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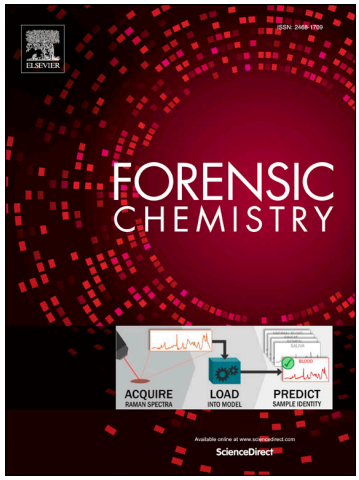

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Alison G. Simon

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Analysis of non-hazardous canine training aids for triacetone triperoxide (TATP)

Alison G. Simon[1*]

[1] School of Forensic Sciences, Oklahoma State University – Center for Health Sciences, Tulsa, OK

*Corresponding author: agsforensics@gmail.com

Abstract

The purpose of this study was to analyze the headspace of non-hazardous canine training aids for triacetone triperoxide (TATP) that are currently available either for purchase or developed by a United States government entity. A total of eight training aids were analyzed using solid phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) to qualitatively identify the volatile compounds present in relative quantities over time until the depletion of TATP. At Time 0, ScentLogix™ had the highest relative abundance (211.2 ± 13.1 analyte/external standard (A/E)). While the SOKKS® aid contained no TATP, the aid with the lowest relative abundance at Time 0 was TOIDS (5.4 ± 0.7 A/E). As expected based on the abundance results, ScentLogix™ lasted the longest (at least 45 weeks) and TOIDS lasted the shortest period of time (2 days). However, ScentLogix™ also had the highest number of headspace components (acetone, diacetone alcohol, diacetone diperoxide, and TATP). Four of the tested training aids contained only TATP in the headspace: Odor Print, TOIDS, Getxent, and TATP POCR. Only four of the aids provided blanks (Getxent, TrueScent®, TA-SPOT®, and POCR), with the Getxent blank resulting in the lowest matrix odor. The manuscript concludes with a discussion of several considerations related to non-hazardous canine training aid selection, including the use of blanks, cost, storage, purity, and longevity.

1. Introduction

Triacetone triperoxide (TATP) is a primary explosive that is shock, heat, and friction sensitive [1]. Because of its hazardous nature, there are several limitations regarding TATP in canine training. Many canine teams do not have access to the material, and those that do are typically restricted to safe, non-operationally relevant scenarios. Such canine teams may turn to a commercially available or non-hazardous training aid in place of or in addition to their exposure to bulk TATP.

There are currently seven commercial training aids for TATP available on the market (Table 1) and one provided by the United States government that were included in this study. Likely due to the proprietary nature of the industry, the information available to the public is sparse. Of the seven aids, only two show related chemical analysis, and three provide information related to canine trials. None of the manufacturers provides completely transparent testing methods and results for either bench chemical analysis or canine analysis. Some of the manufacturers, however, do provide anecdotal evidence. While understandable, this dearth of information can make it difficult for canine handlers and trainers to select the most appropriate materials for training.

*Table 1. Commercial training aids for TATP currently available for purchase. *Training aids not specific to TATP*

Training aid	Manufacturer	Price per aid	Amount of TATP in the aid	Blanks provided?	Storage
TrueScent® [2]	Signature Science, LLC	$159.00	5% by weight	Yes	Room temperature
ScentLogix™ [3]	Polymath Interscience, LLC	$524.98	Not given	No	Cool, dark place
TA-SPOT® [4]	Gallant Technologies	$349.00	0-50%	Yes	N/A
Odor Print [5]	Precision Explosives, LLC	$50.00	Not given	No	Not given
TOIDS [6]	Tripwire Operations Group	$65.00	Not given	No	Refrigeration recommended
Getxent* [7]	Getxent	$290.00 / 50 tubes	N/A	Come blank	Cool, dark, dry place

SOKKS®* [8]	SOKKS®	$1117.90 / 200 aids	Not given	No	Cool, dark place

In addition to the seven commercial aids listed in Table 1, there was one training aid developed by the United States government included in this study. The polymer odor capture and release training aid (POCR) was created at National Institute for Standards and Technology (NIST) [9] and developed at the Federal Bureau of Investigation (FBI) to provide a reliable, long-lasting source of target odor. While these aids are not currently available on the market, they are important points of comparison for the handlers that utilize them. The POCR has a lifetime of almost 270 training hours and can be stored at room temperature. TATP POCRs contain 0.54% mass fraction TATP [10]. There have been several recent publications characterizing other aspects of the TATP POCRs [10,11,12,13,14].

The goal of the current study was to analyze the headspace of the seven commercial aids and the POCR for headspace components and TATP longevity using solid phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS). The purpose of the study is not to recommend one aid over another. Rather, it is simply to provide crucial information to practitioners in the field to help them select the best tools to maintain training and operational efficacy.

2. Method and data analysis

2.1 Samples

The following training aids were purchased for analysis (see Figure 1 for images):

- TrueScent® from Signature Science, LLC, Arlington, VA (purchase dates: December 2020 and July 2021; analysis dates: February 2021 and October 2021, respectively)
- ScentLogix™ from Polymath Interscience, LLC, Annapolis, MD (purchase date: December 2020; analysis date: April 2021)
- TA-SPOT® from Gallant Technologies, Fulton, MD (purchase dates: December 2020 and March 2021; analysis dates: February 2021 and June 2021, respectively)
- Odor Print (non-mini version) from Precision Explosives, LLC, Spotsylvania, VA (purchase date: December 2020; analysis date: March 2021)
- Tripwire Odor Imprint Devices (TOIDS) from Tripwire Operations Group, Gettysburg, PA (purchase date: March 2021; analysis date: October 2021)

- Tubes from Getxent, Neuchâtel, Switzerland (purchase date: January 2021; analysis dates: July 2021 and January 2022)
- SOKKS® K9 Detection Training Aids, Narangba, QLD, Australia (purchase date: December 2020; analysis date: April 2021)

Additionally, the following US government TATP training aid was tested:

- POCR from FBI, Quantico, VA (manufacture dates: June-July 2020 and January 2021; analysis dates: February 2021 and March 2021)

Getxent tubes are an absorption-based training aid that are blank when purchased and require imprinting. The instructions for imprinting the tubes with explosive odors state that the Getxent tube should be exposed to the target odor for 24-48 hours, and that they can be used for up to 6 months after imprinting. Following these instructions, four tubes were placed in a closed 0.47 L (1 pint) arson can with a 1 g sample of TATP synthesized by an FBI Explosives Chemist in a blue antistatic vial. Two of the tubes were exposed to the TATP for 24 hours, and two were exposed for 48 hours. One tube from each exposure time was tested immediately following imprinting; the other was stored for 6 months in a heat sealed Mylar bag before being tested. The two tubes tested immediately after charging are referred to herein as Getxent (24 hr) and Getxent (48 hr), while the two tubes that were stored for 6 months prior to testing are referred to at Getxent (24 hr; stored) and Getxent (48 hr; stored).

A blank training aid was analyzed for those training aids that either provided it along with the TATP aid or had it separately available for purchase:

- TrueScent®
- TA-SPOT®
- Getxent
- POCR

Figure 1. Training aids analyzed in the study: (A) TrueScent®, (B) ScentLogix™, (C) Odor Print, (D) TOIDS, (E), TA-SPOT®, (F) Getxent tube, (G) SOKKS®, and (H) TATP POCR.

2.2 SPME-GC-MS

Each training aid was placed in a separate, sterilized 0.47 L (1 pint) arson can. The cans were left open with no lid to allow the training aids to dissipate. However, a lid was placed on the can for sample extraction when three polydimethylsiloxane/carboxen/divinylbenzene (PDMS/CAR/DVB) SPME fibers (Millipore Sigma, Burlington, MA) were placed simultaneously through a hole in the lid and exposed for 5 minutes. On Day 1, samples were taken at time 0, 1 hour, 4 hours, and 8 hours. Samples were then taken daily for 3-5 days, and weekly until no TATP

was detected. Temperature and humidity were monitored and recorded across the sampling time period using a Lascar EL-USB-2-LCD data logger (Chesterland, OH) (Figure 2).

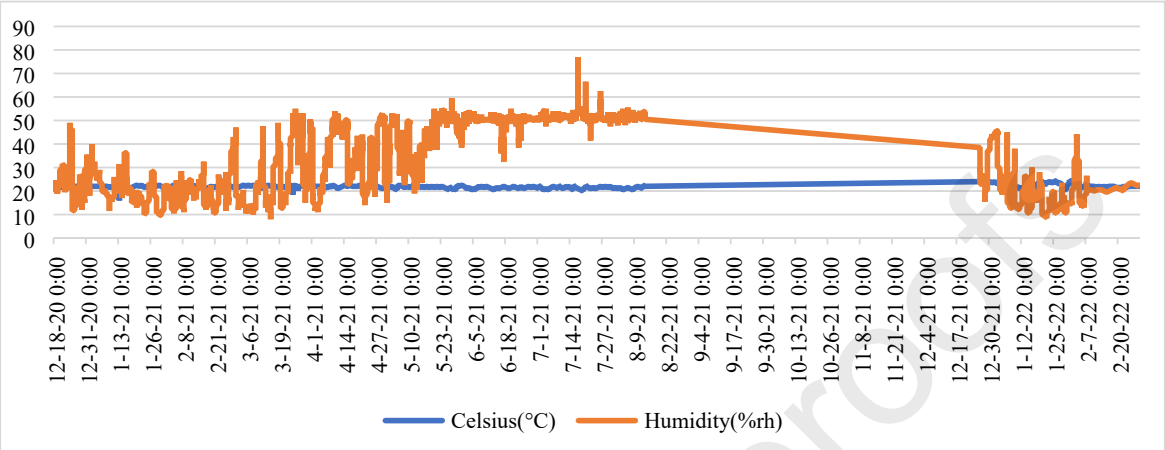

Figure 2. Temperature and humidity in the sampling space, recorded from December 2020 through February 2022.

The SPME fibers were desorbed for 5 minutes at 150°C with a 5:1 split in a gas chromatograph (GC) inlet with a 6 m DB-5MS column (0.25 mm ID; Agilent J&W, Santa Clara, CA). The GC oven was held to 50°C for 1.5 minutes, then ramped to 150°C at 25°C/min, where it was held for an additional minute. All samples were analyzed in triplicate using a mass spectrometer (MS) in scan mode. The instrument was an Agilent 6890N with a 5973 mass selective detector.

Blank samples were analyzed using a similar method to that described above. However, three changes were made to ensure that any heavier, semi-volatile compounds emitted from the blank matrix would be observed. First, blanks were allowed to equilibrate in a sealed pint can for 90 minutes. They were each sampled at 0, 60, and 90 minutes using PDMS/CAR/DVB SPME fibers. Second, the temperature ramp for the GC oven ended at 250°C. Finally, the DB-5MS column was 30 m.

Mass spectral identifications were performed using NIST MS Search 2.0 Library. TATP and DADP were further compared to external standards (AccuStandard, Inc., New Haven, CT for TATP; DADP synthesized by an FBI chemist and diluted with acetonitrile). Retention times of each compound are provided in Table 2.

Table 2. Retention times for each compound identified in the headspace of the TATP training aids.

Compound	Retention time (min)
Acetone	0.57
Diacetone alcohol	0.77
DADP	1.86
TATP	2.26

Results are presented in terms of analyte to external standard ratios (A/E). The external standard was 1mL of 0.1 mg/mL of TATP in acetonitrile (AccuStandard, New Haven, CT) at equilibrium inside a 4 mL amber vial. Because this is an external standard, it was extracted using a separate fiber than the sample fibers. This ratio controlled for any differences in environmental conditions since the experiment occurred over time and stretched a period of 14 months. SPME fibers were monitored daily for signs of degradation and were replaced when deterioration began to minimize the effects of fiber aging. Data was subsequently analyzed using unpaired *t*-tests with GraphPad Software (San Diego, CA).

3. Results

3.1 Blanks

An analysis of the blank training aids is presented in Figure 3. Only three of the seven commercial aids provided blank training aids. The FBI POCR analyzed in Figure 3 was a training aid that had been returned from the field (i.e., used by operational canine teams) and showed signs of age-related degradation. A comparison of this blank POCR and a new, previously unused POCR is given in Figure 4, showing that less odor was detected above the new POCR. Used blanks were not analyzed for the other training aids. The TA-SPOT® and TrueScent® blanks aids each had notable background peaks, while the Getxent tube had the lowest amount of odor.

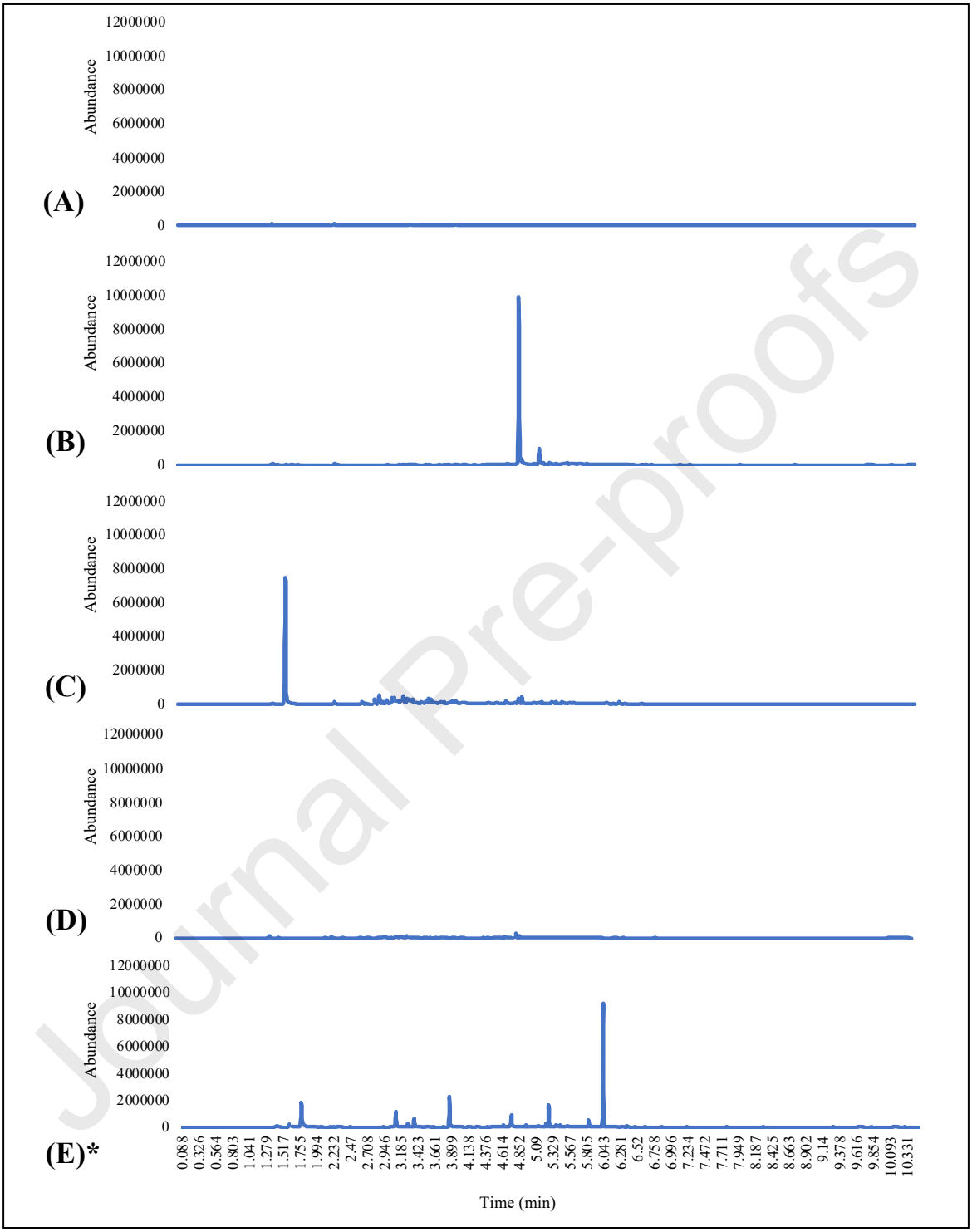

*Figure 3. Chromatograms for each tested blank training aid, adjusted to display equivalent abundances. (A) Control arson can. (B) TA-SPOT®. (C) TrueScent®. (D) Getxent tube. (E) POCR A9-8. *Note: POCR A9-8 was used in Beta-Test A and returned from the field.*

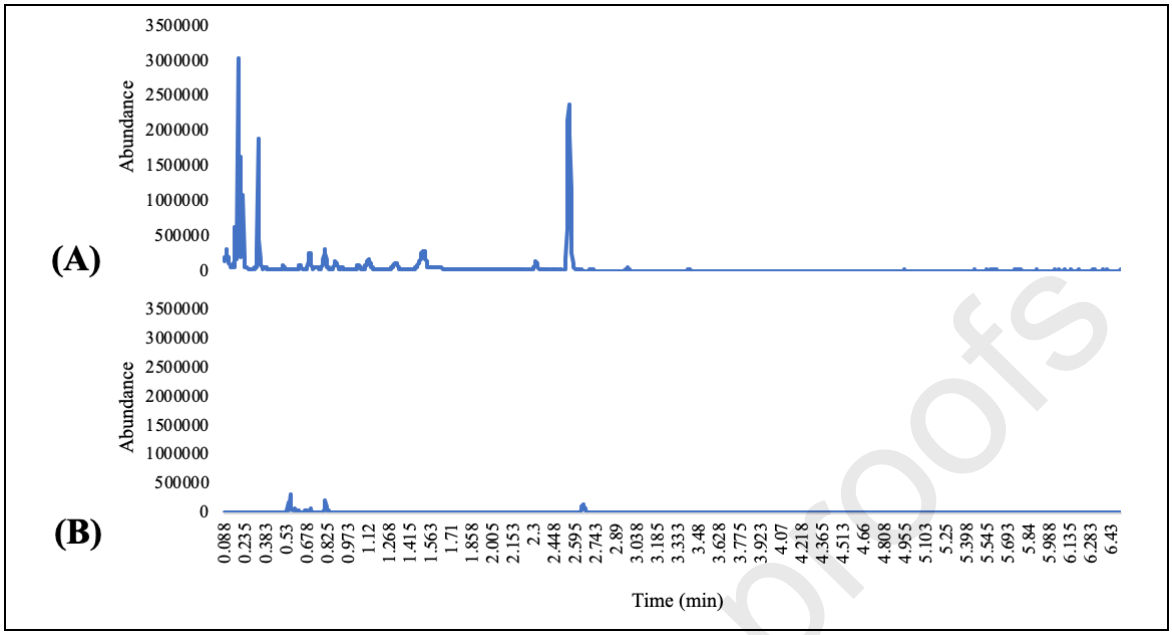

Figure 4. Chromatograms for two separate POCRs. (A) POCR A9-8; returned from the field. (B) POCR C11-6; new and previously unused. This analysis was done using the same instrumental method as the TATP training aid analyses.

3.2 Comparisons at Time 0

Figure 5 shows a comparison of the TATP recovered above each training aid at Time 0. ScentLogix™ had a significantly higher amount than the next closest training aid, TATP POCR (unpaired *t*-test, p = 0.0009). TA-SPOT® and Odor Print had the next highest amounts, respectively. No TATP was detected above the SOKKS® training aid.

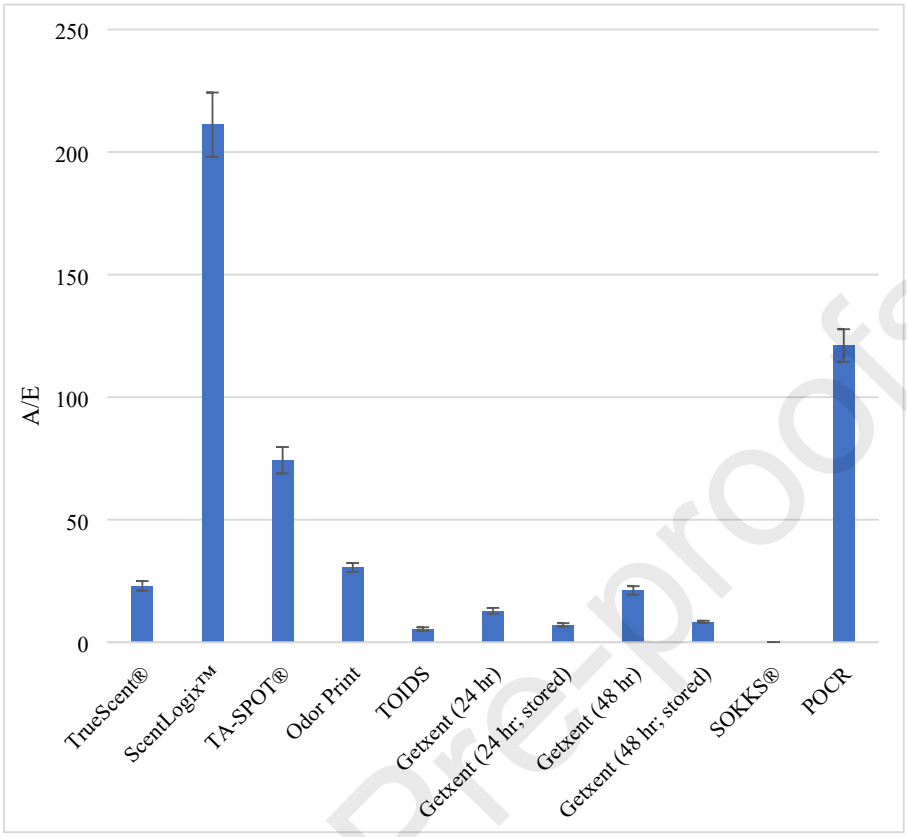

Figure 5. Headspace comparison for TATP recovered from all tested training aid at Time 0.

In addition to TATP, 3 of the training aids contained acetone (TrueScent®, ScentLogix™, and TA-SPOT®). A comparison of the amount of acetone recovered above these aids at Time 0 is given in Figure 6. ScentLogix™ contained both the most TATP and acetone. TrueScent® contained the lowest amount of acetone.

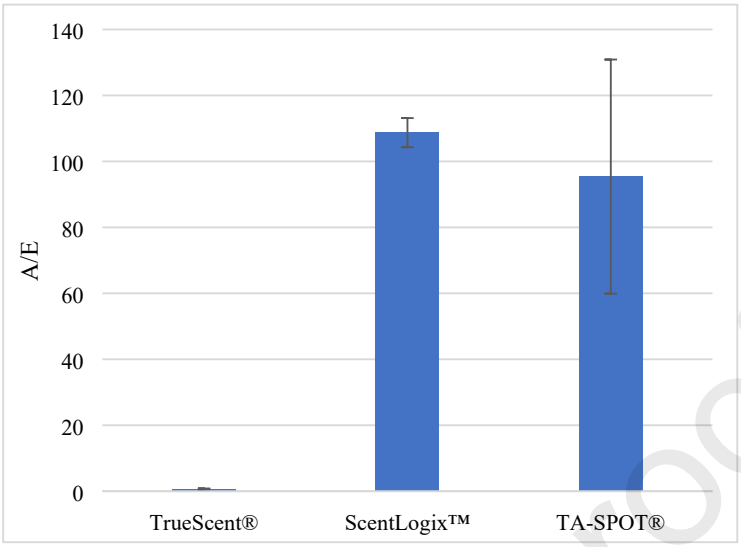

Figure 6. Headspace comparison for acetone recovered from tested training aids at Time 0.

TrueScent®, TA-SPOT®, and ScentLogix™ all contained DADP at Time 0; however, the amount detected was below the signal to noise ratio for all three training aids. DADP was only detected at Time 0 for TrueScent® and was not detected after that sampling period. ScentLogix™ contained one more unique component at Time 0: diacetone alcohol (86.6% probability match).

Purity is an important consideration in training aid selection. The number of headspace components detected above each tested training aid at Time 0 is given in Figure 7. ScentLogix™ had the highest number of contaminants (4 total components: acetone, DADP, TATP, and diacetone diperoxide). TrueScent® and TA-SPOT® each contained 3 components (acetone, DADP, and TATP). The SOKKS® headspace contained 0 TATP or TATP-related compounds. Odor Print, TOIDS, Getxent, and POCR each contained only TATP. This is not a constant for the Getxent tubes because the headspace of a Getxent tube depends on the material used to imprint it. Thus, if the Getxent tubes were imprinted using a less pure form of TATP, the headspace would contain a higher number of contaminants.

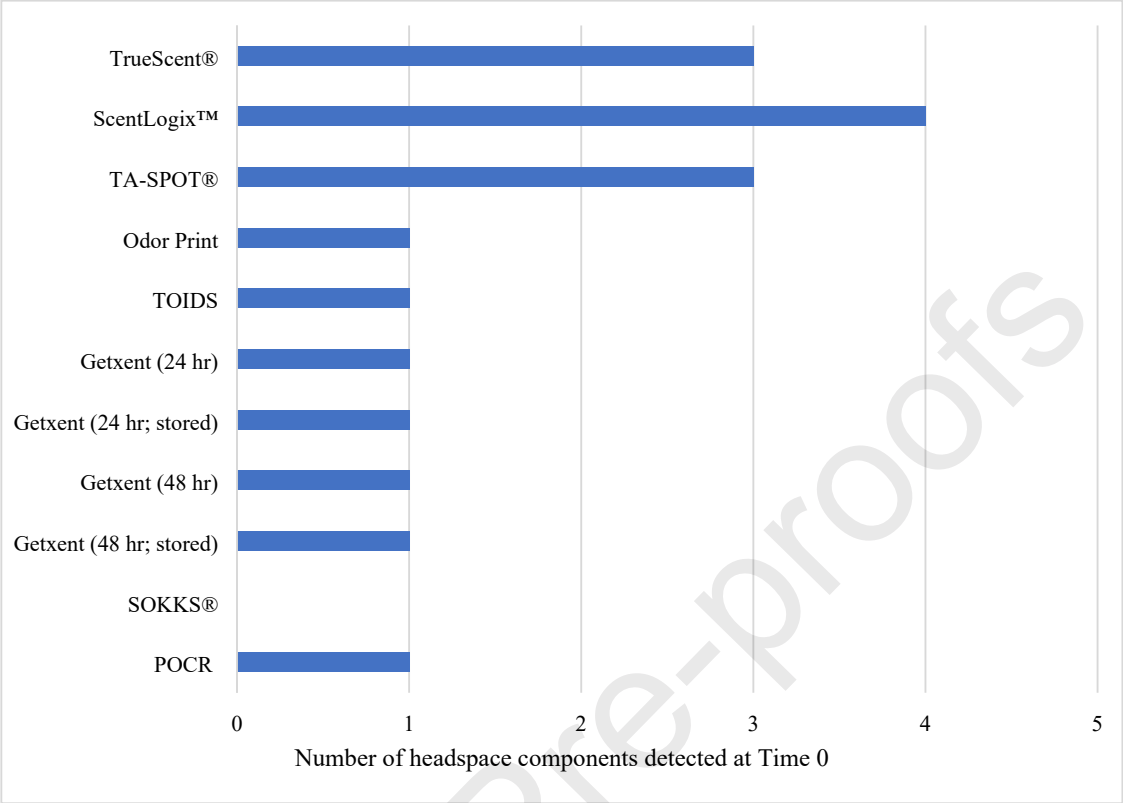

Figure 7. Number of headspace components detected above each training aid at Time 0.

3.3 Comparisons over time

The components detected above each training aid over time is given in Figures 8, 9, and 10. For each training aid, the headspace components detected and their longevity will be discussed. Additionally, unpaired *t*-tests were performed for each training aid to compare the statistical significance of the amount of TATP detected at Time 0 versus 60 minutes and 60 minutes versus 4 hours. These comparisons are important for determining the rates of odor release over the course of an average canine training session. It is expected that at Time 0, the amount of odor will be highest due to the saturation stage of odor release [10]. The saturation stage is caused by the headspace of the training aid building to an equilibrium in a closed system (i.e., during storage). The headspace at equilibrium is then released upon the opening of the training aid. After that time, the rate of odor release will decrease. It is expected that there will be a period of time when the odor is released at a constant rate, known as the steady state. After that point, the rate of odor production will begin to deplete and less headspace will be generated. Thus, if the difference

between 60 minutes and 4 hours is significant, it is likely that the odor is depleting faster and is not in a steady state of odor release, which would be determined by a non-significant difference in odor abundance during that time frame.

Figure 8 shows a comparison of TrueScent®, ScentLogix™, TA-SPOT®, TOIDS, Odor Print, and TATP POCR. TrueScent® contained TATP, DADP, and acetone in the headspace. DADP was detected only at the initial sampling time and was below the signal to noise ratio. Acetone was detected for four days. TATP was detected for 20 weeks. While laboratory conditions prevented the aid from being tested until depletion, the amount of TATP detected was below the signal to noise ratio after Week 7. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0002). Between 60 minutes and 4 hours, the difference was also significant (unpaired *t*-test, p = 0.0018).

The headspace of ScentLogix™ contained TATP, DADP, acetone, and diacetone alcohol, though DADP was below the signal to noise ratio. DADP was detected for 3 days, while acetone was detected for 5 weeks. Diacetone diperoxide was detected for 16 weeks. TATP was detected for at least 45 weeks, though laboratory conditions prevented the experiment from continuing until the target was fully depleted. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0005). Between 60 minutes and 4 hours, however, the difference was not significant (unpaired *t*-test, p = 0.0757).

TATP, acetone, and DADP were all detected upon initial sampling of TA-SPOT®. DADP was under the signal to noise ratio for the two weeks it was detected. Acetone was also detected for two weeks. The amount of acetone detected at Time 0 was higher than that of TATP, which was unexpected. TATP was detected for seven weeks. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0025). However, the difference was not significant between 60 minutes and 4 hours (unpaired *t*-test, p = 0.1246).

Only TATP was detected from the headspace of the Odor Print training aid and was detected for three weeks. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0031). Between 60 minutes and 4 hours, the difference was also significant (unpaired *t*-test, p = 0.0037).

TATP was also the only component recovered from the headspace of the TOIDS training aid and was detected for two days. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0003). The difference was also significant between 60 minutes and 4 hours, (unpaired *t*-test, p = 0.0077).

The TATP POCR headspace contained only TATP, which agrees with previous studies and characterization of the TATP POCRs [10,12,13,14]. TATP was detected for four weeks, which generally agrees with previous data [10]. The amount of TATP decreased significantly after Time 0 (unpaired *t*-test, p = 0.0019). Between 60 minutes and 4 hours, however, the difference was not significant (unpaired *t*-test, p = 0.2114).

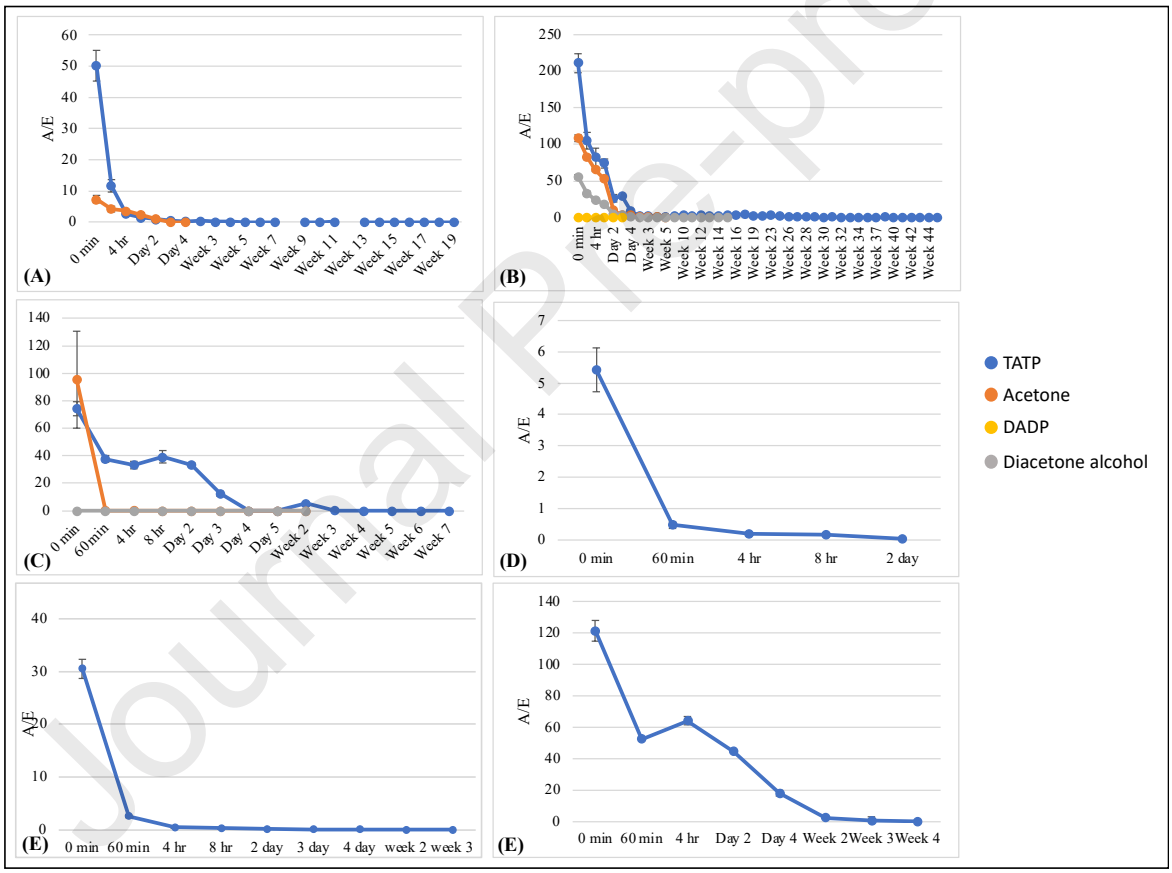

Figure 8. Recovered headspace components for tested training aids (A) TrueScent®, (B) ScentLogix™, (C) TA-SPOT®, (D) TOIDS, (E) Odor Print, and (E) TATP POCR.

The headspace results for all four tested Getxent tubes are given in Figure 9. Only TATP was detected in the headspace. However, because the Getxent tubes are imprinted by each handler or trainer, this is not necessarily always the case and is an artifact of the purity of the TATP used in this study. Both of the unstored tubes had TATP in the headspace for three weeks. The 24-hour and 48-hour imprinted stored tubes lasted for 4 days and 2 weeks, respectively. For both sets, the 48-hour imprint resulted in a higher amount of TATP recovered. Interestingly, after Time 0, the amount of TATP decreased significantly only for the stored samples (Table 3). When used immediately after imprinting, the difference between Time 0 and 60 minutes was not significant.

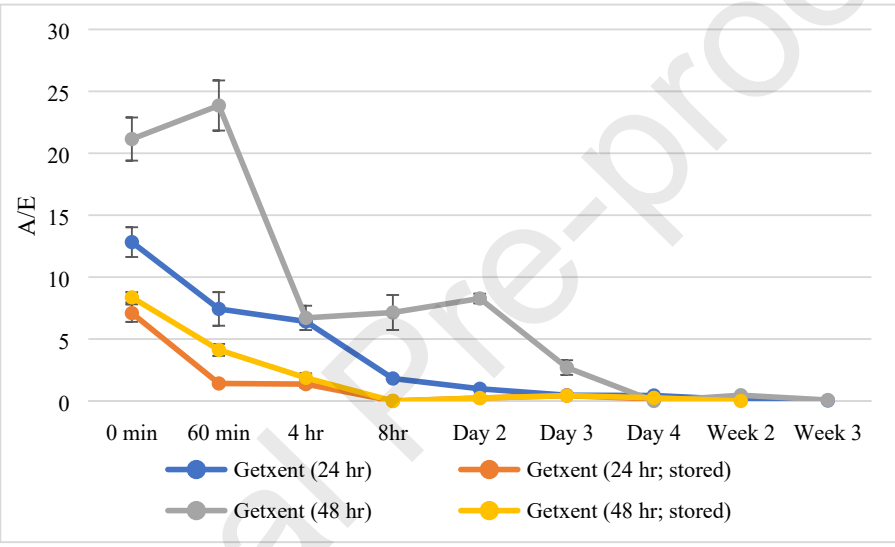

Figure 9. Recovered headspace components for Getxent training aids.

Table 3. Results of unpaired t*-tests comparing the amount of TATP recovered at Time 0 and 60 minutes.*

	p Value	Significant?
Getxent (24 hr)	0.2147	No
Getxent (24 hr; stored)	0.0002	Yes
Getxent (48 hr)	0.1388	No
Getxent (48 hr; stored)	0.0462	Yes

No TATP was detected in the headspace of two separate SOKKS training aids (Figure 10). The retention time of TATP is 2.26 min (see Table 2).

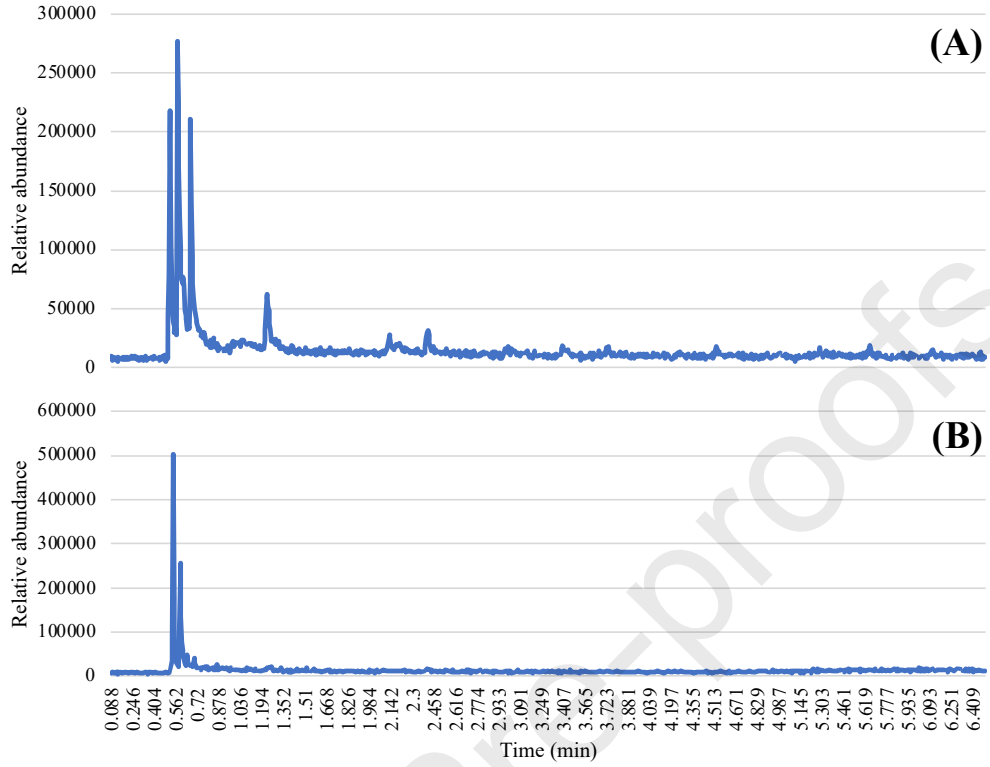

Figure 10. Chromatograms of two different SOKKS training aids, showing no TATP or related peaks. TATP retention time is 2.26 min (see Table 2).

When comparing the amount of TATP recovered at Time 0 and 60 minutes for each aid, all of the aids except for the unstored Getxent tubes were significantly different. This implies that the Getxent tubes did not have a period of saturation, which was expected since the first sampling period occurred immediately after imprinting. There was no storage of the aids to encourage the formation of the saturation stage by limiting dissipation and encouraging equilibrium within a closed system.

Comparing the amounts of TATP detected at 1 and 4 hours can help determine longevity of an aid during a training session. ScentLogix™, TA-SPOT®, Getxent tubes imprinted for 24 hours, and POCR all produced amounts of TATP that were *not* significantly different between 1 and 4 hours, suggesting that they reach of a steady state of odor release during a generic canine training session length of time. TrueScent®, Odor Print, TOIDS, and Getxent tubes imprinted for 48 hours were significantly different, implying that they do not have a steady state of odor release over that same time period.

In addition to the discussions of saturation and steady state odor releases, the longevity of the training aids in general can be useful in selecting an appropriate tool for specific training scenarios. Table 4 shows the longevity of the training aids, meaning how long TATP was detected in the headspace of each. ScentLogix™ was detected for the longest length of time (45 weeks), followed by the TrueScent (20 weeks), although neither of these aids was tested to depletion. TOIDS and the 24 hr stored Getxent tubes lasted the shortest periods of time, 2 and 4 days, respectively.

*Table 4. Length of time TATP was detected in the headspace of each tested training aid. *Not tested until completion*

Training aid	Time TATP was detected
TrueScent®	20 weeks*
ScentLogix™	45 weeks*
TA-SPOT®	7 weeks
Odor Print	3 weeks
TOIDS	2 days
Getxent (24 hr)	3 weeks
Getxent (24 hr; stored)	4 days
Getxent (48 hr)	3 weeks
Getxent (48 hr; stored)	2 weeks
SOKKS®	not detected
POCR	4 weeks

4. Conclusions

This study provided a comprehensive comparison of the headspace above existing non-hazardous TATP canine training aids, including seven commercial aids and one government aid. The results suggest many considerations in training aid selection.

First, each training aid's adherence to the OSAC Dogs and Sensors Subcommittee recommendations for the use of blank training aids should be considered [15,16]. Of those tested, ScentLogix™, Odor Print, TOIDS, and SOKKS® did not provide a blank or have one available

for purchase. Thus, TrueScent®, TA-SPOT®, Getxent, and POCR are the training aids that adhere to this recommendation. Among these four aids, Getxent had the lowest amount of background odor.

In addition to recommendations by the Dogs and Sensors Subcommittee, there are other considerations that handlers and trainers may find important in their selection of a TATP training aid. Cost and storage are among this list (see Table 1), though each organization will have a different budgetary and storage capabilities.

Purity is certainly a main consideration. Odor Print, TOIDS, POCR, and possibly Getxent were the purest, containing only TATP odor. A previous POCR study showed that dogs imprinted on TATP POCR could find bulk TATP at a rate above 90% with < 10% false alert rate [14]. Purity is thus very important, especially for less experienced canine teams. However, less pure training aids may still be important training devices in teaching canines to identify their target odor in differing contexts.

Finally, longevity of each training aid should be considered. The lifetime of an aid chosen for use should be determined by the training scenario(s). Two aspects discussed herein are important: 1) The length of time TATP is released and 2) The rate of release of TATP. Thus, the training aid that lasts longest is not necessarily the most desirable. Further research should be done to explore the effect of storage on the longevity of each aid, as it is expected that proper storage between sessions would extend the lifetime.

Previous research identifying the headspace components of non-hazardous canine training aids for another peroxide explosive, hexamethylene triperoxide diamine (HMTD), found that none of the five available aids was representative of the true material [17]. In contrast, the current study found that all of the non-hazardous training aids analyzed contained TATP in the headspace except for one (SOKKS®). This discrepancy demonstrates the importance of third-party analysis and continuing evaluation of available non-hazardous training aids. Despite similarity between targets, in this case two peroxide explosives, the efficacy of the available training aids to provide representative odors was variable. It is therefore important to provide such chemical analyses and to consider other variables (e.g., the use of blanks, cost, storage, purity, and longevity) for all canine detection targets. By providing comprehensive information to practitioners, handlers and trainers will be able to select the training aid most appropriate for their specific training scenarios.

5. Acknowledgements

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6. Funding

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The author declares no competing interests or relationships.

- Eight non-hazardous canine training aids for TATP were evaluated using SPME-GC-MS.
- Four of the aids were pure, containing only TATP and some matrix odors (Odor Print, TOIDS, Getxent, and POCR).
- Aids lasted from 2 days (TOIDS) to 45 weeks (ScentLogix).