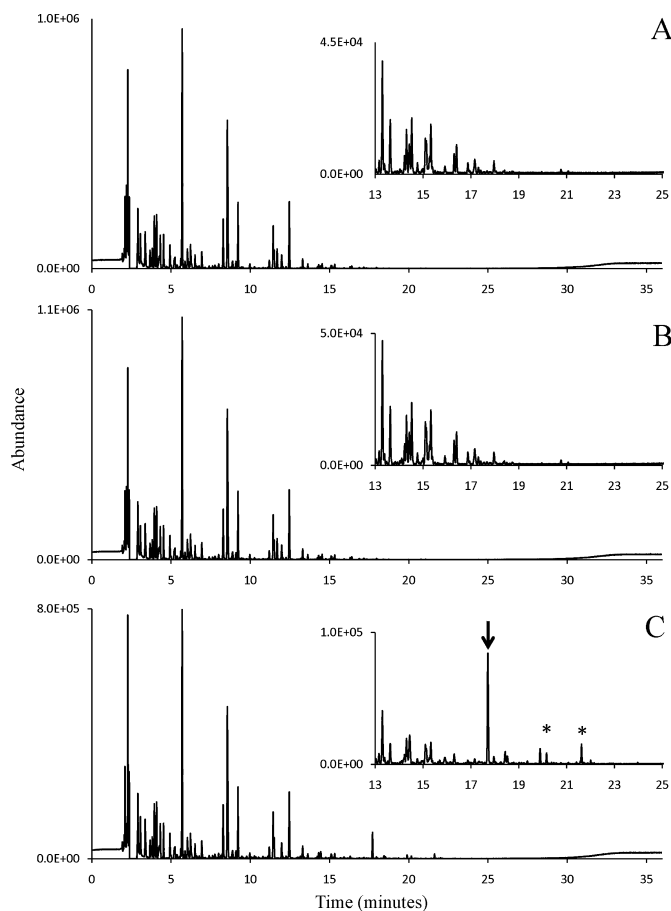


FIG. 3—Chromatograms showing the reaction of inorganic chlorinator with gasoline. (A) Neat gasoline; (B) 30 min; and (C) 1 day after mixing. Inset shows enhanced view of region of interest. * indicates chlorinated alkylbenzenes. The arrow indicates the location of 2-chloro-1,3,5-trimethylbenzene.

reaction was observed (Fig. 4B). After 7 days of reaction, another low abundance peak corresponding to the single chlorination of the C_2 -alkylbenzenes became apparent, as did another cluster of chlorinated C_3 -alkylbenzenes (Fig. 4C). Mirroring the increasing abundance of these peaks over the next 21 days was a relative decrease in the presence of the C_3 -alkylbenzenes, especially 1,2,4-trimethylbenzene. After 28 days, only minor changes were further observed. As with the inorganic chlorinator, no changes were apparent in the alkane and indane EIPs. Despite the change in ratios between the C_3 -alkylbenzenes and additional chlorinated components, each of the chromatograms in Fig. 4 was similar to gasoline. It is important to note that the shifting ratios within the C_3 -alkylbenzene group, a major diagnostic group for the identification of gasoline, might preclude identification of this pattern as gasoline.

When the trichlor product was combined with the gasoline, the temperature of the mixture increased noticeably. The chromatogram of a sample taken after approximately 30 min of reaction showed a pattern similar to that of Chlorinator I after 1 day (Fig. 5A). After approximately 1 day of reaction, the mixture was a bright yellow color. Visible differences were also evident in the chromatogram (Fig. 5B). The C_3 -alkylbenzenes were severely diminished with noticeable changes in abundance within the peak cluster. The C_2 -alkylbenzenes, while present, were also skewed, especially the peak corresponding to the co-eluting *m*- and *p*-xylenes. The C_4 -alkylbenzenes were no longer visible. As expected, peaks corresponding

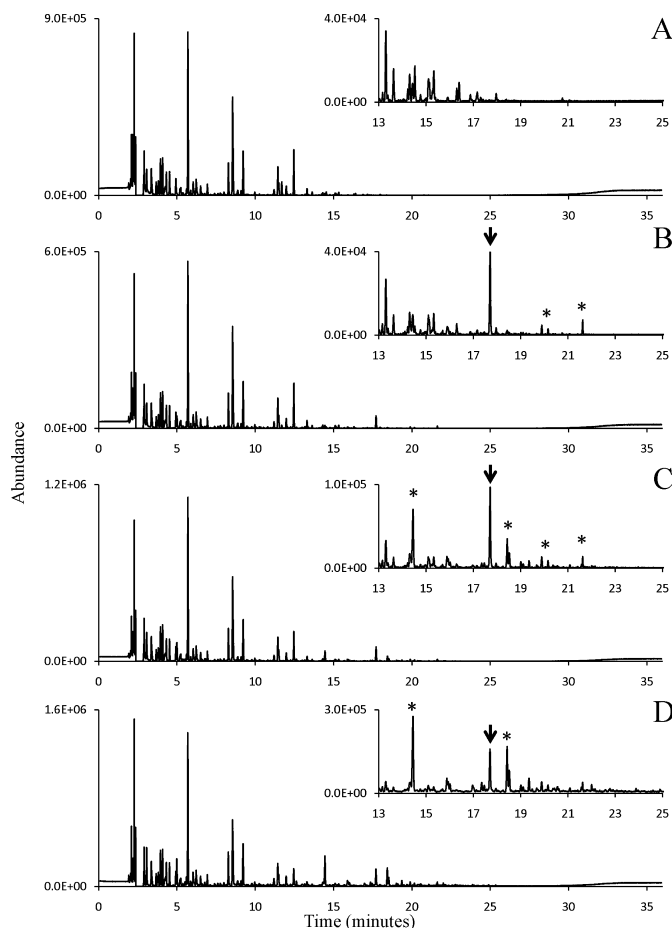


FIG. 4—TICs showing the reaction of dichlor-based chlorinator with gasoline, after (A) 30 min, (B) 3 days, (C) 7 days, and (D) 28 days. Insets show enhanced view of region of interest. * indicates region of chlorinated alkylbenzenes. The arrow indicates the location of 2-chloro-1,3,5-trimethylbenzene.

to chlorinated C_2 -, C_3 -, and doubly chlorinated C_4 -alkylbenzenes were observed. The relative abundance of these peaks was particularly striking when compared with the chlorinated aromatics seen with the other two chlorinators. At this point, the pattern in Fig. 5B is not identifiable as gasoline because of the severe skewing of the C_2 - and C_3 -alkylbenzenes. The degradation of the C_2 -alkylbenzenes continued for approximately 10 days after combination, after which only minor changes were observed (Fig. 5C). By Day 28, the mixture had turned an orange color, which deepened to dark amber with pockets of dark purple by Day 39. As with the other mixtures, no changes were observed in the alkane or indane EIPs.

When no changes were observed in any of the mixtures after 49 days of reaction, a sample of each of the mixtures was weathered to approximately 90% under a stream of nitrogen gas to observe the effect on the observed patterns (Fig. 6). The C_3 -alkylbenzenes are mostly visible in Fig. 6A, which corresponds to the inorganic chlorinator. There are a number of chlorinated aromatics present in Fig. 6A within the dashed box, but the strong presence of the nonchlorinated aromatics demonstrates the relative inefficiency of this chlorinator when compared with the organic chlorinators. The chromatogram of the weathered dichlor mixture (Fig. 6B) had a broad range and a low, poorly resolved envelope of chlorinated aromatic compounds, as well as a few stronger peaks, also corresponding to chlorinated aromatic species. The nonchlorinated aromatics are not prominent within this sample, and the

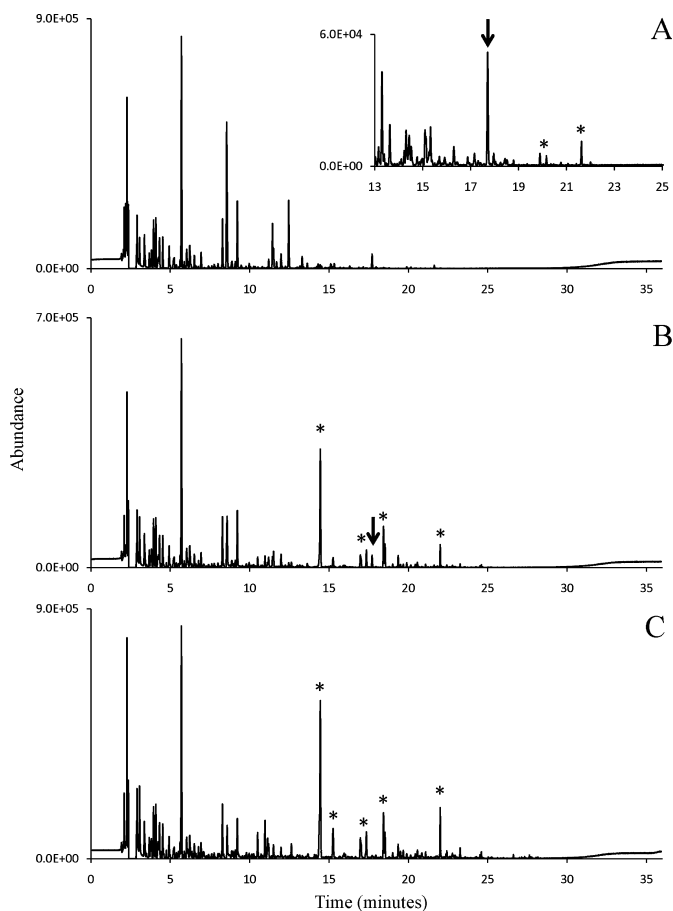


FIG. 5—Chromatograms from reaction of trichlor-based chlorinator with gasoline, after (A) 30 min, (B) 1 day, and (C) 10 days. Inset shows enhanced view of region of interest. * indicates region of chlorinated alkylbenzenes. The arrow indicates the location of 2-chloro-1,3,5-trimethylbenzene.

distribution of chlorinated products between 15 and 25 min is slightly different than seen in Fig. 6A. The chromatogram from the trichlor-based mixture (Fig. 6C) also has a low unresolved envelope, but the TIC is dominated by one large peak and a few smaller peaks. The mass spectrum for the largest peak in Fig. 6C is depicted in the inset. No identity was determined for this peak because of poor comparisons with compounds in the reference library, but the progression of peaks near m/z 188 suggests that this may be a doubly chlorinated C_3 -alkylbenzene.

The chromatograms of the various mixtures were compared. Common components were indicated in the library searches, but the distribution of compounds was quite different. This suggests that while the use of different chlorinators in such a mixture may result in the production of similar products, they may result in dissimilar distributions of said products. It was observed that the spectra of these compounds often contained peaks at m/z 139, 140, 153, 154, and/or 188, suggesting that these masses could be used for extracted ion profiling of chlorinated alkylbenzenes. When the data from the test mixtures are compared with the original case sample, it is apparent that the distribution of compounds is very different, despite the presence of common indicated components. The original case sample consisted mainly of a poorly resolved envelope of compounds instead of the discrete compounds that can be seen in Fig. 6. It is not clear what the cause of this difference is, but it may be possible that proprietary ingredients might affect

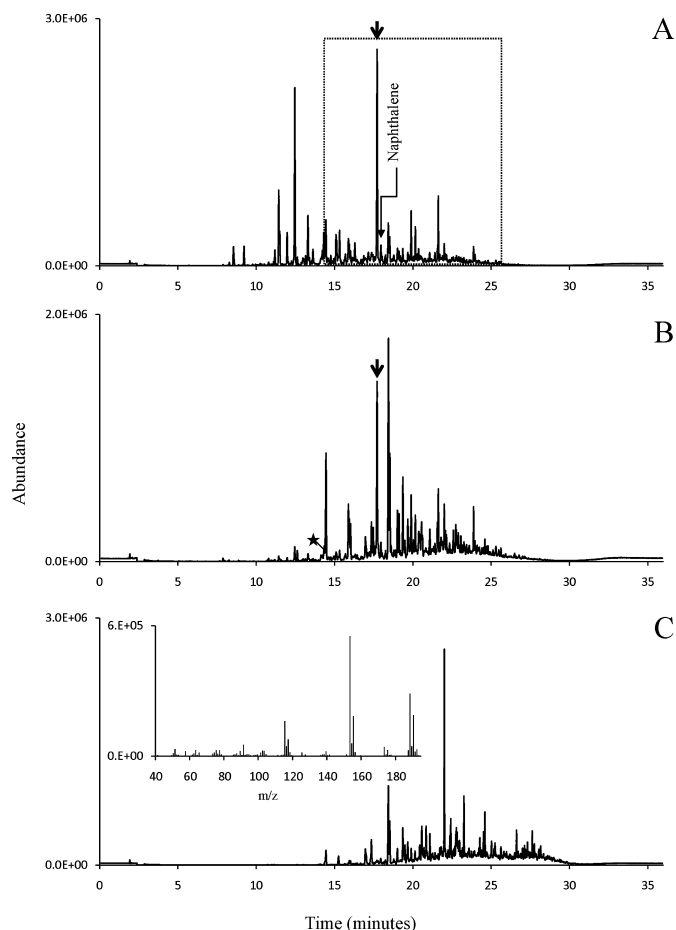


FIG. 6—Chromatograms from 90% weathering of mixtures after 49 days of reaction. (A) Inorganic chlorinator; (B) dichlor-based chlorinator; (C) trichlor-based chlorinator. Area within dashed box in (A) contains mostly chlorinated aromatic species. All major peaks in (B) and (C) correspond to chlorinated aromatics. Inset shows mass spectrum for major peak within (C). The arrow indicates the location of 2-chloro-1,3,5-trimethylbenzene, and the star indicates the location of 2-chloro-1,4-dimethylbenzene.

the chlorinating ability of each individual chlorinator. A number of environmental factors may also contribute, such as exposure to light and moisture.

The strong response of the substituted aromatics to the chlorination may be related to the strong activating effect of alkyl substituents for *ortho*- and *para*-substitution (7). This may particularly explain the rapid degradation of the 1,3,5-trimethylbenzene peak, as seen in Figs 3–5, relative to the other C_3 -alkylbenzenes. In this structure, the positions available for chlorination are *ortho*- to two alkyl groups and *para*- to a third alkyl group. Each alkyl group strongly directs the location of the chlorination and also increases the rate of reaction relative to an unsubstituted ring, provided there is not steric hindrance. While indanes also contain an aromatic ring, the aliphatic ring activates all four aromatic positions for chlorination. The relative abundance of the alkanes and indanes compared with the aromatics, combined with the activating effects of their alkyl substituents, may explain the relative inertness of these two groups to each of the chlorinators.

When comparing the reactivities of the different chlorinators, it is important to consider not only the chemical structures but also the relative amounts of active ingredient. Using the weight percentages listed in Table 1 as a guide, the 10 g of each chlorinator used in the long-term portion of this study contained approximately

0.040 mol of trichlor, 0.029 mol of dichlor, and 0.033 mol of calcium hypochlorite. This, combined with the additional Cl-group on trichlor compared with dichlor, may partially explain the difference in reactivity between the trichlor and dichlor. Future research might include the analysis of equimolar mixtures to explore this possibility. Additional research might also include the use of fuels other than gasoline such as some specialty cleaning solvents, which contain mainly aromatic constituents, to see whether the reaction is affected by the presence of the nonaromatic constituents of gasoline.

It has been suggested that the calcium hypochlorite reacts energetically with brake fluid through the formation of a chlorine radical (Cl[•]) (2). While trichlor may also react through chlorine radical formation, in the presence of Lewis acids, it has been observed to react with aromatic species through charge-transfer complexes (4). As a Lewis acid (8), the presence of the copper sulfate in the organic pool chlorinators may act as a catalyst in the gasoline mixture, allowing reaction through a charge-transfer mechanism that would not otherwise be likely in nonpolar system. As such, the difference in reactivity between the trichlor-based chlorinator and the other two chlorinators may be attributable not only to the direct chlorinating agent, but also to the other ingredients present in the mixture.

Conclusion

During both the small- and large-scale ignitability testing, none of the gasoline-chlorinator mixtures achieved spontaneous ignition, as was reported by online sources. However, the temperature of the mixture increased rapidly and a large amount of vigorous smoke was produced. As such, this mixture is not likely to be used in a self-igniting Molotov, although it may have a potential use in a hoax device.

Also of interest is the formation of a solid mass when the trichlor-based chlorinator was combined with gasoline in sufficiently high proportions. If this mass was received with no background information for analysis, it is unlikely that the presence of cyanuric acid, copper, chlorine and sulfur would suggest the unique mixture from which it originated.

In each of the mixtures of the calcium hypochlorite and dichlor-based chlorinators with gasoline, the presence of gasoline was still indicated in the TIC after 10 days, despite the loss of a

single peak in the C₃-alkylbenzenes and diminished C₄-alkylbenzenes. When the trichlor-based chlorinator was mixed with gasoline, the presence of gasoline was no longer identifiable because of the extremely rapid and efficient chlorination of the C₂- and C₃-alkylbenzenes, which were affected within a day of the initial combination. Interestingly, the relative abundance of toluene seemed unaffected. Additionally, the indane and alkane contributions appeared likewise unaffected by the chlorination.

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Additional information and reprint requests:

Katherine Hutches, Ph.D.
Bureau of Alcohol, Tobacco, Firearms and Explosives
Forensic Science Laboratory – San Francisco
355 North Wiget Lane
Walnut Creek, CA 94598
E-mail: katherine.hutches@atf.gov