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Aircraft Recirculation Filter for Air Quality and Incident Assessment

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The current research examines the possibility of using recirculation filters from aircraft to document the nature of air quality incidents on aircraft. These filters are highly effective at collecting solid and liquid particulates. Identification of engine oil contaminants arriving through the bleed air system on the filter was chosen as the initial focus. A two-step study was undertaken. First, a compressor/bleed air simulator (BAS) was developed to simulate an engine oil leak and samples were analyzed with gas chromatograph-mass spectrometry (GC-MS). These samples provided a concrete link between tricresyl phosphates (TCPs) and a homologous series of synthetic pentaerythritol esters from oil and contaminants found on the sample paper. The second step was to test 184 used aircraft filters with the same GC-MS system: of that total, 107 were standard filters and 77 nonstandard. Four of the standard filters had both markers for oil, with the homologous series synthetic pentaerythritol esters being the less common marker. It was also found that 90% of the filters had some detectable level of TCPs. Of the 77 nonstandard filters, 30 had both markers for oil, a significantly higher percent than the standard filters.

I. Introduction

THIS This study aims to investigate a potential methodology to diagnose the source of an air quality incident after cabin contaminants have dissipated and direct air sampling is no longer possible. Air quality incidents range from a serious smoke event in the cabin to a simple dirty sock smell [1]. Incidents such as a smoke-filled cabin

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occur at very irregular intervals, making direct sampling of cabin air both logistically and economically challenging. The vast majority of U.S. airliners use HEPA recirculation filters for cabin air, and these filters are highly effective at collection of solid and liquid particulates [2]. A possible approach would be to analyze recirculation filters in an attempt to identify the nature of an air quality incident. Primary technical challenges include residues from many hours of service have also been collected on the filters, and some of the target compounds are semi-volatile and could have vaporized from the HEPA filter over time as air continually flows through it.

Previous studies by Spengler et al.[3], and Nagda and Rector [4] reported a wide range of volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) in aircraft cabin air during aircraft operations. It should, thus, be assumed that residue collected on the recirculation filter is a complex combination of normally occurring materials (i.e., clothing fibers and bioeffluents) and "incident" material. Many types of incidents could be selected, but presence of engine oil particulates in the bleed air has been of considerable interest for many years, making it a logical choice [5,6,7,8,9,10,11]. The objective of this study is to show a link between contaminates measured on the filter and a type of air quality incident. If successful, techniques developed could be expanded and applied to other air quality incident types.

The link was investigated through a three-step process. First, a laboratory compressor/bleed air simulator was developed to establish a concrete link between contaminates in the oil and residue collected on the filters. The second step was analysis of 107 standard, used HEPA filters from aircraft. These filters typically had full service life and were from aircraft without chronic air quality complaints. This analysis established a background level for contaminates on the filters. Finally, 77 nonstandard filters were analyzed. A non-standard filter is a filter that was typically removed from an aircraft prior to its normal service life because of chronic air quality complaints or an unspecified air quality incident on the aircraft. In one case the airlines self-reported that the filters were removed after a smoke incident. The airlines were provided results of the analyses of the nonstandard filters without charge. No information was recorded on the type of aircraft the filters were removed from or the reason they were labeled as nonstandard. This anonymity increased participation in the study by the airlines. A comparison of contaminate concentrations in the standard and nonstandard pool provided some unique insight into the nature of air quality incidents.

Bartley et al. [12] conducted research along similar lines but did not focus on the filter as a collection point. They used an internally developed analytical method called direct filter injection/gas chromatograph (DFI/GC) to

measure volatile fractions of lubricating oil contaminations on aircraft environmental control system (ECS) surfaces. The group analyzed oils in both the main engine and auxiliary power unit (APU), determined characteristic traces for each, and found significant differences in the volatile traces. They then obtained wipe samples from the oil-contaminated part of the ECS and were able to determine the source of the contamination was the APU.

Lamb, McGonagle, Cowie, and Cherrie [13] also investigated the buildup of residue on the surface of aircraft. Their objective was to determine potential exposure to organophosphates in aircraft. They looked specifically at tri-n-butyl phosphate (TBP), tricresyl phosphate (TCP), butyl diphenyl phosphate (BDPP), and dibutyl phenyl phosphate (DPP). As part of the study, they showed TCP has a relatively slow decay time on porous materials by documenting almost no decrease in concentration of TCP after 21 days of exposure. It should be noted they did not flow air through this porous material during the exposure test. They found both aircraft and other vehicles had similar concentrations of TBP, BDPP, and DPP. Concentrations of TCP were found to be similar in control vehicles and offices, but were somewhat higher in aircraft although the difference was not statistically significant.

The first sections of this paper outline the facilities and methodologies used in the study. These sections include a broad description of the compressor/bleed air simulator and analytical methodologies used in the GC/MS measurements. The final sections present results of the BAS system, and analysis of standard and nonstandard filters.

II. Compressor/Bleed Air Simulator

The compressor/bleed air simulator (BAS) is an apparatus used to simulate cabin bleed air coming from a jet engine compressor with leaking oil seals. The system works by generating an aerosol stream with Laskin nozzles, and then compressing and heating it to the desired temperature and pressure. The particle-laden mixture is then injected into a cool air stream with an order-of-magnitude larger flow than the BAS mixture flow. The duct air is mixed and sampled downstream, simulating processes that occur in an aircraft cabin.

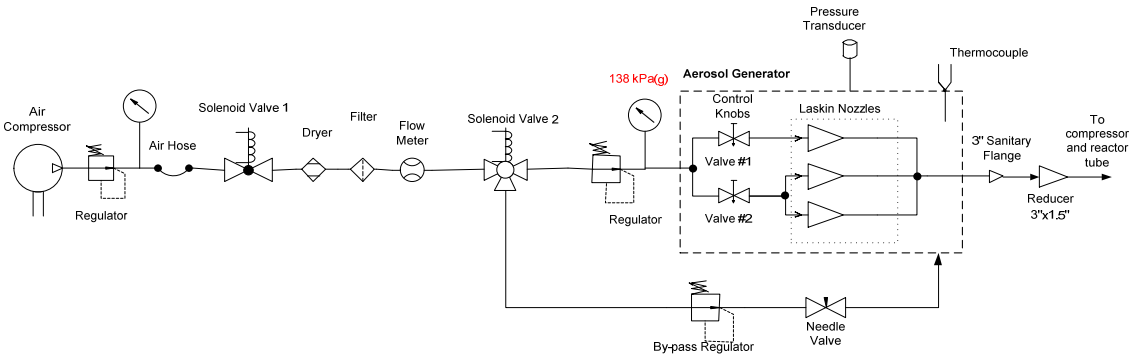

Fig. 1 Diagram of aerosol generator system.

Figure 1 illustrates the droplet generation portion of the BAS. An air compressor was used to pressurize the aerosol generator. This generator had three enclosed Laskin nozzles and two control valves that allowed one, two, or three nozzles to be active. Airflow can bypass the generator if no oil is desired in the flow or to purge the aerosol from the system during shutdown. Oil in the Laskin nozzle was changed at regular intervals to assure the SVOCs in it did not dissipate. The filter and dryer were also replaced as needed.

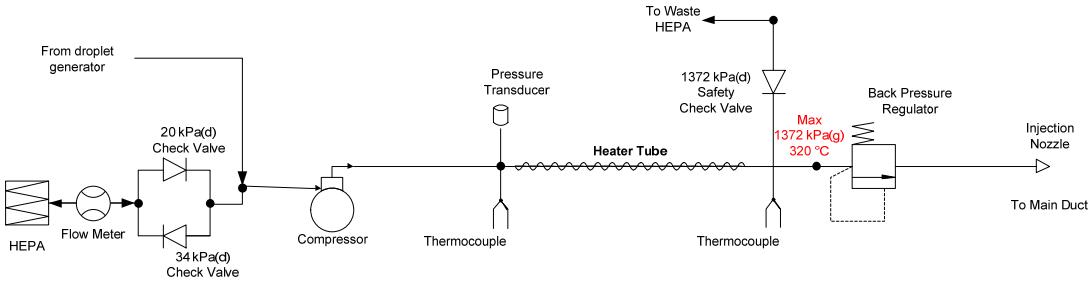

Fig. 2 Compressor and reactor tube assembly.

After the aerosol was generated, it flowed to the compressor and heater tube assembly (Fig. 2). At the secondary compressor inlet, two check valves installed in opposite directions kept the pressure of the aerosol line between 20 kPa above and 34 kPa below atmospheric pressure. A HEPA filter cleaned the air entering or removed oil from exhausted air to preserve the integrity of the aerosol sample. The aerosol then moved through the secondary compressor, which increased the pressure of the stream based on the setting of the backpressure regulator. After leaving the compressor, the flow entered the heater tube—a stainless steel tube wrapped with electric heaters. Four thermocouples were embedded under the heaters along the length of the heater tube, allowing wall temperatures to

be monitored. The heaters provided a pseudo-constant heat flux to the flow, which can be adjusted by changing the voltage on the heater via a variable transformer. A safety-relief valve prevented the pressure in this portion of the system from exceeding 1372 kPa(g). The safety valve exhausted to the outside of the building through a HEPA filter. After the flow was pressurized and heated, it was injected into an air stream in the main duct. Mixing plates were then used to thoroughly mix the two streams. The duct air was sampled downstream of the mixing plates.

The sampling was done with a vacuum pump that drew samples of the particulate-laden air through a 25-mm diameter HEPA paper sample. The sample paper was cut from a clean, cabin air recirculation filter. A three-piece plastic filter cassette was used to hold the filter media in place. An air flow meter monitored the sampled air flow rate and assured the samples were being taken at isokinetic conditions. Clear, flexible, sample transfer lines made of polyvinyl chloride (PVC) hose were used for connections to the various accessories.

III. Used-Filter Sampling

Standard and nonstandard filters were received from airlines in sealed plastic sleeves. Outer housing of these filters were removed in an environmental chamber fitted with both HEPA and electrostatic filters that keep airborne particulate levels to a minimum, reducing the chance of additional material landing on the filter. All tools used in the extraction of samples were dipped in isopropyl alcohol and wiped with a paper towel, or wiped down with isopropyl alcohol. Once the outer housing was removed, smaller panels of the filter were removed, sealed in plastic, and placed in a freezer until delivered to the laboratory for analysis.

IV. GC/MS Procedures

Samples generated from the BAS system, and those cut from standard and nonstandard filters, were analyzed using GC/MS. GC/MS is useful for identifying and determining the concentration of chemical compounds. It is obvious a vast range of compounds are expected on a used HEPA filter. The initial goal was to determine what compounds in the oil would provide unique and reliable markers. Based on analysis of commercially purchased jet oil, TCPs and synthetic fatty acid esters of pentaerythritol and dipentaerythritol that comprise the majority of jet oil [14] were thought to be the most robust markers.

The BAS samples and those extracted from the used filter were analyzed by the same methodology. For the filter samples, a 10-cm by 10-cm square was placed in a glass vial. Forty ml of hexane was added and the filter was sonicated for 30 minutes to facilitate penetration of the hexane into the filter and dust. The liquid was decanted and concentrated to approximately 15 ml and then centrifuged. The supernatant was then transferred to a clean vial,

leaving behind residual filter material particles and dust that did not dissolve in the hexane. The liquid was then concentrated to 0.5 ml. A 1 µl aliquot was injected into an Agilent GC/MSD operated in the electron impaction mode and run in the SIM mode at 368amu, which is sensitive to tricresyl phosphate, and then a second injection was done with the GC/MS operated in the SCAN mode to both confirm the peak identification of the TCP and for the synthetic jet oil pentaerythritol triesters and tetraesters analysis.

Jet oil samples contained four TCP peaks at nominal retention times of 22.0, 22.4, 23.0, and 23.4 min, for the GC conditions used (Fig. 3). These peaks correspond to meta TCP (M-TCP), two isomers of TCP for which commercials standards are not available so positive assignments could not be made (Unk1-TCP and Unk2-TCP), and para TCP (P-TCP) (Fig. 4). The unknown isomers were quantified using the calibration table prepared for P-TCP, since their mass spectra matched that isomer well. Based on the retention time of ortho TCP in the standard (Fig. 4) it was determined that ortho TCP was not present in the commercial oils tested, though it was present in many of the filter samples, thus ortho TCP was not included as a marker for jet oil. Identification of the peaks was based on both matches in retention time and the mass ion fragment pattern. For TCP, key ion fragments were at 369, 367, 369, 165, 91, 198, and 107 amu. Other compounds co-elute with these peaks, which can include the 368 amu ion, so a spectrum subtraction was done to confirm each peak and each mass spectrum was reviewed.

Synthetic pentaerythritol triesters and tetraesters were present in the jet oil sample with an excess of 10 peaks being present starting at retention times in the 20-minute range for GC conditions used. Their mass spectra pattern typically had a base peak at 85 amu and strong ion fragments at 113 and/or 91amu, plus additional ions at 127, 156, and/or 184 amu. These ion fragments are consistent with the mass spectra of a homologous series of pentaerythritol triesters and tetraesters reported to be synthetic turbine oil [15,16]. Presence of these ion fragments in a GC peak with the absence of ion fragments at 71, 99, and 141 amu, common ion fragments in long-chain hydrocarbons also commonly found on the filter extracts, were used to confirm if a peak in the chromatograph of the filter extract was a synthetic pentaerythritol trimesters rather than a saturated, long chain-petroleum hydrocarbon, which elute as similar retention times.

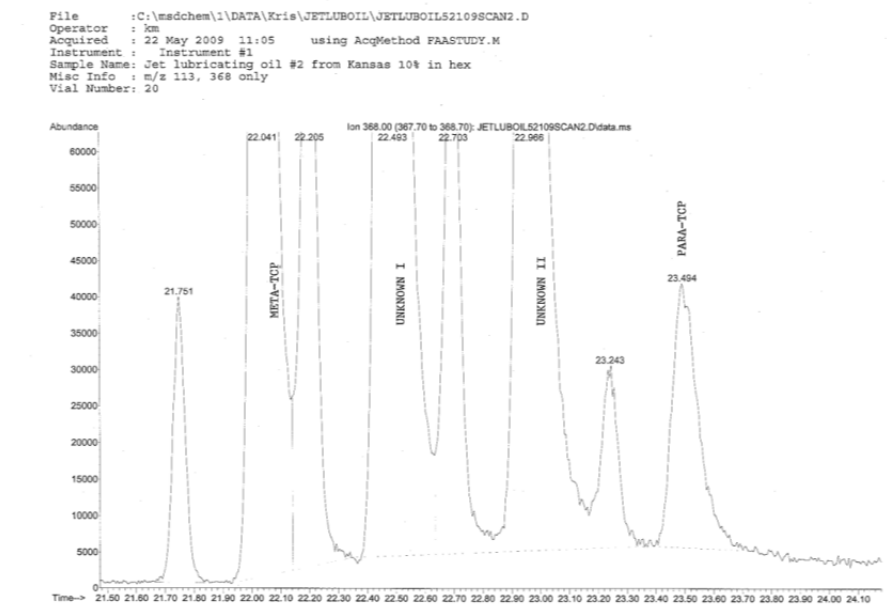

Fig. 3 GC/MS analysis of engine oil.

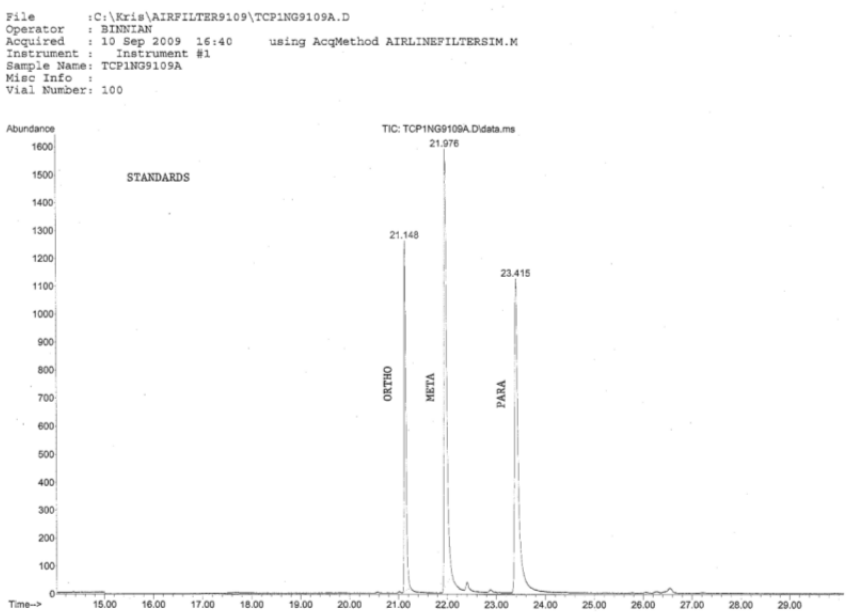

Fig. 4 GC/MS calibration against standards.

It is known that TCPs are present in engine oil, but obtaining a consistent set of isomers was not always possible when testing different jet oils. Based on multiple samples from different oils, M-TCP, P-TCP and the two other TCP peaks were consistently seen in the oil. Although they could not be identified against a standard, presence of these peaks is nonetheless useful as markers for engine jet oil. In the final analysis, four TCP compounds (M-TCP, P-TCP, Unk1-TCP, and Unk2-TCP) and peaks associated with the synthetic esters were identified in the jet oil as potential markers.

V. BAS Results

The goal of the BAS simulation was to mimic conditions of an air/oil mixture in the engine before it enters the bleed air system. Table 1 outlines a typical pressure and temperature range in an engine throughout a flight. The engine has both high pressure and temperature, and low pressure and temperature combinations. The range of conditions shown in Table 2 was selected for BAS experimental runs to include both the high and low combination. The objective of the tests was first to establish a concrete link between contaminants in the oil and contaminants found in particulates collected by HEPA filters. Then the effect of pressure and temperature on the chemistry and morphology of particulates collected was evaluated. For example, pyrolysis may change the nature of the particulates collected by driving the TCP to the gas phase and thus, potentially avoiding any collection on the HEPA filter.

Table 1 Expected engine conditions [17]

Simulated mode of aircraft engine operation	Pressure kPa	Temperature ^{o}C
Initial descent from cruise	200	185
End of descent	460	230
High- to low-pressure switchover	480	280
Cruise	690	250
Top climb	690	310

Table 2 High- and low-pressure test conditions in BAS

Low Pressure		High Pressure	
Pressure kPa	Temperature ^{o}C	Pressure kPa	Temperature ^{o}C
200	130	680	230
200	185	680	250
200	230	680	280
200	250	680	280
200	275	680	310

Table 3 Analytical results from BAS

Pressure	Temp	M-TCP	Unk1-TCP	Unk2-TCP	P-TCP	oil pattern?
kPa	^{o}C	ng	Ng	ng	ng	
209	136	0	0	0	0	NO**
203	130	200	330	260	90	YES
202	186	190	300	230	80	YES
205	231	670	1140	830	200	YES
212.	252	700	1300	1030	250	YES
205.	277	140	220	190	80	YES
689	229	160	260	210	80	YES
711	251	900	1640	1250	280	YES
709	279	250	440	350	110	YES
699	282	700	1270	960	210	YES
719	311	640	1160	890	220	YES
Engine oil in mg/ml		9	16	11	3	YES

**Oil generator isolated. Control sample should be blank.

Table 3 presents the GC/MS analysis of the filter samples generated by the BAS system. The table also shows actual pressure and temperatures obtained in the facility, which are a close match to the desired conditions. The table also includes analysis of a control sample taken with the BAS running, but with the Laskin nozzle isolated. The concentration reported is ng of isomer collected on the 25-mm-diameter HEPA filter sample. It is clear dilute particulates collected from a cabin could contain TCP traces and synthetic jet oil under all pressure and temperature combinations.

The second goal was to access the potential effect of pressure and temperature on the TCP and synthetic jet oil esters signature. It is important to note a uniform sampling time was used for each run represented in Table 3. In addition, the Laskin nozzle system is thought to generate particulates at nearly a constant mass flow for these experiments. Despite these constants, the concentration of TCP in the collected samples appears to have a significant variation with no clear trend appearing with changes in pressure and temperature. One possible explanation for the variation in the results is that flow hydrodynamics in the simulator subsystem are highly variable rather than alterations in chemical changes due to pressure and temperature. Specifically, the reactor tube has significant length and high velocity flow. Thus, it is expected that oil droplets will impact the wall and re-shear into the flow. This

time-dependent process may have an impact on the total concentration of TCP seen on the filter sample. A second cause for variation with temperature and pressure is the degree of pyrolysis of the oil, which will vary, resulting in changes in the amount of original oil collected. Even with this variation in absolute concentration, data from the BAS is useful because the relative concentration of the TCP components is likely of more interest.

It is known that different engine oils have different formulations and different relative concentrations of TCP constituents. In addition, tricresyl phosphates have a number of sources such as a plasticizer in nitrocellulose, acrylate lacquers, varnishes, polyvinyl chloride, and as a flame retardant in plastics, thus complicating the analysis on actual filters. These materials can be used in the interior of aircraft and brought onboard by passengers. When they degrade or abrade from contact, dust can be formed that can be resuspended into the air and contribute to loading on the airplane HEPA filters. Linking individual TCP compounds in samples collected from an aircraft to engine oils may be complicated due to multiple sources of TCP within an aircraft. However, examination of the ratios and presence of all components present in the jet fuel oil could still be a useful indicator of contamination by jet fuel oil. It is also possible the physical process that occurs in the engine and bleed air system may alter the relative concentration of the TCP isomers, thus changing their signature. Figure 5 addresses this question by presenting the ratios of the isomers of TCP using M-TCP as the denominator. Solid lines on the figure represent the ratio of the TCP isomers found in the base oil. It can be seen that Unk1-TCP, Unk2-TCP and P-TCP have a relatively constant relative concentration over the range of pressures and temperatures tested. The low concentration of P-TCP does cause a higher relative percent variation. The conclusion is that M-TCP, Unk1-TCP, and Unk2-TCP exit at nearly constant relative concentrations in contaminants that would be collected by cabin air filters and thus may be potentially robust oil markers.

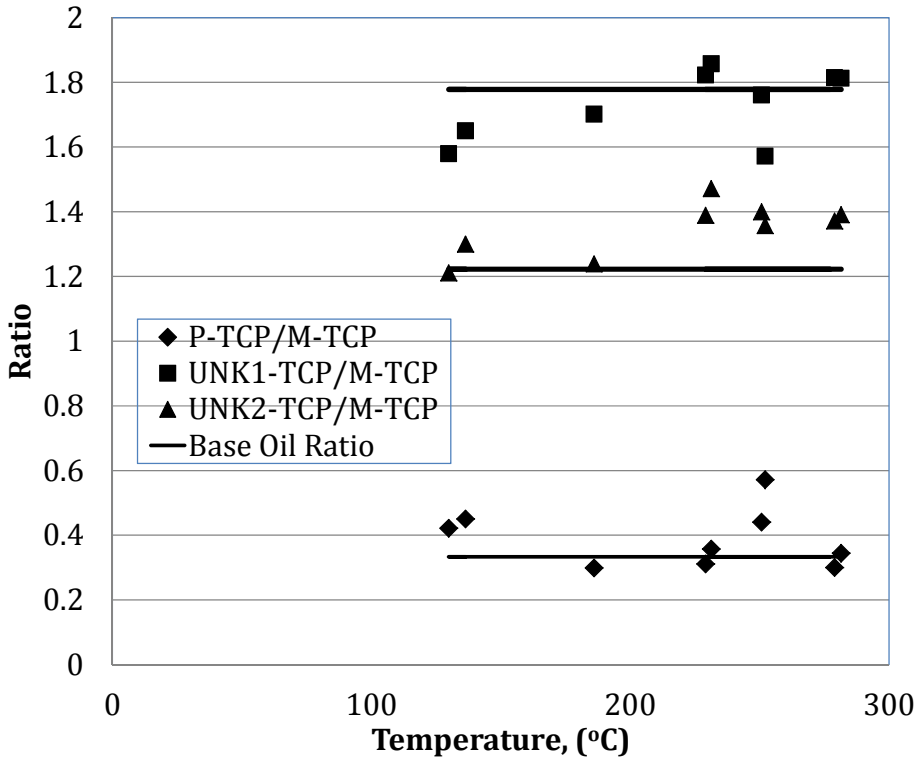

Fig. 5 Relative ratio of TCPs

VI. Filter Analysis

GC/MS analyzed 107 standard filters and 77 nonstandard. Table 4 presents general results of this analysis. It is interesting to note 89 to 95% of filters had detectable levels of M-TCP. Significantly fewer filters had all four TCP markers. For standard filters, 28% had all four TCP markers, while this increased to 45% in nonstandard filters. Standard filters had normal service life, while many of the nonstandard filters were removed early in the service life. TCP is an SVOC, and thus it will evaporate over time. Insufficient information exists at this time to estimate decay curves for TCP on the glass fiber matrix of the HEPA paper, and this was a significant complicating factor in the current study. The combination of TCP and synthetic pentaerythritol triesters and tetraesters is the most robust measure, and only 3% of standard and 30% of nonstandard filters had both markers. Although incident type was not recorded when the nonstandard filters were collected, a significantly higher rate of jet oil signature was seen.

Table 4 Analysis Results

	Standard Filters	Nonstandard Filters
Presence of M-TCP	95%	89%

Four TCP markers	28%	45%
Synth. lub. marker	4%	31%
Both markers	3%	30%

Table 5 shows approximate concentrations of TCPs found by GC/MS analysis. Average concentration of TCPs in nonstandard filters was much higher than in the standard filters, being statistically different for Unk1-TCP and Unk2-TCP at $p<0.05$ and nearly so for M-TCP, and the standard deviation (STDev) was much larger. Careful inspection of the data showed the large standard deviation was driven by a couple of high concentration points in the nonstandard filter set.

Table 5 Average TCP concentration in used filters

	Standard Filter	Nonstandard Filters	T-Test
M-TCP	11.5 ng/cm^2, STDev = 27	23.5 ng/cm^2, STDev = 48	p=0.053
Unk1-TCP	13.6 ng/cm^2 , STDev = 37	29.2 ng/cm^2, STDev = 58	p=0.04
Unk2-TCP	4.8 ng/cm^2 , STDev = 18.9	12.9 ng/cm^2 , STDev = 29	p=0.033
P-TCP	0.7 ng/cm^2 , STDev = 2.8	1.7 ng/cm^2 , STDev = 3.7	p=0.123

An alternate view of concentration distributions in the filters is shown in Fig. 6. A total of 107 standard filters and 77 nonstandard filters are represented in this figure. The histogram shows the percent of total filters that appears in each bin. For example, the graph shows approximately 36% of standard filters had a concentration that falls in the 5 to 10 ng/cm^2. It is interesting that nearly 6% of the standard database had levels of M-TCP of under 1 ng/cm^2, while in the nonstandard filters about 11% fell in this bin. It is also apparent the nonstandard filter set had more filters that fell in the midrange concentration of 10 to 60 ng/cm^2. Finally, five of the nonstandard filters had very large concentrations of more than 300 ng/cm^2, while only three of the standard filters fell in this bin. Figure 7 shows similar information for Unk1-TCP. Similar results were found for all the TCP isomers.

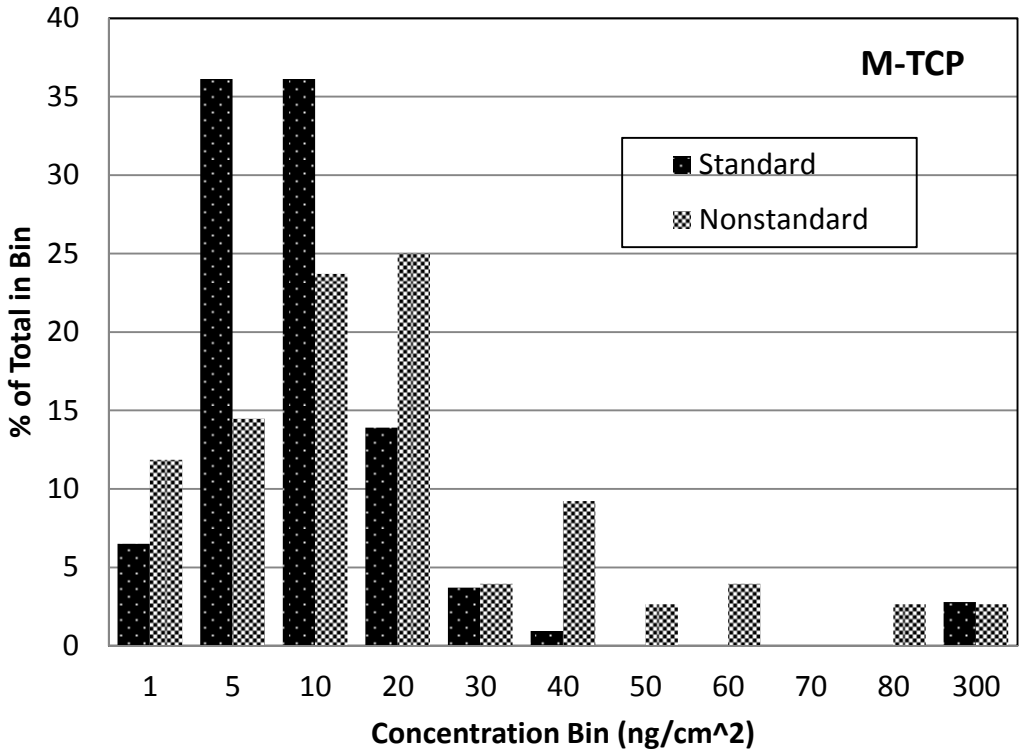

Fig. 6 Histogram of M-TCP concentration for standard filters.

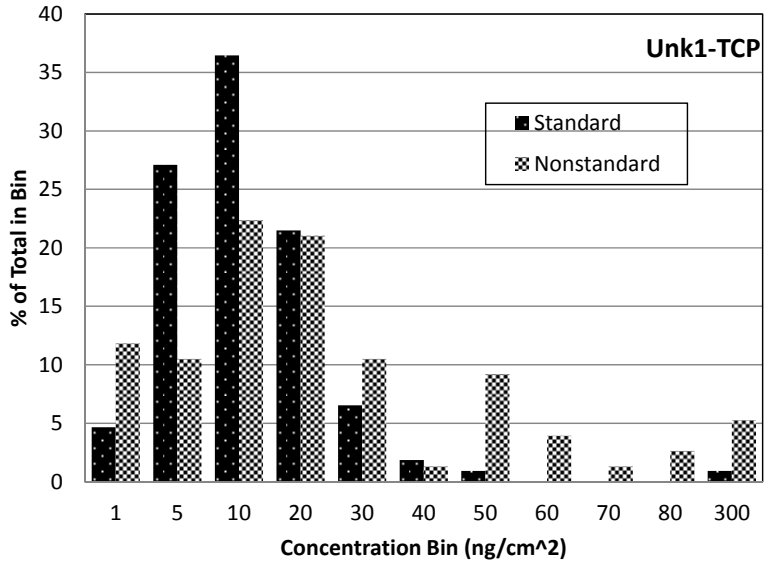

Fig. 7 Histogram for Unknown1-TCP in standard and nonstandard filters.

Standard filter data showed the presence of TCP is not rare and thus may not be a suitable marker. Different brands of jet oils have different formulations, and the lubricant used in the engine and APU are often different as documented by the study of Bartley et al. [12]. The question remains unanswered whether analysis of specific components in jet oil from an aircraft engine could be detected on the filter from that aircraft. Synthetic esters used in jet oil measured on the filters are different from natural oil used for lubrication in motors. It is possible the relative concentration of TCP in different jet oil formulations could be detected on a filter. Fig. 8 and 9 illustrate the relationship between Unkn1-TCP and M-TCP on the filters and in the BAS oil. In all cases, the M-TCP concentration on the filter is at a higher relative concentration than found in the BAS oil. Relative ratio of the two TCPs is nearly constant, regardless of the concentration level on both set of filters which implies a common source. Fig. 10 and 11 show similar results for the other TCP constituents. Relative concentrations of the Unk2-TCP and P-TCP are also strongly correlated with M-TCP but at a significantly different relative concentration than the BAS jet oil we measured. It should be noted the BAS jet oil was one of the most common jet engine lubricants on the market. However, variations in the ratios of TCP isomers across different production lots of the jet oil are unknown.

The strong correlation shown in the figures suggests the TCPs have a common source or a near common mixture of sources. However, it is still possible, though not likely, the jet oil selected in this project produced TCP signatures seen on the filters. Careful application of a constituent mass balance reveals possible loading and decay scenarios where the signatures seen could be produced by the jet oil, and thus it can't be eliminated without further study.

The general method outlined in the paper appears to hold promise, but additional work in which HEPA filters are removed and analysed after a known jet oil leak into the bleed air should be analysed to confirm this is a practical approach. The high rate of TCP occurrence suggests it alone is not the best marker for jet oil and additional signatures are needed. Presence of the homologous series of pentaerythritol triesters and tetraesters are potential signatures for jet oil. Additional additives in jet oil, such as N-phenyl-1-naphthylamine, may be useful to identify whether jet oil has been released into the aircabin and collected on the HEPA filter. For other types of air quality incidents, success can be achieved if unique signatures can be identified. An ancillary conclusion of this study is that TCP at some level is relatively common in aircraft cabin air. No attempt was made in this study to assess the level of exposure reflected by the TCP presence measured.

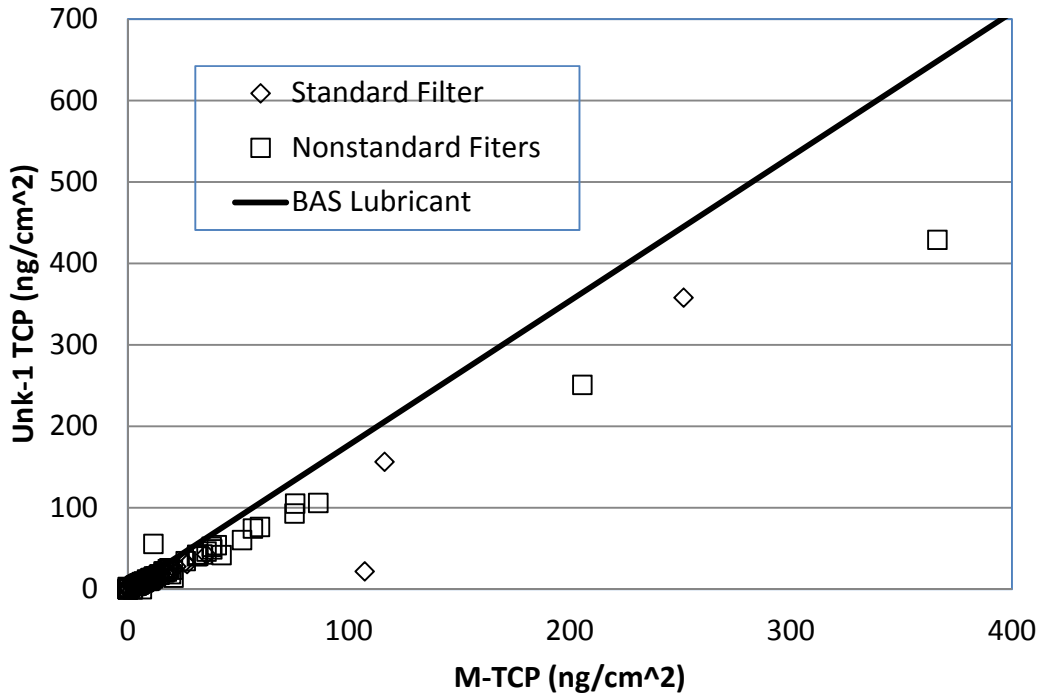

Fig. 8 Unknown 1 versus M-TCP concentration standard and nonstandard filter.

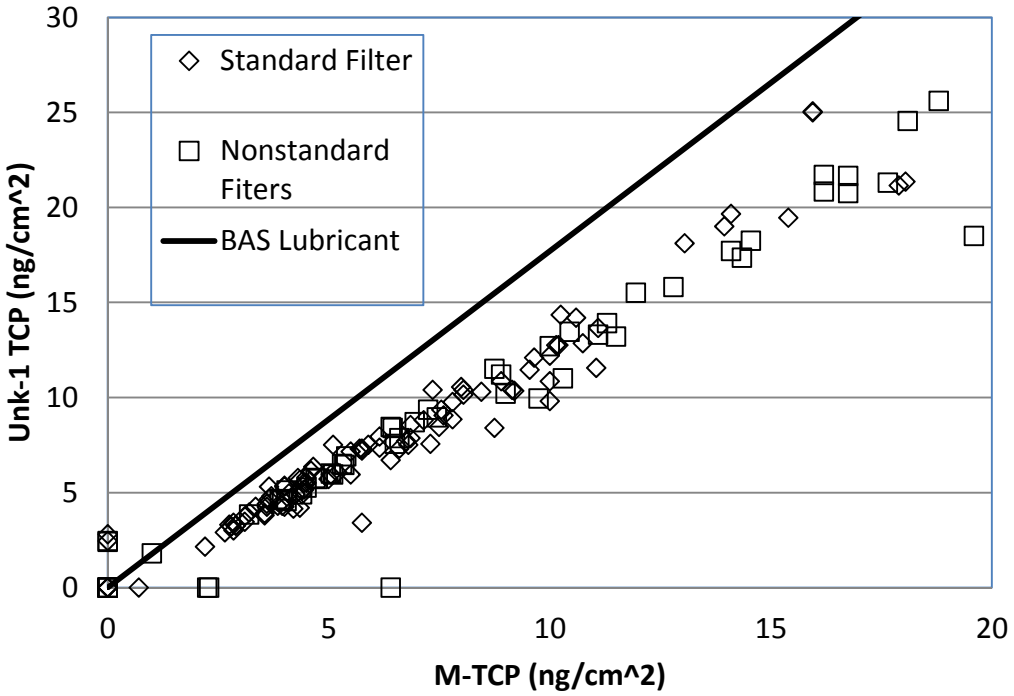

Fig. 9 Unknown 1 versus M-TCP concentration standard and nonstandard filter.

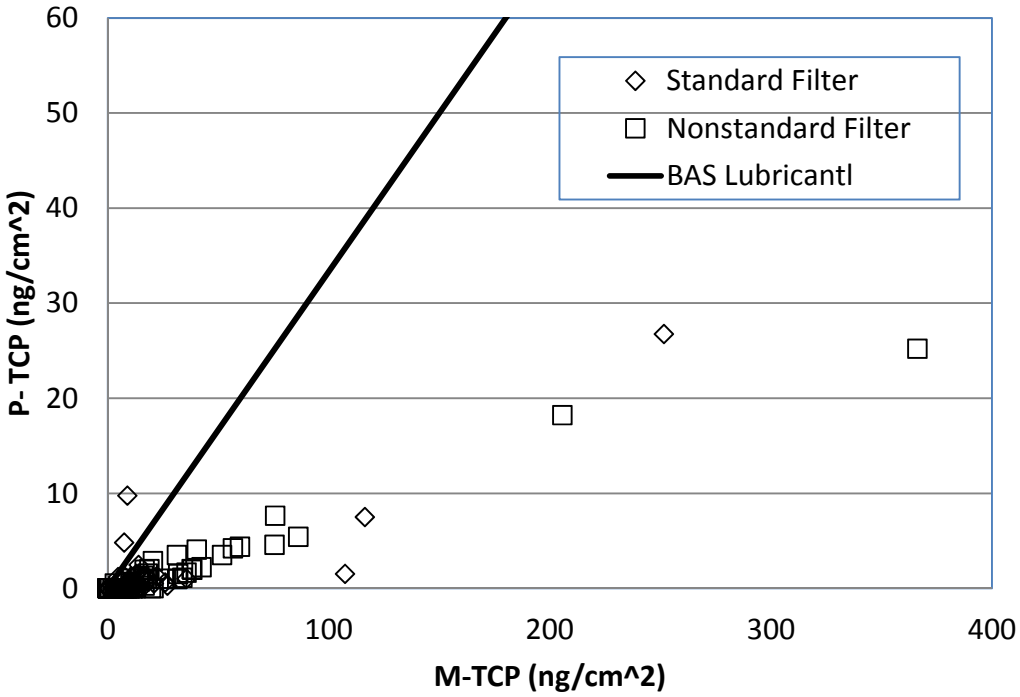

Fig. 10 P-TCP versus M-TCP concentration in standard and nonstandard filter.

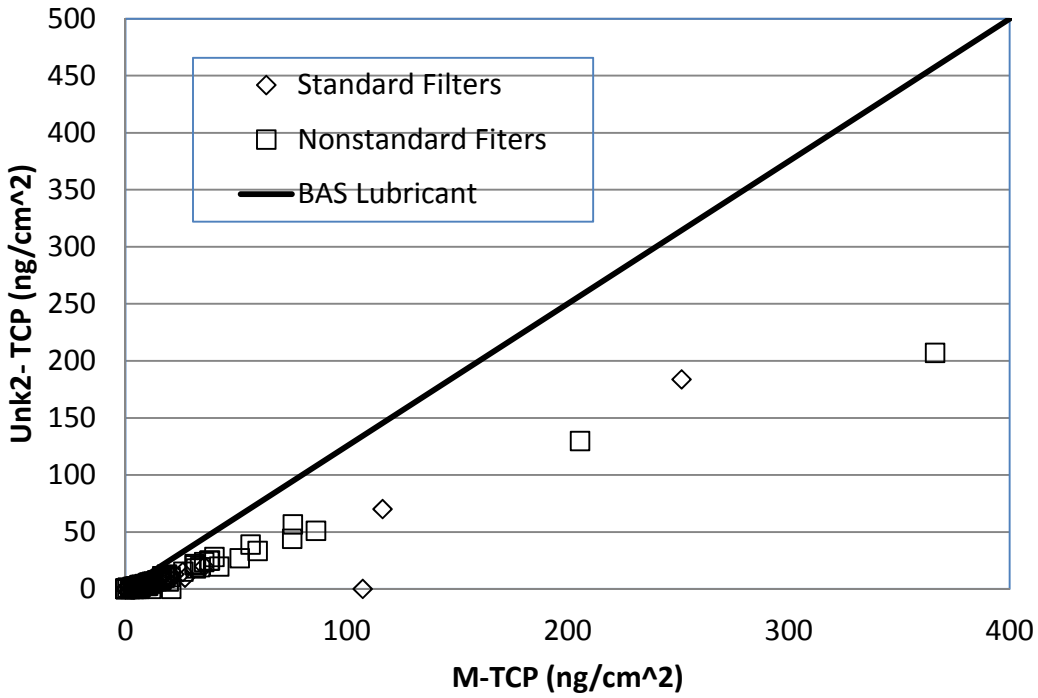

Fig. 11 Unk2-TCP versus M-TCP concentration in standard and nonstandard filter.

VII. Conclusion

The project had two overarching objectives. The first was to determine if recirculation filters from an aircraft can be used to identify the nature of an aircraft air quality problem. As an example of this application, the project focused on bleed air contamination by engine oil, but the techniques could be used for other potential contamination. The second objective was to develop a deeper scientific understanding of bleed air contamination caused by engine lubricant.

Detecting engine oil on HEPA filters proved to be a challenging scientific task. A homologous series of synthetic pentaerythritol triesters and tetraesters, and four TCP isomers were identified in the jet oil and seen on filters. The BAS system proved both contaminates are collected on HEPA filters, and pressure and temperature in the engine do not alter the relative concentrations of these constituents to any great extent. A substantial number of standard and nonstandard filters had measurable TCP loading. Only 4% of the standard filters and 30% of the nonstandard filters had both markers.

This method of documenting air quality incidents holds promise but much additional work would be necessary to provide a practical results. Success is highly dependent on identifying unique signatures for an air quality incident.

VIII. Acknowledgments

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