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DEVELOPMENT OF EQUIPMENT AND PROCEDURES FOR PRODUCING LARGE QUANTITIES OF FOAMED SULPHUR IN THE FIELD

John M. Dale
Allen C. Ludwig

Southwest Research Institute
San Antonio, Texas

Contract AF 29(601)-6408

TECHNICAL REPORT NO. AFWL-TR-65-71

January 1966

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FOREWORD

This report was prepared by the Southwest Research Institute, 8500 Culebra Road, San Antonio, Texas, under Contract AF 29(601)-6408. The research was performed under Program Element 7.60.06.01.D, Project 5710, Subtask 13.157, and was funded by the Defense Atomic Support Agency (DASA).

The program was a continuation of an earlier study entitled "Feasibility of Foamed Sulphur as a Material for Shock-Isolating Large Underground Structures" performed under Contract DA 22-079-ENG-374 (SwRI Project 02-1415).

Inclusive dates of research were 1 June 1964 to 3 June 1965. Captain Edward H. Bultmann, Air Force Weapons Laboratory (WLDC), served as AFWL project officer until September 1964 when 1Lt James A. Eddings, AFWL (WLDC), became project officer. The report was submitted in December 1965.

SwRI personnel who contributed to this program were Mr. John M. Dale, Project Leader, Mr. Allen C. Ludwig, and Mr. Lew W. McNeil.

This technical report has been reviewed and is approved.

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ABSTRACT

Work performed under this contract represents the first practical development of a process and equipment for producing large quantities of rigid sulphur foam. While the sulphur foam formulations and the resultant properties of the foams have not been fully optimized, this new foam material represents a significant advance in the field of foam technology and has applications that measurably exceed the use intended herein. It was demonstrated that foamed sulphur can indeed be produced on a continuous basis at a remote field site by a process and with equipment that is simple and capable of being scaled up to any size. The foam prepared using the procedures and equipment developed herein has mechanical properties comparable to that produced on a batch basis in the preceding laboratory study--namely a density of 27 lb/ft^3, a compressive strength of 130 psi, a constant strain rate deformation to 65-70 percent, and a low water absorption. Since the cost of the raw materials for these sulphur foams is less than two cents per pound, a foam with a density of 27 lb/ft^3 can be produced for only $0.54 per cubic foot. Conventional foams with similar physical properties that can be produced at a remote field site are the rigid urethane foams. The comparative cost of a urethane foam having a compressive strength of 125 psi is approximately $3.00 per cubic foot. During the week of 17 January 1965, the feasibility of producing foamed sulphur in the field was demonstrated for the Air Force at Kirtland Air Force Base, Albuquerque, New Mexico, by filling a 30-cubic foot annular space with rigid foamed sulphur.

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SECTION I

INTRODUCTION

The primary objective of this investigation was to develop equipment and procedures for producing foamed sulphur under remote site conditions. A secondary objective was to demonstrate the feasibility of the final apparatus.

Previous studies (Contract DA-22-079-ENG-374) had shown that sulphur foams have very desirable properties for use as an energy absorbing material. That study had been conducted entirely in the laboratory and no method existed for producing large quantities of the foam. The problems associated with the production of foams are unique to the material produced and foamed sulphur proved to be no exception. However, the knowledge and experience gained from the previous laboratory study proved invaluable in the development of equipment and procedures for producing foamed sulphur in the field.

The program was divided into three phases. The first phase consisted of designing and fabricating the foaming machine. The second entailed placing the machine in operation and developing operating procedures. The final phase consisted of demonstrating the equipment and process by foaming a 30-cu ft section under simulated remote site conditions.

SECTION II

DESIGN, SELECTION, AND FABRICATION OF THE SULPHUR FOAMING MACHINE

At the initiation of this program, it was recognized that the design of a sulphur foaming apparatus could be achieved by two means. One of these was to survey commercially available foaming equipment and components and select those components that could be readily converted to produce sulphur foams. The second was to design and fabricate a unit specifically for the purpose intended.

There are several foaming machines on the market for the production of urethane foams that are adaptable to field application techniques. However, the equipment used for foaming urethane was found to be expensive (the smallest unit sold for more than $7,000.00), complex, and inadequate for foaming sulphur. These units consisted essentially of two tanks, one containing components A, while the other tank contained components B. Each of these materials had to be pumped and carefully metered from the tanks to a mixing head where components A and B were mixed and then discharged into the mold where expansion occurred. The complexity increased as the number of feed streams to the mixing head increased. In addition, the urethane system was designed to operate at room temperature; consequently, the lines, pumps, and tanks are not heated as they would have to be for a sulphur foam system. Thus high initial cost plus cost of conversion eliminated the urethane equipment from further consideration. Components such as melting tanks, heated hoses, air compressors, etc., while readily available and adaptable for use, presented certain limitations which made them unacceptable.

A small experimental apparatus for foaming sulphur was assembled at SwRI from existing tanks, lines, and other equipment. It had first been anticipated that the sulphur formulation would be melted in a heated tank. Then the molten sulphur formulation and the blowing agent would be mixed in a mixing head prior to discharge. The blowing agent upon coming in contact with the molten sulphur, would liberate a gas and expand the melt into a foam. However, experimentation with the assembled apparatus indicated that if the molten formulation and blowing agent were mixed and held under pressure, the material would not expand until the pressure was reduced, as for instance by opening the discharge valve and releasing the material to atmospheric pressure. Thus it appeared feasible to eliminate a complex and costly mixing head or nozzle.

Thus, by using the experience gained from these selected experiments, as well as past experience in handling molten sulphur and sulphur formulations, a system was designed which offered the advantages of simplicity, mobility, and low cost. A layout drawing showing the major components of the sulphur foaming machine as designed is shown in Figure 1. Figure 2 is a photograph of the machine after it was fabricated.

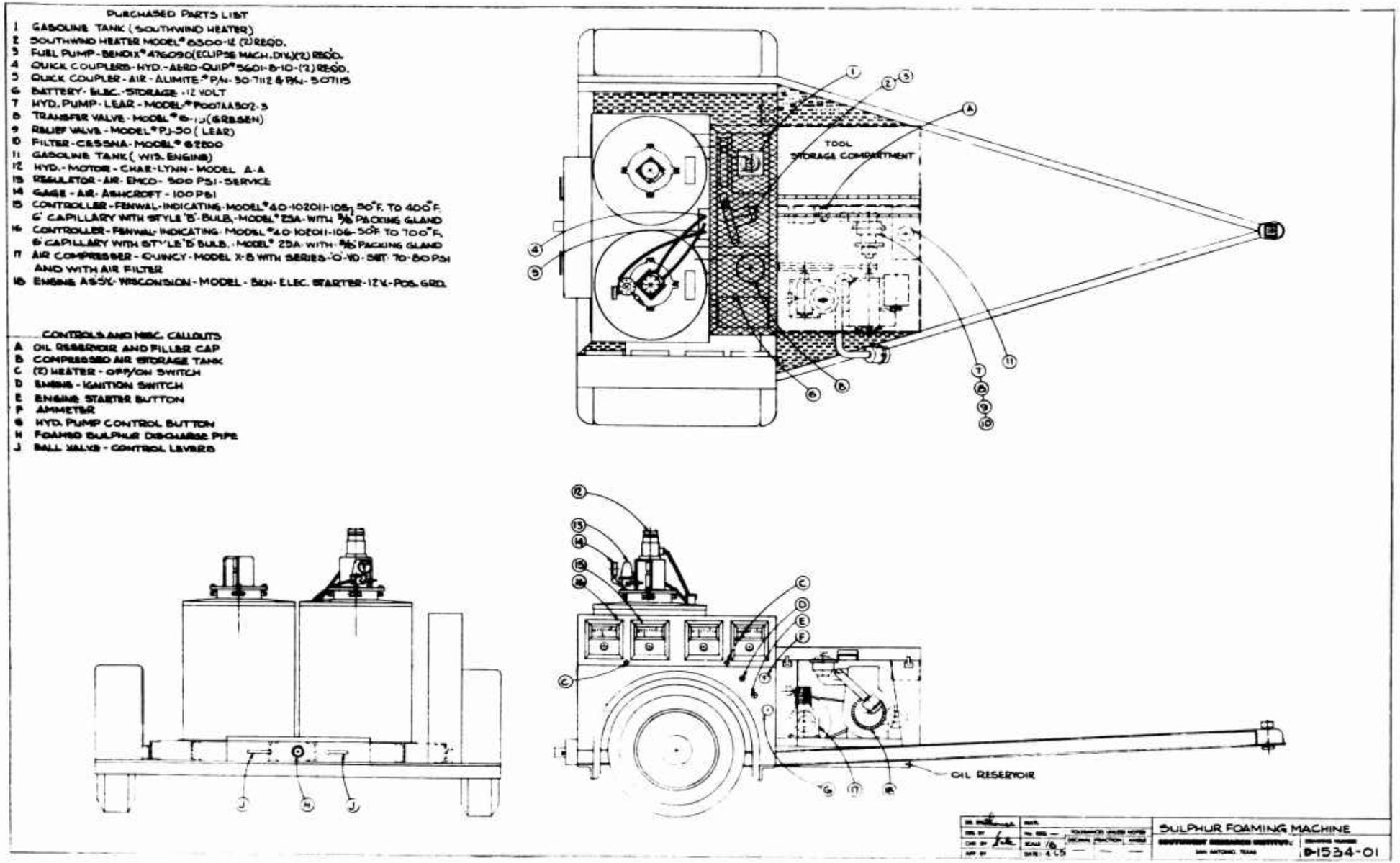

Figure 1

LAYOUT DRAWING OF SULPHUR FOAMING MACHINE

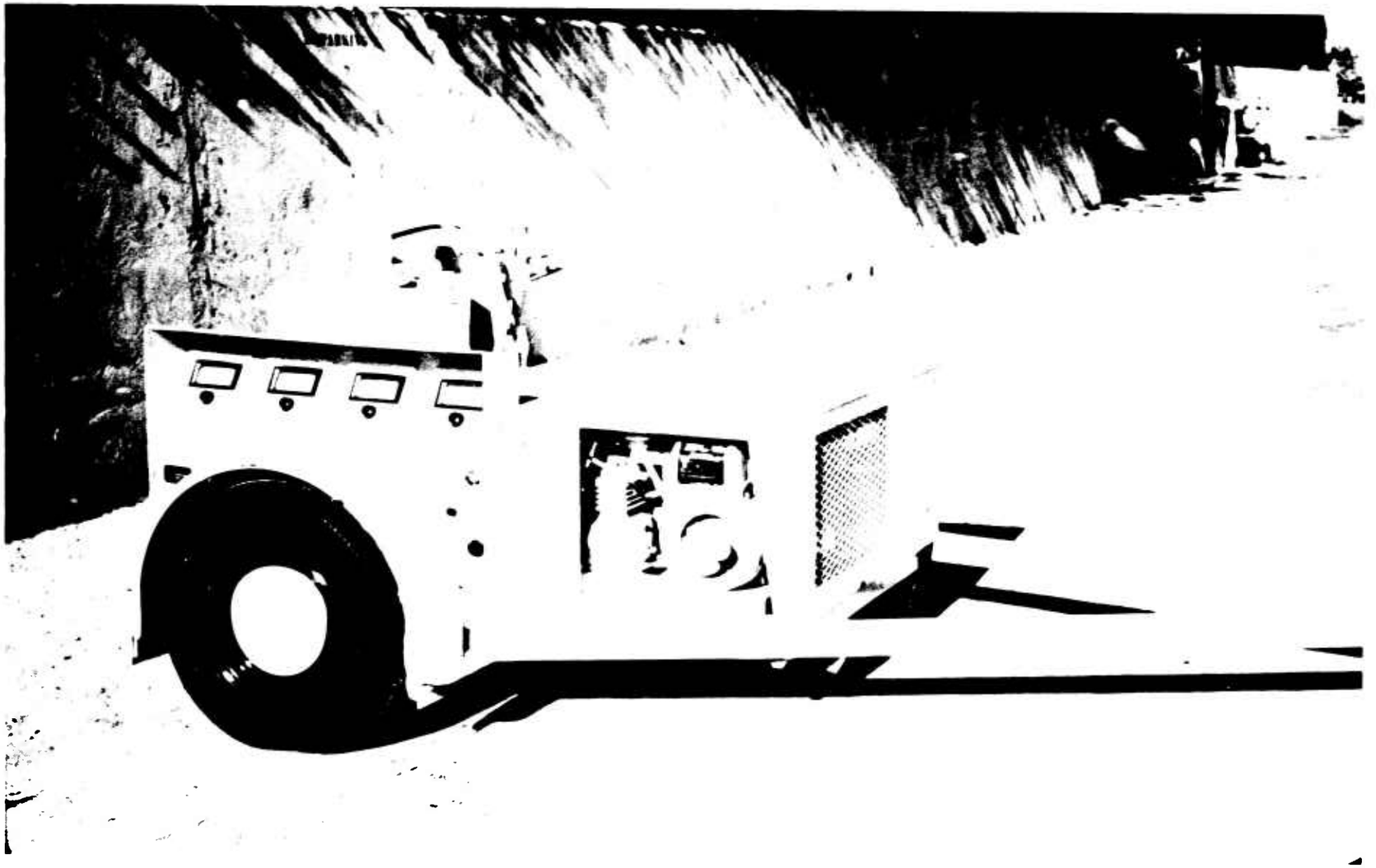

FIGURE 2. SULPHUR FOAMING MACHINE

Since only seven months and very limited funds had been allocated for all research, design, fabrication, and testing of the sulphur foaming machine, a short delivery time and low cost were of critical importance. The lowest bid and shortest fabrication time was from the Charles Machine Works of Perry, Oklahoma, who subsequently fabricated the sulphur foaming machine.

Briefly, the unit consists of two 15-gallon melting tanks equipped with hydraulic agitators. Each tank is heated by a gasoline-fired heater which has its own electric fuel pump and air blower. The sulphur and other chemicals are melted, agitated, and reacted in the melting tanks. Once the formulation is reacted and brought to the proper temperature, the blowing agent is added. Air pressure supplied by an air compressor is applied to the tank and the formulation and blowing agent are agitated. Being under pressure, the resultant mixture does not expand until the discharge valve is opened. The pressure drop across the valve allows the formulation to expand, such that a material with the consistency of shaving cream is discharged into the mold. The air compressor, hydraulic pump, and generator-starter are operated by a small air-cooled gasoline engine. For mobility the entire unit was mounted on a trailer and except for gasoline, the unit is self-contained.

SECTION III

OPTIMIZATION OF PROCEDURE AND FORMULATION USING THE SULPHUR FOAMING MACHINE

A. Process Development

Upon receipt of the sulphur foaming machine experimentation for producing foams was begun. Various formulations developed in the previous laboratory study were used as a starting point. In that study sulphur was reacted with various plasticizers which increased the viscosity of the sulphur. Talc, used as a stabilizing agent, helped retard collapse by preventing drainage of the liquid from the bubble film. A blowing agent such as sodium bicarbonate was dispersed in the molten sulphur formulation which contained the plasticizer and talc. When dispersed in the molten mixture, the sodium bicarbonate decomposed releasing water vapor and carbon dioxide. These gases then expanded the molten mass into a foam which subsequently solidified on cooling. Initial experimentation indicated that foams produced with the pilot model required twice the amount of blowing and stabilizing agents that were required by the smaller batches prepared in the laboratory. One major problem, the formation of voids in specimens having a thickness of eight inches and greater, persisted. A similar problem with specimens of much smaller thickness had also been encountered in the original laboratory study. In the laboratory study, the void problem was solved by a change in the plasticizer. Using the sulphur foaming machine, it was found that the blowing agent and technique for introducing it had a measurable influence on controlling the voids.

The entire laboratory study of sulphur foams was built around the use of chemical blowing agents which decomposed thermally. A chemical or chemicals were dispersed in the sulphur melt. The thermal decomposition, once started, had a tendency to continue for some time. In samples with relatively large thicknesses, the time from pour to the time of solidification of the center mass involved a time lapse of 2 or 3 hours, testifying to the attractive thermal insulation characteristics of the sulphur foams. Study of the voids formed in such samples indicated that the void was not caused by cell collapse and coalescence, which is generally the problem in foam materials, but was the result of continued decomposition of the blowing agent.

For this reason, it was decided to investigate three other techniques for expanding the melt. The first of these was the use of a low boiling point liquid that could be dissolved under pressure into the molten formulation. Upon release of the pressure, as for instance discharge to atmospheric pressure, the liquid would vaporize and expand the melt into a foam. While sound in principle, finding a liquid with a suitable vapor pressure and compatibility with the foam formulation proved more difficult than anticipated. Fair success

was achieved using carbon tetrachloride, heptane, and normal butyl alcohol. Fluorocarbon compounds as a group were considered but ruled out because of their high cost. Ultimately, this area of investigation was abandoned, but it is still worthy of further research effort because of the advantage offered by the complete and immediate formation of a gas.

The second technique investigated was the mixing of a gas with the sulphur formulation. This technique required a mixing head in which the formulation and a gas could be intimately mixed prior to discharge. Consequently, a mixing head was designed and fabricated. Although a series of unsuccessful experimental runs were made, it is anticipated that had a pressure system of 300 to 400 pounds per square inch been employed, this technique could have been made to work. However, the advantages of a lower pressure system (less than 100 pounds per square inch) were too numerous to abandon. Therefore, the use of a mixing head was set aside.

The final means for blowing the melt reverted to the use of blowing agents that involved a chemical reaction. Thus, more emphasis was placed on using the acid-carbonate reaction developed in the laboratory study to effect a more immediate release of gas. Using this approach, it was demonstrated that by careful control of temperature, pressure, formulation, and procedure, rather large specimens could be prepared without the voids in the center. Figure 3 shows two large specimens, one with void spaces and one without.

Also investigated was a procedure that would shorten the reaction time required for the production of foamed sulphur. It was found that reacting the plasticizers with sulphur at 170° C for twenty minutes was equivalent to reacting these materials at 155° C for one hour. However, the time saved by reacting at the higher temperature was offset by the additional time required to sufficiently cool the material such that it could be properly expanded. The one change that did expedite the overall procedure was a one-shot addition of talc, calcium carbonate, and acid. Previously, once the plasticizer had been reacted, the talc and calcium carbonate were added and thoroughly dispersed before the acid was finally added. This involved a 10-15 minute waiting period to mix the talc and carbonate. Also, the lid on the tank had to be removed each time an addition to the tank was made.

It was found that once the material was plasticized and then foamed by the one-shot addition of the talc and blowing agent, the material could be held in the tank under pressure for one hour without any detrimental effects on the foam properties, and held as long as four hours with only slight deterioration of the foam properties. It was also found that the foamed material can be sprayed a distance of 15 feet from the end of the heated flexible discharge hose without adversely affecting the foam.

Another attractive feature of the sulphur foam is the fact that specimens have been poured to a height of three feet, and the uniformity of the

Cross Section of a Specimen 2 feet square by 8 inches high with a void in the center.

Cross Section of a Specimen 2 feet square by 8 inches high without a void in the center.

FIGURE 3. LARGE SPECIMENS WITH AND WITHOUT A VOID

density and cell size from top to bottom has been excellent. Figure 4 is a photograph of a cylinder three feet in height which was filled with foam and cut in half after solidification.

It is important to note that foamed sulphur with a density as low as 25 pounds per cubic foot, which is less than one-fifth the density of elemental sulphur and with a compressive strength as high as 80 psi, can be prepared on a routine basis with the sulphur foaming machine. Attempts to produce such a foam in the original laboratory study were unsuccessful. Thus, there is every reason to believe that further improvements in lowering the density and improving the strength are possible.

As final preparation for the required demonstration of the sulphur foaming machine, a specimen of 30 cubic feet was prepared at Southwest Research Institute. A mold approximately 5 feet square by 14 inches high was used. The specimen was poured in layers, and the bonding between layers, as well as cell uniformity from layer to layer, was found to be excellent.

A number of 2-inch cubes were cut from this 30-cubic foot block of foam which had an average density of 27-lb/ft^3. The compressive stress-strain data of these specimens indicated compressive strengths of approximately 130 psi with relatively constant stress deformations to 65 to 70%.

A total of some 190 experiments were conducted during the course of this project. They have been recorded in a laboratory notebook which is kept on permanent file at Southwest Research Institute.

B. Optimum Foam Formulation

The foam formulation having a 27-lb/ft^3 density, and a relatively constant compressive strength of 130 psi to stress deformation to 65 to 70% is as follows:

Foam Formulation

Material	Parts by Weight
Sulphur	100
P_2S_5 (Phosphorous Pentasulfide)	3
Styrene	3
Talc (Mistron Vapor)	10
$CaCO_3$ (Calcium Carbonate)	1
H_3PO_4 (Phosphoric Acid 85%)	0.7

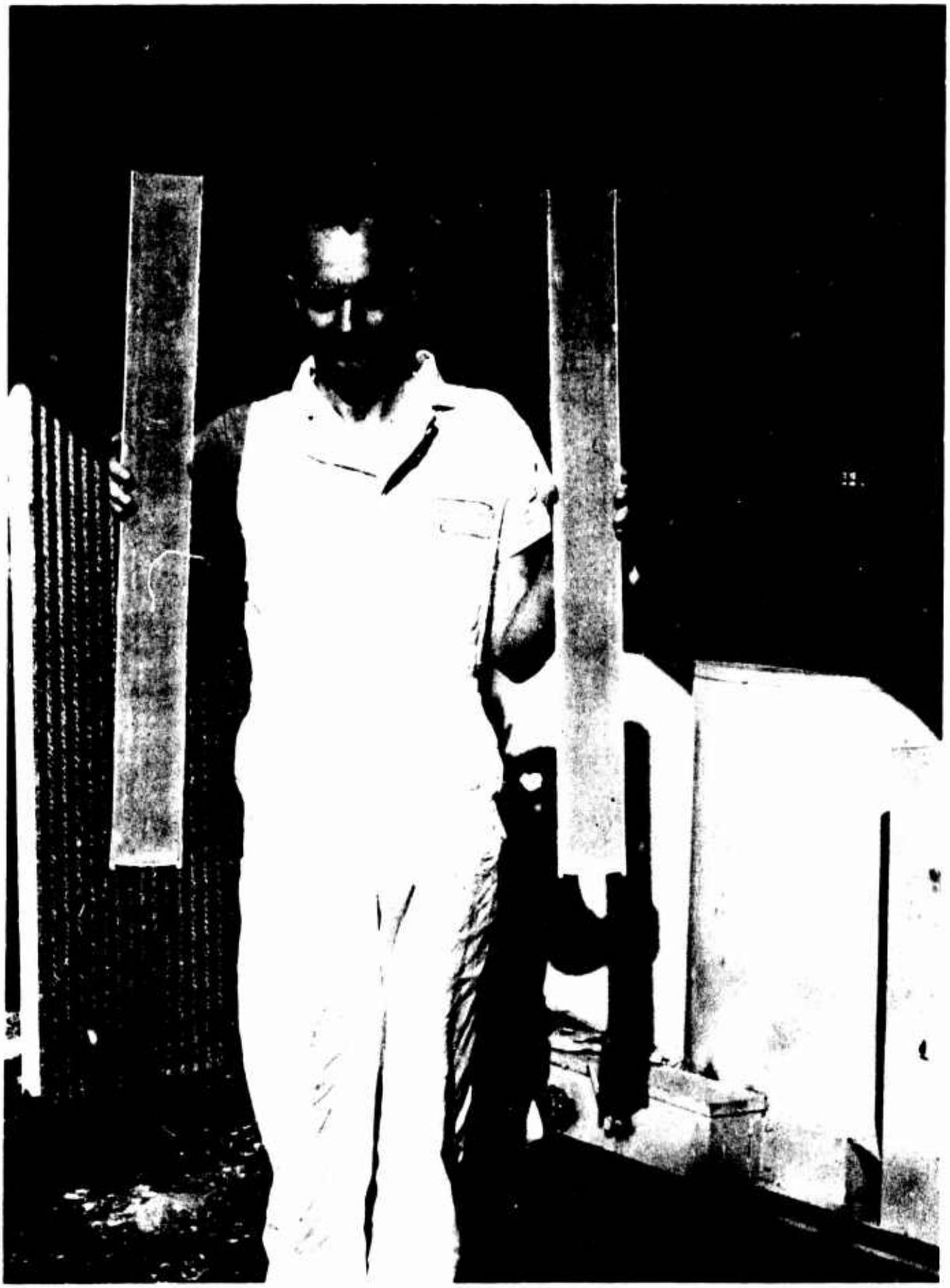

FIGURE 4. FOAMED SULPHUR SPECIMEN 3 FEET IN HEIGHT

A detailed description of preparing foam using this formulation is found in the Appendix. The $CaCO_3$ and H_3PO_4 can be varied to give foams of different densities. Normally, they will not exceed 2 and 1.4 parts, respectively.

The raw materials cost for this formulation using Chemical and Engineering News 1964 Report on "Current Chemical Prices" on extension yields a price of $0.019 per pound. The economic advantage of sulphur foams becomes readily apparent from Table I which is a cost comparison between sulphur foam and urethane foam - the one plastic foam that is capable of being foamed in the field on a large scale.

TABLE I. COST COMPARISON OF RIGID FOAMS

Properties	Urethane[1]			Sulphur		
Density, lb/ft^3	5	9	13	27	45	55
Compressive strength, psi	125	300	500	130	300	500
Approximate cost $/lb	0.60	--	--	0.02	--	--
Approximate cost $/$ft^3$	3.00	5.40	7.80	0.54	0.90	1.10

(1) Ferrigno, T. H., Rigid Plastic Foams, Reinhold Publishing Corporation, pp. 97, 255, 1963.

SECTION IV

DEMONSTRATION

The week of January 17, 1965, was devoted to the demonstration of the sulphur foaming machine to the Air Force at Kirtland Air Force Base, Albuquerque, New Mexico. The sulphur foaming machine, supplies, and two concentric drums which were earlier fabricated to provide an annular space to fill with foam were motor freighted to Kirtland Air Force Base. The equipment was moved to an open area behind the Air Force Shock Tube Facility near Sandia Base, and there the 30-cubic foot annulus was filled with foamed sulphur. This 30-cubic foot mold consisted of one 30-inch diameter cylinder mounted inside a 48-inch diameter cylinder. The length of the cylinders was 4 feet. Each cylinder had spot welded seams for easy disassembly. The cylinders were placed in a horizontal position for filling. The back was totally closed with plywood which also acted as the spacer for that end. Five 2 in. × 4 in. × 9 in. studs were used as spacers on the front end. As the foam level rose in the annulus, boards were placed across the front of the annular space to retain the foam in the mold. Upon solidification of the foam, the boards were removed. Figure 5 shows the foam being discharged from a flexible discharge hose, the end of which is in the operator's hand, and flowing as a stream of molten foam into the recesses of the annulus.

As specified in the contract, upon completion of the demonstration, the sulphur foaming machine was turned over to the U.S. Air Force and moved to the storage yard of the Air Force Shock Tube Facility and covered.

FIGURE 5. A 30-FT^3 ANNULUS BEING FILLED WITH FOAMED SULPHUR

SECTION V

CONCLUSIONS

In the brief span of approximately 3 man-years of research effort, not only has a new foam material been conceived, but the technology associated with it has been developed. In the earlier laboratory study, it was demonstratted that foamed sulphur could be prepared with the desired physical properties for shock-isolation applications.

In the research program described herein, it was proven by demonstration that:

(1) Foamed sulphur can be produced on a continuous basis by a process and with equipment that is simple and capable of being scaled up to any size.

(2) Foamed sulphur can be used to fill the annular space between two concentric cylinders at a remote field site.

(3) Foamed sulphur can be produced from readily available commercial chemicals at a materials cost of less than two cents per pound.

(4) Foamed sulphur prepared by the pilot model has mechanical properties comparable to those produced in the laboratory study - namely a compressive strength of 130 psi with constant strain rate deformation to 65-70%.

(5) Foamed sulphur can be sprayed a distance of 15 feet from the point of discharge without detrimental effects to the foam.

(6) Specimens of foamed sulphur have excellent bonding between layers and excellent cell uniformity.

The areas requiring additional research have made themselves readily apparent during the program. The voids encountered in large specimens of foam are directly related to the foam stability. Thus, additional research into producing more stable sulphur foam formulations should be highly rewarding. Though not detrimental to the physical properties of foam nor to its intended use, the fact that foam formulations do have a certain odor characteristic is considered undesirable. The fact that these odors are generated by the currently used plasticizers indicates that odorless foams could be produced by using more suitable plasticizers. Some preliminary experiments have confirmed the feasibility of such an approach. Other improvements worthy of being pursued would be studies of ways to improve the strength to

weight ratio of the foams as well as ways to reduce the water absorption of the sulphur foams. In addition to their use in shock isolation applications, it is believed that they have many yet unexplored applications in the fields of thermal insulation, structures, buoyancy objects and the like, which might well exceed the application envisioned herein.

APPENDIX

PROCEDURE FOR PRODUCING SULPHUR FOAM

Items to be checked and the operating procedure for producing sulphur foam with the developed sulphur foaming machine are presented as follows:

(1) The oil levels for the engine, air compressor and hydraulic reservoir should be full. SAE 10 is recommended for the hydraulic system and SAE 20 is recommended for the engine and air compressor.

(2) Check the gasoline levels in the fuel tanks.

(3) Check that the hydraulic pump control button (G)* on the control panel is pulled out.

(4) Open the valve on the air storage tank (B) until the engine has been started.

(5) Load the tanks with sulphur, checking that the discharge valves (J) are fully closed.

(6) The engine can now be started by pulling the ignition switch (D) out and engaging the starter button (E) on the control panel.

(7) With the engine started and the tanks loaded, the gasoline heater switch (C) can be turned on at the control panel. The two temperature controllers to the left of the control panel govern the near tank or Tank No. 1. The controllers on the right govern the far tank or Tank No. 2. Open the flue on the appropriate tank to effect a shorter melting time. (With the flue closed, the heated gases are forced out past the discharge line.)

(8) Set the flue temperature controller (700°F full-scale) to 500°F with the control knob. Set the pot temperature controller (400° F full-scale) to 320°F. To avoid damage to the sensing bulb in the melting pot, do not start agitator until the sulphur is completely melted. Once the sulphur is melted, fasten the head securely to the tank, connect the hydraulic lines and engage the hydraulic pump control button to start the agitator. Once the flue temperature has dropped below that of the pot temperature, and the pot temperature has remained constant at

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*Letter refers to callouts in Figure 1.

320°F for 15-20 minutes, the sulphur is at reaction temperature. At this point, shut off the heater.

(9) Disengage the hydraulic pump control button and remove the agitator head. Add all of the styrene and two parts of the P_2S_5. Replace the agitator head and fasten securely. Put the tank under 40 psig and a 'tate for 1 hour.

(10) After 1 hour, reduce the pressure on the tank by opening the air relief valve at the top of the tank, shut off agitator and remove the head. Add the remaining one part P_2S_5, the talc, $CaCO_3$ and H_3PO_4. Replace the head, close the air relief valve and turn on the heaters to heat the discharge line.

(11) Agitate the mixture for 10 minutes, checking from time to time to see that the discharge line has opened. Once the discharge line is open, the heaters can be turned off.

(12) Without having adjusted the air regulator, the pressure gauge will indicate approximately 80 psi. This pressure increase is due to the pressure exerted by the decomposition of the acid-carbonate.

(13) The discharge valve can be opened and the foam poured into place. For a constant foam flow, it is best to have the agitator stopped. From time to time the agitator should be run for a few seconds. Also, the heaters should be turned on intermittently to insure an open discharge line. Once the tank is emptied, let the air blow through the discharge line to clear it. When the line is cleared, close the discharge valve and open the air relief valve and air storage tank valve.

(14) To shut off the engine, check that the hydraulic pump control button is pulled out and the heaters are off. Then push in the ignition switch which turns off the engine.

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13 ABSTRACT

Work performed under this contract represents the first practical development of a process and equipment for producing large quantities of rigid sulphur foam. While the sulphur foam formulations and the resultant properties of the foams have not been fully optimized, this new foam material represents a significant advance in the field of foam technology and has applications that measurably exceed the use intended herein. It was demonstrated that foamed sulphur can indeed be produced on a continuous basis at a remote field site by a process and with equipment that is simple and capable of being scaled up to any size. The foam prepared using the procedures and equipment developed herein has mechanical properties comparable to that produced on a batch basis in the preceding laboratory study--namely a density of 27 lb/ft^3, a compressive strength of 130 psi, a constant strain rate deformation to 65-70 percent, and a low water absorption. Since the cost of the raw materials for these sulphur foams is less than two cents per pound, a foam with a density of 27 lb/ft^3 can be produced for only $0.54 per cubic foot. Conventional foams with similar physical properties that can be produced at a remote field site are the rigid urethane foams. The comparative cost of a urethane foam having a compressive strength of 125 psi is approximately $3.00 per cubic foot. During the week of 17 January 1965, the feasibility of producing foamed sulphur in the field was demonstrated for the Air Force at Kirtland Air Force Base, Albuquerque, New Mexico, by filling a 30-cubic foot annular space with rigid foamed sulphur.

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