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EXPLOSIVE DESTRUCTION SYSTEM'S DRUM FILTER PART 2. SIMULATION

John J. Mahle

RESEARCH AND TECHNOLOGY DIRECTORATE

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14. ABSTRACT Operating conditions for the Explosive Destruction System's (EDS) drum filter including temperature, flow rate and humidity, based on operator logs and other studies, are documented here. The vapor phase feed concentration data has not historically been recorded for EDS operation, so estimated values based on either worst case assumptions or liquid phase equilibria relationships are used. A simulation breakthrough model is shown to provide excellent agreement with the experimental results of the EDS validation study. The efficiency of the two-layer, silica-ASZMT carbon at the nerve agent toxicity level, 0.0001 mg/m ³ , was demonstrated experimentally in that report but was not studied for an all silica bed. A worst case condition was selected corresponding to the vessel vapor equilibrated at 80% of agent GB saturation. The simulation was then used to predict performance for the baseline bed of, 50% of the total bed length, of adsorbents silica and ASZMT equivalent of 8 and 14 continuous MEA and GB ventings, respectively. When this more realistic series of steps is simulated at worst case conditions using a full length of all silica gel only, a minimum of 13 MEA wash cycles can be retained without any elution of chemical agent.					
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PREFACE

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EXPLOSIVE DESTRUCTION SYSTEM'S DRUM FILTER PART 2. SIMULATION

1. INTRODUCTION

The Explosive Destruction System (EDS) is a field deployable device used by PM Non-Stockpile Chemical Materials Agency (CMA) to render chemical munitions safe by a contained detonation followed by chemical treatment. A schematic of the EDS is presented in Figure 1. The residual neutralent is transferred to a waste drum and the residual gas is filtered prior to release. The EDS is installed in an environmental enclosure (EE). A secondary vapor and aerosol filter system, the EE filter, is operated to capture all the emissions from the enclosure. All exhaust of the EDS drum filter flows directly to the EE filter.

The primary function of the EDS carbon drum filter is the capture of chemical agent. The secondary objective is retention of neutralent monoethanolamine (MEA) vapor. A two layer adsorbent configuration is used with silica gel at the front end to capture MEA and the current military impregnated activated carbon vapor adsorbent ASZMT at the back end for chemical agents.

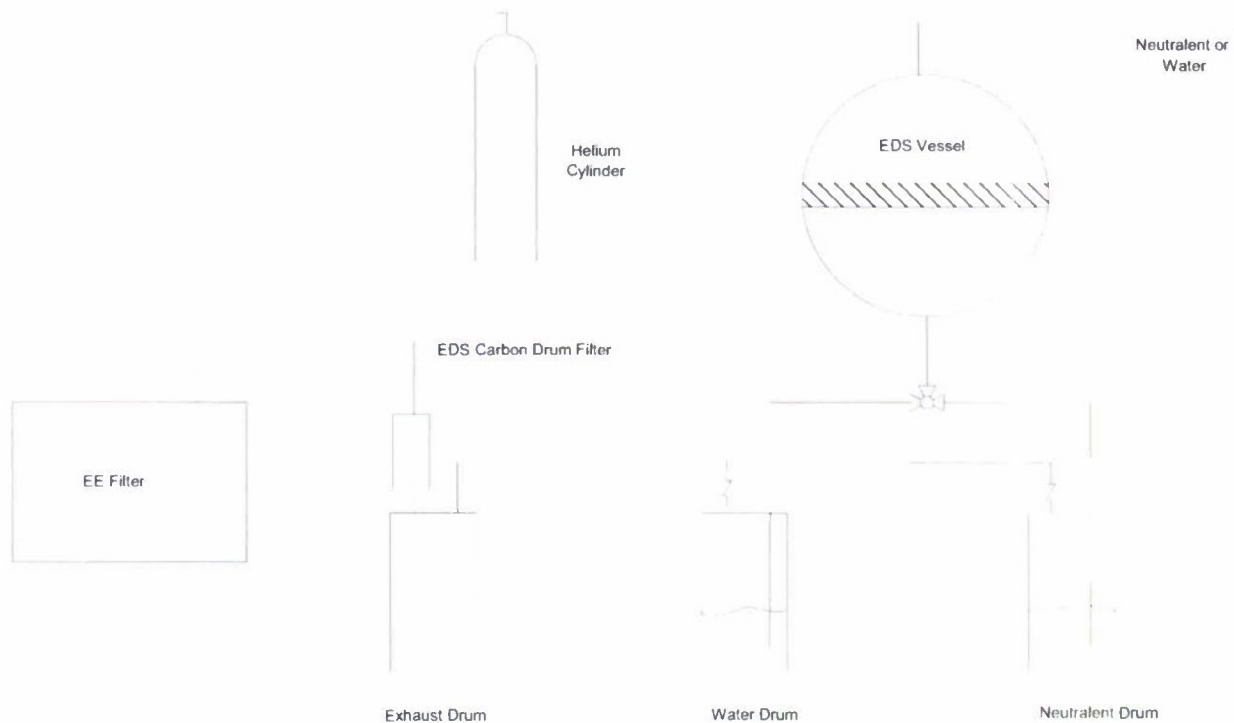


Figure 1. EDS Schematic

The EDS process consists of multiple steps outlined in Table 1. Steps 1-3 are conducted to leak check the seals and sealing surfaces of the EDS vessel. Step 4 is detonation. Steps 5-9 encompass agent neutralization with reagent MEA and subsequent vessel rinsing and purging procedures. The only step that could potentially release chemical agent to the carbon drum filter is the initial helium atmosphere for the helium checking being vented in Step 3, and in this case, chemical agent would only be vented to the carbon drum filter if the target object to be destroyed is leaking. After detonation and some period of reaction the liquid neutralent is analyzed to confirm chemical agent has been destroyed at or below the treatment goal concentration (1 ppm for nerve agents and 50 ppm for mustard agents). Therefore, only negligible quantities of chemical agent are vented as a result of the neutralization step with MEA and all subsequent steps in the process. Two water rinse steps are performed with the rinses sent to a separate drum. Finally, a helium purge is performed where this gas is vented through the neutralent drum. The vapor components that result from Steps 5-8 are water, MEA, and products of combustion/neutralization.

Table 1. EDS Process Steps

Step	
1	Helium Atmosphere
2	Vessel Leak check
3	Vent Helium
4	Detonation
5	MEA Reaction and Vessel Drain
6	Water Rinse 1
7	Water Rinse 2
8	Helium purge through Neutralent

A study was conducted, which optimized the adsorbent design of the carbon drum filter.¹ It was determined that oxidation of adsorbed MEA by the impregnant metals, Cu, Zn, and Mo, on ASZMT can provide enough heat to ignite the activated carbon. Therefore, a filter design was selected with an entrance layer of non-combustible silica gel large enough to retain the anticipated MEA load. A ratio of 33/67 wt% silica gel/ASZMT was recommended. The current baseline bed adsorbent layering of the EDS carbon drum filter system, 50/50 vol% (55/45 wt%), differs from that original proposal. Details of the EDS carbon drum filter with baseline adsorbent configuration are presented in Table 2.

Table 2. Baseline EDS Carbon Drum Filter Design

Parameter	Value
inner dia (in)	4.03
inner dia (cm)	10.2
area (ft ²)	0.089
length (cm)	27.0
length (ft)	0.886
silica length fraction	0.500
carbon length fraction	0.500
silica length (cm)	13.500
Silica length (ft)	0.443
carbon length (cm)	13.500
carbon length (ft)	0.443
silica grade	Davison 408
carbon grade	ASZMT
silica density (g/cm ³)	0.754
carbon density (g/cm ³)	0.630
silica mass (kg)	0.837
carbon mass (kg)	0.700
total adsorbent mass (kg)	1.537

Current practice is that the EDS carbon drum filter is changed after each EDS cycle. There is an interest in conducting further parametric studies on the relationship between carbon drum filter performance and the silica gel to ASZMT bed layering ratio beyond the current design limit. Earlier work validated the filter to provide adequate filtration for three EDS loadings of MEA reagent, where the design limiting condition is MEA not chemical agent capacity. By increasing the ratio of silica gel to ASZMT, it might be possible to extend the service life of the carbon drum filter given the chemical agent concentration has been determined to be much lower than stipulated in the original design.

2. BREAKTHROUGH MODEL

A simulation of the effect of bed layering can be used to identify an optimal design. The code used in this study had been developed earlier to simulate demilitarization incineration post-treatment filters. Details of the simulation development and validation are reported in Goldfarb, et al.^{2,3} This model simulates multicomponent filter breakthrough in the presence of humidity. Adsorption equilibrium is correlated by potential theory, which permits extrapolation for a wide range of chemicals based on vapor pressure. The model was originally validated using a coconut carbon.

To further validate the adsorption bed simulation program for the EDS drum filter, it can be compared to EDS experimental results. Vapor pressure correlations for MEA, dimethyl methylphosphonate (DMMP), and chemical agents were obtained from the literature. Buettner¹ reported breakthrough of MEA to a silica only bed filled to one third total filter length at dry conditions in a 22-42 min window at the concentration of 0.05 mg/m^3 . A simulation of that test is presented in Figure 2 with a calculated breakthrough time of 20 min. Although the data and experiment do agree for this case, there is not a high level of confidence in the level of separation that can be achieved using silica gel (i.e., there is only one data point reported at a concentration below the breakpoint of 0.05 for MEA).

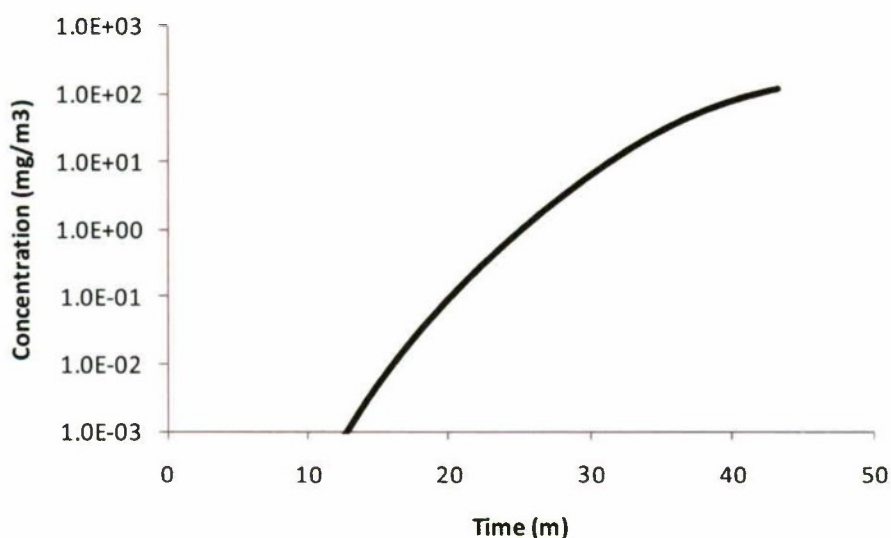


Figure 2. MEA Simulation to Bed of Silica Only, Dry Conditions (equivalent to 1/3 silica wt fraction)

Buettner^{1,3} also reported experimental results of the breakthrough of DMMP for the 33/67 wt% silica/ASZMT design under dry conditions. Results predicted from the simulation are shown in Figure 3 for a silica layer and ASZMT layer. The midpoint breakthrough time for silica is 101 min. The breakpoint (0.0001 mg/m^3) concentration time from the simulation is 209 min for ASZMT. The model cannot simulate multiple layers of adsorbent so an estimate for the layered bed is simply the sum of the two breakthrough values measured for the respective single layers, 310 min. The average breakthrough value from the experiments¹ utilizing the bi-layered adsorbent was 382 min (where there is a 20 min sampling window). Results of these MEA and DMMP simulation versus data comparisons indicate close agreement and serve to validate the models predictive capability.

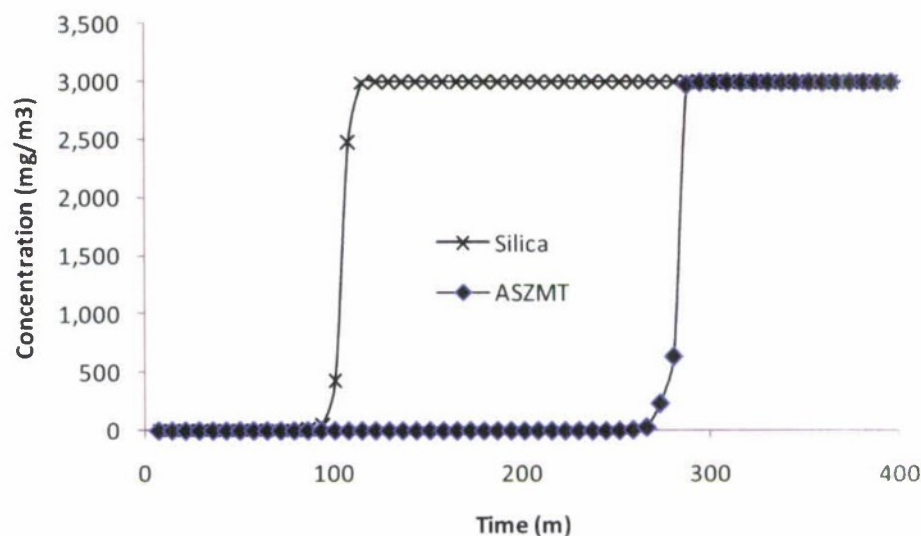


Figure 3. Simulation of DMMP Breakthrough for Standard Filter Layering, 0.33 wt% Silica, 67 wt% ASZMT

To apply the model to actual filter operation, some method must be developed to estimate vapor compositions from liquid phase compositions because only the liquid phase composition of the neutralent is reported, while the EDS drum filter is exposed to vapor concentrations. Therefore, to estimate the vapor composition of various soluble components a vapor-liquid equilibria (VLE) relationship must be used. Any approach to assess the filtration performance of the EDS drum filters will require that the filter inlet concentration profile be estimated because it has not been measured. There is however data reported for the liquid phase composition. Table 3 presents values taken from the NRC MEA Neutralent Report for the HD case.⁴ Standard models to estimate VLE use an activity coefficient, γ , for the liquid phase and assume the vapor phase behaves ideally, which can be expressed as

$$p y_i = \gamma_i x_i p_{\text{sat},i}$$

for each soluble component of the mixture. Belabbaei⁵ described the VLE of MEA and water. Activity coefficients can be estimated for the neutralent composition in Step 5. From Table 3, it can be seen that MEA, water, and HD breakdown products are the only significant components of the EDS neutralent. However, there are no reliable literature values for the vapor pressure of the breakdown products; therefore, the chemical agent HD will be used to estimate the properties of the HD products. At 50 °C and a mass fraction of 0.84 for MEA, the reported activity coefficients of MEA and water are 0.98 and 0.56, respectively. The activity coefficient of the agent decomposition products are taken at 1.0 because this gives the worst case of volatility. Table 4 presents the calculation of the vapor phase composition from the liquid phase composition using reported activity coefficients for MEA and water. The composition of MEA and agent product in the two water wash steps is considered to be insignificant.

Table 3. HD Neutralent Composition

Component Category	Neutralent Component	Concentration *
Reagent component	Monoethanolamine	84 wt%
Reagent component	Water	9 wt%
HD breakdown product	HETM (N-(2-hydroxyethyl)thiomorpholine)	5 wt%
HD breakdown product	HEAES	1 wt%
CWA	HD	< 50 ppb
Chemical Fill Impurity	1,4 dithiane	0.2 wt %
RCRA TCLP Constituent	Trichloroethylene	2 mg/L
RCRA TCLP Constituent	vinyl chloride	5 mg/L
* Estimated from Neutralent Study		

Table 4. Vapor Liquid Equilibria Estimation

	wt fraction	x	γ (Belabbaci ⁶)	psat (Pa @ 50 °C)	P _{total} (Pa)	y _i	p _i	RH	°C (ppm)
MEA	0.84	0.72	0.98	354	2068	0.120	248.6	0.70	2486
water	0.09	0.26	0.56	12412		0.876	1811	0.14	18108
HD Products	0.07	0.02	1	362		0.004	8.3	0.023	83

3. EDS OPERATING CONDITIONS

The steps in the EDS process were presented in Table I, several of which result in potential release of agent or agent reaction products to the EDS collection drums and carbon drum filters. In the case where liquid only is discharged, the equivalent volume of gas would also be discharged through the filter. In the case where a pressure reduction based venting occurs, the volume of vapor is readily calculated based on the reactor volume. The vapor stream exiting the EDS carbon drum filter is vented to the EE exhaust, which is treated through the EE filter. Two different size reactor systems, P1 and P2, are used.

No comprehensive design report is available, which summarizes the EDS system. Therefore, based on conversations^{†‡} and review of the SOP⁶, a detailed description of the process steps and conditions was compiled for this study (see Table 5 and summary in Table 6). In practice, there can be variations in the values reported in Table 5 due to operator preferences and chemical agent treated. Therefore, where possible, average and maximum values are reported. In this study, only those chemical agents that are neutralized by MEA are considered (i.e., HD, GB, and VX).

It is recognized that flow rates of the actual system are non-linear in time. However, there is no data to fully characterize this transient behavior nor is the adsorption model validated for transient pressure and flow. Therefore, only constant flow rates are considered. The Buettner¹ report cited communications with Sandia National Laboratories Albuquerque, NM) that selected 14 cfm as a representative flow rate, which had been determined from observations that the P1 vessel depressurized from 30 psig in ~2 min. Table 5 shows that the computed flow rates for the various steps differ from the original assumption. Step 3 has the largest flow rate, 29 cfm. The flow rates for the three rinse steps are modeling by averaging the vapor and liquid contribution of the step.

In addition to the flow rate, the vapor composition for each step must also be estimated. As stated earlier the only step where chemical agent vapor might be vented to the carbon filter is Step 3. In Step 3, a worst case estimate must be generated because there is no reported data. If it is assumed that a munition to be destroyed leaked agent while in the EDS vessel, then the worst case would be that the vapor vented in Step 3 had been saturated with the agent at its vapor pressure. However, due to temperature fluctuations, a more realistic assumption would be that vapor is at 80% of saturation with agent in Step 3. The vapor for Steps 5-8 that reaches the EDS carbon drum filter has been sparged through either the neutralent drum, Steps 5 and 8 or the water drum, Steps 6 and 7. It is assumed here that this only results in 80% of saturation for the vapor exiting the drum. The VLE for the mixtures in Steps 5-8 can be calculated as discussed in Section 2. If the filter is used for multiple 8 step cycles, a complex multicomponent adsorption profile is developed on the filter as subsequent waves of MEA, humidity, and agent are delivered to the bed.

† Crocker, R. *Updated: EDS Drain Flows for Drum Filter Design*; Sandia National Laboratories: Albuquerque, NM, December 2009; unpublished data.

‡ Hagler, C., personal communication, Pine Bluff CDF AR, July 2010.

Table 5. Details of EDS Steps

Step	Parameter	Units	P1	P2	Notes
*	EDS Reactor Volume	L	205	605	
0	Ordnance and charge placed in reactor and sealed				
1	He initial atmosphere pressure	psig	60	20	
2	Leak check				
3	He vent through carbon filter final pressure	psig	0	0	may have agent but no MEA
	vent time	min	< 1	< 1	
	vented gas volume	L	837	823	
	vented gas flow rate	Lpm	837	823	
	vented gas flow rate	cfm	29.9	29.4	This is greater than the nominal 14 cfm, 53 cfm was found to rupture burst disk
4	explosive charge				
	avg pressure HD	psig	28	10	
5	MEA Rinse liquid volume	L	120	204	gravimetric drain to neutralent drum
	max pressure	psig	50	26	MEA pumped in at this pressure
	avg pressure	psig	28	10	
	avg temperature	°C	50	50	
	time liquid drain	min	5	10	(could be 5-30 min)
	vented liquid flow rate	Lpm	4	20.4	
	vented liquid flow rate	cfm	0.86	.73	
	vented vapor volume	L	289	709	
	time for venting vapor	min	1	1	
	vapor flow rate	Lpm	289	709	
	vapor flow rate	cfm	10.3	25.3	
	total gas volume	L	1245	1736	
	avg vapor flow rate	cfm	2.4	3.0	time averaged liquid + vapor flow rate

Table 5. Details of EDS Steps (Continued)

Step	Parameter	Units	P1	P2	Notes
6	Water Rinse	L	80	133	MEA, no agent
	time	min	5	10	(could be 5-30 min)
	flow rate	slpm	16	13	
	flow rate	cfm	0.57	0.47	
	total gas volume	L	1326	1869	
7	Second Water Rinse	L	65	133	
	time	min	5	10	
	flow rate	slpm	13	13	
	flow rate	cfm	0.46	0.46	
	total gas volume	liters	1391	2002	
8	Helium Purge (1A cylinder delta P)	psig	500	500	Through water drum catching any MEA vapor
	Cylinder volume	L	49.8	49.8	
	vented volume	L	1693.	1693.	
	time	min	5	5	
	flow rate	slpm	339	339	
	flow rate	cfm	12.1	12.1	
	total gas volume	L	3084	3696	

Table 6. Summary EDS P1 and P2 Step Simulation Conditions

Step	Parameter	Units	P1	P2	notes
*	EDS Reactor Volume				
0	Ordnance and charge placed in reactor and sealed				
1	He initial atmosphere				
2	Leak check				
3	He vent through carbon filter final pressure				
	time	min	1	1	
	vented gas flow rate	cfm	29.9	29.4	
	temperature	C	25	25	
	humidity	%RH	0	0	
	P _{HD} ($0.8 \cdot P_{sat}$)	Pa	14	14	
	P _{GB} ($0.8 \cdot P_{sat}$)	Pa	262	262	worst case
	P _{VX} ($0.8 \cdot P_{sat}$)	Pa	0.096	0.096	
4	Detonation				
5	MEA, Neutralization and Drain				
	time	min	6	11	
	vented gas flow rate	cfm	2.4	3.0	
	temperature	C	50	50	
	humidity	%RH	14	14	
	partial pressure MEA	Pa	117	117	
	partial pressure HD reaction products HD	Pa	36	36	worst case
6+7	Water Rinses 1 and 2				
	time	min	10	20	
	vented gas flow rate	cfm	0.52	0.47	
	temperature	C	25	25	
	humidity	%RH	80	80	
8	Helium 1A cylinder purge				
	time	min	5	5	
	vented gas flow rate	cfm	12.1	12.1	
	temperature	C	25	25	
	humidity	%RH	50	50	
	partial pressure MEA	Pa	117	117	

The volumes and flow rates of the P2 operation are larger than P1. Therefore, P2 conditions will only be considered in the following simulations as these represent the worst case flow to the EDS filter.

4. SIMULATED PARAMETRIC BED LAYERING

The adsorption simulation model can be used to predict EDS carbon drum filter performance over a range of untested conditions. Although the simulation has been validated with experimental data, it has limitations. Two conditions appropriate to this problem that are not incorporated into the model are multiple layers of adsorbent and transient feed conditions. Therefore, the design for EDS drum filter must be addressed through a series of separate cases.

The first case considers the capacity for MEA and HD product from combined Steps 5 and 8 on a filter filled at the baseline silica bed depth (50% of column length). The vapor composition calculated for the neutralant is taken from Table 4. The HD mixture is considered worst case because it has the highest MEA concentration. In the Buettner¹ report, the MEA breakthrough concentration was taken as 0.05 mg/m^3 , which was their detection limit. They noted that the TWA concentration is 7.5 mg/m^3 . Here the TWA value will be used. Breakthrough of MEA at 7.5 mg/m^3 occurs at 62 min (see Figure 4). Therefore, given a filter vapor exposure time for combined Steps 5 and 8 of 7.7 min, the silica section could contain ideally eight consecutive MEA loadings. The HD product component breaks through much later than MEA as expected. The high volatility of MEA would result in elution through the bed if clean air is passed through the filter after the chemical challenge is stopped.

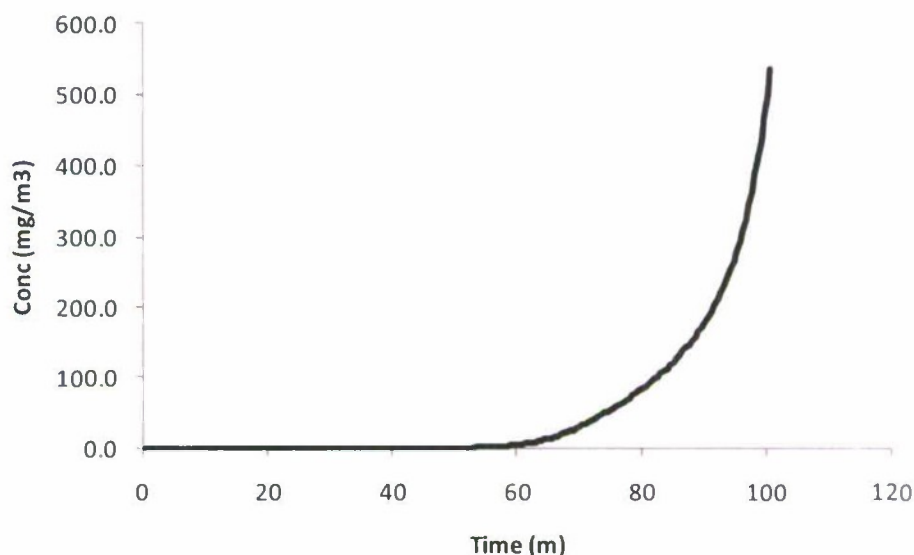


Figure 4. Feed Silica Bed with MEA and HD Product Followed by Helium Sweep

The next simulation case seeks to identify the bed depth of silica and ASZMT required to retain chemical agent from a leaking munition during the helium leak test blowdown.

The worst case would be with chemical agent GB because it has the highest volatility and is most weakly adsorbed, at 80% of saturation. Using the baseline bed depth (50% of column length) and the breakpoint concentration for GB, 0.0001 mg/m^3 , the breakthrough times for a continuous GB challenge are 11 and 14 min for silica and ASZMT at the P2 conditions respectively (see Figure 5). This can be normalized by the feed time, 1 min, shown in Table 6, to give the number of Step 3 events 11 and 14 for silica and ASZMT at the adsorbent's baseline fill. It can be seen that the TWA concentration breaks through at this half-bed length in approximately one half the time that the feed breakthrough occurs. This indicates that 50% half of this bed or 25% of full bed depth corresponds to the mass transfer zone for chemical agent. Once this mass transfer is contained, adding additional bed depth will add correspondingly more than a linear amount to the overall breakthrough time.

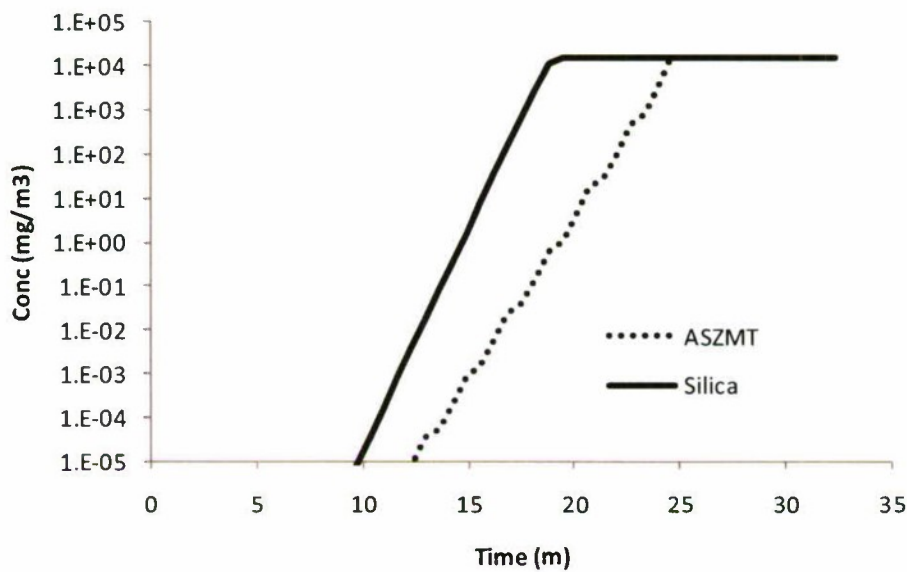


Figure 5. Feed GB to 50% of Full Bed Length of Silica and ASZMT

The task of running a simulation for multiple cycles can be simplified if some worst case assumption is made. The ability of the filter to retain chemical agent is lessened if any contaminants are loaded on the bed prior to the chemical agent challenge. This would favor displacement of the chemical further to the effluent. Therefore, multiple cycles can be best simulated as a series of MEA washes followed by a series of chemical agent ventings. In addition if the helium sweep step occurs after the chemical agent adsorption, it would be able to elute the adsorbed chemical agent from the filter. So a worst case series of steps would be Steps 5 and 8 followed by Step 3. The water wash steps are not considered significant.

It is of interest to consider a silica only bed at these worst case conditions. A full length bed of silica gel is challenged with equivalent of 13 MEA washes followed by 13 equivalent Step 3 GB challenges and finally 13 equivalent water washes. Here, the helium sweep is added to the MEA wash because Step 8 sparges through the MEA drum. Figure 6 presents the results where only MEA appears at the product at a significant level. Although MEA continues to elute after the challenge is stopped, it does not significantly exceed the TWA concentration of 7.5 mg/m^3 . The GB and HD product concentration are predicted to be well below the safety limits. Similarly the concentration of the HD product and GB are not predicted to increase at the product end during the water wash similar to the behavior of MEA. It must be recalled that the model has not been validated with silica gel at the 0.0001 mg/m^3 concentration level. The silica only data from Buettner¹ reported MEA breakthrough at 0.05 mg/m^3 . This is only a concern because silica gel is not as microporous as activated carbon and adsorbs the interferant water strongly.

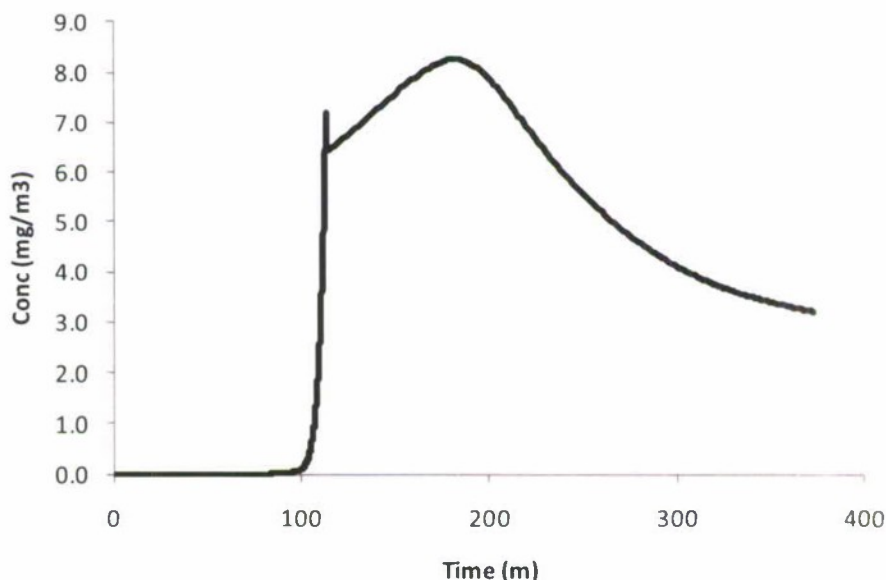


Figure 6. MEA Breakthrough Profile for a 13-Cycle Challenge Performed as 13 Equivalent MEA Washes Followed by 13 Equivalent GB Ventings then 13 Equivalent Water Washings

5. CONCLUSIONS

An analysis has been presented of the Explosive Destruction System (EDS) carbon drum filter capacity at P2 conditions. An engineering model developed in an earlier Chemical Materials Agency study was validated by accurately simulating previously reported EDS breakthrough data. A survey was conducted to compile most current operating conditions of the EDS filter system with the results reported as a series of steps. Worst case conditions were identified for the EDS process from this survey in terms of potential chemical agent and monoethanolamine burden. The agent GB was seen as the worst case for a leaking munition. A

series of simulations was conducted to identify the effects of relevant operating conditions and adsorbent selection for EDS performance. The simulations do not show appreciable difference between silica gel and ASZMT capacity for the leak events so there is not an advantage for including the ASZMT layer. However, the efficiency of silica, at the nerve agent toxicity level, has not been demonstrated experimentally. There is found to be adequate capacity of an all silica bed for ~13 EDS runs.

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