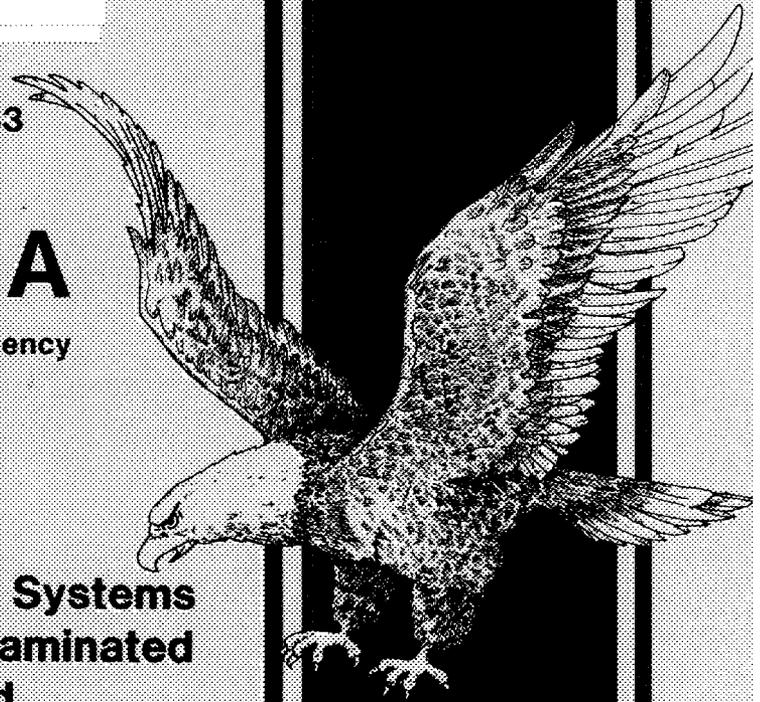


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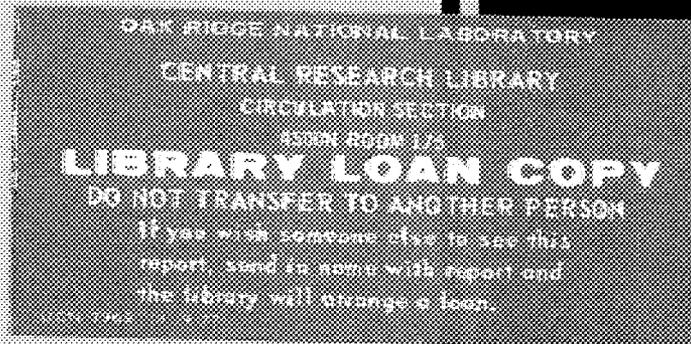
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U.S. Army Toxic and Hazardous Materials Agency



Preliminary Evaluation of Thermal Systems for Regenerating Explosives-Contaminated Carbon: Safety, Cost, and Technical Feasibility

July 1988



Prepared by
OAK RIDGE NATIONAL LABORATORY
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operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
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Engineering Technology Division

PRELIMINARY EVALUATION OF THERMAL SYSTEMS FOR REGENERATING
EXPLOSIVES-CONTAMINATED CARBON:
SAFETY, COST, AND TECHNICAL FEASIBILITY

Final Report to the U.S. Army
Toxic and Hazardous Materials Agency

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ABSTRACT

A preliminary evaluation of the thermal regeneration of nitrobody-contaminated carbon is made with special attention to safety, cost, and technical feasibility. A comparison of the multiple-hearth, rotary-kiln, electric-belt-furnace, and fluidized-bed technologies indicates that the rotary kiln and electric-belt furnace are more appropriate for nitrobody-contaminated carbon at the current low production rates. Of these two, the electric-belt furnace is probably best for Army applications, particularly with regard to safety concerns and cost involved in pilot-scale testing at an Army facility.

Cost estimates for production-scale regeneration and/or treatment at three Army facilities reveal that the economics strongly depend on the individual plant circumstances and the assumed production rates of contaminated carbon. At lower production rates, thermal treatment with disposal is cheaper than regeneration. For the regeneration cases considered, a fixed, centrally located regeneration facility appears to be the least expensive alternative.

Recommendations for future work include (1) laboratory-scale thermal-regeneration and thermal-treatment testing of spent carbon, (2) continued characterization of the explosive properties of the spent carbon, and (3) consideration of additional regeneration strategies including solvent extraction with subsequent co-fired solvent combustion.

1. INTRODUCTION

The U.S. Army produces a substantial amount of wastewater contaminated with explosive nitrobodyes. Much of this wastewater is generated during the manufacturing, loading, assembling, and packing of munitions containing TNT, RDX, HMX, and other similar explosives. In addition to current production wastes, the Army is concerned with the cleanup of

hundreds of nitrobody-contaminated wastewater lagoons created during past operations. One of the most widely used cleanup processes for nitrobody-contaminated wastewater is adsorption of the contaminants in packed beds of granulated activated carbon (GAC). The carbon removes most of the explosive material from the water and retains it for subsequent disposal.

In the past, nitrobody-contaminated carbon has either been open-burned or disposed of as a solid waste. However, it has become clear that these disposal options will no longer be acceptable in the future. As a result, the Army is searching for cost-effective methods that will minimize the volume of waste carbon and provide a more acceptable treatment for the final residue.

This report addresses one generic option for minimizing waste volume: thermal regeneration of the spent carbon. Currently available thermal regeneration technologies are reviewed with emphasis on their relative safety, cost, and technical feasibility. Three levels of detail are considered: (1) a preliminary screening of four widely used thermal regeneration technologies, (2) a more detailed comparison of two specific regeneration units that might be pilot tested at an Army facility, and (3) a brief evaluation of possible operating strategies for full-scale production.

2. SCREENING OF THERMAL TECHNOLOGIES

2.1 Thermal Regeneration Technologies

Initially, four thermal technologies were screened for their general suitability: multiple hearth, fluidized bed, direct-fired rotary kiln, and electric-belt furnace. A brief description of each follows.

2.1.1 Multiple hearth

The multiple-hearth regenerator consists of from four to eight hearths contained in a single refractory-lined steel shell. The hearths are located on top of one another and are connected by a central shaft that drives a series of scrapers known as rabble arms. Carbon entering near the rim of the top hearth is raked toward the center and down a hole into the next hearth. The next set of arms rakes the carbon to the outside, over the rim, and into the hearth below. This is repeated until the material is discharged from the bottom hearth. Burners using either fuel oil or natural gas are mounted on the shell of the lowest two or three hearths and direct the flames tangentially into the space above the hearths. Steam can be added through ports above the third hearth. An afterburner is installed above the top hearth for destruction of the organics in the off-gas.

The multiple-hearth furnace is a well-established technology for wastewater carbon regeneration. As compared to the other three technologies, multiple hearths have distinct advantages for large-scale, large-volume operations. The separate stages in the furnace provide relatively good control over the temperature profile, residence time, and furnace atmosphere. The thick refractory and low surface-to-volume ratio reduce fuel consumption. Area requirements are reduced because additional stages can be stacked on top of each other.

On the negative side, multiple hearths are prone to corrosion and erosion and have high maintenance and operating costs. Temperature cycling caused by feed outages can damage the upper hearths, and the furnaces require long start-up and shutdown times. Because of the

complex design of the furnace, repair downtime can be long, resulting in operating schedule delays. The scraping action of the rabble arms and the large carbon inventory are likely to increase the risk for explosive initiation and propagation.

2.1.2 Fluidized bed

In the fluidized-bed regenerator, GAC particles are fluidized with hot combustion gases that are generated by burning propane or light fuel oil. One or two beds may be employed although the two-bed design is the more common. The carbon can be fed into the bed by a variety of methods, but the most common feed systems utilize gravity or screw feed. The bed is usually discharged through a gravity drain. Cyclones are used downstream to collect entrained carbon particles. An auxiliary-fueled afterburner destroys the organics released in the bed off-gas.

The fluidizing action produces excellent gas-solid contacting, and the lack of moving parts reduces operational complexity and maintenance costs. Like the multiple hearth, stages can be vertically stacked, resulting in reduced area requirements. For nitrobody-contaminated carbon the major disadvantages are (1) the requirement for a high-capacity blower for the fluidizing air, (2) high carbon loss caused by particle attrition and elutriation, (3) large volumes of potentially explosive material in close proximity in a single vessel, and (4) poor control of the particle residence time and temperature profile within each bed.

2.1.3 Rotary kiln

The direct-fired rotary kiln consists of a refractory-lined steel cylinder closed at the ends and mounted on trunnions. The cylinder is mounted at a slope so that the carbon granules will flow through the kiln as it turns. Flights and lifters are typically mounted on the inside of the shell to produce a tumbling action in the flowing carbon. A variable-speed drive coupled to a speed reducer controls the rotation of the kiln, which in turn controls the solids throughput. Wet spent carbon enters through a feed-screw and flows countercurrent to hot flue gas produced by the burner at the opposite end. Reactivating steam is typically added through one or more end-mounted injection nozzles.

The rotary kiln is noted for its ease of operation and short start-up and shutdown times relative to the multiple hearth. Rotary kilns are also relatively easy to adapt to portable or semiportable applications. The technology suffers from several important disadvantages including (1) metal shell corrosion and refractory breakage, (2) relatively poor control of the particle residence time and particle attrition caused by the tumbling action, and (3) relatively poor control of the gas temperature and concentration profiles caused by backmixing and the single air/combustion gas inlet. For large-scale permanent installations, the high kiln plan area can result in higher capital costs.

2.1.4 Electric-belt furnace

Electric-belt furnaces (EBFs) consist of an electrically heated tunnel furnace through which the carbon is conveyed on a metal-mesh belt. The resistance heaters provide a high degree of control over the temperature profile, and the lack of combustion gas permits tight control of the atmosphere in each zone. Reactivating steam is provided by water vaporizing from the wet carbon, reducing or eliminating the need for supplemental steam injection. The reactivated carbon is discharged into a quench tank, and the off-gases are passed through an afterburner and scrubber system.

Like rotary kilns, EBFs are relatively easy to adapt to portable operation. EBFs have the lowest carbon attrition rate of the four technologies considered here because of the gentle handling of the carbon. Minimal operator attendance is required, and short start-up and shutdown times result in flexible operating schedules. The zones in the furnace provide excellent control of the furnace atmosphere, temperature profile, and residence time. The biggest disadvantage to the EBF is corrosion of the conveyor belt. Also, the large plan area and electric power required by a production-scale system can significantly raise the capital costs.

2.1.5 Other electric furnace technologies

In reviewing the status of EBF technology, it was noted that other electric furnace regeneration technologies are now becoming commercially available. One such technology, an electrically heated downflow moving bed, has apparently received prior consideration by the Army [Lee and Toy (1984)]. While some features of this and other electric furnace technologies appear promising, it was concluded that the EBF is the most commercially proven electric furnace technology. For the purposes of this study, only the EBF was considered. Future investigations should reconsider these new technologies.

2.2 Screening Procedure

As a first attempt to eliminate the least attractive options, a qualitative comparison of the prospective thermal technologies was made. The results of this analysis are summarized in Table 2.1. Each of the systems was ranked from best to worst in various categories that were selected for their relevance to the safety, cost, and technical risk involved in regenerating nitrobody-contaminated carbon. In most categories the EBF was judged to be superior, with the rotary kiln being judged the next best option overall. From best to worst, the overall ranking is EBF, rotary kiln, fluidized bed, multiple hearth. The reasons behind our rankings for each technology are summarized in the following sections.

2.2.1 Suitability for remote operation

Several issues were considered in this category: (1) the number and complexity of the furnace controls, including any that may have to be manually adjusted during operation; (2) feed system flexibility and reliability; (3) the possibility of any carbon holdups that would require operator attention; and (4) the physical arrangement of key components and their proximity to dangerous locations during necessary operator attention. The EBF is judged to be the least complex technology, particularly with regard to the absence of a burner and the associated control systems. The multiple hearth, with its complicated

Table 2.1. Relative ranking of the four major thermal regeneration technologies

Category	Rank			
	(Best) 4	3	2	(Worst) 1
Remote operation	EBF ^a	FB ^a	RK ^a	MH ^a
Detonation propagation	RK	MH	EBF	FB
Detonation initiation	EBF	RK	FB	MH
Particle attrition	EBF	RK	MH	FB
Atmosphere control	EBF	FB	MH	RK
Residence time control	EBF	MH	RK	FB
Temperature profile control	EBF	MH	RK	FB
Commercial availability	MH	RK	EBF	FB
Start-up/shutdown time	EBF	RK	FB	MH
Rate flexibility/turndown	EBF	RK	MH	FB
Ease of modification	EBF	RK	FB	MH
Mobility	EBF	RK	FB	MH
Capital cost	EBF	FB	RK	MH
Operating cost	EBF	FB	RK	MH
Maintenance time and cost	FB	RK	EBF	MH
Energy consumption	EBF	FB	MH	RK
Space requirements	FB	MH	RK	EBF
Previous experience	RK	EBF	FB	MH
Totals:	RK = 46, FB = 39, EBF = 62, MH = 33			

^aProcesses: RK = rotary kiln
 FB = fluidized bed
 MH = multiple hearth,
 EBF = electric-belt furnace

design, multiple burners, and numerous moving parts, is expected to require the highest operator attention and control sophistication.

2.2.2 Ease of explosive propagation of detonation

The relationship between the level of carbon contamination and its ability to propagate a detonation wave are still uncertain. Recent tests at Los Alamos National Laboratory indicated that spent carbon typical of that produced at Iowa Army Ammunition Plant (IAAP) does not readily propagate, even when loaded with TNT to levels above the saturation capacity. However, these same tests also indicated significant energetic activity (i.e., energy release without propagation) for explosive levels as low as 11.1 g per gram of dry carbon [Stretz (1988)]. Studies done several years ago at Los Alamos also indicated that the presence of carbon reduces the thermal stability of TNT [Smith (1969)].

Field observations by the authors and discussions with IAAP and Mississippi Army Ammunition Plant (MAAP) indicate that thermal precipitation of solid explosives sometimes occurs in the carbon columns. In view of this, it is prudent to assume that the selected technology must be able to accommodate the possibility of feeding fragments of pure explosive. It is expected that a rotary kiln could be designed similar to the Army's explosive waste incinerators such that the flights would provide barriers to detonation propagation. It is also likely that the floor of each level in the multiple hearth could act as barriers. The continuous carbon layer in the EBF would not offer much propagation resistance, and the close solids contact in the fluidized bed would make detonation barriers essentially impractical.

2.2.3 Ease of explosive initiation

As with detonation propagation, the ease of explosive initiation of nitrobody-contaminated carbon is currently unknown. Thus, until strong evidence is available to the contrary, it is prudent to assume that fragments of pure explosive could occasionally be fed to the regenerator. Equipment producing friction or impaction of the solids is of particular concern with respect to initiation. The rabble arms in the multiple hearth would probably produce a large amount of friction.

Friction and impaction are likely to be least in the EBF. All four technologies use similar feeding systems that would have to be carefully analyzed and probably redesigned to eliminate friction/impaction points.

2.2.4 Particle attrition

The EBF was rated the best in this category because of the gentle handling of the carbon on the conveyor belt. The tumbling action in the rotary kiln causes some carbon attrition, but not as much as in the multiple hearth where the rabble arms are constantly abrading the carbon. The constant particle agitation in the fluidized bed would probably produce the largest carbon losses.

2.2.5 Atmosphere control

It is important to establish a varying oxygen content in the different zones of a regeneration furnace. In general, more oxygen is required in the final zones than in the earlier stages. Too much oxygen is detrimental, however, because of increased carbon loss to combustion. The main source of oxygen in a directly fired furnace is the combustion air. Because the EBF does not require combustion air, air need only be added as required for optimum regeneration. The other furnaces are all direct-fired with fossil fuels, and they require careful burner control to optimize the oxygen in the furnace. The rotary kiln also suffers from air inleakage at the rotating seals, which gives it the worst atmosphere control.

2.2.6 Residence time control

The EBF is judged to have the best residence time control because solids retention is set by the speed of the belt and is constant for all the carbon particles. The particle residence time for the multiple hearth and rotary kiln can be adjusted to some degree, but because of slippage and short-circuiting, the control is not as good as in the EBF. The random particle movement in the fluidized bed makes it very difficult to control the particle residence time.

2.2.7 Temperature control

The distinct zones in the electric-belt and multiple-hearth furnaces permit better temperature control. The electric-belt furnace is easier to control because each zone is directly heated by the infrared heating elements, whereas in the multiple hearth the burners are located only on the lower hearths. The profile is more difficult to manage in the rotary kiln because of the gas and solids backmixing and the injection of the hot combustion gases at one end of the furnace. Fluidized-bed temperatures are almost uniform because of the high solids mixing.

2.2.8 Commercial availability

Carbon regenerators have been used extensively in potable and wastewater treatment, odor control, and sugar processing. Traditionally, multiple hearths have been the technology of choice for large-scale facilities. Rotary kilns and fluidized beds have also been used for carbon regeneration in potable and wastewater treatment, and they are receiving increasing acceptance, especially in medium- and smaller-scale facilities. Although the EBF is relatively new, it has been commercialized by a Dallas, Texas, firm that supplies units ranging in size from laboratory use to 250 tons/d of carbon. For purposes of this screening study, the commercial availability of each technology was ranked according to its general frequency of use in industry.

2.2.9 Start-up/shutdown time

Rapid start-up and shutdown are important for regenerating nitrobody-contaminated carbon because of the need to respond quickly to emergency conditions involving explosive hazards. The electric-belt furnace is insulated with a ceramic fiber blanket that has relatively low thermal inertia and can respond quickly to changes in heat input. In addition, electric heaters can be more quickly adjusted to intermediate heat generation rates than gas- or oil-fired burners. Rotary kilns and fluidized beds are lined with more massive refractory bricks, and start-up/shutdown may take 12 h or more. The multiple hearth, because of its large refractory mass and low surface-to-volume ratio, has the longest start-up/shutdown time of all the furnaces.

2.2.10 Rate flexibility/turndown

It is expected that the volume of spent carbon production and the nature of the contaminants will vary from one Army facility to another and from time to time at any specific facility. Thus the need for regeneration rate flexibility is more important than it is with conventional spent carbons. The EBF is ranked the best in this category because it is very easy to adjust the heating and feed rates and still maintain constant conditions of particle residence time, temperature, and gas composition. The rotary kiln and multiple hearth are more difficult to turn down while maintaining constant conditions. In addition, the burners are limited to minimum firing rates, below which they become unstable. The fluidized bed is the least flexible furnace. If the gas flow is too low, the bed will defluidize, and if the gas rate is too high, the bed will elutriate.

2.2.11 Ease of modification

At this point, the development of a safe and cost effective thermal regeneration process for nitrobody-contaminated carbon involves a number of important technical unknowns. Testing and even production-scale operation are likely to involve numerous modifications to the original equipment until a substantial base of operating experience has been accumulated. In light of this, ease of modification is an important selection parameter. The simple design of the EBF makes it the easiest to modify in terms of changes in the feed system, air flows, heating sources, or control system. The rotary kiln and fluidized bed are somewhat more difficult to change, but they are not as difficult as the multiple hearth with its complex design.

2.2.12 Mobility

Because of the small volumes of carbon currently being produced and the geographical dispersion of the Army facilities, serious consideration is being given to using mobile regenerators for meeting at least some of the Army's needs. Both the EBF and the rotary kiln are available in portable units that are suitable for transport from site to

site. The belt furnace requires less time for setup and dismantling (~7 d for the largest, 250-ton/d system). The multiple hearth is rated the least mobile due to its large size, large amounts of refractory, and the numerous parts that need to be assembled and torn down.

2.2.13 Capital and operating costs for a small-scale system

Whether mobile or site-specific, a nitrobody-contaminated carbon regeneration system is likely to be relatively small scale unless it is used to process the carbon from many facilities. Assuming for the moment that the small-scale option is acceptable, rough comparisons can be made of the costs for the four technologies.

Capital costs for carbon regeneration include the furnace, incinerator, scrubber, steam generator (if needed), instrumentation and controls, site preparation, installation, first carbon replacement costs, engineering, contingency, and contractors' fees. The most significant of these costs is the furnace itself. The EBF is the least expensive due to its simple design (a 100-lb/h EBF is estimated to cost around \$250K). The next higher cost furnace is the fluidized bed followed by the rotary kiln, which is about \$330K for a 100-lb/h furnace. The multiple-hearth furnace is the most expensive because of its complex internal structure and moving parts.

For a small regenerating system (100 lb/h or less), the most significant operating cost is expected to be labor. It is judged that the simplicity and more rapid start-up and shutdown of the EBF would require less total operator time than the other technologies. Likewise, the multiple hearth is expected to require the greatest operator time because it is the most complex and slowest to respond.

2.2.14 Maintenance time and cost

In general, simpler designs with fewer moving parts should be easier to maintain. Therefore, the fluidized-bed furnace is rated highest for maintenance. Even though the rotary kiln has a significant number of moving parts, it involves less overall maintenance risk than the electric-belt conveyor. The complicated multiple hearth is rated lowest. The maintenance costs for the multiple hearth are expected to

be especially high because the internal structures are expensive to replace and access is difficult.

2.2.15 Energy consumption

Energy consumption for a regeneration system consists primarily of the heat input to the furnace itself plus the auxiliary fuel in the afterburner. The EBF is rated the best in this category, largely because the net heat transfer to the carbon is more efficient in the absence of combustion air and large volumes of flue gas. Even though electricity is a more expensive energy source than the combustor fuels, it can be converted to thermal energy in the carbon with a higher efficiency. Typical energy use ranges for the four furnaces have been estimated as follows: (1) EBF: 2300–5100 Btu/lb carbon (2) FB: 3000–6000 Btu/lb carbon (3) MH: 4000–6000 Btu/lb carbon (4) RK: 3500–8050 Btu/lb carbon [Demarco (1983); Hill (1986); Lin and Wagner (1984); Radian Corp. (1983); and Zanitsch and Lynch (1978)].

2.2.16 Space requirements

The vertical orientation and staging of the fluidized bed and multiple hearth reduce the plan areas required. The rotary kiln and EBFs have larger floor area requirements. In general, it appears that electric-belt furnaces require the largest area to accommodate a given carbon feed rate.

2.2.17 Previous experience with nitrobody-contaminated carbon

Of the four technologies considered, none except rotary kilns have been tested with explosive-contaminated carbon. IAAP has experimentally regenerated TNT-laden carbon in a rotary calciner, which is similar in many respects to a rotary kiln [Leeper et al. (1977)]. IAAP also thermally deactivates nitrobody-contaminated carbon in their rotary-kiln explosive waste incinerator (a version of the APE-1236) prior to disposal of the carbon in a commercial hazardous waste landfill. While most of the carbon substrate remains after passing through the incinerator, chemical analyses indicate that most of the contaminants have been removed [Miller (1986)]. MAAP has also been able to thermally

passivate their RDX-loaded carbon [U.S. Army Environmental Hygiene Agency (1986), Frierson (1986)].

2.3 Conclusions

Although the relative rankings are qualitative and in some cases are reasonably arguable, the overall trend of the preliminary screening clearly indicates that the rotary kiln and EBF are more suitable than the multiple hearth and fluidized bed for regeneration of nitrobody-contaminated carbon. In general, the former technologies are more suited because they more adequately accommodate the key features that distinguish the Army's problem from conventional carbon regeneration, namely (1) the possibility of explosions, (2) the relatively small volumes of carbon to be processed, and (3) the need to be flexible and to maintain a high level of process control.

3. EVALUATION OF TWO PILOT-SCALE OPTIONS

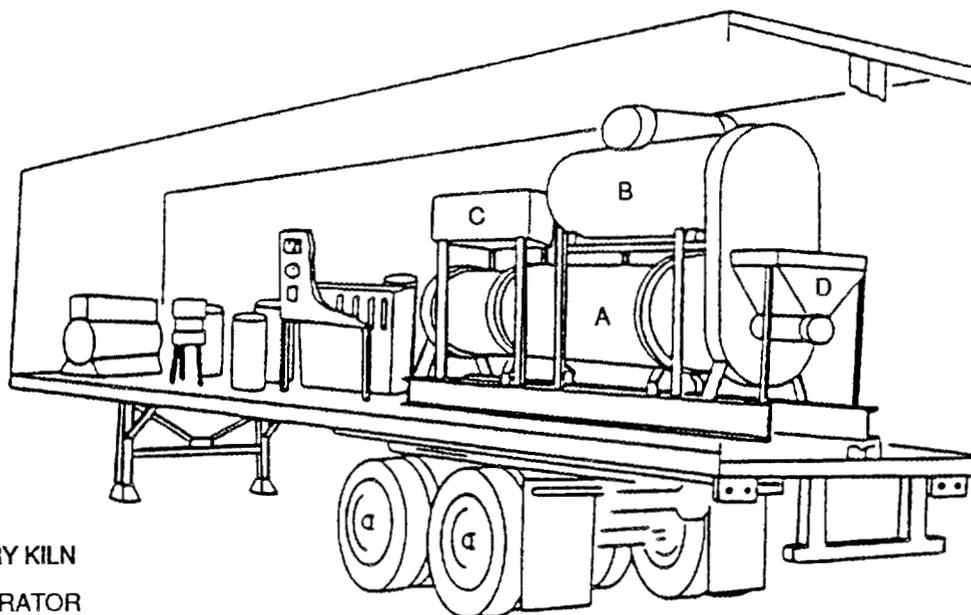
For the next study phase, a team of experts was assembled to make a preliminary evaluation of two specific systems for field testing at an Army facility: (1) the EPA mobile-carbon regenerator (a rotary kiln), and (2) the Shirco infrared regenerator (an electric-belt furnace). These systems were selected as being typical of mobile regeneration units currently available for pilot field testing with conventional spent carbon.

It was recognized at the outset that both of these regenerators were originally designed for conventional carbon regeneration and never intended to be used with potentially explosive materials. Further, it was recognized that the capabilities of each unit are significantly different, and a direct head-to-head comparison is not possible. Instead, the primary focus of the study team was to evaluate the potential modifications that would be required to safely test each unit using explosive-contaminated carbon at an Army facility. In view of the unknown and potentially dangerous properties of explosive-contaminated carbon, personnel safety and testing flexibility were given the highest priority in the study group's evaluation. In commercial operations, other considerations such as unit capacity would also be expected to have major importance.

3.1 EPA Rotary Kiln

The U.S. Environmental Protection Agency (EPA) has designed and constructed a mobile rotary-kiln regenerator. The EPA mobile regenerator is maintained and operated under the EPA's Environmental Emergency Response Unit (EERU). Enviresponse, Inc., of Edison, New Jersey, a subsidiary of Foster Wheeler Energy Corporation, is the current operating contractor for the EERU.

The self-contained regenerator unit is mounted on a semitrailer and includes a rotary kiln, an afterburner, a flue-gas scrubber, and a 60-kW diesel-electric generator. The nominal feed rate of the unit is 45.4 kg/h (100 lb/h) of dry carbon. A simplified diagram of the system is shown in Figure 3.1.



- A. ROTARY KILN
- B. INCINERATOR
- C. SCRUBBER-LIQUID COOLER
- D. CARBON FEDDER

Fig. 3.1. General layout of the EPA mobile carbon regenerator [adapted from EPA (1985)].

Spent carbon slurried with water is fed batch-wise from a storage vessel through a screen to separate the excess water from the carbon. This normally results in an ~50 wt % mixture of carbon and water. The carbon is gravity fed into a weigh tank where it is sampled and then discharged into a screw-feeder hopper. The screw feeder meters the carbon directly into the kiln.

The rotating kiln has an inside diameter of 0.37 m (14.6 in.) and a length of 3.87 m (12.7 ft). The solid residence time is between 20 and 30 min. It is counter-currently fired with propane, and the carbon is heated to a final discharge temperature of 1140–1260 K (1600–1800°F). Steam can also be injected into the breech to aid regeneration and control the temperature. Discharged carbon is dumped into a water-filled quench tank from which it can be later removed for dewatering and storage.

The kiln off-gas is ducted through a propane-fired afterburner that operates at about 1260 K (1800°F), with a gas residence time of ~1 s.

The afterburner flue gas is quenched with a water spray and caustic scrubbed in a packed tower to neutralize the acid gases. An induced draft fan discharges the scrubbed gases into a portable stack. Auxiliary equipment is provided to recirculate the scrubbing liquor and replenish the caustic used in the scrubbing tower.

3.1.1 Key safety concerns regarding the EPA mobile regenerator

The most important safety concerns identified for the EPA mobile regenerator are as follows:

Remote operation - One of the original design objectives of the mobile unit was to make it as compact as possible so that it could be transported on a 2.4-m-wide (8-ft) by 13.7-m-long (45-ft) by 4.1-m-high (13.4-ft) trailer. In order to accomplish this, the controls and recording instruments were located adjacent to the kiln on the same trailer. In addition, many of the controls, such as the screw-feeder drive, were designed for manual operation, requiring an operator to be nearby while making adjustments.

The proximity of the various controls and instrumentation to the process is judged by the study team to pose an unacceptable safety risk to operating personnel during operation with explosives-contaminated feed. This is especially true when the operators are faced with emergency situations such as fire or major equipment failure. Relocating the instruments and controls to a remote, protected location would reduce this concern. In addition to the trailer for the regenerator itself, a second trailer is needed to transport auxiliary equipment (e.g., tanks, dewatering screen) to field test locations. It may be feasible to locate an instruments and controls panel on this second trailer.

Remote sampling - Regular sampling of the carbon, water, and stack gas streams is required to evaluate both the performance and the environmental acceptability of the mobile unit. Standard sampling procedures now require samples to be collected in the immediate vicinity of the kiln while it is operating. Provisions will have to be made to either provide appropriate shielding at the sample locations or relocate the sample lines to a safe, protected area. In some cases, such as the

carbon weigh hopper, relocation would involve considerable equipment modification and may be impractical.

Feed system - The existing spent carbon feed system is known to have intermittent problems with wet carbon bridging in the hoppers and feed chutes and blinding of the dewatering screen. Correction of these problems often requires manual operator attention. Again, this is judged by the study team to pose an unacceptable safety risk to the operators during the feeding of explosives-contaminated carbon. It will probably be necessary to redesign the dewatering and weigh/feed-hopper system in order to allow the feed system to consistently operate without manual attention.

Wiring system - Both the instrument and electric-power wiring on the mobile unit are not explosion-proof for combustible dust or vapors. Although similar wiring installations are common for industrial kilns, it is anticipated that it may be necessary to install explosion-proof wiring in order to meet the required standards at an Army facility. The study team does not consider the potential hazard associated with the wiring to be significantly different with or without the presence of explosive contaminants. Organic vapors (e.g., fuel oil or propane) and dry carbon dust are present to some degree around the mobile unit regardless of the type of contaminant and would represent the greater risk for exposed wiring. However, the result of electrical or dust fires is of such serious consequence that every precaution should be taken to prevent them from occurring.

Oxidizing versus reducing atmosphere - The current standard procedure is to operate the kiln in an oxidizing mode. This appears to be counter to conventional regeneration wisdom and allows the possibility for the formation of explosive vapor/air mixtures near the charge-end of the kiln or in the exhaust duct between the kiln and the afterburner. Similar operation of the APE 1236 and CAMDS furnaces at Tooele Army Depot during agent deactivation tests have raised serious safety concerns regarding vapor explosions [Burton et al. (1987)]. Further evaluations of the mobile unit should be made in order to determine if the air inleakage can be sufficiently controlled to consistently operate with a reducing atmosphere. Installation of a "flame arrestor" between

the kiln and the afterburner would reduce the likelihood of a flashback. Installation of continuous-reading combustibles and oxygen analyzers at the kiln exhaust is also recommended.

Initiation and propagation of detonation - Until more detailed characterization tests are completed on typical explosives-contaminated carbon, it will not be possible to make definite conclusions regarding the likelihood of initiation and propagation of detonation. However, two aspects of the mobile regenerator design have the potential for increasing the hazard: (1) the screw feeder and (2) the longitudinal kiln flights. Friction between the screw-feeder barrel and the screw flights would be a likely cause of initiation if the feed is sufficiently sensitive. The high feed moisture should reduce the danger, but sensitivity and propagation tests of the spent carbon and screw feeder friction measurements are needed to develop quantitative estimates. Once a detonation is initiated, the longitudinal kiln flights would probably not provide a significant barrier to propagation. A better barrier could be provided by modifying the flights to a helical design along the lines of that used in the Tooele APE 1236 furnace. However, the review team consensus is that such a major revision to the kiln would probably not be warranted for a demonstration test as long as the aforementioned personnel protection measures are taken.

In addition to the previous safety concerns, the review team also noted the following technical points regarding recent testing of the EPA mobile unit at Edison in July 1986 with carbon from the Stringfellow, California, landfill site and proposed field testing of the mobile unit at Stringfellow:

1. During the July 1986 test, the mobile unit operated continuously for ~22 h at a nominal feed rate of 45.4 kg/h (100 lb/h), dry basis. In these tests it was estimated that ~5% of the feed carbon was lost as fines. Most of these fines appeared to exit the system through the manually operated drain at the bottom of the charge-end exhaust duct. The virgin carbon appeared to be approximately equivalent to the typical GAC used at Army ammunition plants. Altogether, ~1360 kg (3000 lb) of carbon was received from the Stringfellow disposal site. This carbon was contaminated primarily with chloro-benzenesulfonic acid

(CBSA). After regeneration, the Stringfellow carbon regained only about 60% of its original adsorptive capacity. The mass of carbon returned to Stringfellow was not measured. A major problem in the recent test was the large amount of fines in the as-received carbon. It is not known if the fines were originally present at Stringfellow or were generated during handling and transit. A large fraction of these fines was apparently carried out with the water in the dewatering screw.

2. Some CBSA-contaminated water was released to the sewer during the Stringfellow carbon test. It was indicated that for normal operation, the mobile unit produces a continuous discharge of ~38–45 L/min (10–12 gal/min) of contaminated water when there is no water recirculation and a one-time discharge of about 1900 L (500 gal) when total recirculation is used. The caustic scrubber water must be bled off at ~7.5 L/h (2 gal/h) in both cases. Although the discharge water was not treated in the recent test, treatment measures could be relatively easily implemented in future tests. The posttest decontamination procedure involves washing down all process surfaces with an aqueous detergent solution. After the recent test, between 1900 and 3800 L (500 and 1000 gal) of contaminated detergent solution were produced from decontamination.

3. The inclined dewatering screen used in the recent test was prone to blinding and did not perform very well. A new dewatering screen has been obtained, but it has a similar design and might be expected to have similar problems. The screen opening size is about 0.025 cm (250 μ m or U.S. std. sieve No. 60).

4. Under typical operating conditions with 45.4 kg/h (100 lb/h) carbon feed to the kiln (dry basis), the kiln operating characteristics include the following:

- an estimated 23 kg (50 lb) of carbon holdup on a dry basis,
- an estimated residence time of 30 min,
- 480 K (400°F) gas discharge temperature at the kiln feed end,
- 1150 K (1600°F) gas temperature at the kiln burner end, and
- an estimated 3% O₂ in the kiln exhaust gas not including air inleakage, 6% including the inleakage.

5. The kiln end seals are constructed from an asbestos-like fabric that is clamped on the fixed ends and rides on the rotating barrel with a steel spring holding it in place. It appears that this design, when properly maintained, should be adequate to keep the air inleakage to a relatively low level, but estimates from the recent test indicate a higher than expected air inleakage. One possible source of this inleakage is the spent carbon feed system (i.e., the hopper, transfer chute, and screw feeder). Correlation of performance data with the kiln operation is made much more difficult by the lack of gas composition measurements at the kiln exhaust and the lack of axial temperature and composition measurements. Test analysis and process optimization could be greatly facilitated by installation of a gas sampling and analysis system on the kiln exhaust duct and installation of an axial thermocouple and gas-sampling probe.

6. All sampling and sample analyses in the latest test were done by a subcontractor, Trace Technologies. This includes carbon, process water, scrubber brine, and stack samples. The Enviresponse personnel estimate that experienced boiler operators can be trained to operate the mobile regenerator in only 1-2 d. They routinely hire operators from outside agencies for demonstration tests involving similar equipment. For the proposed demonstration test at the Stringfellow landfill in California, it is expected that three operators will be required per shift.

7. The estimated cost for the proposed Stringfellow landfill field test is \$1.5M (1986). The test is planned to last ~2 months, 21 d of which the mobile regenerator will be operated. A batch of ~5900 kg (13,000 lb) of carbon (dry basis) will be alternately loaded and then regenerated to measure the effectiveness of the regeneration. The \$1.5M includes permitting, equipment preparation prior to transport, transportation to the site, setup and shakedown, testing, sampling and sample analysis, decontamination and dismantling, return equipment transport to Edison, and final report preparation. This estimate is substantially higher than the \$545K given in the original test proposal document [EPA (1985)].

3.2 Shirco Infrared Regenerator (EBF)

The second mobile process evaluated for the pilot-scale testing of explosive-contaminated carbon regeneration is the Shirco electric-belt furnace. Shirco, Inc. of Dallas, Texas, is a relatively new company specializing in the application of its proprietary infrared technology (i.e., electrically heated tunnel furnaces) to the problems of hazardous waste incineration and carbon regeneration. As shown in Fig. 3.2, the infrared carbon regeneration system utilizes a woven-wire conveyor belt to carry the carbon through the tunnel furnace, exposing it to radiant heating under controlled residence time and atmosphere.

The main components of the Shirco electric-belt furnace are the tunnel furnace and conveyor, the feed and discharge equipment, and an off-gas afterburner. Spent carbon is fed with ~50% moisture into the kiln through one of three feed systems: gravity feed from a hopper, an inclined belt feeder, or a screw feeder. The carbon is deposited onto the wire mesh conveyor belt in a layer ~1.9 cm (0.75 in.) thick, and the residence time is controlled by the variable-speed motor driving the belt. As the carbon moves through the furnace, it is heated by infrared radiation from resistance heaters above the belt.

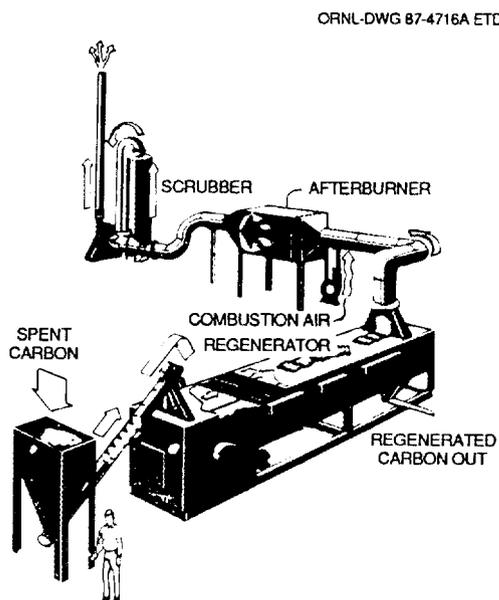


Fig. 3.2. General layout of the Shirco infrared carbon generator [adapted from Hill (1986)].

The three temperature zones within the furnace are independently controlled. A slightly negative pressure at the discharge end causes a co-current flow of off-gas through the furnace. The gas is composed of pyrolysis and combustion products from the volatiles driven off from the carbon and steam produced as the moist carbon dries. Normally, the water vaporized from drying provides sufficient steam for reactivation; however, additional steam ports are available if more steam is required. The off-gases are vented to an afterburner where air is injected and combustion takes place at 1370–1480 K (2000–2200°F), with a gas residence time of about 2 s. The off-gas is quenched and scrubbed with caustic.

3.2.1 Key safety concerns regarding the Shirco EBF

The major findings of the study team regarding the suitability of the Shirco portable regenerators for testing with explosive-laden carbon are as follows:

Remote operation — As in the case of the EPA mobile regenerator, the study team considers it necessary to remove personnel from the immediate vicinity of the furnace during the feeding of explosives-contaminated carbon of unknown or variable properties. Under normal circumstances, the Shirco units are operated from a control panel immediately adjacent to the furnace and some direct manual adjustments are made to the feed system during operation. It is likely that the control panel can be relatively easily relocated into a more protected area, for example, a van located some minimum distance from the regenerator and/or appropriately shielded. It was estimated that the actual rewiring of a separate remote control panel could be accomplished in only a matter of 4–5 h. Most of the important operating controls are located on the existing control panel. A precedent for remote operation of a Shirco system has been set at the DOE Savannah River Plant where there is a Shirco incinerator being used to incinerate radioactive wastes. This incinerator is reportedly operated entirely by remote control.

Feed system — Some minor modifications to the feed system will be necessary to make it remotely operable. Bridging in hoppers and chutes is the principal problem requiring direct operator intervention. Shirco

normally uses one of three feeder systems on the portable unit: simple gravity feed from a hopper, an inclined belt feeder, or a screw feeder. It appears that the inclined belt conveyor would be the best method for dewatering and metering the feed to the furnace. With an enlarged inlet hopper, the inclined belt conveyor could be fed remotely with a carbon-water slurry.

Remote sampling - Water and stack gas sampling requirements for the Shirco portable unit are similar to those of the EPA mobile unit. Provisions should be made to either provide adequate shielding at sample sites near the furnace or relocate the sample lines to a safe protected area. If the carbon slurry/inclined belt feed system is used, feed samples can be taken at the slurry pump.

Wiring system - The instrument and electric-power wiring on the portable unit are not explosion-proof for combustible dust or vapors, but it appears that the appropriate modifications could be made more easily than on the EPA mobile unit. The external resistance heater connections are enclosed in ducts that can be purged to prevent accumulation of dust or vapors, and most of the other wiring is enclosed in conduits that could be sealed or purged. The need for explosion proofing is also less for the Shirco unit because there are no sliding seals that could emit dust and no auxiliary liquid fuels that could spill or release combustible vapors. Proper insulation and grounding of the furnace heating element systems is important because the applied voltages can be as high as 535 V.

Furnace atmosphere - The Shirco portable regenerator is normally operated under reducing conditions. Air inleakage is limited to small flows through the solids feed and discharge chutes and is minimized by maintaining only a slight pressure differential between the furnace and the atmosphere. It is also common practice not to inject activating steam into the furnace, relying instead on the steam produced by the carbon drying. The lack of an oxidizing atmosphere in the furnace greatly reduces the potential vapor explosion hazard and effectively eliminates the possibility of a flashback from the afterburner.

Carbon accumulation in the furnace - Shirco personnel indicated that typically about 1% of the carbon fed onto the belt conveyor is

vibrated off the belt and falls to the bottom of the furnace. Provisions should be made to periodically remove this material from the bottom of the furnace to prevent accumulation of potentially dangerous levels of contaminated carbon. A related problem that should be considered is the possibility and consequences of a belt failure (i.e., the failure of the belt so that the entire carbon load is dumped onto the floor of the furnace).

Initiation of detonation - The inclined belt feeder would reduce the likelihood of initiation due to friction as compared to the screw feeder. Once the carbon is on the woven-wire belt, however, there are two more potential initiation sources: the impact of the cakebreakers and the slow heating of the explosive components in the carbon layer. The relative importance of these potential initiation sources cannot be estimated until the explosives characterization data are available, but it appears that the furnace operation could be modified to accommodate these concerns. For example, the cakebreaker action could be reduced or eliminated and the carbon layer thickness and feed zone temperature adjusted to allow more rapid heating. Slow "baking" of the feed carbon in the feed chute after an emergency shutdown or power failure might also lead to an explosive initiation. Measures could be taken to reduce this possibility, such as shielding the chute from furnace radiation and water cooling the chute walls.

Propagation of detonation - The continuous layer of carbon on the belt conveyor could constitute a favorable path for propagation of a detonation front. There are no barriers present to inhibit propagation, and each solid particle (which could be contaminated carbon or a particle of nearly pure precipitated explosive) lies adjacent to many others. A direct path would be available from the belt up into the feed chute. Until characterization data and operating experience are available to demonstrate otherwise, the feed carbon should be assumed to be capable of propagation and the appropriate design and operating standards instituted. For testing purposes, the uncertainties regarding the possibility of full detonations are best dealt with by insuring that totally remote control and remote sampling are used, that the test system is located sufficiently far from any other flammable or explosive

materials, and that adequate barriers are installed to protect personnel and vital equipment should a full detonation occur.

In addition to the previously described safety concerns, the study team noted the following features of the Shirco portable regenerator:

1. There is relatively little auxiliary equipment required. The lack of a primary combustor eliminates the need for a primary air blower, an auxiliary fuel system, and the primary burner controls. The use of steam generated directly from the spent carbon eliminates the need for a steam generator and steam addition system. In most circumstances, the need for an auxiliary fuel in the afterburner is also minimal because of the fuel value of the furnace off-gases.

2. Control of the temperature profile and residence time are more precise in the Shirco portable unit than in a rotary kiln. There are two heating zones in the Shirco unit, each of which can be individually controlled. By experience it has been determined that good performance is obtained for many carbons with gas temperatures of 1170 to 1230 K (1650 to 1750°F) in both zones. The use of the belt conveyor in the electric furnace creates an essentially plug-flow condition, whereas the tumbling action of the kiln introduces a random axial mixing of the carbon particles.

3. The conveyor belt produces less attrition than the rotary kiln. Typical Shirco experience is that the total system attrition losses are about 5 to 7% (this includes attrition in the screw feeder). Losses due to carbon oxidation are estimated to be about 2 to 3%.

4. From a maintenance and operational reliability standpoint, the wire-mesh conveyor belt is probably the most vulnerable single piece of equipment. Until more operational data are available for analysis, it will be difficult to judge the relative frequency and severity of belt failures caused by erosion and corrosion.

5. The nominal feed capability of the portable Shirco regenerators is 13.5 kg/h (30 lb/h) of carbon (wet). These units can also be configured as waste incinerators. At their testing and development laboratory, Shirco has a test regenerator capable of processing 18 kg/hr (40 lb/h) of dry carbon (i.e., 36 kg/h wet) with up to a 12:1

turndown. They also have bench-scale thermogravimetric (TG) units for batch testing small samples prior to running continuous tests on the tunnel furnaces. The TG furnaces are capable of temperatures up to 1370 K (2000°F) and can be purged with combinations of gases including oxygen, nitrogen, inert gases, and steam. This capability can be used to optimize the regenerator performance.

6. Shirco now has three portable units available for field testing (one in Europe and two in this country). A fourth unit is now under construction. They plan to have six units available by the end of the year. These units are built so that they can fit on a trailer 13.7 m (45 ft) long by 2.4 m (8 ft) wide by 4.1 m (13.5 ft) high. The loaded trailer weighs 12,700 kg (28,000 lb). The approximate cost of each portable unit (trailer ready) is \$330K (1986).

7. One of the Shirco portable units was demonstrated at Twin Cities Army Ammunition Plant the week of January 19, 1987. The purpose was to test the effectiveness of regenerating granular activated carbon loaded with trichlorethylene (TCE), and burning PCB-contaminated soil. Two members of the study team witnessed the first stage of this test. Approximately 114 kg (250 lb) (dry) of carbon was processed during the regeneration. TCE loading on the carbon was measured at 7-10%, and the feed rate to the furnace was 11.4-13.6 kg/h (25-30 lb/h) wet carbon (30% water). The estimated total cost for the Twin Cities test was \$80K, including permitting, site preparation, transportation, decontamination, and data analysis. Normally Shirco prefers that the customer handle the subcontracting for water and stack sampling, and these costs are not included in the \$80K. Federal Cartridge, the contractor for Twin Cities, had arranged with Interpoll, Inc. of Circle Pine, Minnesota, for analyses of stack emissions, feed, and product carbon. Total analysis of the stack gases and product carbon will be included in a report to be issued by Federal Cartridge.

8. To conduct a carbon regeneration demonstration similar in scope to the Shirco test at Twin Cities, it is expected that a portable unit would require

- 30 kw of 480-V electrical power (can be supplied from a portable generator),

- a standard 120-V wall outlet to supply lights and fans,
- a source of process water capable of 10 gpm at 15 psi for initial fillup,
- a 3.8-L/min (1-gal/min) source of makeup water,
- flat dirt or gravel setup area, and
- a 2.8-std-m³/h (100-std-ft³/h) propane supply (45-kg propane bottle).

3.3 Estimated Costs for Pilot-Scale Testing

The scope of a pilot-scale test at an Army facility has not yet been clearly defined. In order to fully determine the effectiveness of the technology, however, it is likely that such a test would probably involve three complete adsorption/regeneration cycles using several thousand kilograms of carbon. The following are estimated costs (1986\$) and time requirements to conduct such a program:

1. Prepare test site at suitable munitions facility: 2 weeks at \$10,000.
2. Negotiate sampling and operating contracts: 4 months at \$20,000.
3. Negotiate EPA permits: 6 months at \$50,000.
4. Transport and set up at suitable facility: 3 weeks at \$8000.
5. Conduct shakedown test (utilities and labor included in item 3): 2 months at \$20,000.
6. Conduct three regeneration/adsorption cycles (utilities, carbon, and miscellaneous labor included in item 3): four months at \$300,00-500,000.
7. Tear down, clean, and transport unit: 3 weeks at \$7500.
8. Data reduction and documentation: 6 months at \$60,000.

In addition to the above, equipment modifications would be necessary to make either of the two candidate systems suitable for use with GAC contaminated with explosives. The estimated costs of these modifications are summarized in Tables 3.1 and 3.2 for each technology.

Table 3.1. Costs for modifying the EPA mobile regenerator for a pilot test (1986\$)

Modification	Cost (\$1000)
Establish remote control panel	100
Rewire trailer to explosion-proof standards	
Labor	10
Materials	50
Carbon feed system development	150
Installation of new feed system	20
Miscellaneous modifications	50
Remote sampling system	50
Engineering (30% of above)	130
Contingency (30% of above)	170
Total	730

Table 3.2. Costs for modifying the Shirco portable regenerator for a pilot test (1986\$)

Modification	Cost (\$1000)
Modify control panel to be remotely located	10
Rewire to meet explosion-proof standards	50
Modify feed system	50
Provide remote sampling system	60
Engineering (30%)	50
Contingency (30%)	70
Total	290

4. COST COMPARISON OF PRODUCTION-SCALE STRATEGIES

4.1 Regeneration/Disposal Strategies

Seven preliminary production-scale regeneration/disposal strategies were evaluated to obtain rough estimates of the cost differences that could exist among reasonable alternatives. Each of these alternatives was evaluated for three separate munitions facilities and compared on the basis of unit cost, that is, the present value of a particular alternative's life-cycle cost divided by the total amount of carbon treated by that alternative during its economic life. A summary of the major economic assumptions is given in Table 4.1.

The Radford Army Ammunition Plant (RAAP), IAAP, and MAAP were chosen as representative facilities because they represent major installations with a broad range of spent carbon production and geographical locations. Considering different facilities permitted estimation of the cost sensitivity to treatment rates and transportation distances. The nominal annual carbon production rates assumed for each are summarized in Table 4.2.

Five of the seven treatment strategies were selected as representing a reasonable range of options for rotary kiln and EBF regeneration technologies. The remaining two strategies were included as "baseline" cases for comparison. The following seven strategies were considered.

Thermal treatment with disposal - Represents the current practice followed at IAAP, with the difference that existing explosive waste incinerators (such as the Army's APE 1236 rotary kilns) are modified to operate with flue-gas control (i.e., afterburning and scrubbing) equivalent to that used on the carbon regenerators. The thermally treated carbon is shipped to a hazardous-waste landfill for disposal.

Fixed, rotary - Assumes a fixed (i.e., nonmobile) rotary-kiln regeneration system at each facility.

Fixed, IR - Assumes a fixed EBF regeneration system at each facility.

Fixed, central - Assumes a fixed rotary-kiln regeneration system located at RAAP, which treats the explosive-contaminated carbon from all three facilities.

Table 4.1. Major assumptions in production-scale cost estimates

All options

- Present value based on 10% and 10 years project life
- Depreciation and other tax considerations not included
- Constant dollars
- Construction time 1 year, therefore no interest during construction
- No insurance, royalties, or property taxes included
- Existing U.S. Army land utilized for thermal treatment and/or regeneration, therefore no land cost
- Systems will be designed, procured, and constructed by third parties
- Landfill disposal costs for hazardous solid wastes are \$100/barrel, including transportation

Thermal treatment with disposal

- Existing explosive waste incinerators modified to include flue-gas control equivalent to that used with the regenerators
- Labor costs include those required to feed carbon to the furnace and collect, drum, and ship desensitized product

Fixed regeneration

- Nominal operation 112 h per 5-d week, two chemical operators, one laborer, and one supervisor per 12 h
- Annual maintenance cost is 5% of capital cost
- Activated carbon is nonregenerable after four regeneration cycles and must be replaced
- Nonregenerable (fourth-cycle) carbon is disposed of as a hazardous waste
- 10% carbon loss for each cycle of rotary-kiln regeneration
- 5% carbon loss for each cycle of EBF regeneration
- Propane, \$2/lb; electricity, \$0.07/kWh

Mobile regeneration

- Two trucks will be required
- Two d travel time between sites
- Twenty-day cycle
- Two chemical operators, one laborer, one supervisor per shift
- Two shifts
- Nonregenerable (fourth-cycle) carbon is disposed of as a hazardous waste

Table 4.2. Assumed annual spent carbon production rates

Facility	Dry pounds per year
Radford	93,000
IAAP	10,000
MAAP	50,000

Mobile, rotary - Assumes a mobile rotary kiln that continuously travels between the three facilities.

Mobile, IR - Assumes a mobile EBF that continuously travels between the three facilities.

Third-party treatment/disposal - Assumes each Army facility transfers their explosive-laden carbon to a third party for treatment and disposal at a specialized explosive-waste facility.

Third-party regeneration by commercial carbon suppliers is another option that has been widely used for conventional carbon but was not considered in the present analysis. Our decision not to include this option was based on (1) the finding that no existing commercial regeneration company is licensed to handle and process explosive materials, (2) the indication that some pure explosive can occasionally be found in the raw spent carbon, (3) the lack of reasonable data on the costs required to upgrade the vendors facilities for explosive handling, and (4) the anticipated difficulties involved in segregating the explosive-contaminated carbon from the conventional carbon being processed by the vendor. If these issues can be resolved in the future, third-party regeneration should be reconsidered.

It should be noted that the results of any regeneration/disposal analysis are highly dependent on the assumed spent-carbon production rates and the ability or inability of commercial regenerators to handle explosive-contaminated carbon. Deviations from the assumptions used here can lead to very different conclusions. This point is clearly demonstrated in the complementary carbon regeneration cost study recently performed by A. D. Little [A. D. Little (1987)].

4.2 Cost Comparison

Conceptual cost estimates were prepared for the seven alternatives at each facility. The total annualized capital and operating costs for each case are summarized in Table 4.3. These results illustrate some important points. For instance, in the fixed-rotary-kiln scenario, the capital costs for the three sites are very close. This is because the only difference in the capital costs for each site is the cost of the

Table 4.3. Summary of the estimated capital and annual operating costs for the seven regeneration/disposal alternatives
(FY-1986 dollars)

Alternative	Facility		
	Radford	IAAP	MAAP
Thermal treatment with disposal			
Capital	97,000	97,000	49,000
Operating	286,000	86,000	176,000
Fixed, rotary			
Capital	1,043,000	1,029,000	1,036,000
Operating	200,000	96,000	140,000
Fixed, IR			
Capital	884,000	870,000	877,000
Operating	203,000	92,000	139,000
Fixed, central			
Capital	640,000	69,000	344,000
Operating	230,000	73,000	172,000
Mobile, rotary			
Capital	782,000	276,000	520,000
Operating	584,000	68,000	312,000
Mobile, IR			
Capital	1,167,000	317,000	727,000
Operating	582,000	68,000	316,000
Third-party specialist disposal			
Capital	0	0	0
Operating	933,000	212,000	560,000

initial carbon inventory. The same is true for the fixed EBF. A comparison of the operating costs of the fixed rotary kiln and the fixed EBF also shows little difference. Utilities for the EBF are generally higher, but this is balanced by anticipated lower maintenance costs. Capital costs for the mobile EBF are higher than those for the mobile rotary kiln, but the operating costs for the two scenarios are again very close.

The final results are summarized in Table 4.4, which shows the unit costs for each alternative at each facility. Recall that the unit cost for an alternative is the present value of the life cycle costs divided by the total carbon treated during the project life. Two unit costs are given for the thermal treatment and disposal option, one including capital costs to improve the flue-gas control of the existing incinerators and one without these capital costs. The relatively small differences in the results for this case indicate that the capital cost is less important than other factors.

Table 4.4. Estimated unit costs for the seven regeneration/disposal alternatives
(FY-1986 \$/lb)

Alternative	Facility		
	Radford	IAAP	MAAP
Thermal treatment and landfill disposal	1.99 (1.89) ^a	6.26 (5.29) ^a	2.26 (2.16) ^a
Fixed, rotary	2.45	16.20	3.79
Fixed, IR	2.29	14.37	3.47
Fixed, service	2.21	5.16	2.80
Mobile, rotary	4.70	6.91	4.87
Mobile, IR	5.10	7.37	5.34
Third-party disposal	6.16	13.04	6.88

^aAssumes no capital cost for incinerator modifications to improve flue-gas control.

4.3 Conclusions

The most important conclusion that can be drawn from the preceding analysis is that the cost effectiveness of each strategy varies considerably from plant to plant depending on the spent carbon production rate and/or transportation requirements. These results confirm that the most cost-effective treatment option is dependent on the individual circumstances of each facility and the basic management approach adopted, that

is, whether regeneration will be treated as an Army-wide concern or a local concern. These issues should be carefully considered prior to selection of any particular strategy.

In general, it appears that the thermal treatment with disposal option is less expensive than the other alternatives at relatively low spent carbon production rates. This option is even more attractive if the Army ammunition plants (AAPs) are allowed to use their existing explosive waste incinerators without additional flue-gas controls (which, in fact, is being done at IAAP and seriously being considered at MAAP). However, it also appears that public pressure to move away from land disposal will continue to increase and make this solution less acceptable, unless the thermally treated carbon can be decertified as a hazardous waste.

Of the regeneration strategies, a fixed central unit with carbon shipped from the other sites appears to be the least costly option, especially as production rates increase. The mobile option is expected to be more expensive than the fixed, site-specific option because of the higher cost for transportation and equipment setup, dismantling, and decontamination.

Treatment and disposal by a third party at a specialized waste explosives facility appears to be the most expensive option at the moment. At least one commercial firm, Emergency Technical Services Corporation (ETC) of Schaumburg, Illinois, has experience with other explosive wastes and has expressed an interest in treating and disposing of explosive-contaminated carbon. While such firms are unlikely to have expertise in activated carbon, they are much more likely to have explosives expertise than commercial carbon regenerators.

One issue that was not specifically addressed in this study, but is common to all regeneration options, is the need to ultimately dispose of the waste carbon (i.e., the carbon that can no longer be regenerated). Even with the most optimistic estimates of carbon regeneration efficiency, it is clear that waste must be removed from the regeneration cycle and disposed. If the waste is landfilled, it must be rendered nonexplosive with a procedure similar to the current IAAP thermal treatment. Unless the desensitized waste can be delisted as a RCRA hazardous

waste (not done at IAAP), landfilling will be costly, and the basic issue of land disposal will still exist. Complete incineration of waste carbon is the only clear alternative to landfilling. Even incineration will not completely destroy the waste, because approximately 10% of the carbon is noncombustible mineral ash. Incineration will greatly reduce the volume, however, and it is much more likely that the ash residue can be delisted.

5. BASIC ENGINEERING DATA ON ACTIVATED CARBON

More detailed consideration of regeneration, treatment, and disposal options will require basic information about the carbon's physical and chemical properties. Some important studies of the production rates and properties of explosive-contaminated carbon not covered in the references are summarized in Appendix A. Additional characterizations of activated carbon were carried out in conjunction with the present study and are summarized in Appendixes B, C, and D. While by no means exhaustive, Appendix B summarizes important supplemental background on typical virgin GAC including size distributions, bulk density and water retention characteristics, proximate and ultimate chemical analysis, nitrobody retention capacity, and combustion behavior. Appendix C summarizes the adsorption and desorption characteristics of dinitrotoluene (DNT), which is a major by-product of TNT manufacture that is not addressed in the previous studies. Appendix C also provides data on the extraction of nitrobodyes from carbon, which is a key step in the quantitative analysis of spent carbon samples. Appendix D summarizes the results of preliminary propagation of detonation tests that were performed on samples of IAAP spent carbon at Los Alamos National Laboratory.

6. OVERALL CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The overall conclusions regarding the thermal regeneration of explosives-contaminated carbon are:

- The most important commercial technologies for thermal regeneration of activated carbon are the rotary kiln, the multiple-hearth furnace, the fluidized-bed furnace, and the electric-belt furnace.
- Of the four major commercial technologies, the rotary kiln and the electric-belt furnace appear to have important advantages over the other processes for the regeneration of activated carbon contaminated with explosives. The electric-belt furnace is generally more adaptable than the rotary kiln, and it seems to be more appropriate for regenerating carbons at relatively low production volumes and highly variable or unknown properties. The rotary kiln is more suitable for higher production volumes and for carbons that require less precise control over the regeneration conditions.
- Following extensive modifications, the EPA mobile rotary kiln regenerator could probably be used to safely field test the regeneration of explosives-contaminated carbon. The necessary modifications would include changes to the control, electrical, feeding, and sampling systems so that personnel would be protected from exposure to potential explosions of the contaminated carbon. The estimated cost of these revisions including engineering and contingency is estimated to be \$730,000. The total required time for modification is estimated to be 6 to 8 months.
- A portable electric-belt regenerator could also probably be modified to safely field test the regeneration of explosives-contaminated carbon. Like the EPA mobile unit, a portable commercial electric-belt furnace would require modifications to the control, electrical, feed, and sampling systems to protect personnel from exposure to potential explosions. For a typical portable system these modifications are estimated to cost \$290,000, including engineering and contingency. The total required modification time is estimated to be 3 months.

- For low production rates, thermal treatment with disposal is expected to be less expensive than regeneration. However, it also appears that public pressure to move away from land disposal will continue to increase and make this solution less acceptable, unless the thermally treated carbon can be delisted as a hazardous waste.
- Of the regeneration strategies considered, a fixed central unit with carbon shipped from the other sites is expected to be the least costly option. The mobile option is more expensive than the fixed, site-specific option because of the higher cost for transportation and equipment setup, dismantling, and decontamination. The advantages of a central regeneration facility are expected to increase at higher production rates.
- Ultimate disposal of unregenerable waste carbon is an important issue for all potential regeneration strategies, which has not yet been adequately considered. Thermal treatment to the point of decertification (up to total incineration of the waste carbon to ash) appears to be the only viable solution other than landfilling.

6.2 Recommendations

It is recommended that the Army pursue the following:

- Continue refinement of engineering and cost information regarding the possibility of conducting a pilot field test with a mobile regenerator at an Army facility producing explosives-contaminated carbon.
- Continue characterization of typical explosives-contaminated carbon to better determine the possibilities of initiation and propagation during regeneration.
- Fully characterize the thermally treated carbon from IAAP and determine the technical merits of seeking delisting of this waste as it is.
- Conduct small-scale thermal treatment tests on explosives-contaminated carbon to determine the conditions required to reach a level of decontamination that would provide a clear technical basis for delisting.

- Continue to develop more detailed cost and technical information regarding the most promising production-scale alternatives and the economic incentives for regeneration. The alternatives investigated should include (1) nonthermal options such as in situ solvent regeneration with combustion of the solvent as a supplemental fuel and (2) direct burning of the carbon to ash with no regeneration.
- Continue discussions with potential third-party regenerator/treatment companies to determine if these options would ever be technically or economically justifiable.

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Appendix A

ADDITIONAL REFERENCES RELATING TO THE PRODUCTION AND
PROPERTIES OF EXPLOSIVE-CONTAMINATED CARBON

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Appendix B

TYPICAL PROPERTIES OF VIRGIN GRANULAR ACTIVATED CARBON

Materials

Two commercial activated carbons (Filtrisorb 300 and Filtrisorb 400) produced by Calgon Corporation were chosen for analysis because (1) they are widely used for munitions wastewater treatment, and (2) they appear to be the basis for the standard purchasing specifications used by most ammunition plants. Representative 1-kg samples of each carbon were obtained directly from Calgon Corporation in Pittsburgh. Smaller representative samples were riffled from these original samples for each of the various analyses.

Physical Properties

The results of the various physical measurements made on the two Calgon samples are reported in Table B.1. The basic methods used in each case are summarized below. Where appropriate, American Society for Testing and Materials (ASTM) reference procedures are cited [American Society for Testing and Materials (1987)].

Particle-size distribution

The basic screening procedure outlined in ASTM method D 2862 was followed, except that the mechanical shaker used was a Gilson testing screen model TS-1. The sieves used had opening dimensions of 3360, 2380, 2000, 1700, 1180, 850, 600, 500, 425, 300, and 212 μm . Figures B.1 and B.2 graphically illustrate the the particle-size distributions listed in Table B.1. The unusual shape of the Calgon FS 400 graph indicates a bimodal distribution.

Bulk and water-displacement density

Bulk (apparent) density was measured by a procedure based on ASTM method D 2854. The volume displaced by a known mass of carbon was measured by pouring the carbon through a funnel inserted in a 100-mL

Table B.1. Physical Characteristics of
Calgon Filtrasorb 300 and 400

<u>Particle size distribution</u>		
U.S. std sieve size, μm	Oversize (wt %)	
	FS 300	FS 400
3360	0.00	0.00
2380	9.00	0.00
2000	27.19	0.49
1700	47.58	30.41
1180	73.73	67.16
850	90.14	68.27
600	97.43	87.96
500	98.96	93.94
425	99.27	97.07
300	99.33	99.44
212	99.39	99.51
Weight mean diameter, μm	1641	1304
Harmonic mean diameter (μm)	1308	1008
<u>Density</u>		
	(g/cm ³)	
	FS 300	FS 400
Bulk: Unpacked	0.50	0.45
Packed	0.55	0.48
Water displacement	1.24	1.25
<u>Moisture</u>		
	(Percent of total)	
	FS 300	FS 400
As received	3.45	3.58
Drained	55.00	63.00
<u>Nitrogen BET</u>		
FS 300:	950 m ² /g	
FS 400:	1050 m ² /g	
<u>Abrasion No.</u>		
FS 300:	75%	
FS 400:	75%	

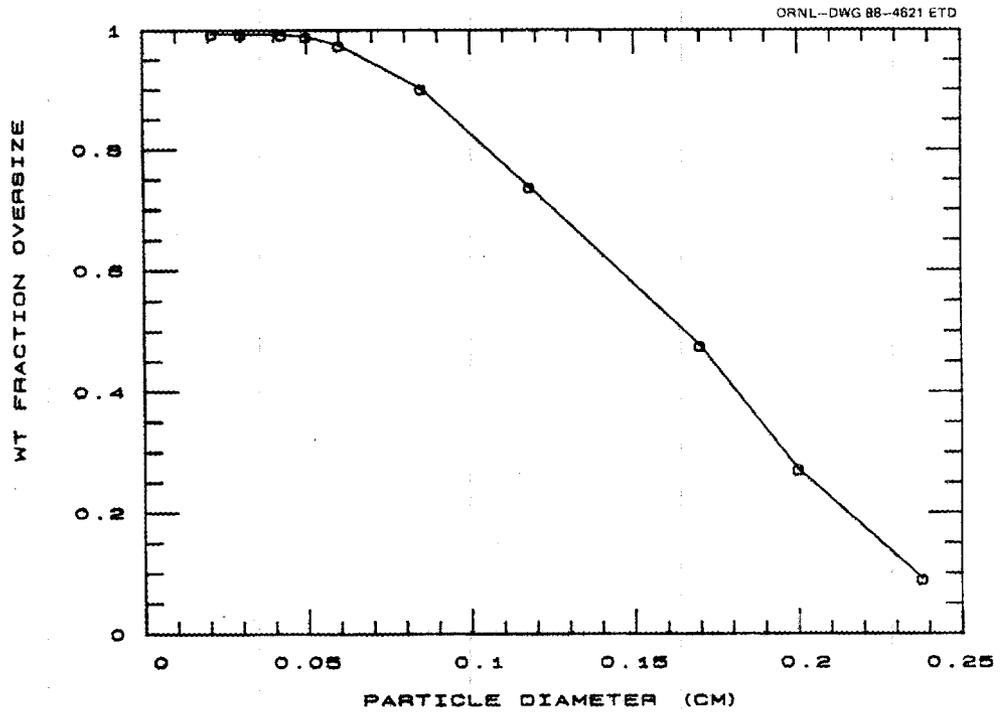


Fig. B.1. Particle-size distribution of typical "large" granular activated carbon.

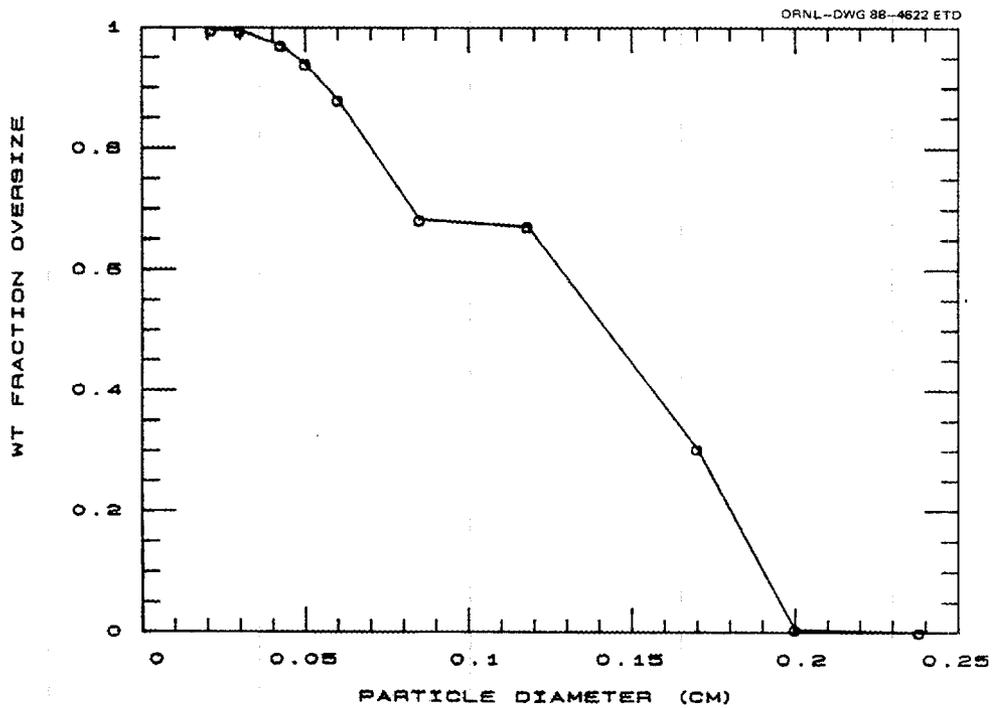


Fig. B.2. Particle-size distribution of typical "small" granular activated carbon.

graduated cylinder. Instead of using the metal feed vibrator recommended in the ASTM standard, however, carbon was gently spread into the funnel manually while the funnel and cylinder were slowly rotated. The reported unpacked bulk density is based on the carbon volume in the cylinder without tamping. Packed bulk density was determined by vigorously tamping the cylinder on the table until no further change in volume was observed.

Water-displacement density was measured by determining the volume of water displaced by a known mass of carbon in a graduated cylinder. The water/carbon mixture was carefully stirred until no more air bubbles were visible and the carbon was thoroughly wetted.

As-received and drained moisture

As-received moisture was determined by heating the carbons to 110°C according to the standard method for coke and coal (ASTM method D 3173).

Drained moisture was determined by pouring a water slurry of each carbon through a 150- μ m screen. After the water had completely drained, the carbon on the screen was dried as before at 40°C for 24 h.

Nitrogen BET surface area and abrasion number

Typical values for these properties were obtained from Calgon [Rogers (1986)]. The cited values were reportedly calculated from nitrogen adsorption data following the method of Brunauer et al. (1938) and ASTM method D 3802, respectively. Both of these measurements can be somewhat subjective and not directly correlatable with processing behavior, but they can still be useful for making relative comparisons with other carbons and other solids handled in bulk processes (e.g., coal and limestone).

Chemical Properties

The chemical characteristics of Calgon FS 300 and FS 400 are summarized in Table B.2. The basic methods used in each case are summarized below. As in Sect. 3, ASTM reference procedures are cited where appropriate.

Table B.2. Chemical Characteristics of Calgon Filtrasorb 300 and 400

<u>Ultimate analysis</u>		
	wt %	
	FS 300	FS 400
Carbon	87.73	87.36
Hydrogen	0.74	0.69
Nitrogen	0.55	0.39
Chlorine	0.0027	0.0022
Sulfur	0.91	0.54
Moisture	3.45	3.58

<u>Proximate analysis</u>		
wt %	FS 300	FS 400
Volatile Matter	2.55	2.87
Fixed Carbon	88.41	86.51
Ash	5.60	7.06

Freundlich adsorption parameters

$$X/M = a C_f^b$$

X/M = adsorbate loading on carbon, mg/gC
 C_f = aqueous concentration of adsorbate, ppm

Carbon	Adsorbate	a (mg/gC)	b
FS 300	DNT	210	0.171
	TNT	320-202	0.120-0.143
	TNT (with RDX)	193	0.123
	RDX	28.2	0.441
	RDX (with TNT)	26.9	0.078
FS 400	DNT	300	0.223
	TNT (with RDX)	290	0.127
	RDX (with TNT)	41.3	0.021

Combustion kinetics parameters

$$R_c = k_w P_{O_2}^n m_c$$

where

R_c = intraparticle-controlled burning rate, gC/s
 k_w = rate constant = $8.48 \times 10^8 \exp(-21,410/T)$,
(s⁻¹ atm⁻ⁿ)
 P_{O_2} = oxygen partial pressure at particle surface, atm
 m_c = mass of carbon present, g
n = reaction order in oxygen (-)

Proximate and ultimate analysis

Proximate and ultimate analyses were made of both carbon samples according to the standard procedures for coal and coke (ASTM methods D 3172 and D 3176, respectively). Duplicate analyses were run for each carbon and the results averaged to obtain the reported values.

Freundlich parameters for nitrobody adsorption

The Freundlich parameters reported are those for the virgin carbon. In general, pore plugging during regeneration tends to reduce the value of the parameter "a," while the value of the parameter "b" is affected little or not at all. The parameters for the adsorption of DNT were measured as described in Appendix C. The parameters for TNT and RDX adsorption were obtained from previous Army studies [Leeper, et al. (1977) and Haberman (1983)]. Ranges are given where these latter two references differ.

The parameters listed for TNT and RDX include values for both the pure contaminants and conditions in which competition for adsorption sites occurs between TNT and RDX. In general, TNT tends to displace RDX when both are present [Haberman (1983)]. This competition can result in previously adsorbed RDX being flushed from a carbon column as TNT is loaded.

Combustion kinetics

The low-temperature combustion kinetics of FS 300 were determined by thermogravimetric combustion of finely divided samples under controlled conditions. Riffled FS 300 samples were ground and double screened to 75-150 μm . Weighed samples of ~20 mg were heated in a Netzsch thermal analyzer at 10°C/min heating rate under gas-flow conditions previously determined to ensure chemical kinetic control of the particle burnout. Oxygen concentrations between 2 and 21 vol % were run. The burnout curves (particle weight vs time and temperature) were analyzed according to the procedures reported previously by Daw and Krishnan (1983) and Daw and Mitchell (1986) to determine the reaction order (in oxygen), the Arrhenius pre-exponential factor, and the

effective activation energy. The resulting Arrhenius rate expression for the intraparticle combustion kinetics is listed at the bottom of Table B.2.

Applying the standard methods developed for coal, it is possible to predict particle burnout times as a consequence of both intraparticle kinetics and external oxygen transport. Figure B.3 illustrates the estimated burnout time for FS 300 particles as a function of temperature and oxygen concentration. For purposes of conservative estimation, it has been assumed that the gas surrounding the particles is stagnant, with oxygen transport occurring solely by diffusion (i.e., the Sherwood number is equal to 2). In actual practice, oxygen convection is usually significantly greater than diffusion, and the actual burnout time will be correspondingly less. The other assumptions used to construct

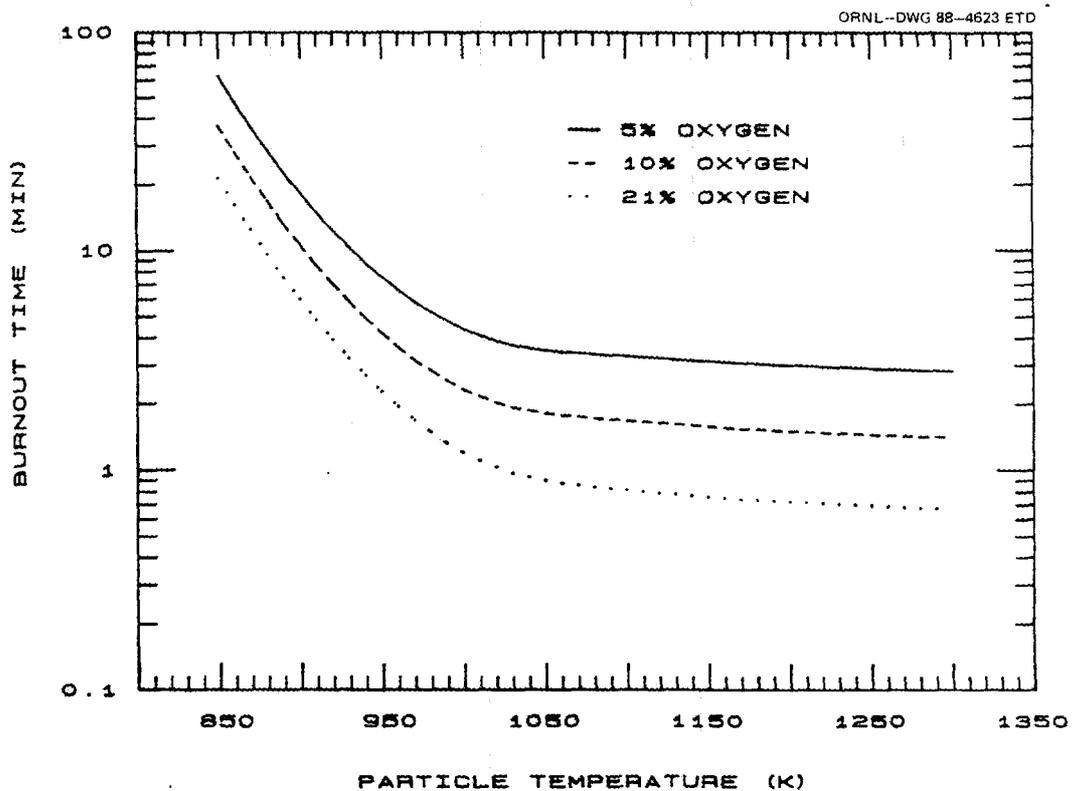


Fig. B.3. Estimated burnout time for "large" granular activated carbon.

Fig. B.3 are

- the effective initial carbon density in the particles is 1.09 g C/cm^3 ,
- particle burnout is essentially complete when 2% of the original carbon remains,
- the weight mean particle size of FS 300 ($1641 \text{ }\mu\text{m}$) is appropriate for estimating the burnout of the overall particle distribution, and
- particles burn effectively with a constant diameter (i.e., with a high degree of oxygen penetration) when the rate is controlled by the intraparticle kinetics.

An important point illustrated by Fig. B.3 is that the particle burning rate becomes almost independent of temperature above 1150 K (1600°F). This "flattening" of the burnout curve occurs because the combustion becomes controlled by oxygen transport from the bulk gas to the particle surface. External oxygen transport is only a weak function of temperature, and further increases in temperature produce little additional benefit.

Because the mean particle size of FS 400 is less than that of FS 300, it is expected that the burnout time of the former will be less than the estimates depicted in Fig. B.3.

Appendix C

ADSORPTION AND DESORPTION OF DINITROTOLUENE
ON ACTIVATED CARBONMaterials

2,4-Dinitrotoluene (Aldrich, 97%) was purified as described in Ho (1986). Methanol (Fisher, HPLC grade) and acetone (Fisher, certified ACS) were used as received without further purification. Water was deionized and double distilled. Two commercial granular activated carbons as described in Appendix B (Filtrisorb FS 300, nominal size 0.8-10 mm, and Filtrisorb FS 400, nominal size 0.55-0.75 mm) were obtained from Calgon Corporation. The FS 300 was used in the as-received size as well as being ground to $90 \times 212 \mu\text{m}$, while the FS 400 was used only in the as-received size. Both granular carbons were washed several times with distilled water to remove the fines and then dried at 105°C for 3 d.

Adsorption characterization

The adsorption capacities of the virgin and extracted carbons were measured by batch isotherm tests. In each case, a known weight of carbon was added to a measured weight of aqueous dinitrotoluene (DNT) solution (10 to 20 g with 25- to 120-ppm DNT) and the mixture placed in a small glass bottle (~30 mL) with a glass stopper. The glass bottles were then shaken for at least 3 d (at about 23°C) on an Ederbach shaker. After contacting, the samples were centrifuged, and the aqueous phase was analyzed by either high-performance liquid chromatography (HPLC) or by ultraviolet spectrophotometer.

Desorption characterization

Some of the desorption experiments were conducted with carbon loaded in the batch adsorptions. In addition, larger amounts of DNT-loaded carbon were produced for the desorption experiments by passing concentrated aqueous DNT solutions (at 4 mL/min) through a small vertical glass column packed with carbon (2 to 3 cm high, 0.50 cm in

diameter). The carbon was held in place with a porous Teflon plug and glass wool on the bottom. The spent carbon was then filtered, rinsed with a small amount of water (<1 mL), and dried on filter paper in a ventilated fume hood for at least 3 d. Typically, the spent carbon prepared in this fashion contained 30 to 40 wt % DNT (~430- to 665-mg DNT/g dry carbon).

Desorption experiments were conducted using column, batch, and Soxhlet procedures. In the column procedure, the spent carbon was packed in the small glass column described above and eluted with 3-5 mL/min of solvent. The solvents used were water, acetone, and methanol. In the batch procedure, 20 to 100 mg of carbon was shaken at room temperature with ~10 g of solvent. The shaking times used were 7 d for water and ≥ 1 d for acetone and methanol. Higher-temperature batch extractions were also carried out by placing the samples in a water bath and stirring them occasionally. In the Soxhlet procedure, ~100 mg of carbon was refluxed with ~300 mL of solvent at atmospheric pressure for 1 d. The extracts resulting from the desorption procedures were characterized by HPLC and GC/MS. Acetone extracts of the virgin uncontaminated carbon were run as controls.

Analytical methods

The concentration of DNT in water was analyzed by either a high-performance liquid chromatograph (HPLC) equipped with a Varian Vari-Chrom UV-VIS detector (reverse-phase column 25 cm long, 4.6 mm I.D., packed with Alltech C18) or by a Perkin-Elmer 559 UV-VIS spectrophotometer. The instruments were calibrated using standard solutions of DNT in water. DNT solutions in acetone and methanol were first diluted with water, and then the organic solvents were evaporated before HPLC or spectrophotometer analysis.

Compounds desorbed from the carbon were characterized by HPLC and gas chromatography/mass spectroscopy (GC/MS). For HPLC, the extracts were diluted with water prior to injection into the instrument. For GC/MS, the extract solutions were filtered, the solvent was evaporated, and the residue was dried under nitrogen for several hours. The dried

residue was then silylated with MSTFA (N-methyl-N-trimethylsilyl-trifluoroacetamide) (Pierce) before injection into the GC/MS. The GC/MS used was a Hewlett-Packard model HP 5995A fitted with a Durabond I narrow-band fused silica column (J. W. Scientific). The column temperature was programmed from 90°C (1-min hold) to 300°C with a 10°C/min temperature ramp.

Adsorption isotherms for virgin and regenerated carbon

The adsorption capacities of DNT on FS 300, FS 400, powdered FS 300, and regenerated FS 300 and FS 400 were measured by the batch method and fitted as Freundlich isotherms:

$$X/M = k C_f^{1/n} , \quad (C.1)$$

or

$$\log X/M = \log k + 1/n \log C_f , \quad (C.2)$$

where X/M is the amount of DNT (mg) adsorbed per gram of carbon at equilibrium, X is the amount of DNT (mg) adsorbed by carbon, M is the weight (g) of the carbon, C_f is the concentration (ppm) of the aqueous phase at equilibrium, and k and n are constants for a given adsorption system.

For the DNT concentrations in this study, Eq. (C.2) appears to hold true, and log-log plots of X/M vs C_f give straight lines with slope $1/n$ and intercept k at $C_f = 1$ ppm. The intercept k is an indication of overall adsorptive capacity. The slope $1/n$ indicates the characteristic energy of adsorption, with smaller values reflecting a lower energy.

Figures C.1 and C.2 show the linear least-square fits for each of the two virgin carbons and the corresponding acetone-regenerated carbons, respectively. Figure C.3 shows the adsorption isotherm for the powdered virgin FS 300. From the intercept values (i.e., X/M at $C_f = 1$ ppm), the adsorption capacities of virgin FS 300, virgin FS 400, and virgin powdered FS 300 are ~210, 300, and 250 mg DNT/g C, respectively. The corresponding isotherm slopes are 0.171, 0.223, and 0.333, respectively ($n = 5.83, 4.49, \text{ and } 3.33$). The measured $1/n$ value of

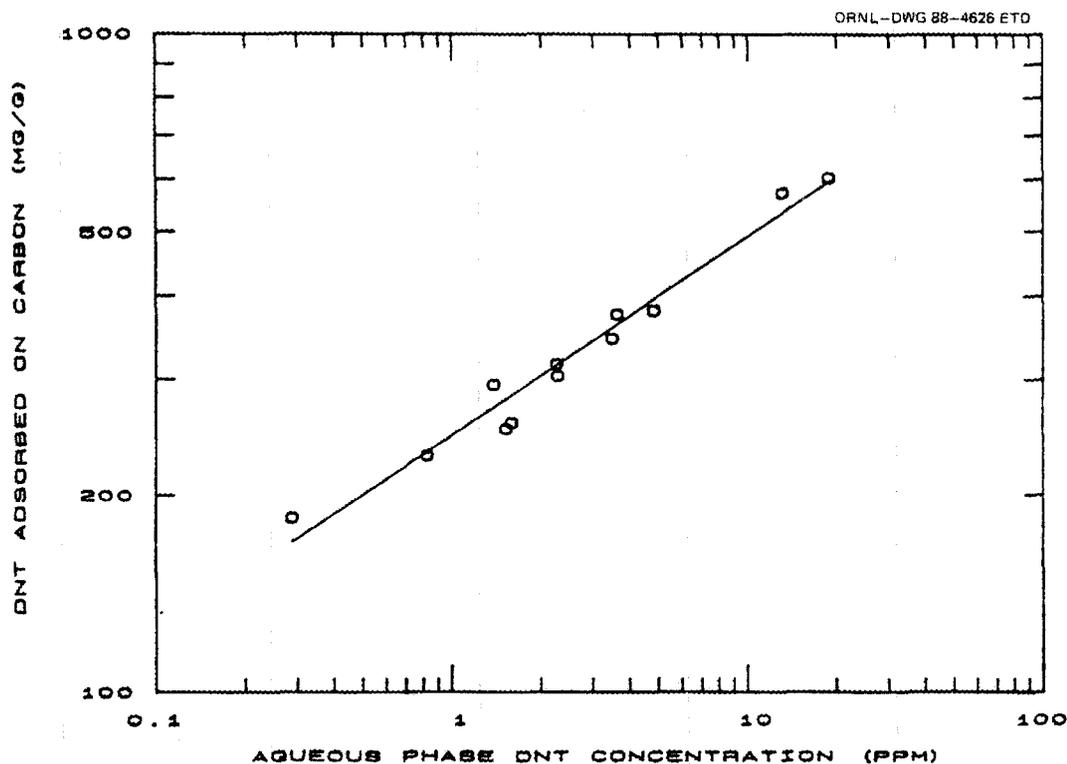


Fig. C.3. Equilibrium adsorption isotherm for DNT on crushed granular activated carbon.

0.333 for powdered FS 300 is roughly in agreement with a reported value of 0.31 for a similar activated carbon (10). Overall, it appears that granular and powdered FS 300 have lower DNT adsorption capacities than FS 400.

Two complicating factors in using the above adsorption data to estimate DNT loadings on spent carbon are (1) the DNT levels are likely to vary considerably in actual AAP wastewaters, and (2) when other nitroaromatics are present, the equilibrium DNT loadings can also be less than those reported above because of the competition for adsorption sites. In spite of these complications, however, a reasonable upper limit for the DNT loading on spent carbon can still be estimated. One approach is to make the conservative assumptions that the column input is saturated in DNT and that the DNT loading on the carbon approaches its equilibrium value as the carbon becomes spent. At 25°C the saturation concentration of DNT is ~250 ppm, yielding equilibrium loadings of

540 and 1030 mg DNT/g C for virgin FS 300 and virgin FS 400, respectively (this translates to about 35 and 51% of the total dry weight).

After DNT adsorption, the spent carbons were regenerated by acetone extraction. Three adsorptions and two desorptions were performed on each carbon (cycle 1: adsorption on virgin carbon; cycle 2: first desorption and second adsorption; cycle 3: second desorption and third adsorption). Desorption was accomplished by the Soxhlet method in cycle 2 and the room-temperature batch method in cycle 3. Figures C.1 and C.2 show that the adsorption capacities of FS 300 and FS 400 after Soxhlet extraction are nearly as great as the virgin carbon, whereas the capacities after batch extraction are drastically decreased.

The relative efficiency of the Soxhlet and batch extraction methods is further demonstrated by the Freundlich isotherms in Figs. C.4 and C.5. In this case, two portions of the same spent carbon were regenerated, one portion being extracted by the Soxhlet procedure and one portion being extracted by the batch procedure. The Soxhlet-extracted

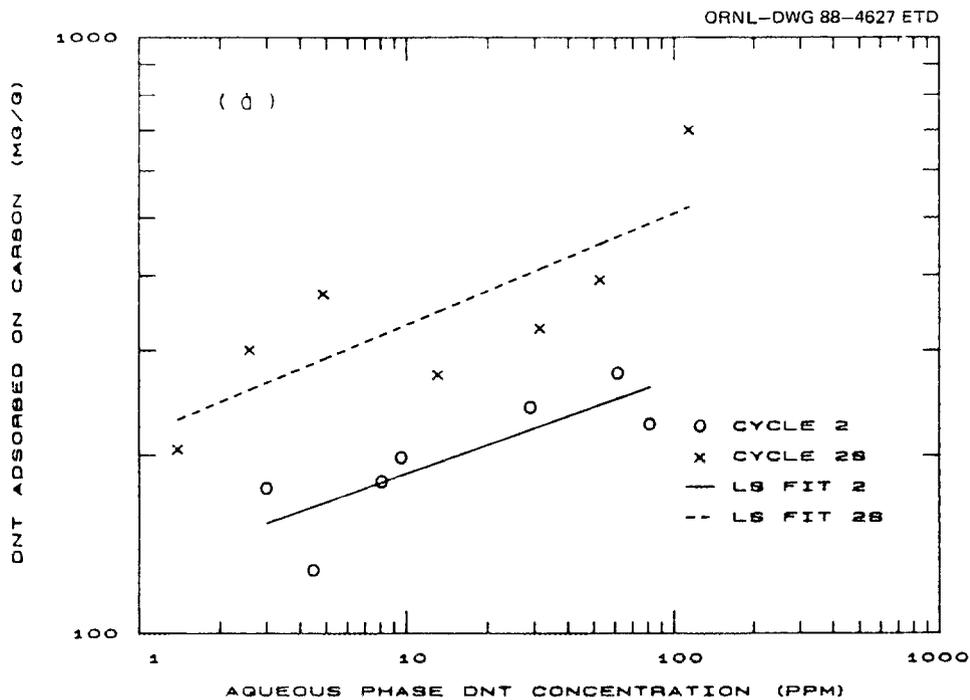


Fig. C.4. Acetone extraction efficiencies for DNT on "large" granular activated carbon.

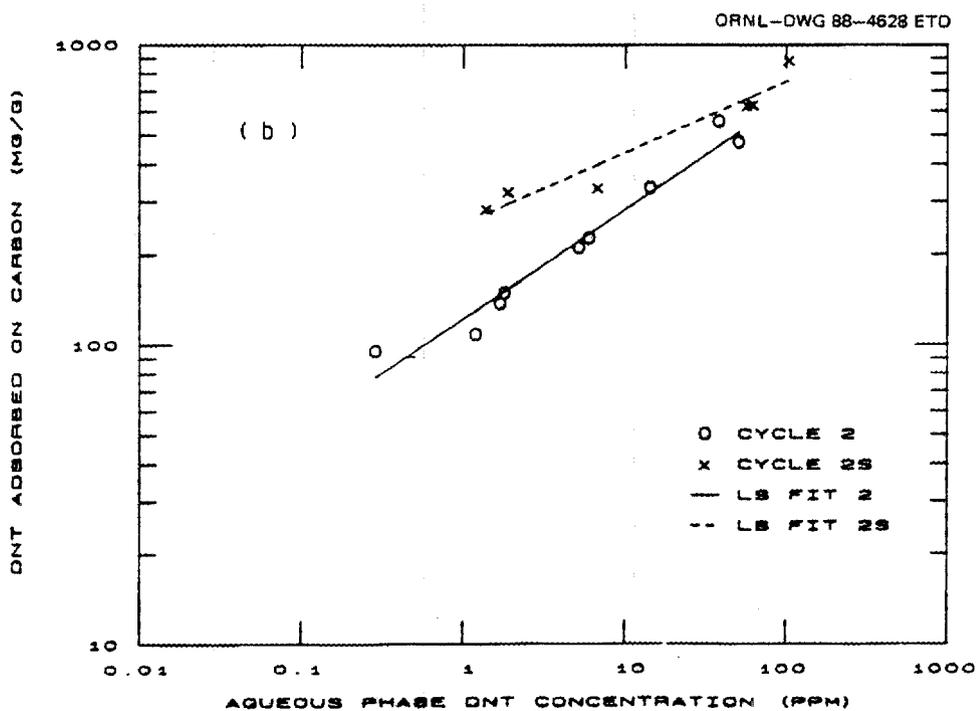


Fig. C.5. Acetone extraction efficiencies for DNT on "small" granular activated carbon.

carbon exhibited at least a 40% better recovery of adsorptive capacity than the batch-extracted carbon.

Desorption with different solvents

DNT-loaded FS 300 and FS 400 were extracted with water, acetone, methanol, and their mixtures by column, batch, and Soxhlet procedures separately. Table C.1 compares the percentage DNT removal achieved for each solvent system with the Soxhlet method. As expected, both acetone and methanol proved to be better solvents than water.

Figure C.6 illustrates the effects of solvent/spent carbon weight ratio and temperature on the degree of DNT removal by water using the batch extraction method. Based on this figure, the desorption of DNT in water appears to increase linearly with the water/carbon ratio and also increases significantly as temperature is increased from 23 to 70°C. These trends are expected, at least in general, because the DNT carrying

Table C.1. Effectiveness of DNT extraction by the Soxhlet method

Solvent	Carbon	DNT removed (%)
Water	FS 300	21.2
	FS 400	35.8
Acetone	FS 300	84.1
	FS 400	94.6
Acetone/water (1:1 by weight)	FS 300	83.3
	FS 400	90.7
Methanol	FS 300	86.9
	FS 400	90.9

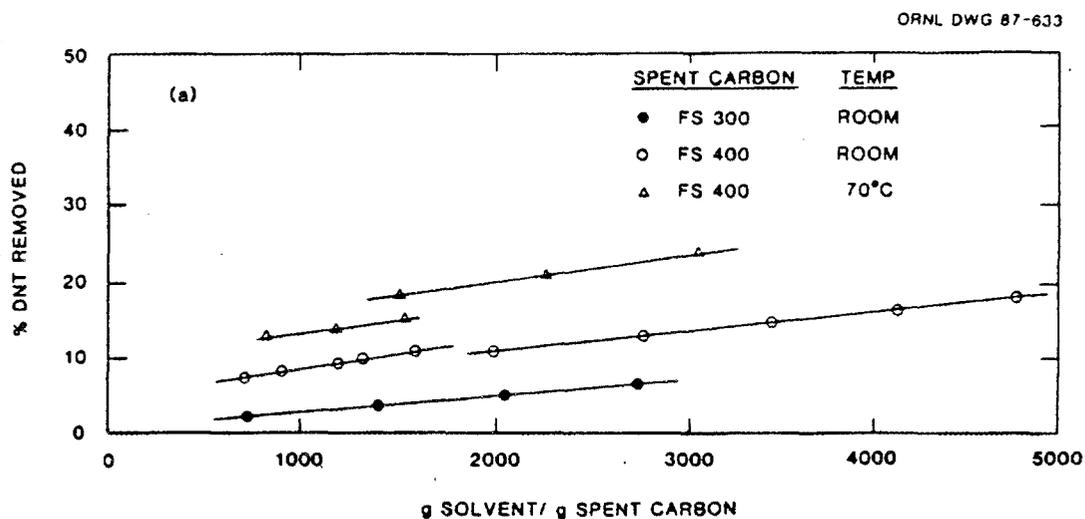


Fig. C.6. Effect of solvent/carbon ratio and temperature on the batch extraction of DNT with water.

capacity of the aqueous phase increases as the water/carbon ratio and temperature increase.

The magnitude of the temperature effect on extractability is supported by Fig. C.7, which illustrates the rapid increase of DNT solubility in water with temperature. Even with the large temperature effect, however, DNT extraction with water is rather inefficient. At

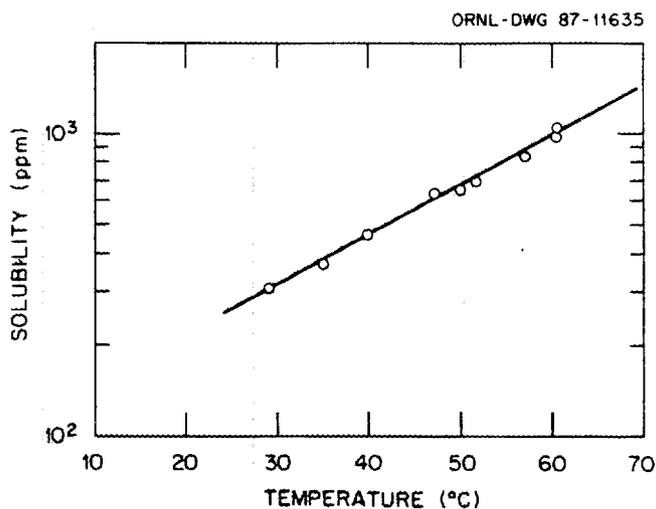


Fig. C.7. Solubility of DNT in distilled water.

70°C (the melting point of DNT), a water/carbon ratio of 3000 results in only about 25% extraction of the adsorbed DNT from FS400.

Figure C.8 illustrates the much greater extraction efficiency of acetone and methanol. Under good contacting conditions, ~85% of the DNT can be extracted from FS 300 and FS 400 at an acetone/ carbon ratio of 500. Beyond ~75% removal, the incremental extraction efficiency decreases, and 100% removal is approached asymptotically.

The large variation between the different experimental runs in Fig. C.8 illustrates the importance of contacting efficiency between the carbon and the solvent. Column desorption always involves some "bypassing" of the solvent, especially in short columns similar to the one used here. (Although not done in this case, such bypassing inefficiency can be at least partly compensated by solvent recycle.) Some of the differences between the methanol and acetone column extraction efficiencies may have been due to differences in the column voidage and the flow rate. The batch experiments provided more efficient contacting and gave, correspondingly, the highest extraction yields. While the batch extraction efficiencies of both acetone and methanol appear similar, acetone appears to be more efficient for removing the by-product species.

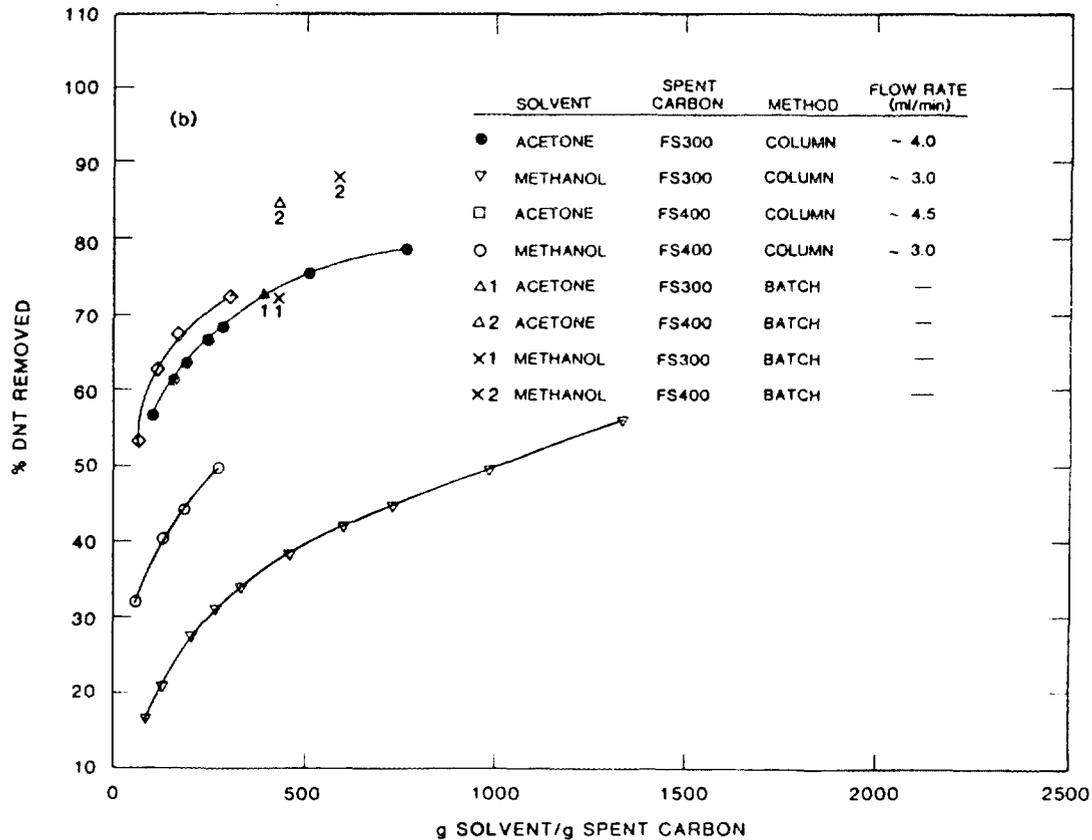


Fig. C.8. Effect of solvent/carbon ratio, temperature, and contacting procedure on the extraction of DNT with acetone and methanol.

Analysis of desorption extracts

The HPLC and GC/MS analyses revealed significant differences among the various extracts and the presence of previously unreported chemical by-products (in contrast to the control extracts of the virgin carbons). Figure C.9(a) is a HPLC spectrum for a methanol extract of spent FS 400 (first cycle, column procedure), and Fig. C.9(b) is a HPLC spectrum for a subsequent batch acetone extract of the previous methanol-extracted sample. These spectra indicate that most of the DNT was removed by the initial methanol extraction and also that some of the DNT was converted to other chemical by-products, which were subsequently removed by the acetone.

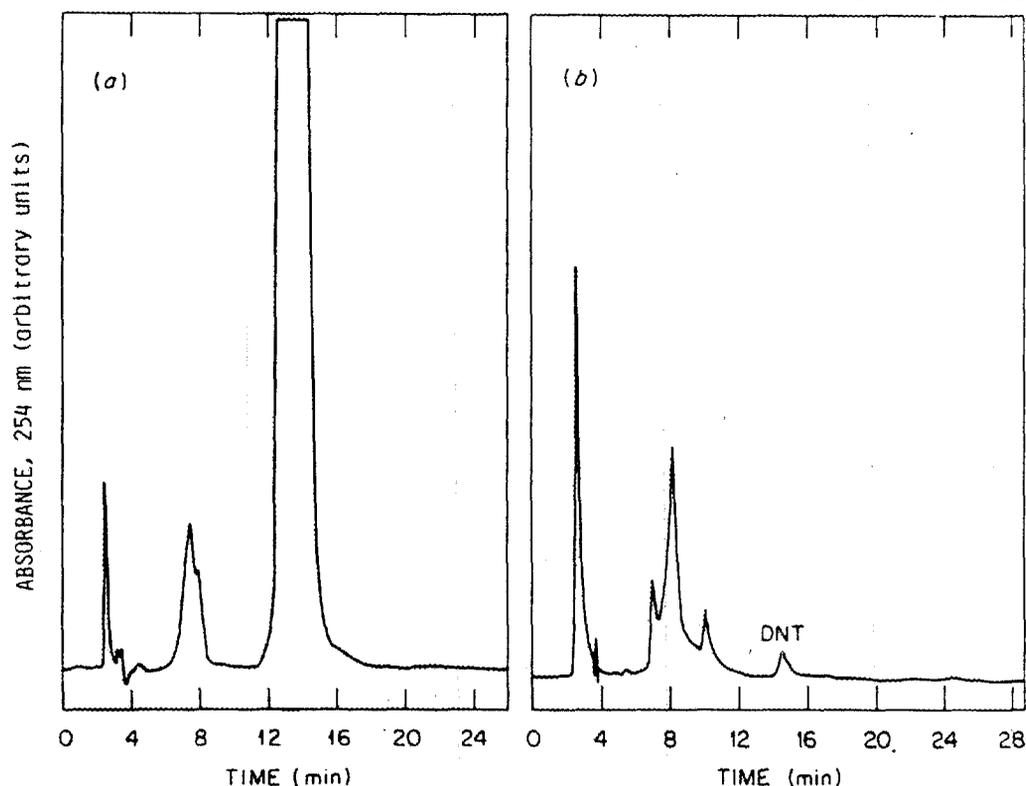


Fig. C.9. HPLC/UV chromatograms of extracts from carbon contaminated with DNT.

The variety and relative abundance of by-product species in the extract appear to depend on the solvent used and the number of adsorption/desorption cycles the carbon experiences. Acetone is the most efficient for by-product extraction, followed by methanol and then water. In the experiments reported here, there were typically three to six distinct by-products appearing in the extracts for carbon that had only undergone one adsorption cycle, while there were as many as eight in acetone extracts from second and third cycles. When water was the solvent, the number and abundance of the extracted by-products increased with temperature.

Figure C.10(a)-(c) illustrates HPLC spectra of water, methanol, and acetone extracts, respectively, of DNT-loaded FS 400 (first extraction, batch method). Based on HPLC spectra with pure reference standards, 2,4-dinitrobenzoic acid is a major by-product appearing in all three

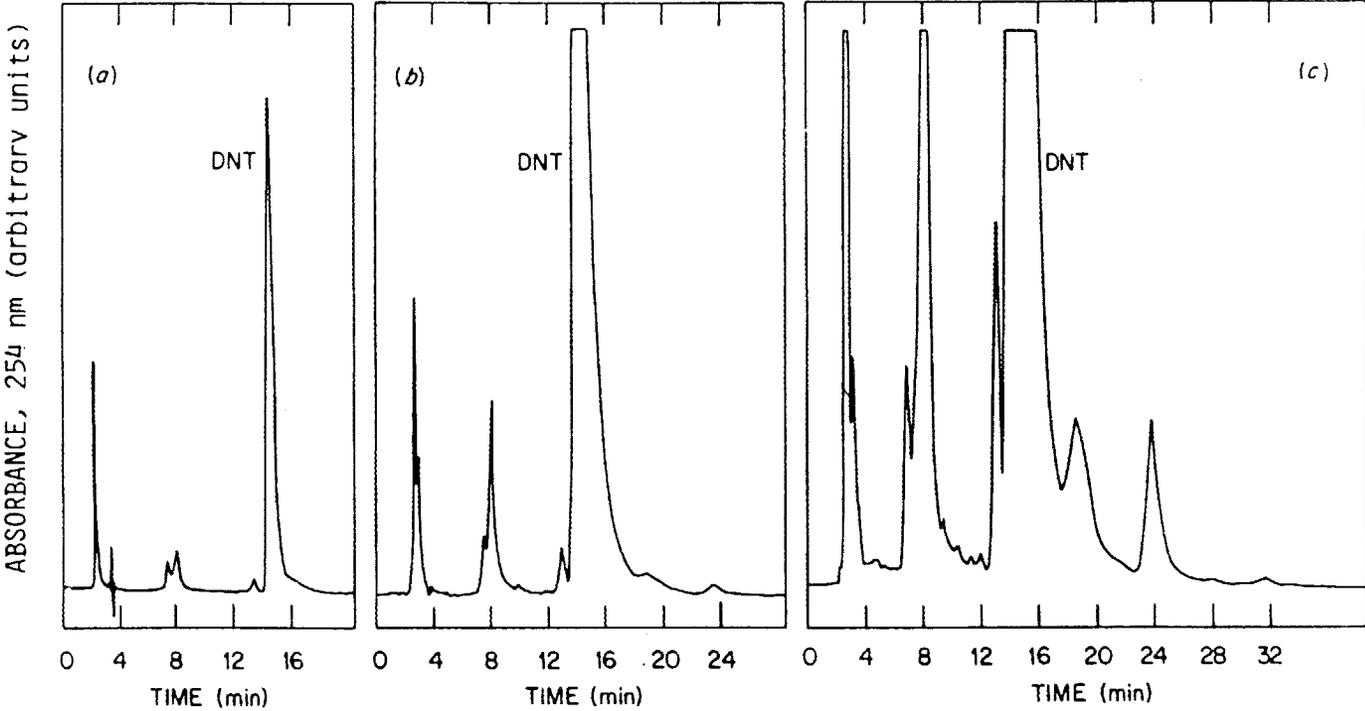


Fig. C.10. HPLC/UV chromatograms of extracts from carbon contaminated with DNT.

extracts. The compound 2,4-dinitrobenzaldehyde also appeared in all three extracts, although its level was rather low in water. Other compounds that could be identified were 2,4-dinitrobenzyl alcohol in the methanol and acetone extracts and 2,4-dinitromethylbenzoate in the acetone extract. The other compounds present have not yet been identified.

Hypothesis for by-product formation

The presence of the chemical by-products suggests that DNT can undergo chemical reactions after it becomes attached to the carbon surface. The prevalence of partially oxidized DNT derivatives further indicates that one of the major reactions is side-chain (methyl-group) oxidation. This is consistent with the observation that activated carbons have large surface areas containing various reactive groups such as carboxyl groups, oxides, and oxygen-containing free radicals.

Because the carbon surface is highly heterogeneous, both physical and chemical adsorption are possible. We speculate that some of the chemically reactive sites serve as catalysis centers, where the DNT can be partially oxidized and then desorbed into the solvent. Highly reactive centers may involve polymerization or direct reaction between the DNT and the carbon substrate, producing compounds that cannot be extracted. Further elucidation of the reaction mechanisms may reveal methods for controlling and enhancing DNT oxidation, producing compounds that might be less dangerous and/or more easily removed from the spent carbon.

Appendix D

PRELIMINARY PROPAGATION-OF-DETONATION TEST
USING IAAP SPENT CARBONSample preparation and analysis

Spent carbon was received at Los Alamos National Laboratory from IAAP in two 55-gal drums. The carbon inside the drums was saturated with water. Sample preparation consisted of draining the free water from the carbon, air drying the carbon in shallow trays, and blending the dried carbon. Samples of the water and blended carbon were analyzed for explosives.

The water drained from the carbon had a relatively low level of contamination including 9.5 ppm RDX, 13.3 ppm HMX, and 1.5 ppm TNT. Two carbon samples were Soxhlet extracted with acetonitrile for 10 h. The extract was then analyzed for both explosives and decomposition products. The latter was found in only trace quantities. The average explosive concentrations were:

<u>Explosive</u>	<u>Grams per 100 cc solvent</u>	<u>Grams per 100 g clean carbon</u>
HMX	0.19	0.31
RDX	1.27	2.04
TNB	0.28	0.45
TNT	5.17	8.31
<u>Total</u>	<u>6.91</u>	<u>11.11</u>

A pyrolysis test was also run on the air-dried spent carbon. This showed a 2.5% water weight loss followed by a slope change in the pyrolysis profile at 250°C (9.5% explosive weight loss) and a net 10% explosive weight loss at 275°C. These results confirm an explosive content in the original spent carbon of about 10 wt % (11 g/100 g clean carbon).

Propagation tests

Propagation tests were conducted to determine if the air-dried spent carbon would sustain a detonation in a 15.2-cm-diam (6-in.) steel

tube. The basic test apparatus employed has been used extensively in previous tests and is described in detail by Lackey (1988). Briefly, the sample was loaded into a 0.61-m-long (24-in.), schedule 40 stainless-steel pipe that was then placed (standing vertically) on a 5.1-cm-thick (2-in.) carbon-steel witness plate. A 5.1-cm-thick (2-in.), 15.2-cm-diam (6-in.) Comp-B pad (60% RDX, 40% TNT) was placed on the top end. Detonation was initiated using a SE-1 exploding-bridgewire detonator and a 9205 booster (92% RDX) on top of the Comp-B pad. The witness plate was inspected after each test to check for indication of detonation propagation. Partial reaction of the explosive contaminant was evaluated by examining the recovered pieces of the pipe.

The initial test consisted of a series of seven shots fired on the standard test assembly. In the first shot, the pipe was filled with virgin Calgon Filtrasorb-300 carbon to establish a control. In the remaining six shots, the pipe was filled with the air-dried IAAP spent carbon. No evidence for propagation of detonation was observed after any of these shots. However, comparison of the pipe fragments for the spent carbon tests with the fragments of the virgin control shot clearly indicated a more severe reaction in the former. Thus explosive reactions were apparently initiated in the spent carbon but failed to propagate down the pipe.

Additional tests were conducted in an attempt to find the explosive loading required to sustain detonation. For these tests both virgin carbon and air-dried spent carbon were artificially loaded with explosive. This was accomplished by dissolving a mixture of HMX, RDX, and TNT (in the original ratios found in the spent carbon) in acetone, spraying the acetone solution onto the carbon, and then allowing the acetone to evaporate. Several explosive loadings were tried up to a maximum of 51.1 g/100 g clean carbon. Although the reaction severity clearly increased with increased loading, propagation to the witness plate was never observed. The test conditions and results are summarized below:

<u>Number of shots</u>	<u>Loading (g/100 g carbon)</u>	<u>Carbon</u>	<u>Propagate?</u>
1	11.1	virgin	No
6	11.1	spent	No
1	16.1	virgin	No
1	16.1	spent	No
1	31.1	virgin	No
1	31.1	spent	No
1	36.1	spent	No
1	41.1	virgin	No
1	46.1	virgin	No
1	46.1	spent	No
6	51.1	virgin	No
6	51.1	spent	No

Conclusions

Propagation tests with spent and artificially loaded carbon did not show propagation in a 15.2-cm-diam (6-in.) pipe, even at explosive loadings significantly in excess of those expected for carbon from munitions wastewater treatment. However, these same tests also clearly show that explosive reactions can be initiated to some degree. Such initiations even appear to occur with unaltered spent carbon from IAAP, which contains ~10 wt % explosive.

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