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CARBON DIOXIDE SCRUBBING CAPABILITIES OF TWO NEW NON-POWERED TECHNOLOGIES

by

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Released by: G.A. Higgins, CAPT, MSC, USN Commanding Officer Naval Submarine Medical Research Laboratory Box 900 Groton, CT 06349-5900

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ABSTRACT

Current guidance for survivors aboard a disabled submarine (DISSUB) recommends the use of the "stir-and-fan" method of carbon dioxide (CO₂) scrubbing in which the contents of canisters of lithium hydroxide (LiOH) are dispersed onto horizontal surfaces. This technique is objectionable because it releases large quantities of fine, caustic LiOH dust and it utilizes LiOH inefficiently. This report presents the results of laboratory studies of the CO₂ scrubbing capabilities of two new products that might improve on "stir-and-fan", the Battelle Curtain (BC) and the Micropore Reactive Plastic Curtain (RPC). Experiments took place within a sealed hyperbaric chamber. CO₂ was added to the chamber at a known mass flow that reproduced what might be encountered in a "worst-case" DISSUB scenario. Natural convection alone circulated gas within the chamber. The mass of BCs or RPCs necessary to limit CO₂ to 3% for about 2 days was determined. The total scrubbing capacity (mass of CO₂ scrubbed per unit mass of agent) of the BC was 0.756 ± 0.012 (mean \pm SD), and the comparable value for the RPC was 0.808 ± 0.007 . Both products provided a scrubbing capacity that is close to the stoichiometric limit of the reaction (0.919). Neither product released sufficient caustic dust to prevent handling by a trained individual wearing no personal protective equipment.

ADMINISTRATIVE INFORMATION

This investigation was conducted under work unit # N0002401WR03301-51004 entitled "Battelle Curtain" and work unit # N0002401WR03302-51003 entitled "Micropore Curtain". The views expressed in this report are those of the authors and do not reflect the official policy or position of the Department of the Navy, Department of Defense, or the U.S. Government. This report; was approved for publication on 28 August 2003, and designated as Naval Submarine Medical Research Laboratory Technical Report #TR1228. [BLANK PAGE]

INTRODUCTION

Fleet Need Addressed by this Project

The preferred method of recovering the crew of a submerged, disabled submarine (DISSUB) is by rescue with a system such as the Deep Submergence Rescue Vehicle.(3) To be rescued, crew must survive in the submarine long enough for rescue assets to arrive on the scene and begin operations. Alternatively, conditions may deteriorate to the point that escape from the DISSUB directly through the water column is necessary. Even when escape is ultimately the best option, a limited capability for survival within the DISSUB may be desirable to optimize conditions for escape (e.g., to wait for daylight, for search-and-rescue assets to arrive, or for better weather). In most scenarios, the factor that limits survival time within the DISSUB is the capacity of equipment and supplies for removal of carbon dioxide (CO_2) from the atmosphere.

 CO_2 can be chemically scrubbed from a submarine's atmosphere via the following overall reaction with lithium hydroxide (LiOH):

$$2\text{LiOH} + \text{H}_2\text{O} + \text{CO}_2 \Leftrightarrow \text{Li}_2\text{CO}_3 + 2\text{H}_2\text{O}$$

When this reaction proceeds to completion, 0.919 grams of CO_2 react with each gram of LiOH. This datum is termed the stoichiometric limit of the reaction. Another noteworthy characteristic of this reaction is that it is highly exothermic, i.e., LiOH crystals get hot as they react with water vapor and CO_2 , releasing 21.4 kcal per mole of CO_2 scrubbed.(2)

Currently, when a nuclear submarine's regenerative CO₂ scrubbing plant is not available, canisters of LiOH can be utilized for CO₂ scrubbing via two methods: 1. placement of canisters in an electrically powered forced-air blower termed a hopper; 2. when a functioning hopper is not available, canisters can be opened, their contents spread on available horizontal surfaces, and compartment air manually fanned over the LiOH granules with periodic stirring of the bed of granules - the so-called "stir-and-fan" method. Because the power requirements of the current hopper (Model 744001D, Air-A-Plane Corp., Norfolk, VA) are significant (4 amperes at 115 volts), and the alternating-current electrical power which it needs will probably not be available aboard an actual DISSUB, non-powered methods will probably be required in this circumstance.

The "stir-and-fan" method of CO_2 scrubbing is objectionable because it releases large quantities of fine, caustic LiOH dust into the DISSUB atmosphere. Furthermore, the Disabled Submarine Survival Guides(3) (a.k.a. Guard Books, documents written for each class of submarine that detail emergency procedures for a DISSUB casualty) specify that a given mass of LiOH will scrub only half as much CO_2 when used with the "stir-and-fan" method than when placed in a powered hopper. This specification has profound implications on the load of LiOH that must be carried by a submarine to meet the demands of a large contingent of survivors for several days. Clearly, a non-powered method of CO_2 scrubbing that releases less dust than "stir-and-fan" while utilizing LiOH stores more efficiently is needed. This report discusses the results of studies performed at the Naval Submarine Medical Research Laboratory (NSMRL) on the CO_2 scrubbing capabilities and caustic dust release of two new products, the BC and the RPC, when utilized with a non-powered method.

The Battelle Curtain

The BC has been developed by the Battelle Memorial Institute, Columbus, OH (figure 1). It is a flexible container similar in size and configuration to an air mattress that can be filled with conventional LiOH granules. The BC utilizes LiOH from the canister currently procured by the Fleet (i.e., manufactured per MIL-L-20213E). The design of the BC incorporates two innovations to reduce dust release:



Figure 1. Four Battelle Curtains. Each curtain is attached to a conventional canister of LiOH, and the contents of the canister have been poured into the curtain. The spacing between curtains at their closest point is 5 cm. This configuration forms a "convective engine".

1. The curtain is manufactured from KimGuard[™] Sterile Wrap (Kimberly-Clark, Roswell, GA), a material that is commonly used to wrap surgical instruments prior to sterilization. This

material has an effective pore size of about 3 microns, much smaller than the bulk of LiOH manufactured per MIL-L-20213E.

2. The curtain has a sleeve which, prior to filling the curtain with LiOH, permits the curtain to be attached to a conventional canister of LiOH, forming a closed system. When the contents of the canister are poured into the curtain, dust is contained within this closed system.



Figure 2. Reactive Plastic Curtains. Each curtain is 31.8 cm wide and approximately 1.5m long. Four "Hangings" of nine curtains each are apparent, two in the foreground and two in back. The spacing between curtains at their attachment point to the frame is 5 cm. This configuration forms a "convective engine".

Micropore Reactive Plastic

Micropore, Inc., (Newark, DE) has developed a method to incorporate LiOH in a polymer matrix, producing a flexible sheet of material termed a Reactive Plastic (figure 2). This material contains over 95% LiOH by weight. It can be rolled into cartridges (ExtendAir® cartridges) which can be placed into a conventional powered hopper. The bulk density of LiOH in these cartridges (mass of LiOH per unit volume of cartridge) is approximately 30% greater than that of conventional LiOH canisters. Cartridges of Reactive Plastic can also be unrolled into a continuous sheet 31.8 cm wide x 7.6 m long that can be cut into subsections as desired and hung vertically as RPCs. Release of dust or larger particles from this material during handling and cutting is, in the experience of the authors, not troublesome to an operator wearing no protective equipment.

METHODS

Overview of the Experimental Approach

As stated above, an objective of this study was to investigate means of CO_2 scrubbing that do not require electrical power. We refer to this as "passive scrubbing". Ideally, these methods also function without any musculoskeletal activity by the crew (such as manually pumping air through the scrubbing material or swinging the material through the air) that would increase the crew's collective metabolic rate and CO_2 production. We refer to this as "static scrubbing". A method that requires no electrical power and no crew exertion is termed a "static, passive" technique. This study addressed first the ability of the BC and RPC to function in a static, passive mode since, if this ideal were reached, studies of alternative, less optimal approaches such as curtain swinging would be unnecessary. In fact, both products functioned well using a static, passive scrubbing technique. Consequently, this study addresses the performance of the BC and RPC when used in this mode only.

The overall approach of this study is similar to that of previous work at the Admiralty Marine Technology Establishment, Alverstoke, UK.(5) Experiments took place within a sealed hyperbaric chamber. CO_2 was added to the chamber at a known mass flow. Atmospheric CO_2 was monitored. Batches of BCs and RPCs were hung within the chamber. Experiments began at a specified atmospheric CO_2 and, when the CO_2 level returned to this maximum limit, the scrubbing capacity of the curtains was considered exhausted. Since the total mass of CO_2 admitted to the chamber during the experiments was known and the mass of BCs or RPCs that was hung was also known, the capacity of these products (i.e., mass of CO_2 scrubbed per unit mass of product) could be determined.

Because CO_2 scrubbing with static, passive curtains depends on natural convection rather than forced ventilation to circulate air within a compartment, modeling the convective conditions that might be found in a DISSUB was important. The experimental approach of this study was "sized to fit" a rather specific, operational scenario that represents a worst-case from the standpoint of CO_2 scrubbing demands:

- U.S. Navy 688-Class submarine, forward compartment;
- 120 survivors occupy 20% of this compartment (283 m³ galley and crew mess);

- maximum acceptable atmospheric $CO_2 = 3\%$ surface equivalent volume (SEV);
- initial $CO_2 = 3\%$ (i.e., the protocol studied CO_2 scrubbing alone, not CO_2 scrubbing plus the time necessary for CO_2 buildup to reach 3%);
- rate of metabolic CO₂ production = 0.1 pounds/hr/man (0.38 l·min⁻¹ at 0 °C and 1 atmosphere absolute pressure, dry).

DISSUB conditions were reproduced in the inner lock of NSMRL's hyperbaric chamber which has a volume of 21.2 m³. Consequently, a "1:13" scaling factor (21.2:283) was applied to the total CO₂ production arising in the above operational scenario, and a mass flow of CO₂ was added to the inner lock that simulated the CO_2 production of 9 men (120 men / 13). Batches ("Hangings") of BCs or RPCs were hung in the inner lock according to a routine that seemed operationally achievable. Each Hanging consisted of about 5.6 kg of product. Curtains were arranged to form a "convective engine" that harnessed the energy released by the underlying chemical reaction to induce natural convection. Gas circulation within the lock was not assisted by any means such as fans or induced curtain movement. The major performance goal was to *limit* (not maintain) atmospheric CO₂ to 3% SEV (throughout this manuscript, all CO₂ percentages are expressed as SEV, i.e., the volume percentage of a gas at sea level pressure which would have the same partial pressure as the actual gas at the elevated or reduced pressure), and additional curtains were hung as necessary to meet this goal. Since the total mass of CO₂ admitted to the chamber during the experiments was known and the mass of BCs or RPCs that was hung was also known, the capacity of these products (i.e., mass of CO₂ scrubbed per unit mass of product) could be determined.

Major outcome data were:

- 1. atmospheric CO_2 as a function of time;
- 2. mass of BC or RPC necessary to limit CO₂ to 3% for about 2 days when used according to the specified procedure;
- 3. scrubbing capacity of the BC or RPC in terms of mass of CO₂ scrubbed per unit mass of BC or RPC.

These data can be applied directly to the ultimate operational question addressed by this project: How much scrubbing agent must be loaded on a submarine with a crew complement of "X" to support "Y" days of survival?

Carbon Dioxide Addition

CO₂ was admitted into the chamber interior via a fenestrated copper tube routed along the inner circumference of the chamber at a height 1 m above the floor. CO₂ addition was metered through a flowmeter (Model GE606, Gilmont Instruments, Barrington, IL) connected to cylinders of compressed CO₂. The flowmeter was calibrated against a chain-compensated water-seal gasometer (350 liter model, Collins, Braintree, MA). System integrity was checked before and after each experiment by attaching a calibrated syringe (3 liter model, Rudolph, Kansas City, MO) to the chamber penetration within the chamber. In all experiments, a constant CO₂ mass flow of 6.69 g/min was utilized, corresponding to the CO₂ production of 9 men, assuming each man produces about 45 g of CO₂ per hour. In some experiments this mass flow was

independently verified by tracking declines in the weight of the cylinders supplying CO₂ to the chamber (Model Champ SQ scale base with Model CD-11 Indicator, Ohaus, Florham Park, NJ).

Routine for Curtain Deployment

The specifics of this routine involve important underlying assumptions of the experimental protocol. The capacity of LiOH for scrubbing CO_2 (in terms of mass of CO_2 scrubbed per mass of LiOH) is a function of how rapidly CO_2 enters the LiOH bed – if the mass flow of CO_2 processed by a given LiOH bed is relatively high, then some measures of the capacity of the LiOH bed will be relatively low.(1) This characteristic of LiOH renders capacity data sensitive to the experimental design. Consequently, the experimental protocol was chosen carefully to reflect operational conditions.

An additional critical aspect of the routine for hanging curtains in this study is that new batches of curtains were hung to *supplement, not replace,* existing curtains. At the time that existing curtains "failed" (i.e., atmospheric CO_2 had risen to a limit), these curtains were still scrubbing some CO_2 , but at a rate that could not keep up with the "load" (i.e., mass flow of CO_2 into the chamber). Consequently, existing curtains shared the "load" with new curtains, enhancing the scrubbing capacity of both.

BCs and RPCs were hung within the inner lock of the chamber according to a routine that seemed operationally convenient: curtains were hung in batches ("Hangings") that were projected to meet the scrubbing demands of a 12-h period based upon the specification in the Atmosphere Control Manual(4) for the capacity of granular LiOH placed in a powered hopper (0.75 pounds of CO_2 per pound of LiOH).

Prior to deployment of the first batch of curtains (Hanging 1), the chamber atmosphere was adjusted to 3% CO₂ and > 85% relativity humidity by adding CO₂ to the atmosphere and placing warm water in the chamber's bilge. The chamber's life support blower was run during this adjustment of initial conditions, but was subsequently shut down for the remainder of the experiment. CO₂ addition was begun when Hanging 1 was deployed and continued throughout the experiment. In each experiment, four Hangings of curtains were hung and run to "exhaustion". The scrubbing capabilities of each fresh Hanging resulted in a decline of atmospheric CO₂ below 3%, followed by a slow rise as the LiOH was consumed. Exhaustion was defined as a rise in atmospheric CO₂ to 3%. Consequently, the entire experiment began and ended at 3% CO₂, as did each Hanging. The times when Hangings were hung and when they reached exhaustion were recorded.

Pressure within the chamber was adjusted to 0.6 m of seawater (msw) by the addition of compressed air. This slight positive pressure served to seal the chamber and to deliver gas through sample lines to exterior CO_2 analyzers. In all experiments the chamber had no discernible leaks over the course of two days of operation, exceptional performance for a large chamber system.

Hangings of fresh curtains were accomplished by divers wearing scuba gear and full-face masks. Because locking divers into the chamber under pressure would have required the assembly of a complete chamber operating team, sometimes at odd hours, the chamber was decompressed to sea level prior to entry of the divers. Divers entered the inner lock of the chamber through the outer lock, closing intervening hatches during ingress and egress, and minimizing the time each hatch was open. Because the atmosphere of both the outer and inner locks was adjusted to 3% CO₂ during the establishment of initial conditions at the start of an experiment, the outer lock served as a "buffer" to minimize the impact of diver passage on the atmosphere of the inner lock. Following egress of the divers, the chamber was repressurized to 0.6 msw. In separate studies of mock lock-ins during which divers cycled through the chamber but did not actually deploy curtains, the atmosphere of the inner lock was observed to change by less than 0.1% CO₂. Data were adjusted for the calculated mass of CO₂ vented from the chamber as a result of depressurization associated with deployment of new curtains.

Each Hanging of RPCs consisted of approximately 5.58 kg of material in nine sheets 31.8 cm wide and about 1.5 m long. These sheets were obtained from the contents of 1.5 canisters of product cut into strips with scissors. A paper hole punch was used to create hanging points at each corner of the strips. Individual strips were hung with twisted paper clips from a rack such that the strips were spaced 5 cm apart (figure 2). This configuration formed a "convective engine" that used the energy from the exothermic reaction of LiOH with CO_2 and water vapor to heat adjacent chamber air, causing gas to rise between the strips and circulate within the chamber.

Each Hanging of BCs consisted of approximately 5.73 kg of material contained in two curtains. Each curtain was prepared by opening one canister of LiOH (Lot 784LH, Tangram Co., Holtsville, NY) with a screwdriver, attaching the BC to the canister, and pouring the granules into the curtain. Curtain/canister units were suspended from pre-fabricated grommets along the edge of the curtains with twisted pieces of 12-gauge copper wire attached to a rack. The two units of a Hanging were hung "back-to-back" such that the minimum space between curtains was approximately 5 cm (figure 1). Units were 1.8 m in length, and the bulk of the LiOH in filled curtains was contained in five tube-shaped sections with a diameter of roughly 2.5 cm.

Data Acquisition

Gas sample lines were situated about 1 m above the floor, one 1 m from the curtains ("Near CO_2 "), and one 2 m away ("Far CO_2 "). Lines were also placed above the curtains ("High CO_2 ") and in the bilge beneath them ("Low CO_2 "). An additional line was located in the outer lock. These lines were lead to CO_2 analyzers (Model CD-3A, Ametek, Pittsburgh, PA) located outside the chamber. These analyzers were calibrated with appropriate zero and span gases prior to each experiment and were checked for drift periodically throughout the experiments.

Atmosphere temperature was sensed with thermistors (Series 400, YSI, Yellow Springs, OH) located 1 m above the floor, 1 m and 2 m from the curtains. A thermistor was also placed above the curtains. A surface contact thermistor was attached to one curtain of each Hanging in experiments with the BC; this thermistor was available only during one Hanging of the RPC studies. The temperature of the atmosphere within the chamber room was also recorded.

Chamber pressure and humidity were monitored with instruments incorporated in the chamber's systems. Barometric pressure in the local area was obtained from the National Weather Service.

Data were manually logged at least every 30 min. Hangings were considered exhausted when the mean of Near and Far CO₂ readings equaled 3%.

Protocol

The protocol for an experiment can be summarized as follows:

- 1. Establish initial conditions: 3% CO₂, >85% relative humidity
- 2. Lock in divers; hang Hanging 1; begin CO₂ addition at 6.69 g/min; lock out divers
- 3. Pressurize chamber to 0.6 msw
- 4. Collect data regarding temperature at four locations, CO₂ at four locations, pressure, and humidity
- 5. When the mean of the Near and Far CO_2 readings reaches 3%:
 - a. depressurize chamber
 - b. lock in divers; hang Hanging 2; lock out divers
 - c. repressurize chamber to 0.6 msw
- 6. Repeat steps 4 and 5 until Hanging 4 is exhausted
- 7. Terminate experiment

Four experiments were completed with the BC and four with the RPC.

Chemical Analyses

At the conclusion of each experiment, samples of scrubbing material were taken from the following locations in each Hanging:

- RPC: from one strip outermost in the group of nine strips that constituted a Hanging at a location as high and as low as possible ("outer high" and "outer low"); from the middle strip in the group of nine strips at a location as high and low as possible ("middle high" and "middle low").
- BC: from one curtain at a location as high and as low as possible ("high" and "low" locations).

Samples were analyzed for LiOH content (percentage LiOH by weight) by an adaptation of U.S. military specification MIL-L-20213E(SH). Specifically, approximately 5 g of sample were ground to a fine powder with either a mortar and pestle (BC) or a small hand-powered rotary food grinder (RPC). This material was dessicated at 120 °C for 4 h under an inert gas purge. Approximately 1g of the dried sample was weighed (Model 1712MP8, Sartorius, Gottingen, Germany), placed in 100 ml of distilled water, cleared of carbonates by the addition of 10% BaCl₂ until precipitation ceased, and titrated with 1.0 N HCl, using phenolphthalein as the endpoint indicator. Samples were handled in a manner that minimized exposure to atmospheric CO₂. The non-LiOH content of fresh samples of granular LiOH and RPC was also determined. Data were converted to the sample's percentage by weight of the LiOH content that was initially present in fresh curtains as follows (see appendix A for details):

$$\% \text{LiOH}_{\text{initial}} = \frac{\% \text{LiOH}_{\text{sample}} * (15430 - [54.3*\% \text{Inert}_{\text{initial}}])}{10000 - (100*\% \text{Inert}_{\text{initial}}) + (54.3*\% \text{LiOH}_{\text{sample}}) - (0.543*\% \text{Inert}_{\text{initial}}) + (54.3*\% \text{LiOH}_{\text{sample}})}$$

where %LiOH_{initial} is the percentage by weight of the LiOH content that was initially present in fresh curtains, %Inert_{initial} is the percentage by weight of the non-LiOH content of fresh granular LiOH or RPC, and %LiOH_{sample} is percentage by weight of LiOH in the sample. For example, this analysis would yield a value of 100% for fresh, unreacted curtains; a value of 50% for curtains in which half of the initial LiOH had been depleted; and 0% for curtains that no longer contained unreacted LiOH.

Studies of the Performance of the BC in Cold Conditions

The chamber was cooled by placing about 220 kg of ice in the bilge of the inner lock one day prior to an experiment, turning off the heat in the chamber room, and opening all windows and doors to winter weather (the temperature within the chamber room was 4.4-9.4 °C). Three Hangings were deployed in each experiment according to the protocol previously described for studies in warm conditions. Three such experiments were performed.

Statistical Methods

Comparisons between Hangings in capacity and endurance were accomplished with analyses of variance followed by Tukey's method for multiple comparisons, and paired t-tests, as appropriate. Temperature and relative humidity data were also examined with analyses of variance. A probability level of $\alpha < 0.05$ was considered significant.

Chemical analyses were examined with two-way analyses of variance with hanging number and location within a Hanging as factors. To assess the effect of location on LiOH content in RPCs, data from "outer high" and "outer low" groups were collapsed as were "middle high" and "middle low" data, and a pair-wise comparison of outer vs. middle location was accomplished with the Wilcoxon signed rank test. Similarly, "outer high" and "middle high" groups were collapsed, as were "outer low" and "middle low" groups, permitting a comparison of high vs. low locations. The probability level of significance for these multiple, *a priori* comparisons was adjusted to maintain an overall probability level of $\alpha < 0.05$.

RESULTS

Atmospheric Carbon Dioxide Levels

Figure 3 presents chamber atmospheric CO_2 content throughout the course of four experiments with the RPC and the BC. Each point depicts the mean of the Near and Far data at a given elapsed time. Each curve in figure 3 contains five maxima, one at zero elapsed time, and one at the time each of the four Hangings reached exhaustion. Note that, in figure 3, each Hanging was considered exhausted when CO_2 reached 3%, but, since this occurred at slightly different times in each experiment, and these curves represent the mean of four experiments, the combined results are "unfocused" and, in most cases, do not quite reach 3%. Each curve in this figure also contains four minima, the point at which the rate of CO_2 scrubbing equaled the rate of CO_2 addition. These minima occur at a substantially lower CO_2 with the RPC than with the BC, suggesting that, at a given CO_2 level, the rate of CO_2 scrubbing with the RPC was higher than that of the BC. These graphs also demonstrate that Hanging 1 of the BC became exhausted more quickly than Hanging 1 of the RPC, but that, over the course of four Hangings, the total endurance times of the two products were nearly identical.



Figure 3. Chamber atmospheric CO_2 content throughout the course of experiments with the BC and the RPC. Each point depicts the mean of the data from four experiments. Error bars depict 1SD.

Hanging Endurance

Although information regarding the endurance of Hangings can be gleaned from figure 3, these data are more conveniently presented in figure 4. Hanging 1 lasted much longer in experiments with RPCs than with BCs. The converse is true for Hangings 2 and 3, and differences in endurance are not apparent in Hanging 4.

Scrubbing Capacity

Figure 5 presents the capacity of these two products for CO_2 scrubbing. The total capacity of four Hangings of RPC (0.808 ± 0.007 ; mean \pm SD) is larger than that of four Hangings of BC (0.756 ± 0.012), but this difference is small.

Atmosphere Mixing

Because CO_2 is more dense than air, it will tend to concentrate in the bilge of a chamber if bled slowly into an undisturbed atmosphere. In experiments with the RPC, CO_2 levels in the bilge (mean Low CO_2) were somewhat higher than those in other locations (figure 6). In regions other than the bilge, CO_2 was distributed homogeneously, indicating that the "convective engine" formed by the curtains was circulating air within the chamber. The BC was less capable in this regard (figure 7); CO_2 tended to accumulate in the bilge throughout these experiments. It should be recognized, however, that the bilge sample line was located within centimeters of the lowest level in the chamber, a region that represents a small fraction of the total volume of the chamber and an area from which crew would be unlikely to breathe. Gas in the "Near", "Far", and "High" regions was well mixed with both products.





Temperature

The temperature of the atmosphere within the chamber room, the temperature of the atmosphere within the chamber at the conclusion of each of the four Hangings, and the maximum temperature derived from a contact thermistor applied to a curtain are depicted in table 1 (temperatures during cold studies of the BC are discussed separately below). Chamber atmosphere temperatures represent the mean of values from thermistors located 1 m and 2 m from the curtains. Because only one value for contact temperature was obtained during studies with the RPC, statistical analyses of contact temperature were precluded. Temperatures obtained with the RPC (other than contact temperature) are lower than those with the BC, although the magnitude of these differences is small. The RPCs themselves got hotter than the BCs. Indeed, the divers who hung the curtains while wearing latex examination gloves reported that the RPC felt quite hot immediately upon exposure to the chamber atmosphere (3% CO₂), hotter than the BC and nearly too hot to handle.

Table 1. Chamber Room Temperatures (°C), Chamber Atmosphere Temperatures During FourHangings, and Maximum Strip Contact Temperatures.

	Room	Hanging 1	Hanging 2	Hanging 3	Hanging 4	Strip
RPC	25.1 <u>+</u> 1.3	28.1 <u>+</u> 0.9	29.1 <u>+</u> 0.8	29.5 <u>+</u> 0.3	29.9 <u>+</u> 0.6	53.0
BC	26.1 <u>+</u> 0.5	28.2 <u>+</u> 1.5	30.4 <u>+</u> 1.1	30.8 <u>+</u> 0.7	31.3 <u>+</u> 0.5	40.0 <u>+</u> 3.6

Data are means \pm SD (only one value of strip contact temperature with the RPC is available). RPC = Reactive Plastic Curtain. BC = Battelle Curtain. Atmospheric temperatures with the RPC are lower than those with the BC (p = 0.002).



Figure 5. Capacities of the BC and the RPC for CO_2 scrubbing. Data are means + 1SD. Statistical analyses indicate that, for the BC, Hangings 2 and 3 have the same capacity, but that all other comparisons of the Hangings of that product are different. For the RPC, Hanging 1 is different from all other Hangings, and all other Hangings are the same. For Total capacity over the course of the complete experiments, the capacities of the BC and RPC are different.

Humidity

In all Hangings of all experiments, the relative humidity (RH) at the conclusion of a Hanging was 100%. The mean minimum RH observed with the BC was 98.5%, with a range of 96% - 100%. The mean minimum RH observed with the RPC was 90.1% (p < 0.001 compared to BC), with a range of 87% - 92%.

Mechanical Properties and Ease of Handling

A total of 144 RPC strips from 24 canisters was produced by one of the investigators (WN) while working in a conventional laboratory environment without personal protective equipment. He noted during this work occasional skin irritation beneath a watch band and no eye, nose, or throat irritation. Although the RPCs were quite friable, all of these curtains were successfully hung in the chamber without breakage. No curtain spontaneously broke during the course of the experiments. At the termination of the experiments the used curtains were brittle and were easily broken.



Figure 6. Chamber atmosphere CO_2 content at four locations during experiments with the Reactive Plastic Curtain. "Near", "Far", and "High" curves are largely superimposed, indicating that gas was well mixed across these three locations. As Hangings became exhausted, values for "Low" CO_2 , taken from a sampling port located deep in the chamber's bilge, tended to be higher than other values, indicating that , at those times, the atmosphere in the bilge was not well mixed with the rest of the chamber atmosphere. Error bars are omitted for clarity.



Figure 7. Chamber atmosphere CO_2 content at four locations during experiments with the BC. "Near", "Far", and "High" curves are superimposed, indicating that gas was well mixed across these three locations. Values for "Low" CO_2 , taken from sampling port located deep in chamber's bilge, tended to be higher than other values, indicating that the atmosphere in this location was not well mixed with the rest of the chamber atmosphere. Error bars are omitted for clarity.

A total of 50 BCs was prepared by one of the investigators (WN). Episodic releases of LiOH dust occurred during this work that necessitated moving the operation outdoors, although, with practice, these releases were eliminated. Curtain preparation involved first prying the lid, mesh screen, and felt filter out of a conventional LiOH canister with a screwdriver. If this was not performed slowly and carefully, a plume of dust was released into the face of the operator. When transferring the contents of a canister into the curtains, the curtains occasionally ripped near the location of ultrasonic welds, releasing dust and granules. Two curtains failed beyond use during filling and were discarded. While hung in the chamber, two curtains ripped near the location of ultrasonic welds close to the grommets from which they were hanging. These failures did not release granules or cause the curtains to fall.

Chemical Analyses

Figure 8 depicts unreacted LiOH content, expressed as percent by weight of the LiOH initially present when the curtains were hung, of samples taken at the end of the experiments from four Hangings of RPC. The LiOH contents of Hangings 1 and 2 were the same, while the content of Hanging 3 was greater than those of Hangings 1 and 2, and the content of Hanging 4 was greater than that of Hanging 3. Little unreacted LiOH was present in the first two Hangings by the end of the experiments. LiOH content at outer locations was higher than at middle locations, and

content at high locations was higher than at low locations. Analysis of variance of these data also revealed a significant interaction between hanging number and location within a hanging, indicating that location within a Hanging influenced LiOH content, although, as Hangings became exhausted, these regional differences were obscured.



Figure 8. LiOH content, expressed as percent by weight of the LiOH initially present when the curtains were hung, of samples taken at the end of the experiments from four Hangings of RPCs. Data are means + 1SD; n.s. = differences not statistically significant. The LiOH contents of Hangings 1 and 2 were the same, while the content of Hanging 3 was greater than those of Hangings 1 and 2, and the content of Hanging 4 was greater than that of Hanging 3. Content at outer locations was higher than at middle locations, and content at high locations was higher than at low locations.

Figure 9 presents data for the BC in a similar manner. As with the RPC, the LiOH contents of Hangings 1 and 2 were the same, while the content of Hanging 3 was greater than those of Hangings 1 and 2, and the content of Hanging 4 was greater than that of Hanging 3. Content at the "high" location was higher than at the "low" location in Hangings 3 and 4.

The results of these experiments with the BC prompted a separate, additional set of studies to determine how completely granular LiOH reacts with CO_2 under severe conditions. A bed of fresh granules less than 3 mm thick was placed into a sealed Erlenmeyer flask. A jet of CO_2 was injected into the flask onto the granules, maintaining the gas within the flask at nearly 100% CO_2 for 24 h, after which chemical analyses were performed for LiOH content. Three such experiments were completed. The unreacted LiOH content of these samples was $5.9 \pm 2.7\%$. Additional studies were performed in an identical manner except that the LiOH granules were ground into a fine powder prior to placement in the flask; in these studies, the LiOH content was

below the limit of detection of the assay, 0.5%. Consequently, one might interpret figure 9 with the perspective that, for practical purposes, about 6% of the LiOH within a BC can not react with CO_2 . When viewed in this fashion, these figures indicate that Hangings 1 and 2 contained little "accessible" LiOH by the end of the experiments.



Figure 9. LiOH content, expressed as percent by weight of the LiOH initially present when the curtains were hung, of samples taken at the end of the experiments from four Hangings of BCs. Data are means + 1 SD; n.s. = differences not statistically significant. The LiOH contents of Hangings 1 and 2 were the same, while the content of Hanging 3 was greater than those of Hangings 1 and 2, and the content of Hanging 4 was greater than that of Hanging 3. Content at high locations was higher than at low locations. The dotted line is at 6%, the content that was observed after granular LiOH was subjected to 100% CO₂ for 24 h.

Studies of the Performance of the BC in Cold Conditions

The temperature within the chamber ranged from 7.9-10.7 °C at the start of the experiments, and plateaued at 12.2-13.3 °C. Ice was present in the bilge at the end of the experiments, indicating that the rise in temperature was a result of liberation of heat by the curtains rather than exhaustion of the ice supply. The capacity of three Hangings of BCs under these cold conditions was 92.0% of that reported above for Hangings 1-3 in a warm chamber.

DISCUSSION

This study investigated the CO₂ scrubbing capabilities of two new products, the BC and the RPC, when utilized with a non-powered, "passive, static" method (i.e., convection was not

induced by stirring the atmosphere or moving the curtains). Curtains were hung in batches sized to last approximately 12 h while exposed to a constant mass flow of CO₂ (6.69 g/min). Initial conditions in the chamber included 3% CO₂, and exhaustion of a Hanging was defined as a return of the chamber atmosphere to 3% CO₂. In each experiment, four Hangings were deployed over the course of about 45 h. The total scrubbing capacity (i.e., mass of CO₂ scrubbed per unit mass of product) of four Hangings of BC was 0.756 ± 0.012 , and the comparable value for the RPC was 0.808 ± 0.007 . These capacities compare favorably to that assumed by the Guard Books for conventional granular LiOH utilized in a powered hopper (0.75). They also compare favorably with that dictated by the stoichiometric characteristics of the underlying chemical reaction (0.919), indicating that roughly 85% of the total mass of LiOH placed in the chamber had reacted with CO₂ by the time the experiments were terminated. Hence, the first of two major findings of this study. Both the BC and the RPC, when used according to the passive, static method of this study, provide a scrubbing capacity that is comparable to that of granular LiOH in a powered hopper.

A major shortcoming of the "stir-and-fan" method of CO₂ scrubbing is that it releases clouds of highly caustic dust. In our study, both the RPC and BC were handled by an individual wearing no personal protective equipment without difficulty, except in the unusual instance when the BC ripped. In separate studies, during vigorous handling of both products, the surrounding atmosphere contained LiOH dust below the limit of detection of the method used, 17 ug/m³ (P. Hennessey, personal communications). Therefore, the second major finding of this study: Both of these products represent a great advance in terms of dust release over the current method, "stir-and-fan".

A useful application of the results of this study is estimation of the mass of scrubbing material that must be on hand to deal with the demands of a multi-day DISSUB scenario when the BC and RPC are used according to the experimental protocol. The approach to this problem that involves the least assumptions is to estimate scrubbing capacity on the basis of the total results obtained over the course of the entire two-day protocol. The capacity observed in a longer timeline may exceed this estimate slightly, but it will not be less. To conservatively adjust these estimates by accounting for variability in the experimental results, two standard deviations can be subtracted from the mean data. Capacity estimates derived in this fashion are presented in table 2.

Product	Capacity
RPC	0.794
BC	0.732

Table 2. Capacity Estimates for the RPC and the BC in a Multi-Day Scenario.

Data are means - 2SD. Capacity is defined as mass of CO₂ scrubbed per unit mass of product.

The Guard Books state that the scrubbing capacity of LiOH with the "stir-and-fan" method is half that of the powered-hopper method. This specification has profound implications on the load of LiOH that must be carried by a submarine to support a large contingent of survivors for several days. The BC and RPC, primarily by virtue of their relatively high scrubbing capacities, offer the potential for halving scrubbing agent "load-outs". For example, per the methods of the

Guard Books, the mass of LiOH required to provide six days of "stir-and-fan" CO₂ scrubbing for 120 men is 2095 kg, whereas, utilizing the capacity data in table 2, the required mass of RPC is 989 kg, a savings of 1106 kg, and the required mass of BC is 1073 kg, a savings of 1022 kg. Stated another way, the number of LiOH canisters currently required to support six days of "stir-and-fan" scrubbing for 120 men is 733 canisters, whereas, utilizing the capacity data in table 2, 376 conventional canisters are needed with the BC method (a savings of 357 canisters), and 257 RPC canisters are necessary (assuming the manufacturer's most current estimate of the final configuration of their product: 3.856 kg of product per canister, in a package with dimensions comparable to those of conventional granular LiOH canisters).

The experimental approach of this study involved addition of a known mass flow of CO_2 to a sealed chamber containing a known mass of scrubbing material. Calculation of scrubbing capacity was, therefore, straightforward. An alternate approach used in some investigations is to track changes in the weight of a LiOH bed placed in an atmosphere "clamped" at a specified CO_2 content. This approach does not require access to a sealed chamber. The difficulty with this method is that anhydrous LiOH (the form of LiOH contained in current canisters) can gain weight in such an environment by two means: reaction with water to form LiOH monohydrate, and reaction with carbon dioxide to yield lithium carbonate. Unless samples of the bed are removed and baked in a CO_2 -free atmosphere prior to weighing to eliminate free water and lithium monohydrate, the extent of carbonate formation in the bed is not known. The experimental method reported herein avoids such ambiguity.

Our experimental design did involve several assumptions about conditions within the DISSUB including the number of survivors, the maximal acceptable CO₂ content, and the mass of scrubbing material that is deployed in each Hanging. These assumptions determined the rate at which CO₂ was added to the chamber and the mass of scrubbing material that was present, or, by analogy to an electrical circuit, the CO2 "load" on the curtains. Previous investigations have demonstrated that the scrubbing capacity of a given bed of LiOH through which gas is forced is a function of this load, assuming that exhaustion is defined as CO₂ "breakthrough" through the bed.(1) For example, a heavily loaded bed will have a lower capacity than a lightly loaded bed. The implication of this concept to our study is that our capacity data are strictly valid only for the loading conditions present during the experiments. These capacity data also bound what would be encountered in a more lightly loaded scenario, i.e. a capacity greater than or equal to that observed in this study would be found. However, this study can not predict what would happen in a more heavily loaded scenario since rapid addition of CO₂ might quickly exceed the rate at which CO₂ is being scrubbed, even though a great deal of unreacted LiOH remains. Further experiments at a range of loadings would generate a family of capacity curves that could specifically address a wider variety of DISSUB conditions.

The effect of CO_2 load on curtain endurance may explain the pattern of atmospheric CO_2 levels depicted in figure 3. Each trace includes four minima, the points in time at which the rate of CO_2 addition equaled the rate of CO_2 scrubbing. These minima are much lower with the RPC than with the BC, suggesting that, at a given atmospheric CO_2 concentration, the RPC scrubbed more rapidly. This may be a consequence of a relatively greater surface area of LiOH exposed to the atmosphere with this product, a notion consistent with the fact that each Hanging of RPC deployed nine curtains, whereas each Hanging of BC consisted of two curtains (the height and

width of both types of curtains were roughly equal, as was the mass of LiOH involved in each Hanging).

An important assumption underlying this study is that excursion of the atmospheric CO₂ up to, but not exceeding, 3% is acceptable. Currently, no explicit, authoritative guidance is available to establish precisely the CO₂ limit that applies aboard a DISSUB to guide LiOH utilization. The Guard Books do state that rescue or escape should be complete before 6% CO₂ is reached. Also, they specify that, if a powered hopper is used, outlet CO₂ should be monitored for "breakthrough" that indicates canister depletion, and, if "stir and fan" scrubbing is necessary, all available LiOH should be spread out immediately. None of these recommendations specifically addresses the CO₂ level at which curtains of LiOH should be deployed. The current version of the Atmosphere Control Manual(4), as amended on 12 February 99, establishes 1.5% CO₂ as a limit for 24-h exposures, but 0.7% CO₂ as a limit beyond 24 h. A revision of this Manual currently under review states that, if a powered hopper is used, "… 3% CO₂ concentration is recommended for DISSUB situations in order to maximize the canister utilization with minimal impact on crew health" (R. Hagar, personal communication).

The CO₂ limit chosen to govern curtain deployment must not be too high, because untoward physiologic effects will result (e.g., exercise tolerance will be limited, thereby compromising the ability to deal with a new casualty or to perform escape operations.) This limit must not be too low, because LiOH curtains will be declared exhausted when, in fact, much unreacted LiOH remains. In our judgment, a 3% limit seems to be an effective compromise between these competing concerns, and we incorporated this limit into the experimental design of this study. It should be borne in mind that the resulting capacity data are strictly valid only with this 3% CO₂ limit. These data also bound what would be encountered with a higher limit, i.e. a capacity greater than or equal to that observed in this study would be found. However, this study can not predict what would happen with a lower limit since curtains might be declared exhausted when a great deal of unreacted LiOH remains. Further experiments at a range of CO₂ limits would generate a family of capacity curves that could specifically address a wider variety of DISSUB conditions.

The individual curtains in a Hanging were spaced about 5 cm apart. Recognizing that the reaction of LiOH with CO₂ and water vapor is exothermic, we postulated that such an arrangement would form a "convective engine" that would circulate the atmosphere in the chamber. CO_2 levels monitored at three of four locations were homogenous (above the curtains, and 1 m above the deck at near and far points; some CO₂ accumulation did occur deep in the bilge), indicating that this arrangement was effective at circulating gas. A variety of human-powered scrubbing methods have been developed over the years (e.g., Prenderville 1981(5)), but these devices increase the metabolic activity of the operator and, therefore, decrease the survival time of the crew based upon CO_2 limits.(6) The curtain spacing we chose was based upon a crude estimate of optimal spacing tempered by a desire to minimize the habitable volume occupied by the curtains. Whether a different spacing might be more effective is a question worthy of further study.

An important aspect of the experimental design is that exhausted Hangings were not removed from the chamber – they were supplemented, not replaced, by fresh Hangings. We anticipated

that four Hangings, conducted over about 48 h, would provide sufficient data to predict the LiOH requirements of longer timelines such as a 7-d survival scenario. Indeed, with the RPC, a "plateau" in the data was reached – the endurance and capacity of Hanging 1 were less than those of Hangings 2-4, but Hangings 2-4 were not statistically different from each other. Additional insight into whether a "plateau" in performance had been reached after four Hangings is provided by the chemical analyses: if a plateau is reached, then Hangings hung early in the experiments should have contained little unreacted LiOH. In fact, chemical analyses of RPC samples revealed that the LiOH contents of Hangings 1 and 2 were small and equal, while the content of Hanging 3. Consequently, it is reasonable to predict that in a longer scenario, Hanging 5 and all subsequent Hangings would have a capacity and endurance comparable to those of Hangings 2-4. Furthermore, this information suggests that, to conserve habitable space within the compartment without decreasing Hanging endurance or capacity, Hanging 1 can be taken down when Hanging 5 is hung, Hanging 2 can be taken down when Hanging 6 is hung, and so on.

With the BC, no such "plateau" in the endurance and capacity data was reached. The scrubbing capability of Hanging 1 was rapidly overwhelmed. However, once Hanging 2 was hung, a large mass of unreacted LiOH was present in the chamber that was able to handle the CO₂ "load" far longer than if Hanging 1 had been removed when Hanging 2 was hung. This "load sharing" between fresh and old Hangings continued throughout the experiment, resulting in a total endurance and capacity that were nearly as great as those of the RPC. Chemical analyses of samples of the BCs taken at the end of the experiments revealed a pattern identical to that of the RPC data: the LiOH contents of Hangings 1 and 2 were small and equal, while the content of Hanging 3 was greater than those of Hangings 1 and 2, and the content of Hanging 4 was greater than that of Hanging 3. It seems likely that, in a longer scenario, as with the RPC, Hanging 1 can be taken down when Hanging 5 is hung, Hanging 2 can be taken down when Hanging 6 is hung, and so on, without significantly decreasing endurance and capacity.

Samples for chemical analyses were taken from multiple locations within the Hangings to gain insight into the regional patterns of CO₂ scrubbing activity. We speculated that arranging the curtains as described would form a "convective engine" that would circulate gas within the compartment by natural convection. If this occurred, then discrete regions of the curtains would be exposed to non-uniform concentrations and mass flows of CO₂. Indeed, significant regional differences in LiOH content were seen with fresher Hangings of both products (Hangings 3 and 4). High regions contained more LiOH than low regions, presumably because low regions scrubbed out some of the CO_2 before it flowed to high regions. With the RPC, peripheral regions contained more unreacted LiOH than core regions, perhaps because convective flow was higher in these core regions, delivering more CO₂ to these regions and exhausting them more quickly (the BC Hangings consisted of only two curtains and, therefore, had no "core" curtains). With depleted Hangings (Hangings 1 and 2), these regional differences became obscured, perhaps because of dissipation of the "convective engine" and subsequent homogenous exposure to CO₂. Further studies might directly compare the performance of curtains arranged as described in this study versus solitary curtains to determine the importance of curtain spacing and arrangement. This issue is of operational importance since DISSUB survivors may wish to

conserve habitable space by arranging curtains in a more dispersed fashion – for example, by hanging them against walls or other structures, thereby "getting them out of the way".

We were able to monitor, but not control, temperature within the chamber. Like most hyperbaric chamber systems, atmospheric cooling in this chamber is normally accomplished by a heat exchanger in an external life support loop, and functioning of this cooling system requires operation of a forced air blower. Such forced convection would have disturbed the DISSUB-like conditions we were attempting to produce. Placing ice in the chamber's bilge was effective in cooling the chamber, permitting cold (or, at least, cool) studies with the BC.

Although ice was present in the chamber throughout the cold experiments and the chamber room was cool, the chamber temperature rose during theses experiments, reflecting liberation of heat by the curtains. Chamber air temperature also rose during warm studies, reaching about 5 °C above room temperature with both products. Whether heat liberation by curtains could significantly impact compartment temperature aboard a DISSUB is an important survival issue worthy of further analysis and experimentation.

All experiments were performed at a chamber pressure of approximately 1 atmosphere absolute. Whether hyperbaric conditions (as would occur in a partially flooded DISSUB) would modify these results is also a topic worthy of future study.

Figure 3 demonstrates that the minimum atmospheric CO_2 content achieved was much lower with the RPC than with the BC. It is interesting to speculate whether DISSUB survivors are better off in a CO_2 atmosphere that cycles dramatically versus one that features a relatively constant, moderately elevated CO_2 content. Deep cycling could attenuate metabolic compensation of respiratory acidosis. Many studies have investigated the physiologic effects of acutely and chronically elevated inspired CO_2 , but none has directly addressed cycling CO_2 . We speculate that the impact of CO_2 cycling in our study is likely to be negligible because the maximum CO_2 content achieved in our study was 3%, a level that produces only mild physiologic effects.(7)

In summary, the total scrubbing capacity (i.e., mass of CO₂ scrubbed per unit mass of product) of four Hangings of BC was 0.756 ± 0.012 (mean \pm SD), and the comparable value for the RPC was 0.808 ± 0.007 . These capacities compare favorably to that assumed by the Guard Books for conventional granular LiOH utilized in a powered hopper (0.75). The preparation and use of both products did not release sufficient caustic dust to prevent handling by a trained individual wearing no personal protective equipment.

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The opinions and assertions contained herein are the private ones of the authors and are not to be construed as official or reflecting the views of the Navy Department or the naval service at large.

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APPENDIX A: Calculation of LiOH content:

The chemical analysis directly yielded the percentage by weight of LiOH in the sample:

(1) %LiOH_{sample} = $\frac{\text{LiOH}_{\text{sample}} * 100}{\text{Inert}_{\text{sample}} + \text{LiOH}_{\text{sample}} + \text{Li}_2\text{CO}_{3\text{sample}}}$

where %LiOH_{sample} is the percentage by weight of LiOH in the sample, LiOH_{sample} is the mass of LiOH in the sample, Inert_{sample} is the mass of inert substances in the sample derived from non-LiOH material present in the fresh LiOH granules (or RPC), and Li₂CO_{3sample} is the mass of Li₂CO₃ in the sample resulting from reaction of LiOH with CO₂. Note that, since the processing of the sample included cooking the sample, the sample included no free water or lithium hydroxide monohydrate at the time it was weighed. Note also that %LiOH_{sample} is not equal to the remaining percentage by weight of the LiOH that was present in the fresh curtain since the mass of the curtain increases as it reacts with CO₂ and water vapor. Additional calculations are necessary to obtain the remaining percentage of LiOH:

As a sample of fresh LiOH granules (or RPC) reacts with CO₂, the mass of inert ingredients (Inert_{initial}; consisting of non-LiOH substances present in the fresh granules or RPC) does not change. Each mole of LiOH that reacts with CO₂ yields 0.5 moles of Li₂CO₃. For example, if 100g of fresh granules contains 2.9% inert ingredients, then, by definition, Inert_{initial} = 2.9g, and the percentage by weight of the LiOH initially present (%LiOH_{initial}) is 100%. This fresh sample contains 97.1g of LiOH. If this sample reacts with CO₂ such that 70% of the initial LiOH is consumed, then, at that point, %LiOH_{initial} = 30%, and the sample contains the following substances:

 $LiOH_{sample} = 29.1g$ $Li_2CO_{3sample} = 104.9g$ Inert_{sample} = 2.9g

Substituting these data into (1), %LiOH_{sample} = 21.3%, which is the datum that would be directly yielded by a chemical analysis of this sample. Reversing this computational processes, such that, given %LiOH_{sample}, %LiOH_{initial} is determined, can be accomplished through substitution and algebraic manipulation of (1), yielding the expression stated in the text.

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scrubbing capacity that is close to the stoichiometric limit of the reaction (0.919). Neither product released sufficient caustic dust to prevent handling by a trained individual wearing no personal protective equipment.