

PAPER

CRIMINALISTICS

Mark Fitzgerald,¹ Ph.D. and Daniel Bilusich,² Ph.D.**Sulfuric, Hydrochloric, and Nitric Acid-Catalyzed Triacetone Triperoxide (TATP) Reaction Mixtures: An Aging Study**

ABSTRACT: The organic peroxide explosive triacetone triperoxide (TATP) is regularly encountered by law enforcement agents in various stages of its production. This study utilizes solid-phase microextraction (SPME) and gas chromatography/mass spectrometry (GC/MS) to examine sulfuric acid-, hydrochloric acid-, and nitric acid-catalyzed TATP syntheses during the initial 24 h of these reactions at low temperatures (5–9°C). Additionally, aging of the reaction mixtures was examined at both low and ambient temperatures (19–21°C) for a further 9 days. For each experiment, TATP could be readily identified in the headspace above the reaction mixture 1 h subsequent to the combination of reagents; at 24 h, TATP and diacetone diperoxide (DADP) were prominent. TATP degraded more rapidly than DADP. Additionally, chlorinated acetones chloroacetone and 1,1-dichloroacetone were identified in the headspace above the hydrochloric acid-catalyzed TATP reaction mixture. These were not present when the catalyst was sulfuric acid or nitric acid.

KEYWORDS: forensic science, analytical chemistry, triacetone triperoxide, diacetone diperoxide, gas chromatography mass spectrometry, improvised explosive

Triacetone triperoxide (TATP), or 3,3,6,6,9,9-hexamethyl-1,4,7-cyclononatriperoxane (Fig. 1), is a poorly stable peroxide explosive that was first reported in 1895 by Wolfenstein (1). TATP readily decomposes to form the explosive homolog diacetone diperoxide (DADP, Fig. 1) and as such has never been seriously considered for mainstream military usage (2–4). Additionally, both peroxide explosives (TATP and DADP) are very sensitive to initiation because of heat, impact, and frictional stimuli (5). However, despite the hazards involved in producing TATP and incorporating it into an explosive device, it has become a popular explosive for backyard chemists and terrorists alike because of its ease of manufacture and the availability of the precursor compounds (6,7). TATP has been utilized in a number of criminal and terrorist activities and is regularly encountered by law enforcement authorities in clandestine laboratories in various stages of its production (8,9).

The acid-catalyzed degradation of TATP to DADP has been well documented. It has been shown that when either sulfuric, hydrochloric, or nitric acid is utilized in the synthesis, acid inclusion within the TATP crystals may occur (6,10). It has also been demonstrated that the susceptibility to degradation of isolated TATP (to DADP) depends on the choice of acid catalyst and the rigor of subsequent purification procedures (10). Furthermore, isolated, purified TATP in intimate contact with sulfuric acid, or in the presence of vapors of this acid, decomposed to form DADP and acetone (11).

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In the presence of hydrochloric acid, isolated TATP also degraded to form chlorinated acetones (11).

The aim of this study was to gain insight into the formation and chemical degradation of TATP within aged reaction mixtures. This initially involved three separate syntheses of TATP, which employed a different acid catalyst, i.e., sulfuric, hydrochloric, and nitric acids. Each of these master syntheses was then split into multiple sealed samples that were stored under controlled conditions and the headspace sampled at defined periods by solid-phase microextraction (SPME) and analyzed using gas chromatography/mass spectrometry (GC/MS). It was intended that these experiments would show after what duration subsequent to the combination of reagents, TATP and DADP may be detectable in the reaction headspace. It was also the intent of this study to identify by-products and/or decomposition products of the reactions and to examine longevity characteristics of aged TATP and DADP when allowed to remain in reaction mixture.

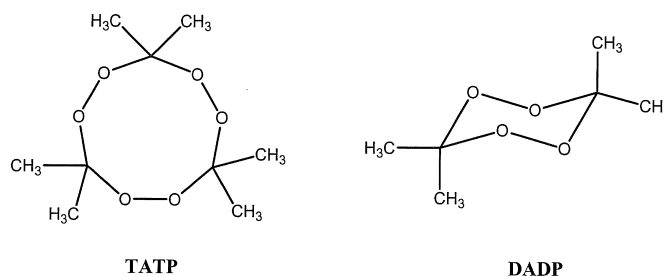


FIG. 1—Molecular structures of triacetone triperoxide and diacetone diperoxide.

Materials and Methods

The synthesis of the peroxide explosive detailed in this report was carried out in specialized facilities by chemists experienced in the production and handling of explosives. TATP is a very sensitive and powerful explosive and as such should only ever be handled by those properly equipped and qualified.

Reagents

TATP and DADP standards were synthesized following established procedures and the purity confirmed by differential scanning calorimetry and GC/MS (12,13); 37% w/v Spectroscopic grade hydrochloric acid (Merck, Kilsyth, Victoria, Australia), 98% w/v AR grade sulfuric acid (APS Group, Seven Hills, NSW, Australia), 70% w/v nitric acid (APS Group), AR acetone (Merck), 30% w/v AR grade hydrogen peroxide (Sigma-Aldrich, Castle Hill, NSW, Australia), >95% chloroacetone (Merck), and 1,1-dichloroacetone (Sigma-Aldrich) were used as supplied.

Synthesis and Reaction Mixture Sampling

Syntheses of TATP were adapted from literature procedures (10). Sulfuric acid (1.9 M, 15 mL) was added to a stirred, chilled (5°C) mixture of acetone (5 mL, 68.2 mmol) and 30% w/v hydrogen peroxide (1.5 mL, 13.2 mmol). Immediately after addition, and before TATP precipitation, the reaction mixture was divided into 15 0.5-mL portions in sealed, 20-mL crimp-top SPME sample vials and refrigerated (5–9°C). At 1, 3, 5, 7, and 24 h, an individual reaction vial was removed from the refrigerator and allowed to warm to room temperature (20 min). Its headspace was then absorbed onto a SPME fiber and immediately analyzed by GC/MS. Also, 24 h after the initial combination of reagents, five of the 10 remaining refrigerated samples were removed from the refrigerator, protected from light and aged at ambient room temperature (19–21°C). Then, after 2, 3, 4, 7, and 10 days (relative to the initial combination of reagents), an individual sample aged at 5–9°C and a sample aged at 19–21°C were examined by SPME GC/MS.

For the experiments detailed herein utilizing alternate mineral acids, the synthetic procedure was repeated using either hydrochloric acid (1.9 M, 15 mL) or nitric acid (1.9 M, 15 mL). These reaction mixtures were also divided, stored, and sampled as described for sulfuric acid. All experiments were repeated to confirm consistency of results.

Instrumentation

Solid-phase microextraction headspace sampling was performed using polydimethylsiloxane/carboxen/divinylbenzene (Supelco, Bellefonte, PA) fibers (coating thickness 50/30 µm). The crimp-top SPME vials (70 × 22 mm) were fitted with silicone/Teflon septa and extracted for a period of 30 sec. A CombiPAL Autosampler (Varian, Palo Alto, CA) was employed to ensure consistency of absorption and desorption times.

Mass spectrometric experiments were carried out using a Varian 4000 ion trap GC/MS instrument (Varian) maintaining a 500:1 sample split ratio and a injector temperature of 120°C. SPME desorption time was 5 min onto a Restek RTX[®]-5 SILMS, 15 m × 0.25 mm × 0.25 µm capillary GC column (Chromatolytic Technology, Boronia, Victoria, Australia). The carrier gas was helium (1 mL/min). For each GC run, the initial oven temperature was maintained at 50°C for 2 min, increased at 10°C/min to 90°C and

held for 3 min, and then increased at 20°C/min to 200°C and held for 2 min. Total run time was 16.5 min.

The mass spectrometer utilized an internal electron ionization source in positive ion mode with 70 eV electron energy and 10 µA current. The ion trap conditions were target TIC 20000, maximum ion time 2500 µsec, multiplier offset 300 V, and scan time 0.54 sec/scan. The transfer line to the mass spectrometer was heated to 170°C. The ion trap temperature was 150°C. The scan range was *m/z* 40–400 with a 0.35-min filament delay. The NIST mass spectral library (version 2d, April 26, 2005) was used for tentative compound identification, with the identity of each compound subsequently confirmed by comparison of spectra and retention times to authentic standards (14).

Results

Three syntheses of TATP were performed using different acid catalysts, i.e., sulfuric, hydrochloric, or nitric acid. Typically, TATP synthesized clandestinely is produced using one of these three acids. Additionally, the relative quantities and concentrations of reagents are consistent with reaction mixtures which may reasonably be expected to be encountered by law enforcement agents. Each of the three reaction mixtures was separated into 15, 0.5-mL portions in sealed containers, aged under controlled conditions, and analyzed once at chosen intervals using SPME and GC/MS (see Materials and Methods section for full details). Relative, rather than absolute, quantitation was employed to elucidate reaction characteristics (or trends) to facilitate more meaningful comparisons to reactions that adopt different synthetic procedures. The sampling timeframes were chosen to emulate the various states of TATP reaction mixtures that may be encountered by law enforcement, from the initial combination of chemicals through to aged and/or neglected syntheses. SPME was chosen as it has been shown that for TATP syntheses, the reaction headspace is representative of the volatile products in the reaction mixture (11).

The gas chromatograms of the headspace above the sulfuric acid-catalyzed TATP synthesis that was stored at 5–9°C and analyzed at 1, 3, 5, 7, and 24 h are shown in Fig. 2. Subsequent gas chromatograms of the reaction mixture aged at 5–9°C and at 19–21°C, which were then analyzed at 2, 3, 4, 7, and 10 days, are shown in Fig. 3 parts (A) and (B), respectively. One hour subsequent to the combination of reagents (see Fig. 2), both TATP and DADP are readily identifiable in the headspace above the reaction mixture. It is also apparent (Fig. 2) that the ratios of the areas under the peaks of TATP and DADP (*c.* 80% and 100% respectively) remain largely unchanged over the first 24 h of the experiment.

The gas chromatograms of the headspace above the sulfuric acid-catalyzed TATP synthesis, aged at 5–9°C and sampled at 2, 3, 4, 7, and 10 days (Fig. 3A), show little change in the relative abundances of TATP and DADP over the duration of the experiment. In contrast, the chromatogram of the reaction mixture aged at 19–21°C and measured at 10 days (including an initial 24 h at 5–9°C) displays a smaller relative peak area for TATP (5%) compared to DADP (100%) than what was apparent at 7 days (TATP; 59%, DADP; 100%) (Fig. 3B). The peak in the chromatogram owing to DADP remains prominent for the duration of the experiment.

Chromatograms of the headspace above the hydrochloric acid-catalyzed TATP synthesis that were measured during the first 24 h of the experiment are shown in Fig. 4. GC/MS analysis of the reaction headspace measured 1 h subsequent to the combination of reagents reveals a relatively major TATP peak (100%) compared to the DADP peak (3%), which is barely visible above the baseline; however, at 3 h, the DADP peak (8%) is more pronounced.

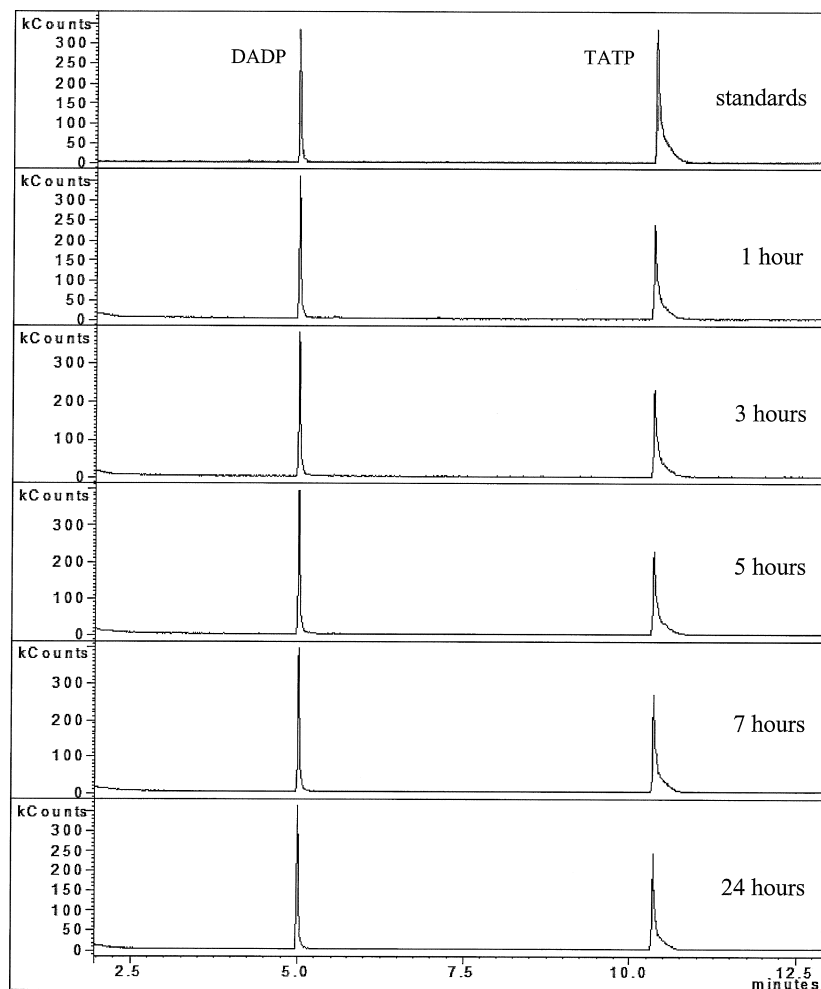


FIG. 2—The gas chromatography/mass spectrometry chromatograms of the headspace above the sulfuric acid-catalyzed triacetone triperoxide synthesis that was stored at 5–9°C, sampled using solid-phase microextraction and analyzed at 1, 3, 5, 7, and 24 h subsequent to the initial combination of reagents.

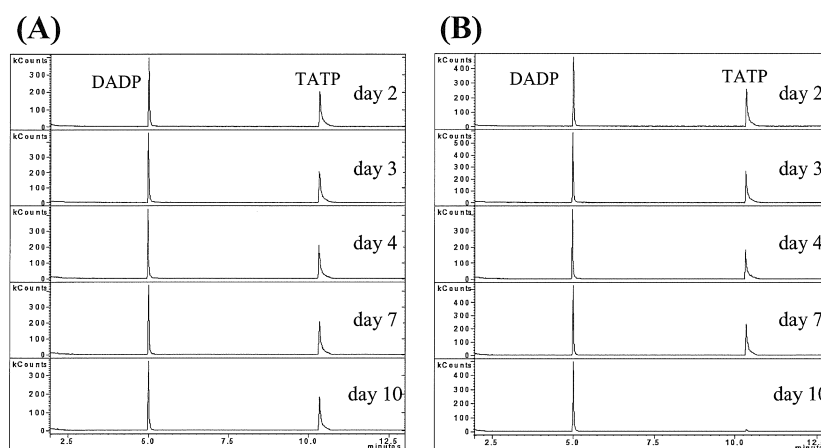


FIG. 3—The gas chromatography/mass spectrometry chromatograms of the headspace above the sulfuric acid-catalyzed triacetone triperoxide syntheses that were aged at 5–9°C (A) and 19–21°C (B) (after an initial reaction time of 24 h at 5–9°C), sampled using solid-phase microextraction and analyzed at 2, 3, 4, 7, and 10 days subsequent to the initial combination of reagents.

The DADP peak is again enlarged relative to the TATP peak in the trace measured after 5 h. The chromatogram recorded at 7 h continues the trend of the DADP (20%) peak increasing in size relative to TATP (100%); however, there is now also a minor peak at 2.7 min, which is attributed to chloroacetone. After the reaction has

proceeded for 24 h at 5–9°C, the base peak in the trace is TATP (100%), with DADP (54%) prominent, and a clearly visible peak owing to chloroacetone (1%).

The gas chromatograms of the headspace of the hydrochloric acid-catalyzed TATP synthesis, aged at 5–9°C and analyzed at 2,

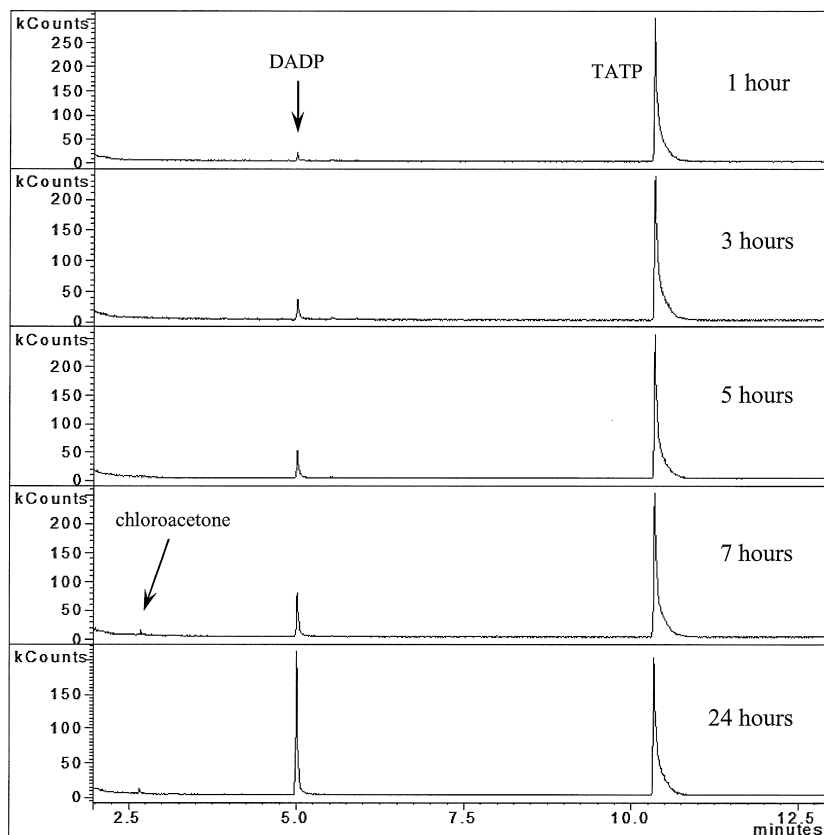


FIG. 4—The gas chromatography/mass spectrometry chromatograms of the headspace above the hydrochloric acid-catalyzed triacetone triperoxide synthesis that was stored at 5–9°C, sampled using solid-phase microextraction and analyzed at 1, 3, 5, 7, and 24 h subsequent to the initial combination of reagents.

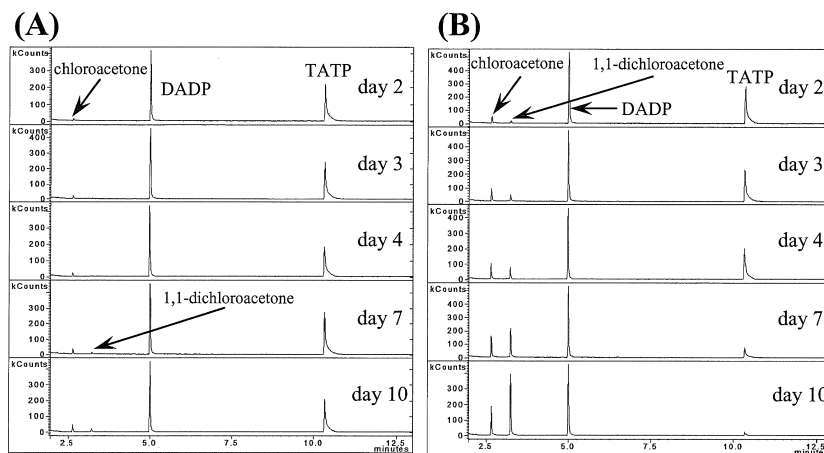


FIG. 5—The gas chromatography/mass spectrometry chromatograms of the headspace above the hydrochloric acid-catalyzed triacetone triperoxide syntheses that were aged at 5–9°C (A) and 19–21°C (B) (after an initial reaction time of 24 h at 5–9°C), sampled using solid-phase microextraction and analyzed at 2, 3, 4, 7, and 10 days subsequent to the initial combination of reagents.

3, 4, 7, and 10 days, are shown in Fig. 5A. Analogous experiments carried out on the reaction mixture aged at 19–21°C are included in Fig. 5B. For the chilled reaction mixture (Fig. 5A), the relative peak areas of TATP and DADP remain largely unchanged for the duration of the experiment. However, the chloroacetone peak does increase in line with the incremental chronology of the experiment, and at the 7-day mark, a second chlorinated acetone, 1,1-dichloroacetone, becomes visible above the baseline of the trace at 3.2 min. The peak owing to 1,1-dichloroacetone follows the trends of the other degradation products and is larger at 10 days.

Not surprisingly, the trends apparent in the chromatograms taken of the aged, chilled reaction mixture are more apparent in the samples stored at 19–21°C, conditions where reaction rates would be expected to be higher (Fig. 5B). At the elevated temperature, both chloroacetone (7%; compared to DADP [100%]) and 1,1-dichloroacetone (2%) are clearly visible in the chromatogram at the day 2 stage. The peaks owing to the chlorinated acetones increase relative to the other peaks in the traces as the experiment progresses. After the reaction mixture has aged for 10 days, the relative (compared to DADP) areas under the peaks of the chlorinated acetone

peaks are now *c.* 32% and 74% for chloroacetone and 1,1-dichloroacetone, respectively. It is also apparent that TATP has significantly degraded after 7 days in the reaction mixture, and after 10 days, only a minor peak owing to TATP is visible in the chromatogram.

The chromatograms of the headspace above the nitric acid-catalyzed synthesis of TATP at 5–9°C (first 24 h) are shown in Fig. 6. The data for the second phase to these experiments, where five 0.5-mL samples of the reaction mixture were allowed to remain at 5–9°C while another five were aged at 19–21°C, with analyses at 2, 3, 4, 7, and 10 days, are summarized in Table 1. One hour subsequent to the combination of reactants, TATP (100%) is present in the reaction headspace and the peak owing to DADP (4%) is minor (Fig. 6). During the first day of the reaction, DADP becomes more abundant in the headspace above the reaction mixture (relative to TATP), and at the 24 h mark of the experiment, the relative areas under the peaks of TATP and DADP are 100%, and 49%, respectively. After another 24 h at 5–9°C (Table 1, day 2), the relative areas of TATP and DADP are 77% and 100%, respectively, and these ratios remain largely unchanged for the remainder of the experiment. This is in contrast to the reaction mixtures aged at 19–21°C, which displays significant degradation of TATP at 7 days and only a minor peak owing to TATP at 10 days (Table 1, days 7 and 10).

Discussion and Conclusions

The simplicity and the ready availability of methods to synthesize TATP that may be found in the open literature, online, and

in clandestine explosives manuals, coupled with the ease of sourcing the precursor chemicals, have resulted in the peroxide explosive being utilized in numerous, recent, illegal activities. Consequently, law enforcement groups and other related agencies are encountering illegally manufactured TATP in various stages of its production. These stages may include the individual, separate precursor compounds; the combined reagents (all or possibly just some); early stages of the synthesis; the recently isolated product or a stored sample; or old, neglected syntheses still in reaction mixture form. Despite the variations in TATP procedures and the stages at which they may be encountered, little work has been carried out previously to examine the characteristics of the synthesis during these various stages. In this study, the different stages of sulfuric acid-, hydrochloric acid-, or nitric acid-catalyzed TATP syntheses were examined, from the initial combining of the reagents to 10-day-old syntheses. Under the chosen reaction and aging conditions, the timeframes for the appearance (and in some

TABLE 1—Relative percentages of TATP to DADP (100%) for nitric acid-catalyzed TATP syntheses (5–9°C and 19–21°C).

| Percentage Peak Area Owing to TATP Relative to DADP (100%) | Days Following Synthesis | | | | |
|--|--------------------------|----|----|----|----|
| | 2 | 3 | 4 | 7 | 10 |
| Stored temperature (°C) | | | | | |
| 5–9 | 77 | 77 | 72 | 81 | 85 |
| 19–21 | 68 | 59 | 59 | 19 | 5 |

DADP, diacetone diperoxide; TATP, triacetone triperoxide.

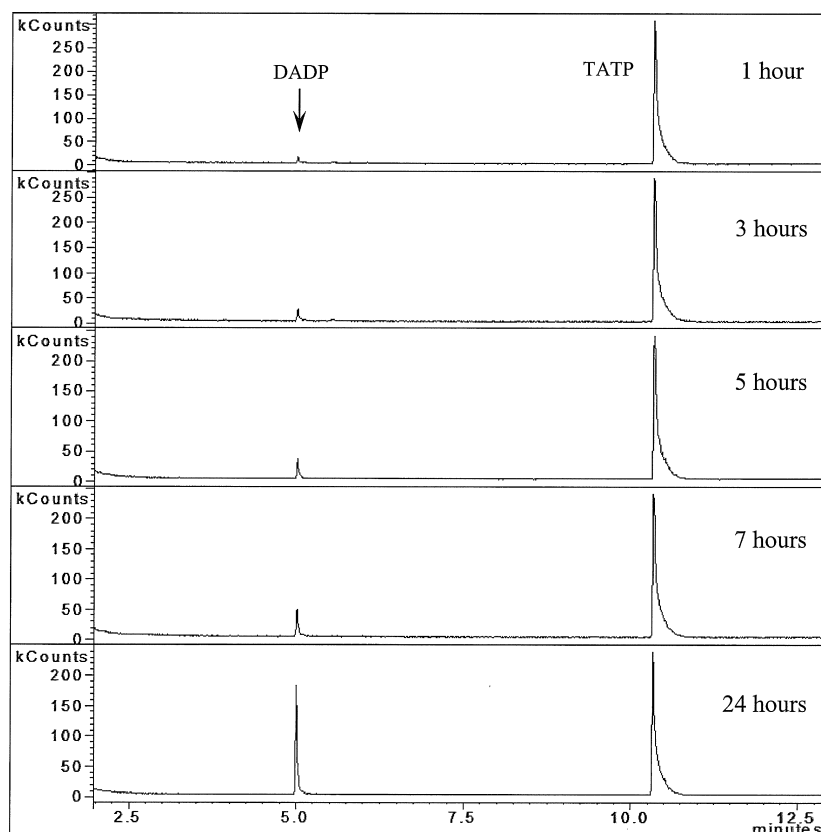


FIG. 6—The gas chromatography/mass spectrometry chromatograms of the headspace above the nitric acid-catalyzed triacetone triperoxide synthesis that was stored at 5–9°C, sampled using solid-phase microextraction and analyzed at 1, 3, 5, 7, and 24 h subsequent to the initial combination of reagents.

cases degradation) of TATP, and its analog DADP, were examined, as were the presence of any additional products of the reaction (or degradation), which may be characteristic of the type of acid used.

In each of the syntheses detailed herein, be it either the sulfuric, hydrochloric, or nitric acid catalyzed, TATP was able to be readily identified in the headspace above the reaction mixture using SPME and GC/MS 1 h subsequent to the combination of reagents. Typically, at this stage of the reactions, little TATP had precipitated from solution, i.e., the presence of TATP was able to be identified before an appreciable quantity of the explosive could be produced or isolated. Naturally, this has ramifications for the development of future TATP detection techniques. It was also noted that while DADP was prominent in the GC/MS trace measured 1 h subsequent to the addition of the sulfuric acid catalyst, in the reactions utilizing the other catalysts (hydrochloric acid and nitric acid), the DADP peak was relatively minor at the same stage, typically taking several hours to become prominent in the trace. In all reactions studied, TATP was prominent in the GC trace during the first 24 h of the experiments, and at 24 h, DADP was also prominent.

When each of the differently catalyzed reactions was aged at 5–9°C and analyzed by GC/MS at 2, 3, 4, 7, and 10 days, the ratios of the areas under the peaks in each trace for TATP and DADP were similar (see Figs. 3A and 5A and Table 1). For example, at day 2, the ratios of the areas under the peaks in the trace of the sulfuric acid-catalyzed reaction for TATP and DADP were 75% and 100%, respectively, compared to 83% and 100%, respectively, when the catalyst was hydrochloric acid. Likewise, when the three reaction mixtures had aged at 5–9°C for 10 days, the ratios of the area under the peaks in the GC/MS trace for TATP and DADP were also similar (sulfuric acid catalyzed, TATP [81%] and DADP [100%]; hydrochloric acid catalyzed, TATP [77%] and DADP [100%]; and nitric acid catalyzed, TATP [85%] and DADP [100%]). Under the chosen experimental conditions, each of the three reactions reached equilibrium with respect to the interconversion of TATP and DADP after 2 days, and the relative abundances of these species had changed little after aging for 10 days at 5–9°C. Conversely, when the reactions were stored at 19–21°C, after an initial 24 h reaction time at 5–9°C, the TATP peak in the GC/MS trace of the headspace above the sulfuric acid-catalyzed reaction was reduced (compared to DADP) at 10 days, whereas for the hydrochloric acid- and nitric acid-catalyzed syntheses, the TATP peak was reduced at 7 days. The DADP peak remained prominent at 10 days for each of the three syntheses. Not surprisingly, the duration and conditions under which a TATP reaction is stored can greatly influence what is able to be identified in the headspace above the reaction mixture. It is possible that SPME and GC/MS analysis of the headspace of a neglected TATP synthesis may show little or no TATP present; however, DADP may continue to be readily identifiable.

The SPME GC/MS headspace analysis of the reaction mixtures that utilized sulfuric or nitric acid did not identify the presence of any side products or degradation products that were specific to either of these acids. However, when TATP was synthesized (as shown in the Materials and Methods section) using hydrochloric acid and analyzed at 7 h, a peak owing to chloroacetone was just visible above the baseline in the trace at 2.7 min. The chloroacetone peak was larger again, relative to the DADP and TATP peaks, in the trace measured at 24 h, the duration of a typical TATP synthesis. Upon further aging of the reaction mixture at 5–9°C (up to 10 days [total]), the peak owing to chloroacetone became more pronounced in each subsequent trace, and at 4 days, a second

chloroacetone, 1,1-dichloroacetone, was just visible above the baseline at 3.2 min. At 10 days, chloroacetone and 1,1-dichloroacetone were readily identifiable in the GC/MS trace. Both chloroacetones were also readily identifiable in the headspace above the hydrochloric acid-catalyzed TATP synthesis, which was stored for 24 h at 19–21°C (after an initial 24 h at 5–9°C). These peaks became more prominent, compared to TATP and DADP, at each testing increment, until at 10 days, they were measured at chloroacetone (32%), 1,1-dichloroacetone (74%), DADP (100%), and TATP (5%). These results show an easily identifiable, catalyst-specific difference in the chemical composition of the reaction mixtures which may be exploited when analyzing unknown samples to distinguish hydrochloric acid-catalyzed TATP syntheses from those which utilize either sulfuric or nitric acid.

In summary, TATP is a poorly stable peroxide explosive that is commonly synthesized clandestinely and is frequently encountered by law enforcement agents and other first responders. An aging study of sulfuric acid-, hydrochloric acid-, and nitric acid-catalyzed TATP syntheses revealed that TATP was present in the headspace above each of the reaction mixtures 1 h subsequent to the combination of the reagents. It has also been shown that TATP may rapidly decompose in the reaction mixture; however, DADP, an explosive analog and decomposition product of TATP, may be more stable and appear more prominently in the reaction headspace for a longer period. It has also been demonstrated that chlorinated acetones, particularly chloroacetone and 1,1-dichloroacetone, are produced during the hydrochloric acid-catalyzed synthesis of TATP. Collectively, these findings enhance the current understanding of the formation and degradation of TATP, provide a method that may assist to distinguish the catalyst employed in unknown syntheses, and provide insight that may be exploited for future detection techniques.

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