

Plasma Abatement of Volatile Organic Compounds in Nitrogen and Carbon Dioxide Background Gases

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Thermal conversion of crew waste to gases that can be vented from spacecraft for mass reduction has been investigated by NASA and others within the Trash-to-Gas program. One concern with operating thermal waste processors onboard spacecraft is the potential for contamination of cabin atmosphere with volatile organic compounds (VOCs) from gaseous reactor effluent. Recent efforts at Kennedy Space Center's Applied Chemistry Laboratory have developed and reported on a recirculating plasma gas system for the rapid reduction of VOCs. In this case, a direct-current thermal plasma was used to break down VOCs into simpler, and preferably more benign chemical species. Previous work reported the application of this system on gases with carbon dioxide as the background. Here the authors report the results of plasma VOC abatement with an improved recirculation system. The VOCs studied included a set in a nitrogen background (benzene, pentane, ethanol, and Freon-113) and a set in a carbon dioxide background (benzene, pentane, ethanol, and acetone). Each of these analyte species is included in NASA's Spacecraft Maximum Allowable Concentrations list. The plasma abatement system showed a greater than 99.999% reduction in VOC concentrations within 2 minutes of plasma treatment for the nitrogen background, and greater than 95% VOC reduction within 2 minutes for the carbon dioxide background. This is a significant improvement over what was reported previously. All analytes obeyed first-order rate kinetics in agreement with the previous work.

Nomenclature

cm ²	=	square centimeters
CO	=	carbon monoxide
CO ₂	=	carbon dioxide
D	=	diffusivity
EMI	=	electromagnetic interference
GC-MS	=	gas chromatography mass spectrometry
HCN	=	hydrogen cyanide
HV	=	hand valve
<i>k</i>	=	rate constant

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L	=	liters
min	=	minutes
min ⁻¹	=	per minute
mL	=	milliliters
N ₂	=	nitrogen
O ₂	=	oxygen
OSCAR	=	Orbital Syngas Commodity Augmentation Reactor
ppm	=	parts per million
psig	=	gauge pounds per square inch
PT	=	pressure transducer
QD	=	quick disconnect
RV	=	relief valve
s ⁻¹	=	per second
slpm	=	standard liters per minute
SMAC	=	Spacecraft Maximum Allowable Concentrations
t _{1/2}	=	half-life
TC	=	thermocouple
VOC	=	volatile organic compound
μL	=	microliter
°C	=	degrees centigrade

I. Introduction

ONE of the most critical aspects of crew safety aboard spacecraft is the atmosphere in which they live and work. With no natural means of air ventilation and recycling, the closed volume of a spacecraft has the potential to retain and concentrate airborne contaminants such that they may become a hazard to the crew. Thus much effort has been applied toward methods of either physically removing hazardous materials from the cabin atmosphere (e.g., filtering) or chemically converting it to a benign form (e.g., catalysis, combustion, etc.).¹⁻⁴ The authors have recently reported on the use of a plasma for the reduction of several potentially hazardous gaseous compounds within a carbon dioxide matrix.⁵ In that work the authors reported rapid plasma decomposition of several test compounds including benzene, acetone, ethanol, and pentane as representative of different chemical types (aromatics, carbonyls, alcohols, and aliphatics, respectively). Here the authors describe the continuation of this work to include plasma treatment in a nitrogen matrix, as well as processing of effluent gases from the pyrolysis of simulated crew trash.

In this work the authors included an additional target compound, namely 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113). As with the other target compounds in this and the previous study, Freon-113 appears on NASA's Spacecraft Maximum Allowable Concentrations (SMAC) list.⁶ While Freon-113 represents an important class of compounds for spacecraft air quality, it was avoided in the previous study due to the potential formation of phosgene, a very hazardous gas that has been used as a chemical warfare agent.

Additionally, the authors report here experiments performed with opposite polarity. While the previous study utilized only a negative plasma electrode bias (relative to the grounded system exterior), the work presented here also includes results from the application of a positive plasma electrode bias.

II. Methods and Procedure

While the experimental apparatus has been described in detail previously, the principal components are reproduced here for convenience.⁵ However, it should be noted that one important difference between the two systems is that the recirculating pump was upgraded from that used in the previous work. As described there, the recirculating pump suffered from significant electromagnetic interference (EMI) from the plasma source which degraded its efficiency. The pump described below for use in this study displayed significant improvement in flow efficiency with little EMI. As such, the target gas concentrations decreased much more rapidly than in the previous work. Therefore, runs were shortened with samples collected at times of 0 min, 0.5 min, 1 min, and 2 min (as opposed to 0 min, 1 min, 2 min, 5 min, 10 min, and 15 min for the previous study).

A schematic of the plasma recirculating system is shown in Figure 1. As shown and noted in Figure 1, the primary components of the system are a KNF PU3243 recirculating pump with a rate of 15 slpm (A), an Entech™ 100 mL gas sample canister (B), a Swagelok™ 500 mL in-line tank (C), a 103 L lecture bottle with a test gas standard (Boggs Gases, Titusville, FL) and regulator (D), a plasma chamber (E) with a thermocouple (TC) on the outside wall and a

quartz view-port (F), a tube baffle (G) to collect potential soot produced, and a 10 mL sample tube (H) to periodically collect aliquots of gas sample for analyses via a gas chromatograph mass spectrometer (GC-MS, see below). Also noted in Figure 1 are various hand valves (HV, Swagelok™), pressure transducers (Omega, Inc. MMA050V10P4J6T4A5CE), quick disconnects (QD, Swagelok™), and a pressure relief valve (Apollo RVA 15-15-112); some of these components will be referenced in the procedure described below.

To process a test gas, the system was first purged by filling with either CO₂ or N₂ gas (through QD1) to 25 psia (as measured by the PTs), and then evacuated with all HV's open (also through QD1); the purge/evacuation was performed a minimum of three times. The same purge/evacuation procedure was performed for two Entech™ canisters and four 10 mL sample tubes. Next, the system was charged with test gas to 25 psia (typically 100 ppm of the analyte in a CO₂ or N₂ balance) using a gas standard from a lecture bottle (D) also through QD1, with all HV's open except HV2, HV5, and HV6, which were closed. The system charge was performed with one of the Entech™ canisters in place such that the Entech™ canister was also charged with the test gas; this sample was subsequently analyzed as the start (T = 0 min) sample. The T = 0 Entech™ canister was then secured by closing its inherent valve and then replaced with the other evacuated Entech™ canister (which would eventually be used to collect the T = 5 min gas sample, see below). After charging, HV1 was closed, as were HV5 and HV6; the rest of the HV were opened. Next, the recirculating pump (A) was turned on, forcing the test gas through the system (in the direction of the blue arrow in Figure 1). The plasma (E) was then turned on at the same moment that a timer was started. The plasma was

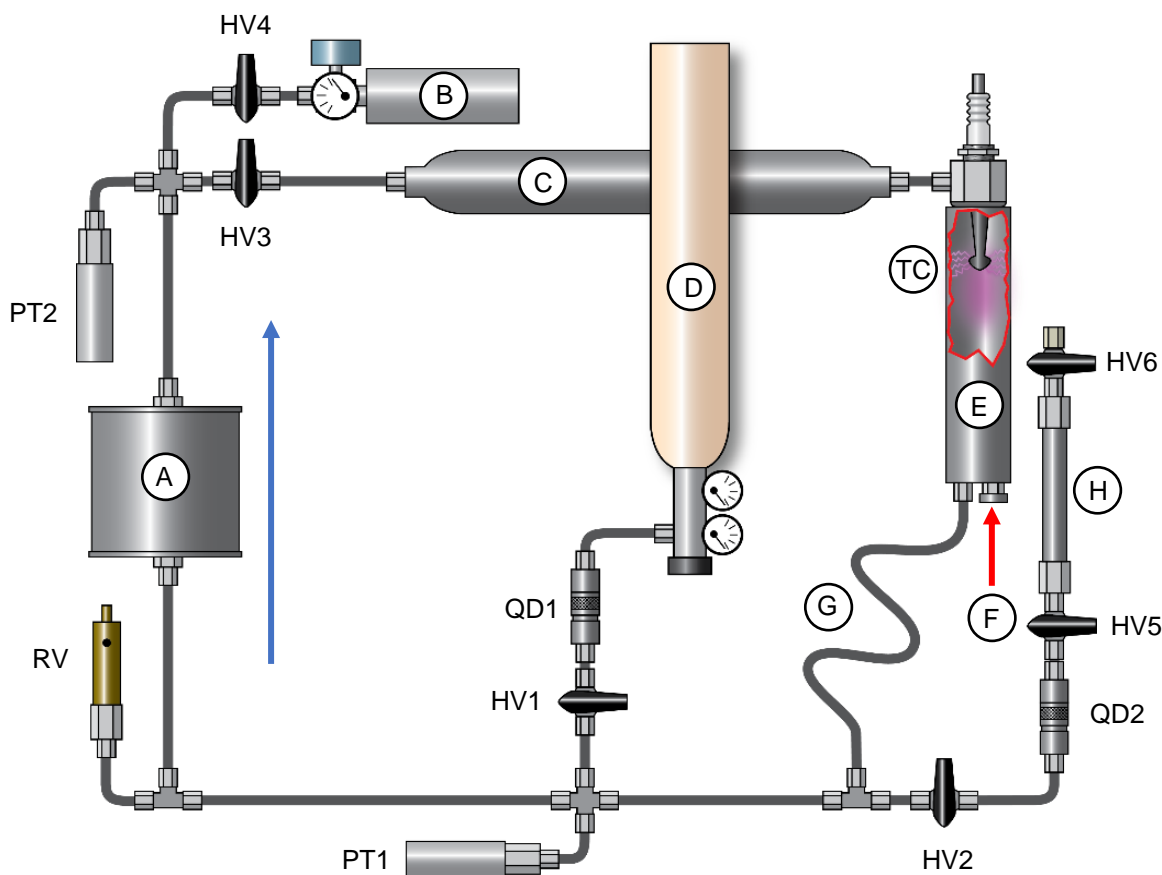


Figure 1. A schematic of the recirculating plasma gas system. In this diagram HV = hand valve, PT = pressure transducer, QD = quick disconnect, and RV = pressure relief valve. Components of the system are as follows: A = recirculating pump, B = 100 mL Entech™ gas sample canister, C = 500 mL in-line tank, D = supply gas lecture bottle with regulator, E = plasma chamber and system (with a cut-away view showing the plasma electrode), F = plasma chamber viewport, G = baffles, and H = 10 mL sample tube. The flow direction of the recirculating pump is noted by the blue arrow.

a DC arc plasma powered by an Applied Technologies Corporation Plasma Pilot PD-E-051 high voltage DC power

supply. The discharge arrangement was a DC arc plasma over a ~3 mm gap with gas flow entering and then performing a 90° turn through the plasma discharge. The electrode was a modified spark plug with a prolate spheroid attachment on the end to assist with heat distribution. A visual inspection was made through the viewport (F) to ensure that the plasma was present.

In general, the appearance of the plasma was identical for all analytes tested with the color produced according to their supply gases – in this case the primary gas was either CO₂ or N₂. It is also important to keep in mind that the concentrations of the VOCs were low enough that they would not appreciably contribute to the visual appearance of the plasma. The TC output was recorded before, during, and after each plasma exposure to provide a measure of the temperature resulting from the plasma itself. Note that the EMI from the plasma caused significant noise in the PT signals, thus not allowing pressure data collection during the runs. However, when the plasma was turned off, the system pressures were measured to be consistent with the starting pressures with some loss due to the sample tube collections. The sample tubes (H) were used to collect gas aliquots at plasma exposure times of 0.5 and 1 min. This was accomplished by opening HV5 for 5 seconds, then closing HV5 followed by closing HV2. The 10 mL sample tube was then removed at QD2 and replaced with the next (evacuated) 10 mL sample tube, followed by opening HV2. After an exposure of 5 minutes, the plasma was switched off. Then HV4 and the Entech™ valve were opened, and HV 3 was closed in order to force the gas into the Entech™ canister to a pressure of about 10-15 psig (as read on the Entech™ pressure gauge); the sample in the second Entech™ canister thus represented the 5 min exposure time. The Entech™ valve was then closed as HV3 was again opened, and the recirculating pump (A) was turned off, thus completing the plasma exposure. The Entech™ canister was removed for subsequent analysis. Also, the recirculating system was again purged three times with CO₂ or N₂ (as appropriate for the balance gas of the subsequent test gas) in preparation for its next run.

All collected samples were then analyzed with an Agilent™ model 6890N gas chromatograph with an Agilent™ model 5973 mass spectrometer (GC-MS). Gas samples of 250 µL were collected into a 1 mL gas-tight syringe from the sample tubes through septa that were connected exterior to HV6 (shown by the taupe-colored nut by HV6 in Figure 1). The small head-space between HV6 and the septum remained evacuated during sample collection. HV6 was then opened (thus filling the head-space with analyte gas) immediately before sample collection and analysis via GC-MS. The gases were collected from the Entech™ canisters through a septum that was attached directly to the canister Swagelok™ fitting (after purging the head-space with analyte gas). The 250 µL samples were directly injected into the GC-MS from a 1 mL gas-tight syringe with a split injection. The chromatograph used a DB-624 fused silica GC column. The temperature profile started at 35°C for 5 min, then ramped to 170 °C over 20 min where the oven temperature was held for a total run time of 35 min. The mass spectrometer used electron ionization (EI) with a quadrupole mass analyzer collecting from $m/z = 33$ to 450. Quantification was performed using the Agilent™ quantification tool with the peak area of the most abundant non-interfering ion for each analyte. For each run, the T = 0 min sample was used for a single-point calibration at 100 ppm.

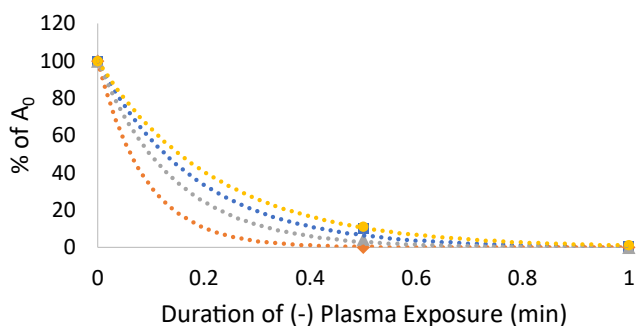
The T = 0 min and T = 2 min samples (i.e., the start and end samples collected in the Entech™ canisters) were also analyzed for primary gases (CO₂, CO, and O₂) with an Inficon™ Fusion micro-gas chromatograph (after sample collection for GC-MS). Here the sample canisters were connected directly to the GC input. External standards (Boggs Gases, Titusville, FL) were used for calibration.

This same procedure was used for the plasma treatment of effluent gases collected from the pyrolysis of crew waste simulant. The waste simulant was identical to that used in the Orbital Syngas Commodity Augmentation Reactor (OSCAR) described previously.⁷⁻¹⁰ In this case, the waste simulant was loaded into a stainless-steel tube which was placed into a tube furnace and the temperature ramped up to 600°C while nitrogen was flowed through it. The resulting effluent gases were collected and treated as stated above.

III. Results and Discussion

Figure 2 shows the results for the CO₂ gas background. This was performed for comparison to the previous study. Recall that Freon 113 was not treated with the CO₂ background to avoid the potential formation of phosgene as mentioned above. Figure 2(a) shows the average ppm of each analyte as a function of duration of the plasma treatment. The average concentration at each sampling time was determined from triplicate tests of each individual analyte. Figure 2(b) shows the logarithm of the average concentration versus the plasma treatment time; the linear behavior in the logarithm plot indicates first-order rate behavior which is the same as observed in the previous study.⁵ The resulting first-order rate constants (k) and half-lives ($t_{1/2}$) are given in Table 1.

(a) Exponential Decay of VOC Analytes



(b) First Order Rate Comparisons

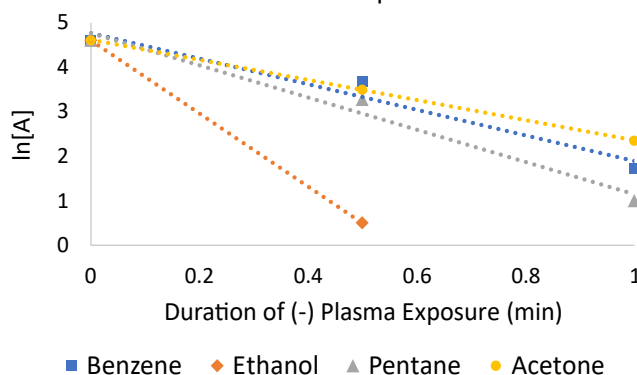


Figure 2. (a) VOC concentration in CO₂ for 2-min treatment. (b) The logarithm of the concentration vs treatment time.

stalls; in essence, while the pump was stalled, the target compound was quickly reacted to completion within the plasma volume, whereupon the plasma would spend some fraction of its time with a diminished concentration of the target compound (i.e., only what diffusion could supply). This is corroborated by a comparison of the first-order rate constants (k) with the diffusivity for each compound. Table 2 shows the values for k and diffusivity (D , Torr·cm²·s⁻¹) taken from literature values, and Figure 3 shows a correlation plot.¹¹ While the correlation is not linear, it is monotonic indicating a potential relationship. It should be noted that these hypotheses are speculative at this time, and there are no plans to explore this effect further at the time of this writing.

Table 2. Rate constants and diffusivities for VOC target compounds in a CO₂ background.

Analyte	k (min ⁻¹)	D (Torr·cm ² ·s ⁻¹)
Ethanol	9.43	0.074
Acetone	3.94	0.176
Pentane	6.88	0.101
Benzene	5.90	0.118

Table 1. Rate constants and half-lives for VOC target compounds in a CO₂ background.

Analyte	k (min ⁻¹)	$t_{1/2}$ (min)	k_{2023} (min ⁻¹)
Ethanol	9.43	0.074	3.061
Acetone	3.94	0.176	1.010
Pentane	6.88	0.101	0.825
Benzene	5.90	0.118	0.913

Also shown in Table 1 are the first-order rate constants reported in the previous study (listed as k_{2023}). It is immediately apparent from these data that the increased recirculating flow resulted in a significantly enhanced rate of VOC breakdown. This is interesting because, based on pump convection alone, an increased flow rate should not change the duration that a test compound spends in the volume of the plasma itself. Thus, this difference must be due to some other physical phenomenon. One possibility is that the circulation used in the previous study was slow enough and/or intermittent that diffusion became an important contributing factor. That is, the reaction products were diffusion-limited for exiting the plasma volume such that they maintained a higher local concentration. Whereas with the greater convection of this study, the reaction products were swept from the plasma volume where they diluted in the total volume of the gas and were thereby less likely to induce a reverse reaction. Another hypothesis is that the intermittent nature of the previous pump resulted in a diffusion-limited case during the pump

Correlation plot, k vs D

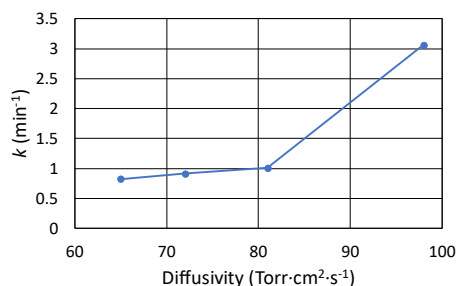


Figure 3. Correlation plot of first-order rate constant (k) vs. diffusivity (D).

It's interesting to see that the order of rate constants is different with the higher flow. In the previous lower-flow study, the rate (from highest to lowest) was: ethanol > acetone > benzene > pentane. However, the arrangement of rate in the results presented here are (from highest to lowest): ethanol > pentane > benzene > acetone. Attempts were made to correlate the reaction rates to different physical properties of the molecule as was considered in the previous study (enthalpy of combustion, flashpoint, ignition temperature, and ionization energy), however no correlation to a physical property was observed. At the time of this writing, the authors have not formulated a satisfactory explanation for this behavior.

Prior to full analyses of treated gases, the authors performed screening for possible toxins based on the elements present in the systems. For target compounds with nitrogen as the background gas, one possible product was hydrogen cyanide (HCN), a known poisonous gas. Thus, before applying the full procedure, each target gas was plasma treated for 15 min with the entire system in the hood. Then the product gases were measured using a Draeger™ HCN sampling tube. None of the product gases showed any HCN with the exception of acetone which showed at least stoichiometric concentrations of HCN. Plasma production of HCN from acetone has been reported previously.¹² With this in mind, the authors did not perform analyses on acetone with nitrogen as the background gas. Thus, under nitrogen, analyses were limited to benzene, pentane, ethanol, and Freon 113.

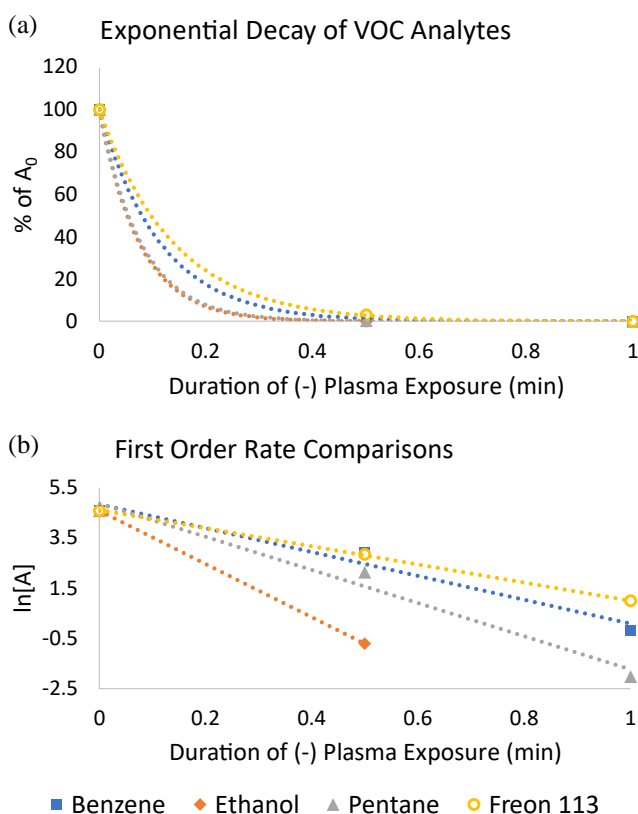


Figure 4. (a) VOC concentration in N₂ for 2-min treatment. (b) The logarithm of the concentration vs treatment time.

generally endothermic whereas combustion is exothermic, it would be reasonable to assume that a combustion-based process would be more rapid. However, with only 10% O₂ production from the plasma, it may be that the combustion is slowed by the reduced oxygen concentration.

The authors also performed the same experiment with the opposite polarity for the plasma electrode. For the measurements described above, the plasma electrode was negative relative to the grounded external wall of the system. Figure 5 shows the results for the target compounds in the CO₂ background gas, but with the plasma electrode biased positive relative to the grounded external wall of the system. Figure 5(a) shows the average ppm of

Figure 4 shows the results for the target compounds in the N₂ background gas. Figure 4(a) shows the average ppm of each analyte as a function of duration of the plasma treatment. The average concentration at each sampling time was determined from triplicate tests of each individual analyte. Figure 4(b) shows the logarithm of the average concentration versus the plasma treatment time; the linear behavior in the logarithm plot also indicates first-order reaction rate behavior. The resulting first-order rate constants (*k*) and half-lives (*t*_{1/2}) are given in Table 3.

Table 3. Rate constants and half-lives for VOC target compounds in an N₂ background.

Analyte	<i>k</i> (min ⁻¹)	<i>t</i> _{1/2} (min)
Ethanol	7.99	0.0868
Freon 113	7.47	0.0928
Pentane	11.13	0.0623
Benzene	7.52	0.0921

As can be seen from Figure 4 and Table 3, the VOCs were more rapidly removed from the gases for the N₂ background than for the CO₂ background. This is somewhat surprising considering the observation (reported in the previous study and elsewhere) that the CO₂ plasma generates up to 10% O₂ which would presumably enhance the combustion (and thus removal) of the VOCs. Under an N₂ background, the principal mechanism for the breakdown of the VOC is presumably plasma pyrolysis. Since pyrolysis is

each analyte as a function of duration of the plasma treatment. The average concentration at each sampling time was determined from triplicate tests of each individual analyte. Figure 5(b) shows the logarithm of the average concentration versus the plasma treatment time; the linear behavior in the logarithm plot also indicates first-order reaction rate behavior. The resulting first-order rate constants (k) and half-lives ($t_{1/2}$) are given in Table 4.

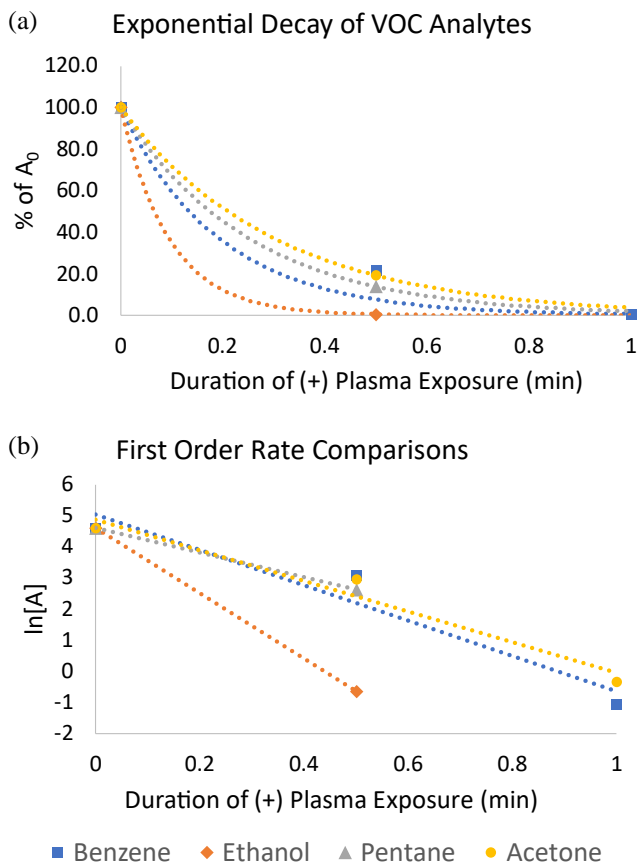


Figure 5. (a) VOC concentration in CO_2 for 2-min treatment under positive bias plasma conditions. (b) The logarithm of the concentration vs treatment time.

has used GC-MS to analyze effluents as part of the Trash-to-Gas project; as such, it is calibrated to analyze for several VOC constituents including alkanes (nonane, octane, heptane, pentane, and butane), aromatics (xylenes, ethylbenzene, toluene, benzene), isoprene, carbohydrates (butanol, isopropyl alcohol (IPA), ethanol, methylisobutyl ketone (MIBK), methyl ethyl ketone (MEK, also butanone), and acetone), and methylene chloride (the only calibrated halogen to be observed in the pyrolysis effluent). The results of the analyses of each plasma-treated aliquot are shown in Figure 6 and corresponding numerical data are shown in Table 5. The sections of Table 5 are separated by chemical classes of alkanes, aromatics, and carbohydrates+methylene chloride. As can be seen in these data, the majority of components generally decrease over the duration of the plasma treatment. However, there are some notable exceptions especially among small carbohydrates including isopropyl alcohol (IPA), ethanol, and acetone that are somewhat erratic, but all increase at some point during the plasma treatment. These results demonstrate that plasma treatment is able to significantly reduce the constituents of complex VOC mixtures such as those resulting from thermal treatment of mixed trash material.

Table 4. Rate constants and half-lives for VOC target compounds in an N_2 background with a positive voltage plasma.

Analyte	k (min^{-1})	$t_{1/2}$ (min)
Ethanol	8.74	0.079
Acetone	3.86	0.180
Pentane	5.29	0.131
Benzene	5.24	0.132

A comparison of the results from Table 4 and Table 1 shows that in all cases the rate of VOC removal was greater for a negative-bias plasma than for the corresponding positive-bias plasma (2 min exposure with CO_2 background gas). Interestingly, the relative rate constants are in the same order and similar spacing with ethanol showing the highest rate of removal, then pentane and benzene with similar rate constants, then acetone showing the lowest of the rate constants among the target VOCs. The authors hypothesize that the reason for the lower rate constants observed with a positive plasma bias is that the majority of the chemistry is driven by energetic free electrons that are capable of attaining higher energies with less loss to thermal processes. The negative bias plasma directly emits these free electrons whereas the positive plasma relies upon emission of secondary electrons within the plasma/gas system. This results in higher kinetic rates for the negative-bias plasma.

Trash pyrolysis effluent is known to contain numerous VOC compounds. The Applied Chemistry Laboratory at Kennedy Space Center

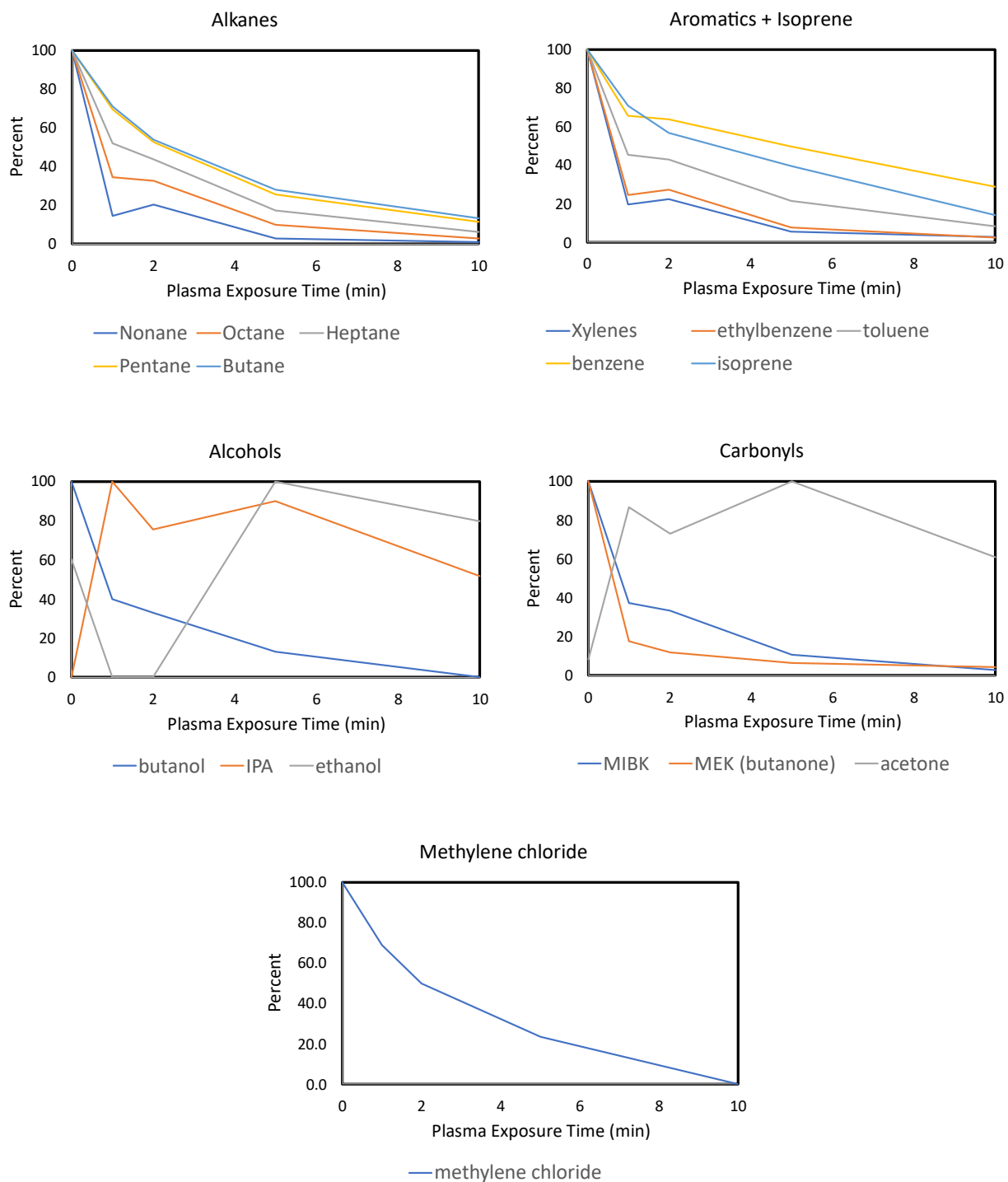


Figure 6. Plots of percent maximum versus time for the analytes measured for plasma treatment of pyrolysis effluent of simulated crew trash. For clarity, plots are separated by class of compound.

Table 5. Percentages of measured analytes for plasma treatment of gas effluent from simulated crew trash pyrolysis in nitrogen. Values are given as percent of the highest measured value.

Time (min)	nonane	octane	heptane	pentane	butane
0	100.0	100.0	100.0	100.0	100.0
1	14.7	34.5	52.0	69.5	70.9
2	20.3	32.6	43.7	52.7	54.0
5	3.0	10.1	17.4	25.7	28.0
10	1.0	2.8	6.1	11.6	13.4

Time (min)	xylenes	ethylbenzene	toluene	benzene	isoprene
0	100.00	100.00	100.00	100.00	100.00
1	19.77	24.51	45.32	65.58	70.77
2	22.48	27.33	42.95	63.83	56.60
5	5.58	7.61	21.54	49.57	39.59
10	2.77	2.35	8.17	28.88	14.06

Time (min)	butanol	IPA	ethanol	MIBK	MEK (butanone)	acetone	methylene chloride
0	100.00	0.07	60.05	100.00	100.00	8.27	100.00
1	40.00	100.00	0.00	37.20	17.75	86.69	68.76
2	32.88	75.42	0.00	33.30	12.02	72.82	49.70
5	12.95	89.90	100.00	10.62	6.35	100.00	23.30
10	0.00	51.81	79.64	2.79	4.23	60.96	0.00

It is also important to note that, despite the apparent formation of these three reaction products, all of them display subsequent decreases in concentration suggesting that continued treatment would eventually remove them as well. Methylene chloride is the only calibrated halogen to be observed in these data, showing relatively rapid removal within 10 minutes of plasma treatment.

The behavior of individual components in the mixture of gas effluent does not appear to obey first-order kinetics (or necessarily any simple rate law). This is likely due to complex interactions between components where certain analytes are either products and/or intermediates of other constituents. Thus, no rate constants were calculated from the effluent results as they might be misleading regarding the relative rates of removal.

IV. Conclusions

The results presented here show that plasma is a potentially very useful approach to abatement of VOCs in a crewed breathable atmosphere. It has several potential advantages over other common methods such as filtering (which requires consumable filters) or catalytic systems (whose catalysts can become poisoned over time). However, it may be that plasma will work best in combination with one or more additional abatement methods. The treatment of the pyrolysis effluent presented here is an excellent proof-of-concept showing the capabilities of this approach.

In the context of previously reported results, it is apparent that the recirculation rate for the gases is a very important parameter that can affect the predominant process of action. This can be very profound such as seen in the comparison shown in Table 1 where the higher recirculation rate corresponds to a much higher rate of reaction. As mentioned above, it's important to note that, for a given volume of gas in the system, the amount of time the target compound spends within the plasma is theoretically the same regardless of the recirculation rate (assuming a recirculation of at least one volume equivalent during a test). This is equivalent to the fraction of plasma volume ($\sim 0.981 \text{ cm}^3$) to the total gas volume ($\sim 1 \text{ L}$) which is equal to 9.81×10^{-4} . This indicates that for the higher recirculation rates, the target compounds are eliminated after just 0.118 s of direct plasma exposure (corresponding to 2 min of total treatment time). This observation is consistent with a very rapid reaction within the plasma. Thus, for slower recirculation rates, diffusion becomes a more important contributing factor in the overall observed reaction rates since diffusion becomes a more important contributing factor (versus convection) for the movement of the gases through the plasma volume which then degrades it nearly instantaneously.

There are some questions yet to be answered. While plasma treatment of the pyrolysis effluent did show chemical intermediates (ethanol, IPA, and acetone), the authors have still not demonstrated a thorough mass balance for these systems. This will be required to ensure that some other, as yet unobserved hazard is not produced with the plasma treatment. Once these questions are answered, it will be possible to move forward with developing these promising technologies.

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