DOWNEY PLANT

RESEARCH AND ENGINEERING DIVISION

AGENT PROTOTYPE MUNITION DISSEMINATION SYSTEMS

A Report On

Contract DA-18-035-AMC-117(A)

To

U.S. Army Chemical Center
Edgewood Arsenal, Maryland

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AEROJET GENERAL

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AGENT PROTOTYPE MUNITION DISSEMINATION SYSTEMS

Progress Report for March through May 1964

0843-01(01)QP

Prepared Under

U.S. Army Chemical Center

Contract DA-18-035-AMC-117(A)

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1. INTRODUCTION

This first quarterly progress report, covering the period from March through May 1964, is submitted in partial fulfillment of Contract DA-18-035-AMC117(A).

The objective of this program is to experimentally evaluate chemical agent dissemination techniques and concepts as applied to prototype munition systems, establish performance data and the technical feasibility thereof, and provide optimized system design criteria for agent utilization.

2. SUMMARY

The work performed to date under the contract includes efforts in the following areas:

a. Monitoring of the design of test facility which is being designed and built with corporate funds for use in performance of the contract.

b. Studies of intimate mixes of high explosives and agents.

c. Production of small spherical particles of solid agents.

The design of the facility is approximately 95% completed and preparations are being made to obtain contractors' bids for construction costs. The facility is scheduled for completion by the end of September, with an additional 30 days following that date to be used for chamber calibration and checkout. Thus far, the schedule outlined for the design, construction, and checkout of the facility is being adhered to.

Intimate mixtures of high explosives and cinnamic acid (used as a simulant for agent CS) have been prepared and tested to determine the optimum mixture ratios. These tests have been completed and the results indicate that agent-explosive ratios of approximately 1:1 produce the best agent recoveries. Mixtures of RDX and agent CS will be prepared in agent-explosive ratios of 1:1 and 3:2 (by weight) and will be shipped to CRDL for testing.

Experiments to produce small spherical particles (10 μ or less) of solid agent have been conducted and the results show that molten CS can be atomized in a fast-moving cold-gas stream to create spherical particles. The spheres created thus far have been larger than the desired size and the individual spheres show growths of CS-crystal whiskers on their surfaces. It is expected that the desired sizes can be achieved by improving the atomizing apparatus, and steps are being taken to experimentally investigate the controlling parameters affecting the crystal-whisker growth.

3. PROGRESS DURING REPORT PERIOD

3.1 FACILITY DESIGN

Immediately following contract go-ahead, the design of the Munition Evaluation Laboratory (toxic facility) was undertaken by the Aerojet-Downey Plant Engineering Department with design requirements being supplied by project personnel.

On 16 March 1964, the responsibility for completion of the design of the facility was transferred to AETRON (the Aerojet division specializing in architect engineering/management services).

Shortly after undertaking the design of the facility, AETRON outlined a schedule for completion of the facility, Figure 1. It is estimated that approximately 30 days, following the completion date of September 28, will be required for facility check out and chamber calibration. This will result in an operable facility by the end of October.

AETRON has completed the design and drawings of the 20- by 20-ft test chamber and requests for bids have been sent to several tank fabricators in order that preprocurement of the test chamber can be made. These bids are scheduled for opening on 11 June 1964, and contract award will be made shortly thereafter.

On May 31, AETRON estimated that the remainder of the facility design was 95% complete and that requests for bids for construction of the facility are scheduled to be mailed on or about 8 June 1964. At this report period the schedule shown in Figure 1 is being adhered to.

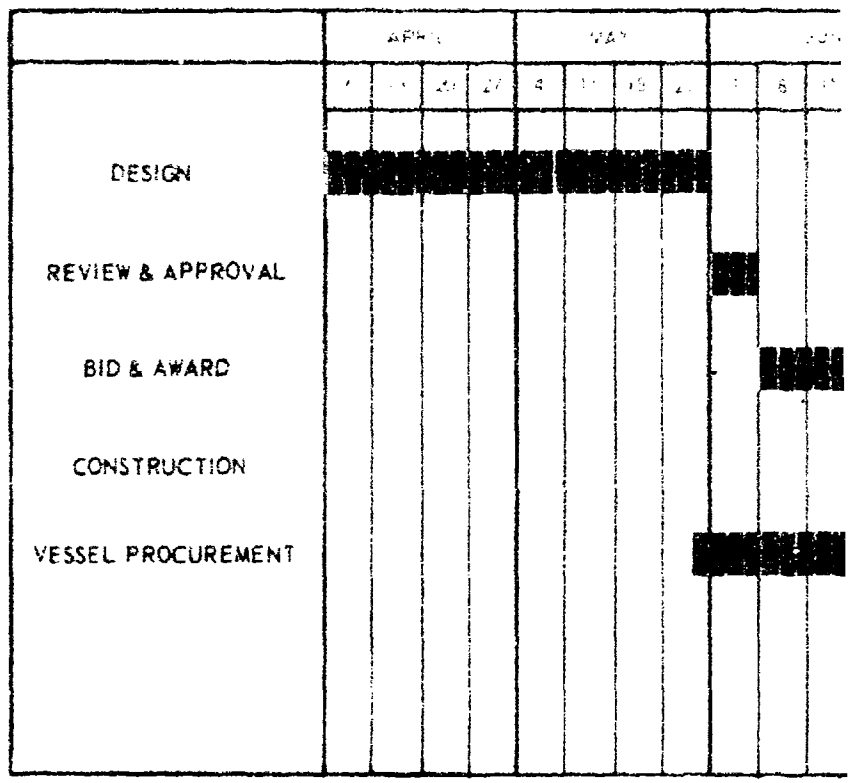
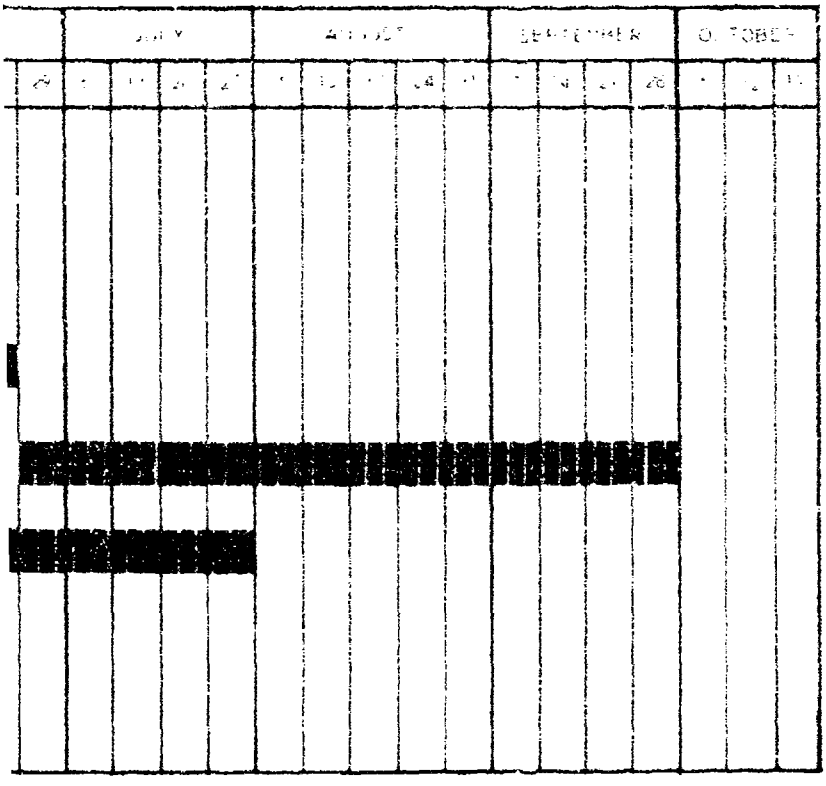


Figure 1. Planning Schedule for



nition Evaluation Laboratory.

A photograph of a model of the facility, made from the latest design drawings, is shown in Figure 2. The existing Chemical Laboratory has been incorporated into the facility design to provide more efficient facility usage and to provide concentration of laboratory personnel in one location. The addition (labeled "High Energy Laboratory" in Figure 2) will be utilized to support work not connected with this contract.

No contract funds are being expended on the design and construction of the toxic facility other than the time required of project personnel to provide design input and to monitor the progress of the facility.

3.2 INTIMATE MIX STUDIES

The purpose of this study was to produce intimate mixtures of high explosives and agent CS in ratios which would contain the maximum recoverable quantity of CS, while maintaining a detonable mixture.

Transcinnamic acid (CA) was used as a simulant for CS in the first groups of mixtures in order to avoid the more obvious handling problems which would be encountered through the use of CS.

The similarity between the various properties of the two materials is shown below:

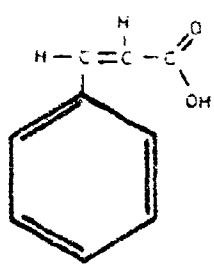
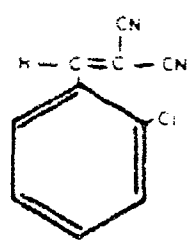
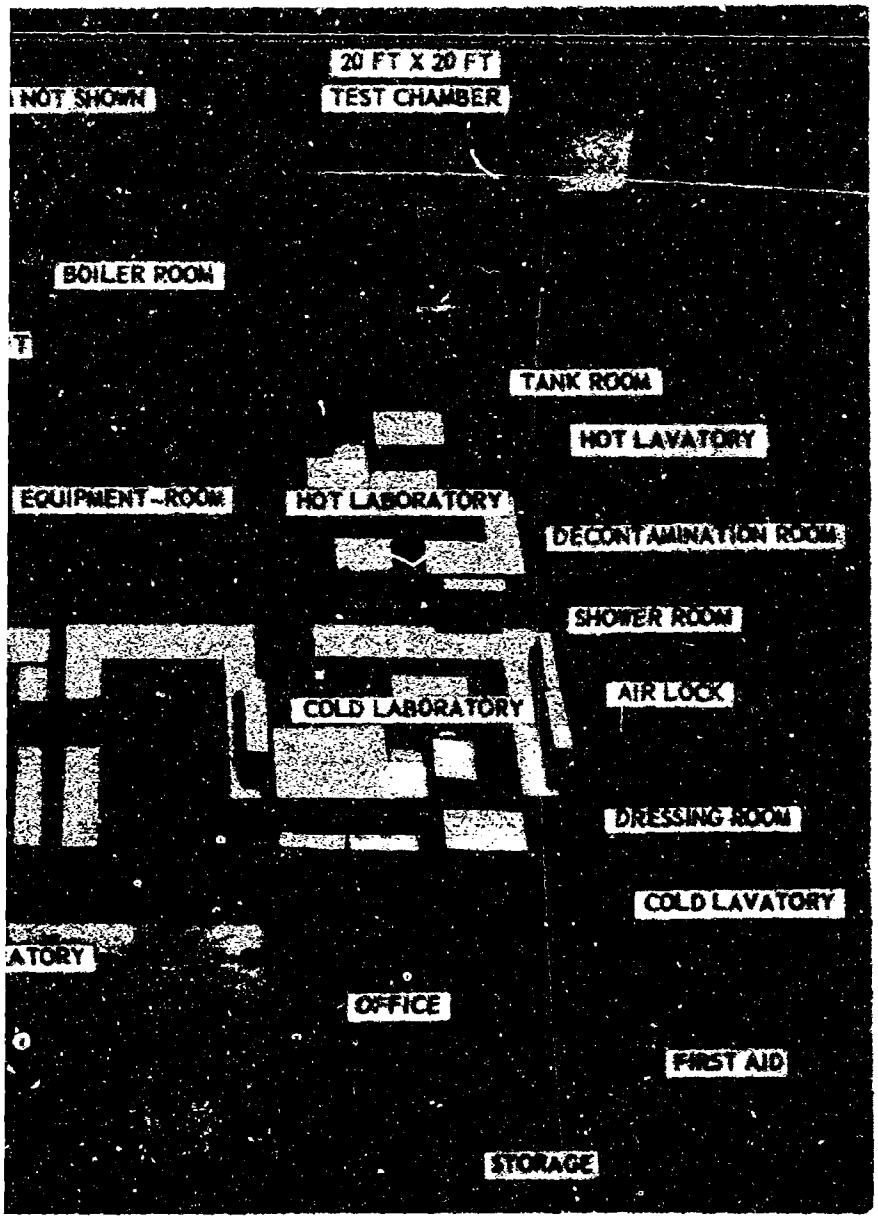
	Cinnamic Acid	CS
Formula		
Boiling Point	300°C	310 - 315°C
Melting Point	133°C	93 - 95°C
Density	1.25 gm/cc	Not available
Solubility	Slight in H_2O	Insol. in H_2O

Figure 2. Model



of Munition Evaluation Laboratory.

3.2.1 Mixture 1

Cinnamic acid and explosive Composition B were melted together in various proportions and cast in glass tubes, 1 in. in diameter by 6 in. long, for use in determining detonation velocities by means of streak camera techniques.

Drop sensitivity data (by the modified Bureau of Mines method) on the various proportions of HE/simulant are shown in Table 1, along with sensitivity data on RDX and Composition B (included for reference purposes).

Table 1. Drop Sensitivity Values for Mixture I Castings.

Material	Sample Size (mg)	50% Point (in.)
RDX	10	14.25
Composition B	10	21.10
Mixture Ratio (HE/Simulant, by weight)		
1:1	10	20.10
2:3	25	No Detonation
3:7	25	No Detonation
1:4	25	No Detonation
1:9	25	No Detonation

Detonation tests were run on 3/4-in. diameter by 1-1/2-in. long cylinders of the 1:1 mixture of Composition B and Cinnamic acid. A number 6 detonator failed to detonate the mixture; however, a number 6 detonator in combination with a 1/4 by 1/4-in. tetryl booster, or a DuPont number 83 detonator alone, caused detonation.

Differential thermal analysis records of the 1:1, 2:3, and 3:7 ratio mixture castings indicate detonation temperatures ranging between 187° and 197°C.

Casting of the intimate mixes was abandoned because of the difficulty in preventing the inclusion of voids in the mix during the casting process.

3.2.2 Mixture II

Crystalline explosive RDX with 2% graphite and cinnamic acid were also mixed in the same proportions listed in Table 1. These were pressed into 3/4-in.-diameter by 6-in.-long cylinders for detonation velocity tests. Streak-camera shots of the detonation of these mixtures showed that the detonation velocities were generally in the range expected, however, this work was discontinued because of lack of reproducibility of detonation velocities between identical samples because of mixture inhomogenities.

High-speed motion pictures taken of the detonation of mixes ranging from 50% CA - 50% RDX through 80% CA - 20% RDX showed that the 50% CA - 50% RDX mix appeared to produce the best results, based on cloud size and geometry. Some of the higher agent-explosive mix ratios appeared to flash after detonation.

Drop sensitivity tests resulted in no detonations for any of the mixture ratios. Various samples of these mixtures were also detonated in the large aerosol chamber at the Chino Hills Laboratory in order to obtain samples of the resulting aerosol cloud. Material balances were made by collecting fallout samples at the floor of the chamber on trays which had a total area of 81 ft^2. This is equivalent to 0.223 times the area of the tank floor. Assays of the fallout samples were made spectrophotometrically by comparing the optical absorbance, at 271 millimicrons wavelength, of an ethanolic (95%) solution of the sample with that of standard solutions of cinnamic acid in ethanol. Figure 3 illustrates the absorption spectrum of cinnamic acid in ethanol.

Intact pieces of the undetonated mixture were collected separately and weighted in the cases where detonation was incomplete. Recoveries from the various mixtures are shown in Table 2.

These experiments indicated that organic molecules, similar in structure to CS, would survive the conditions encountered in detonation when intimately mixed with the explosive RDX. The data in Table 2 shows that the best recoveries and most complete detonations were obtained with the 1:1 and 3:2 agent to explosive mass ratios. Dissemination efficiency has not as yet been determined for the cinnamic acid because the relatively large difference between the initial size range of the cinnamic acid (MMD ca. 130 μ) and the CS which will be used in future experiments (MMD < 5 μ) make efficiency comparisons questionable.

As a result of these preliminary experiments, intimate mixtures of RDX and CS will be prepared in agent-to-explosive ratios of 1:1 and 3:2, and shipped to CRDL for testing.

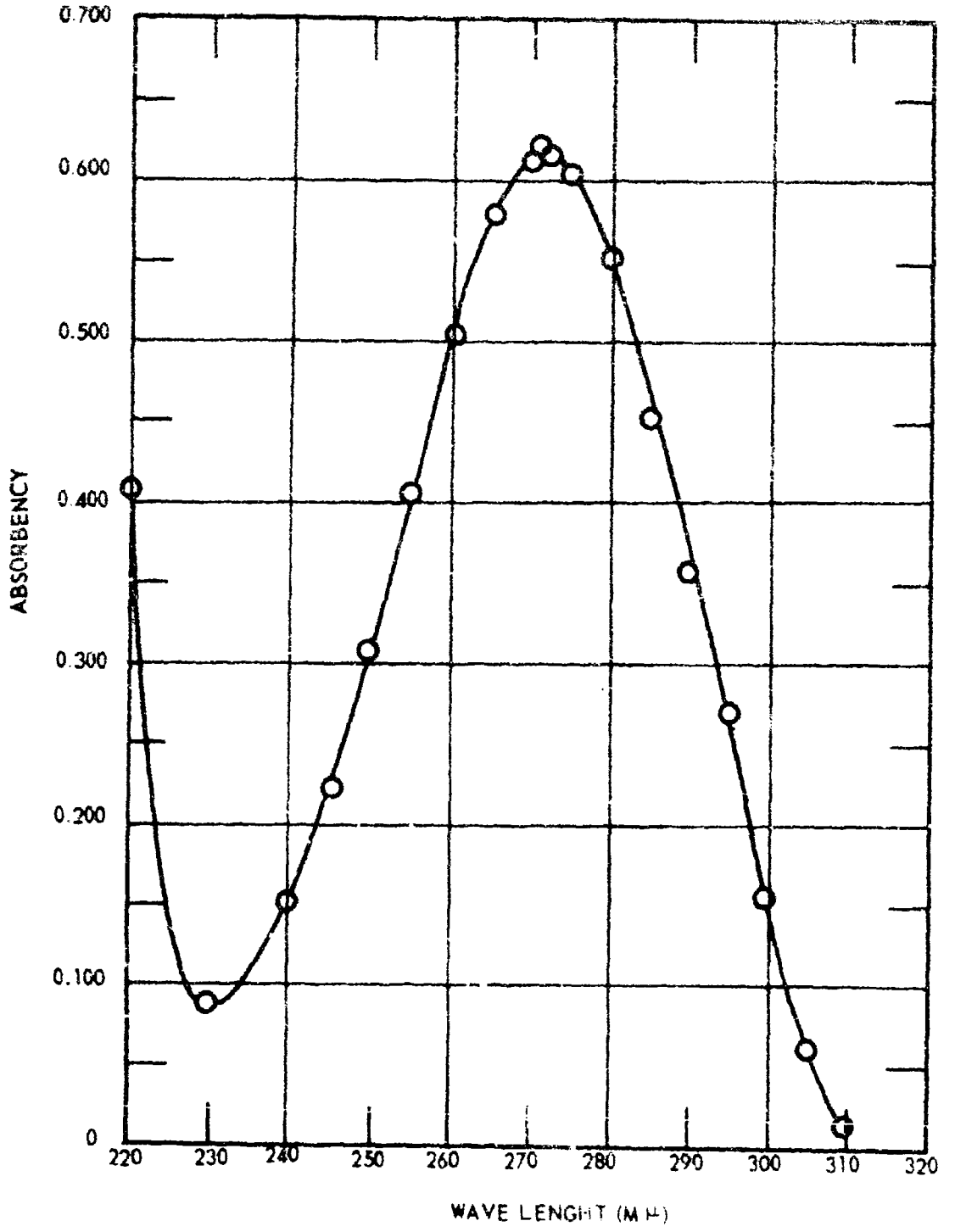


Figure 3. UV Spectrum, Cinnamic Acid.

Table 2. Recovery of Cinnamic Acid fro

	A	B	A/B	C	D
Shot No.	Material in Mixture		A/B Ratio	CA Recovered From	
	CA (gm)	Expl. RDX (gm)		Detonated Material (gm)	Undetonated Fragments (gm)
1	10.74	10.74	1:1	9.07	--
2	12.18	8.12	3:1	7.83	2.02
3	16.24	6.96	7:3	5.91	5.76
4	17.60	4.40	4:1	7.07	7.43

Intimate Mix Detonations.

$\frac{C}{A-D}$	$\frac{C+D}{A}$	$\frac{C}{A}$
% Recovery of CA Initially in Mixture Which Detonated	% CA in Original Mixture Recovered From Det. and Undet. Mixture	% CA in Original Mixture Found in Detonation Products
84.45	84.45	84.45
77.07	80.87	64.40
56.61	71.86	36.40
67.98	82.39	40.20

3.3 PREPARATION OF MICROSPHERES OF ORGANIC SOLIDS

The purpose of this work is to produce spherical particles of solid agents with diameters of 10 μ or smaller. Before starting the experimental work on this portion of the program, a brief literature survey was made to study the techniques previously explored for producing spherical particles. The most promising approach appeared to be that of atomizing a molten material (assuming thermal stability) to the desired size range, followed by rapid cooling of the particles to cause solidification before coalescence or bonding of particles could occur.

Several rough experiments were conducted using two techniques. The first technique employed a hot liquid carrier medium at a temperature sufficient to melt the organic material to be spheroidized. The hot organic liquid was dispersed in the carrier medium using a blender operated at a speed high enough to achieve the desired particle size. The two phase system was cooled rapidly by pouring the hot dispersion into additional carrier medium which was maintained at a low temperature.

Paraffin was used as the organic material in one test because its low melting point would allow the use of hot water as the carrier medium. A wetting agent (Aerosol O. T.) was also added to the mixture to reduce agglomeration. Observations made with a microscope indicated the majority of particles were spherical in shape and of the order of 5 μ in diameter. Attempts to isolate the particles as a dry powder resulted in excessive agglomeration.

A similar test was conducted using cinnamic acid as the solid (mp 133°C) and mineral oil as the carrier medium. The resultant particles exhibited a great deal of crystal growth because of the apparent solubility in hot mineral oil.

The second technique consisted of atomizing the molten material in the air at ambient temperatures. The first experiments using this procedure were made by atomizing molten paraffin with a paint spray appartus, and collecting the spray in liquid nitrogen. Figure 4 shows the resulting particles after evaporation of the liquid nitrogen. The resulting sphericity and particle size range indicates that this procedure might be used satisfactorily.

Based on the results of the paraffin experiments, a crude device was fabricated to melt the organic material and dispense it from a hypodermic needle into a cold gas stream to produce the desired atomization. Calculations indicated that the experimental parameters could be varied to

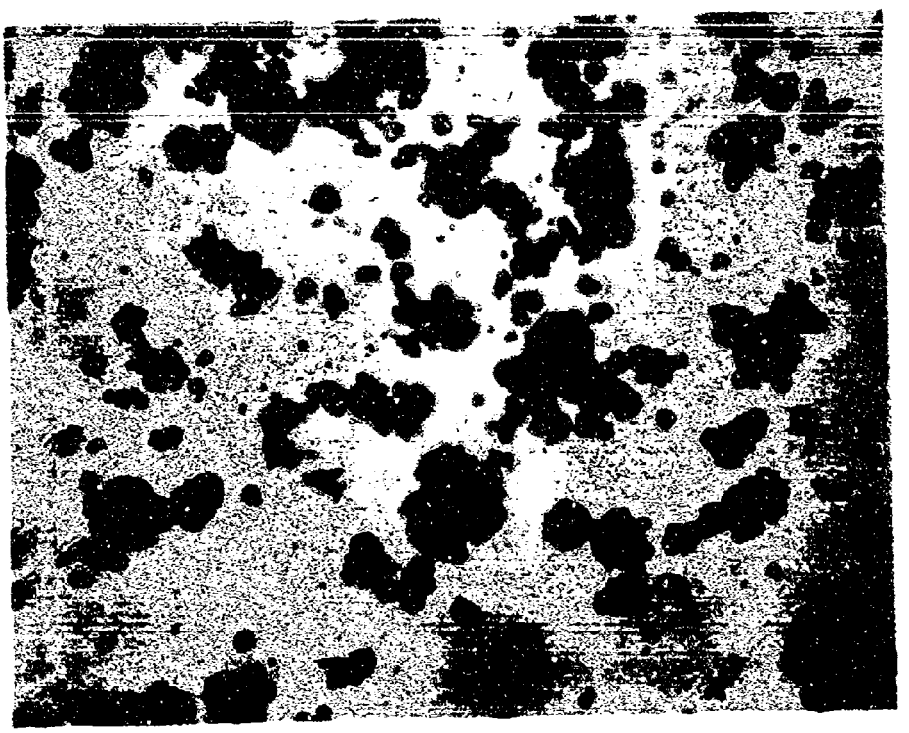


Figure 4. Atomized Paraffin.

assure reasonable success in the production of particles in the desired size range. The gas stream was directed into a tube approximately 2 ft in diameter by 9 ft in length in order to permit the aerosol to settle on microscope slides, placed along the bottom of the inside of the tube.

Cinnamic acid was used in the first tests with the apparatus because it is nonirritating and is similar to agent CS in many of its properties. Figure 5 shows the resultant particles to be generally spherical, however, the appearance of the surface of the spheres is somewhat rough. The spheres, when dispersed in a liquid medium, disintegrated into particles smaller than 2 μ, many of which appeared to have crystalline structure. This suggests that the cinnamic acid crystallized and that the spheres contained an unstable grain structure, possibly stressed. Agent CS was then atomized in the same apparatus under essentially identical conditions.

The resulting particles were generally larger in size than for the cinnamic acid. This was attributed to a relatively strong breeze which caused the molten-liquid filament to enter the gas stream off-center where the air velocities were lower, resulting in less breakup.

Although the particles appeared spherical, they had grown needle-like crystals similar to thorns on a cactus. Figure 6 shows an example of the resulting particles.

From microscopic observation of slides over several hours, no visible additional crystal growth was detected. This was expected because agent CS is reported to have a low vapor pressure (5.6 μ at 65°C). Therefore, it was concluded that the formation of the crystals occurred during the atomization and cooling treatment. This problem may be minimized by either using more rapid cooling systems or additives to prevent crystallization.

From the work conducted thus far, it appears that spherical particles can be produced, in sizes below 10 μ, by atomization of the molten material and close control of the experimental apparatus. Additional rough experiments, oriented in this direction, will be conducted.

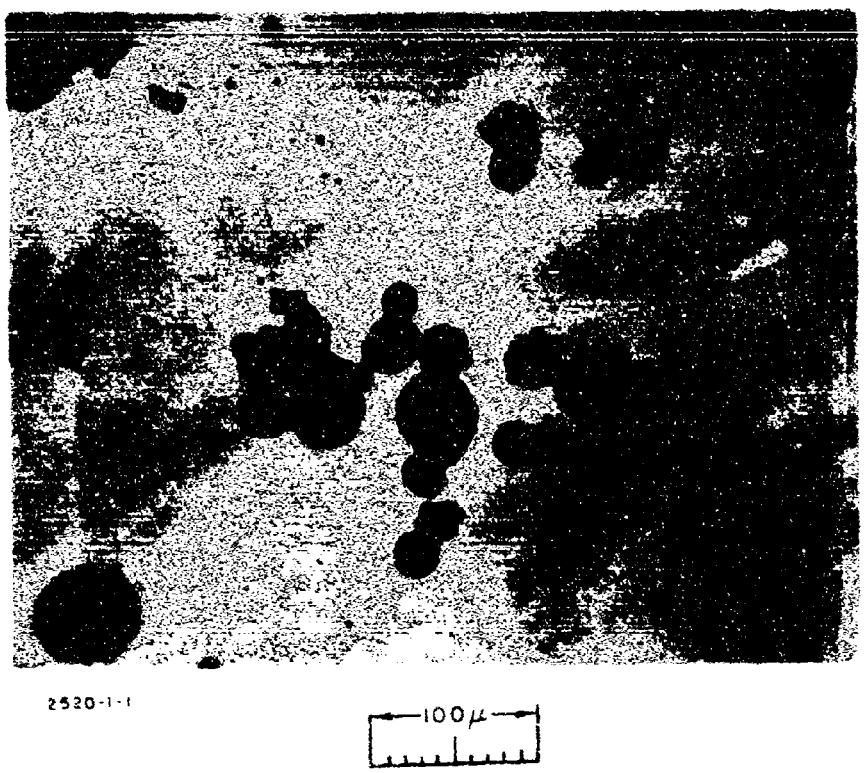


Figure 5. Atomized Cinnamic Acid.

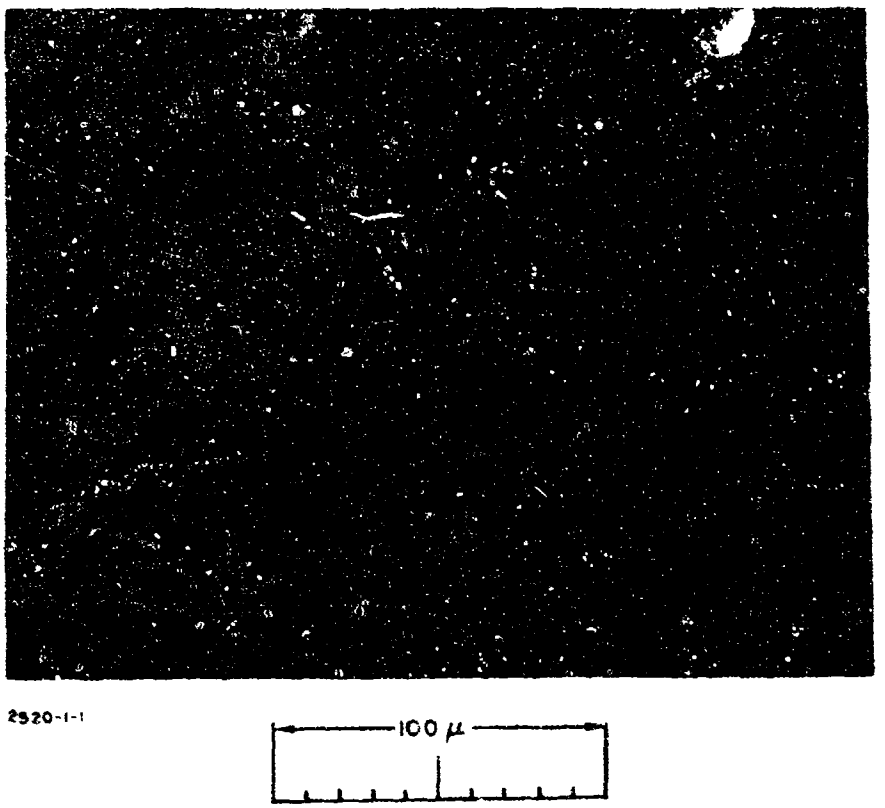


Figure 6. Atomized CS Showing Crystal Whiskers.

4. FUTURE WORK

4.1 MUNITION EVALUATION LABORATORY

Project personnel will continue to monitor the progress on the completion of the design of the facility and the construction work which is expected to begin before July 1.

4.2 INTIMATE MIX STUDIES

Mixtures of RDX and agent CS will be prepared in agent-explosive ratios of 1:1 and 3:2 (by weight), and will be shipped to CRDL for testing.

4.3 PREPARATION OF MICROSPHERES

Experiments will be continued to further reduce the particle size and to learn more about the parameters affecting crystal whisker growth on the surface of the CS particles.