**Characterization of Volatile Markers in Electronic Components via Solid-Phase Microextraction (SPME) and Comprehensive Two-Dimensional Gas Chromatography with Electron Ionization Time-of-Flight Mass Spectrometry (GCxGC/EI-TOF-MS)**

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Abstract

The development of novel non-destructive methodologies for the verification of electronic components authenticity is imperative to reduce the economic and safety risks associated with the use of counterfeited products. Currently, inspection methods rely on a series of electrical and physical tests that usually are labor intensive, destructive, and still not 100% effective given their dependence on human interpretation. This paper presents an alternative method using solid-phase microextraction (SPME) and comprehensive two-dimensional gas chromatography with electron ionization time-of-flight mass spectrometry (GCxGC/EI-TOF-MS) to chemically characterize electronic components. The novel approach outlined in this study utilized the high sensitivity of GCxGC/EI-TOF-MS systems to consistently identify more than 300 volatile markers in two different groups of 14-pin 16-bit microcontrollers and one group of 16 GB microSDs from three different manufacturers. Results show that the chemical characterization of the electronic components is effective, repeatable, and non-destructive. The significant volatile markers are found to be dependent on the manufacturer, age, and type of the electronic component. Thus, the list of significant volatile markers of each electronic component could potentially serve as basis to verify the source of an electronic component. Compared to existing physical verification methods, this novel approach is faster and less ambiguous avoiding the overlook or false identification of counterfeits. In fact, this methodology could potentially indicate if further inappropriate manipulations have taken place on the component based on the presence and quantity of 1,3,5-cycloheptatriene, thereby reducing the risk of counterfeit components being installed in electronic systems. Using GCxGC/EI-TOF-MS and SPME techniques may be helpful in reducing the quantity of counterfeit electronic components found in warfare systems used by the United States Department of Defense (DoD) which is especially at risk due to the rapid aging of deployed systems and a vast span of interconnected logistics supply chains that provide components for installation in such warfare systems.

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*Keywords:* Two-Dimensional Gas Chromatography, Solid Phase Microextraction, Non-Destructive Electronics Authenticity, Trusted Electronics, Secure Defense Logistics

Introduction

Electronic components are prevalent in many consumer products and throughout most warfare systems utilized by the United States Armed Services. Ensuring that these systems reliably function is mission-critical to the Army, Air Force, Navy, and Marine Corps (Petel, 2014). Thus, there is an incentive for the United States Department of Defense (DoD) to ensure that the components and electronic devices used in complex electronic military systems function correctly even in the most adverse and strenuous environments. This condition represents a huge challenge for DoD since the failure of electronic components is difficult to predict and usually caused by many conditions. However, failure can be highly prevented (Semiconductor Industry Association SIA, 2011) if the installation of counterfeit parts, i.e., remarked / repackaged / recycled / refurbished or even cloned devices are avoided (Mura, Murru & Martines, 2020).

Given the evolving modern sophisticated counterfeiting techniques, it is pertinent for the DoD to develop an effective method to identify the authenticity of electronics to ensure National Security. Common techniques currently available in the industry to identify counterfeits include a series of electrical and physical tests that must be performed by highly-qualified experts usually involving multiple analytical techniques. On the one hand, analyzing contact degradation, device parameter distributions, chip curve trace are recommended practices to evaluate the electrical performance of a component (Ahmadi, Tavousi, Favata, Shahbeigi-Roodposhti, Pelapur & Shahbazmohamadi, 2018). Physical characterization, on the other hand, encompasses the inspection of packaging quality, labeling in the integrated circuits (ICs), and analysis of the structure of the die through simple visual inspection and high-tech imaging techniques such as X-ray, infrared, acoustic microscopy, scanning electron microscopy, and focused ion beams (Chen, Zhang & Zhang, 2014). Nevertheless, these methods, especially those based on visual physical assessments, are time consuming, most of them destructive (Hillman, 2011), and still far to be 100% effective since they rely on human interpretation.

Despite all efforts, the amount of counterfeit electronic devices that have found their way into the DoD supply chain increased from 3,868 in 2005 to 9,356 in 2008 (U.S. Department of Commerce, 2010). Although this trend decreased over the next years thanks to the creation of the Counterfeit Prevention Policy in 2013, the DoD has been unable to eradicate the issue in full (United States Government Accountability Office, 2016). The introduction and proliferation of counterfeit electronic devices in DoD systems are attributed to two causes: (1) many systems within the DoD are used far beyond their expected end-of-service dates, and (2) the development of the globally integrated economy prevents logistically sourcing products produced solely within the United States (Gansier, Lucyshyn & Rigiliano, 2013). These factors have made it easier for counterfeit products to find their way into the DoD logistics supply chain. According to the Alliance for Gray Market and Counterfeit Abatement, about 100 billion dollar loss are reported worldwide by electronic companies every year due to counterfeited products (Pecht & Tiku, 2006). Analogously, it is estimated that “as many as 15 percent of all spare and replacement semiconductors purchased by the Pentagon are counterfeit” (Semiconductor Industry Association SIA, 2011).

Sources for electronic components can come from four primary sources: the original manufacturer, the original manufacturer's authorized distributors, independent distributors or brokers, or aftermarket manufacturers (Sullivan & Wilson, 2017). As a preventative method, the DoD always seeks to purchase components from a "trusted source" which is a source known to produce genuine electronic components. This "trusted source" is typically the original manufacturer. However, because some systems have been extended far past their end-of-service date, components may have to be purchased from sources other than the original manufacturer because they may no longer manufacture the component (Livingston, 2010). The DoD may be forced to purchase from the original manufacturer's authorized distributors or even independent brokers who may not be as completely vetted. This less-than-desirable situation creates an opportunity for counterfeit electronic components to find their way into DoD systems, creating a weakness in the Nation's security as components are not constructed to required specifications or components are manufactured with a vulnerability to malicious malware through backdoor applications (Petel, 2014).

Under this perspective, it is imperative to develop alternative methods to non-invasively and unambiguously determine the fidelity of electronic components such as semiconductors, microcontrollers, and processors used in warfare systems before their actual installation and use (Livingston, 2010). Preliminary alternative approaches to identify counterfeits include (i) the analysis of the electromagnetic fingerprint of the IC as a contactless side channel to measure its transient activity (Huang, Boyer & Ben Dhia, 2015), and (ii) the chemical characterization of ICs through flowing atmospheric-pressure (helium plasma) afterglow mass spectrometry (FAPA-MS) complemented with chemometric techniques (Pfeuffer, Caldwell, Shelley, Ray & Hieftje, 2014). Nevertheless, the robustness and complete development of the electromagnetic method (Huang, Boyer & Ben Dhia, 2015), and the variable response of FAPA-MS to different types of analytes (alkanes, alcohols, aldehydes, ketones, carboxylic acids, and organic peroxides) and the effects of further oxidation processes occurring in the afterglow region (Brüggemann, Karu & Hoffmann, 2016) are still matter of discussion.

One-dimensional gas chromatography (GC) coupled with solid-phase microextraction (SPME) is a proven method for identifying trace volatile organic compounds (VOCs) in the headspace surrounding solid and liquid samples of unknown composition. SPME-GC/MS has been used to study the feasibility of canine detection of mass storage devices (DeGreeff, Cerreta & Rispoli, 2017). Additionally, these analytical techniques have been commonly used in chemical forensics to identify unknown compounds at extremely low concentrations (Brasseur, Dekeirsschieter, Schotsmans, de Koning, Wilson, Haubruge, & Focant, 2012; Gordin & Amirav, 2000). Aiming to complement these alternatives, a non-target approach based on comprehensive two-dimensional gas chromatography with electron ionization time-of-flight mass spectrometry (GCxGC/EI-TOF-MS) and solid-phase microextraction (SPME) is proposed here to characterize the chemical fingerprint of electronic components. GCxGC separates at least an order of magnitude more compounds compared to conventional one-dimensional gas chromatography (GC) (Reddy & Nelson, 2013). This capability broadens the possibility of studying complex chemical mixtures usually found in forensic (Stadler, Stefanuto, Byer, Brokl, Forbes & Focant, 2012), environmental, fuels, food, fragrances, and biological applications (Prebihalo, Berrier, Freye, Bahaghighat, Moore, Pinkerton & Synovec, 2018). However, to the best of our knowledge, GCxGC has not being used to study volatile markers present in electronic components. Thus, this work aimed to develop an alternative and non-destructive method for identifying VOCs in electronics as a potential approach for assessing the authenticity of microcontrollers and micro storage devices in the short future. This study showed that different types of electronic components with different manufacturers, countries of origin, and ages have different chemical signatures, and demonstrates that it is possible to differentiate between equivalent components that differ along with these variables.

Materials and Methods

*Samples --* For this study, three different types of electronic components were selected for sampling: two types of 14-pin 16-bit microcontrollers (Microchip Technology Incorporated, 2009; Texas Instruments, 2004), and a 16 GB microSD card (SanDisk, 2019). Thirty replicates of each component type were performed along with 16 controls designed to capture the baseline composition of environmental and instrument VOCs adequately. Controls consisted of one run relative to a column blank, two runs aiming to study the natural chemical elution of the SPME fibers, and thirteen runs of the SPME fibers exposed to empty vials. Compounds that are found in more than half of the control group samples were automatically excluded from consideration for possible VOCs of significance in the electronic components. The sample size composition is detailed in Table 1. Once each population was sampled, the chemical signatures of the electronic components were compared to the volatile compounds identified in the controls to determine the common and unique VOCs of each group. It is worth mention that the comparison with certified counterfeit components was not possible in the present study given the uncertainty related to their procurement process.

*Table 1*. Sample information

|  |  |  |  |
| --- | --- | --- | --- |
| Product classification | 14-pin 16-bit  Microcontroller | | 16 GB  MicroSD Card |
| Manufacturer | Microship Technology | Texas Instruments | SanDisk |
| Country of Origin | Thailand | Malaysia | China |
| Date of Manufacture | October 2015 | May 2017 | 2019 |
| Samples Collected | 30 | 30 | 30 |

*Sample Preparation --* Prior to sampling, 20 mL glass headspace vials were placed in an oven for 8 hours to clean the vials of any VOCs remaining from manufacturing. Component samples were removed from the original manufacturer's packaging and placed directly in the cleaned vials. The vials were then purged with nitrogen for 10 seconds to remove ambient environmental VOCs before submersion in a water bath at 70℃. The temperature of 70℃ was selected to protect the electronic components from being destroyed during the sampling process. This temperature is within the safe operating temperature defined by the manufacturers (Microchip Technology Incorporated, 2009; SanDisk, 2019; Texas Instruments, 2004). Divinylbenzene/ carboxen/ polydimethylsiloxane (DVB/ CAR/ PDMS) solid-phase microextraction (SPME) fibers (DeGreeff, Cerreta & Rispoli, 2017) were preconditioned at 250℃ for ten minutes prior to sampling each day. The fibers were manually exposed to the samples within the vial for 50 min before being manually injected into the inlet of the GCxGC/EI-TOF-MS, set at 250℃ for desorption of the VOCs.

Sample Analysis -- In this study, GCxGC/EI-TOF-MS was used. Pegasus GC-HRT 4D (EI) High-Resolution TOF/MS (LECO Corporation, Saint Joseph, MI) with an Agilent 7890B gas chromatograph, a non-moving quad-jet dual stage thermal modulator cooled with liquid nitrogen, and an Agilent G4513A auto-injector was used for qualitative analysis of the samples. Chromatographic conditions for GCxGC/EI-TOF-MS are shown in Table 2. The ion source temperature was set to 250 °C, and the electron energy was 70 eV. ChromaTOF (Version 1.90.60.0.43266) software was utilized for data collection (with an m/z of 50-1000), processing, and analysis. The signal-to-noise ratio was set to 3. Identification of the compounds was achieved by matching the measured mass spectra (similarity value of > 600) with Wiley (2011) and NIST (2011) mass spectral databases. This analysis method was selected to yield a broad spectrum of VOCs for consideration.

*Table 2*. Chromatographic Conditions for Sample Analysis

|  |  |
| --- | --- |
| Parameter | Description |
| Columns | Primary: ZB-35HT Phenomenex (60 m × 0.25 mm × 0.25 µm)  Secondary: ZB-1HT Phenomenex (1.9 m × 0.25 mm × 0.25 µm) |
| Carrier gas | UHP helium, 1.20 mL/min |
| Inlet | Purge time: 60 sec  Purge flow: 85 mL/min |
| Oven Temperature | Isothermal 40 °C for 2 min, followed by a linear gradient of 8 °C/min to a temperature of 280 °C, being held isothermally for 1 min |
| Modulation Period | 2.0 s with a 0.6 s hot pulse time |
| Temperatures | Inlet: 250 °C  Transfer line: 250 °C |

*Data Processing* -- Peak tables generated by individual samples were manually analyzed to generate a list of reliable VOC markers for each sample group tested. For this study, only compounds that were named using the mass spectral databases listed above were considered. Duplicate named compounds within a given sample were removed from consideration. Further, compounds were required to meet three criteria: (i) they must be present in at least 25 of the 30 replicates within each sample group, (ii) be present in less than half of the control samples, and (iii) have reliable retention times. Due to instrumental drift, similarly named compounds within sample groups were selected as reliable markers if they fell within a reliable retention time range (Table 3).

Results and Discussion

More than 300 compounds were in average correctly identified for each group, exhibiting the success of the SPME technique and the great resolution achieved through GCxGC/EI-TOF-MS. Microchip technology microcontroller counted the less (319) while the microSD counted the highest (405) amount of volatile compounds. Inversely, the 14 pin 16-bit microcontroller manufactured by Microchip Technology exhibited the largest standard deviation (70) among samples while the microSD counted the lowest (20). The 14-pin 16-bit microcontroller by Texas Instruments lied in between with 378 VOCs with a standard deviation of 24 compounds. The lower amount of VOCs and higher standard deviation of the microchip developed by Microchip Technology (Thailand) compared to the one manufactured by Texas Instruments (Malaysia) can be attributed to either different (i) origins/manufacturing processes or (ii) aging conditions among them, being this last one 19 months newer. Aging, in fact, has been a key factor previously identified that induced detectable changes in the electromagnetic fingerprint of ICs (Huang, Boyer & Ben Dhia, 2015) and it is expected to induce changes in the chemical fingerprint as well.

After narrowing down the list of identified VOCs to those unique to each component type, retention time analysis was used to identify the significant VOCs across samples. Table 3 displays the list of significant VOC markers for each sample group that met the required selection criteria while achieving excellent consistency in terms of retention times. Across the three sample groups, there were two significant VOC markers identified in the Microchip Technology sample group, six in the Texas Instruments sample group, and twenty-one in the SanDisk sample group. These compounds can be considered as the chemical fingerprints of each electronic component and manufacturer representing potential key VOCs to be used for further comparison to counterfeit suspects.

*Table 3*. Significant VOCs Having Consistent Retention Times

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| VOC | Sample Group | Retention Time Range  (s) | Average Peak  Area (un) | Peak Area Standard Deviation (un) |
| 1,3-dimethyl  benzene | Microchip Technology | 626-632 | 3.7E+05 | 2.0E+05 |
| 1,3,5-cycloheptatriene | Microchip Technology | 496-522 | 5.3E+05 | 5.2E+05 |
| Phenol | Texas Instruments | 836-840 | 7.7E+05 | 2.4E+05 |
| 1,1'-(1,2-cyclobutanediyl)bis-, trans-benzene | Texas Instruments | 1682-1686 | 2.9E+05 | 7.6E+04 |
| Benzaldehyde | Texas Instruments | 846-850 | 3.1E+05 | 1.1E+05 |
| Hexanal | Texas Instruments | 546-550 | 1.1E+05 | 4.7E+04 |
| 1,3-dimethyl-benzene | Texas Instruments | 628-632 | 2.4E+05 | 9.0E+04 |
| 1,3,5-cycloheptatriene | Texas Instruments | 498-502 | 5.6E+05 | 2.4E+05 |
| Hexamethylene diacrylate | SanDisk | 1492-1496 | 7.9E+06 | 2.2E+05 |
| 4-acetyl- morpholine | SanDisk | 1180-1184 | 1.3E+06 | 4.4E+05 |
| 2,3-dihydro-5-methyl-furan | SanDisk | 370-376 | 6.5E+06 | 2.9E+06 |
| 1,6-Hexanediol | SanDisk | 1062-1068 | 6.5E+06 | 1.9E+06 |
| 2,2,4-trimethyl-3-hydroxy-, isobutyl ester pentanoic acid | SanDisk | 1228-1232 | 5.5E+06 | 9.3E+05 |
| 1,3,5-Cycloheptatriene | SanDisk | 496-514 | 2.9E+06 | 1.4E+06 |
| 2-methyl-, 1-(1,1-dimethylethyl)-2-methyl-1,3-  propanediyl ester propanoic acid | SanDisk | 1430-1432 | 6.6E+06 | 2.0E+06 |
| 2,4-Di-tert-butylphenol | SanDisk | 1390-1394 | 2.6E+06 | 1.1E+06 |
| Phenol | SanDisk | 838-842 | 6.5E+06 | 4.0E+06 |
| 4-(methylthio)- benzaldehyde | SanDisk | 1678-1682 | 7.6E+05 | 4.6E+05 |
| butyl dodecanoate | SanDisk | 1582-1586 | 7.3E+05 | 3.5E+05 |
| 1,2-benzene-dicarboxylic acid, bis(2-  methylpropyl) ester | SanDisk | 1732-1736 | 2.5E+06 | 1.5E+06 |
| 1,2,3,5-Tetramethyl-benzene, 2-oxime- | SanDisk | 1426-1430 | 1.0E+06 | 4.2E+05 |
| Tetrahydropyrrolo[2,1-c] [1,4] oxazin-4-one | SanDisk | 1392-1396 | 2.4E+06 | 7.6E+05 |
| Tetracosamethyl-cyclododeca- siloxane | SanDisk | 1952-1956 | 2.0E+05 | 6.8E+04 |
| N,N,N- Triethylamine | SanDisk | 354-368 | 2.5E+06 | 1.6E+06 |
| 2,4,6-trimethyl- benzoic acid | SanDisk | 1356-1362 | 4.2E+06 | 2.9E+06 |
| 2,4,6-trimethyl- benzophenone | SanDisk | 1736-1740 | 9.7E+04 | 5.0E+04 |
| 2-(1-  methylethyl)- 9H-Thioxanthen-9-one | SanDisk | 1970-1978 | 4.8E+06 | 3.7E+06 |
| (2-decyldodecyl)-benzene | SanDisk | 1460-1472 | 2.4E+05 | 1.2E+05 |
| 1,2,3-trimethyl-benzene | SanDisk | 812-814 | 8.1E+05 | 1.5E+05 |

Most VOCs present in the microSD group were found to be around one order of magnitude greater than the VOCs found in either of the microcontroller sample groups. This condition allows identifying the total surface area of the electronic device sampled as a key factor affecting the diffusion rates of VOCs into the headspace during the SPME (Pawliszyn, 2012). The total surface area of components shows that the surface area of the sampled microSDs (380.2 mm2) was slightly more than twice as large as the total surface area of the sampled microcontrollers (188.4 mm2). Differences among different ionization efficiencies can also account for the differences in the peak areas among different chemical compounds. However, the difference in surface area, age, and manufacturer of the electronic component are clearly identified as critical factors when comparing the peak areas for 1,3,5-cycloheptatriene, the only VOC marker identified as significant across all three sample groups. This compound, 1,3,5-cycloheptatriene, is a cycloolefin monomer commonly used as a polymerization retarder in the manufacturing process of cross-linkable resins widely found in electronic components due to their excellent electrical insulation properties, mechanical characteristics, and heat resistance (Sugawara, 2007).

Based on these findings, further work should be focus on quantifying significant VOCs as a function of the aging process under both (i) virgin and (ii) expected normal use technical conditions. This mapping, to be unique for each electronic component produced by each manufacturer, could potentially serve as the chemical fingerprint needed for unambiguously identifying counterfeit products present in the DoD logistics supply chain. Given the high resolution and repeatability achieved through SPME and GCxGC/EI-TOF-MS techniques, it is reasonable to expect that any use or modification by means of relabeling, repackaging, or refurbishing (Sood et al., 2011), will modify both the chemical fingerprint and the quantity of 1,3,5-cycloheptatriene of the electronic component allowing the identification of the counterfeited product before jeopardizing more complex electronic systems.

Conclusions

This study demonstrated a viable method for identifying VOC signatures of electronic components utilizing SPME and GCxGC/EI-TOF-MS. The high sensitivity of this sampling method allows for the identification of hundreds of potential VOC markers for any given electronic component. This methodology exhibited the possibility of differentiating electronic components that vary in their manufacturing, countries of origin, and production dates based on their VOC signatures. It is possible that other VOC markers can be distinguished by further optimizing the methods outlined above to capture more indicative VOC markers for additional electronic components. Table 3 outlined multiple VOC markers that were consistently identified in each sample group. These significant VOCs can serve as chemical fingerprints for future research focused on identifying VOC markers present in such devices. 1,3,5-cycloheptatriene was identified as a common significant VOC present in the 14-pin 16-bit Microcontrollers and 16 GB microSD here studied. Aging and surface area of the electronic component were identified as critical parameters affecting the final VOCs and their quantity, especially for 1,3,5-cycloheptatriene. Therefore, further work should be related to compare these findings to counterfeited products to evaluate the differences in terms of significant VOC markers and quantity of 1,3,5-cycloheptatriene as a function of aging, under virgin and expected normal use conditions.

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