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ULTRASONIC DISPERSION OF PARTICULATE HIGH DENSITY FUELS



ENERGY & MINERALS RESEARCH COMPANY  
964 E. Swedesford Road, P.O. Box 389  
Exton, Pennsylvania 19341

May 1981

Technical Report AFWAL-TR-81-2033  
Final Report for Period September 1979 - September 1980

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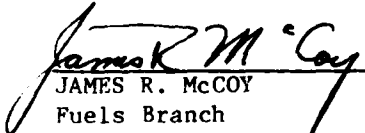
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This technical report has been reviewed and is approved for publication.

  
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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br><br>The high energy-density slurry fuels being developed to increase missile range may present settling and gelation problems in use. This project was performed to demonstrate ultrasonic dispersion techniques, with the goal of deagglomerating to more uniform, smaller particle-size solids distribution and increased storage stability. Batch and continuous process laboratory-type arrays were used at various power levels (nominal 200 to 1000 watts), on carbon and boron/JP-10 slurries of interest to the Air Force. |                                      |                                                                                        |

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✓ Analytical techniques, difficult with the particulate sizes involved (down to the 300-millimicron range), showed some measure of ultrasonic deagglomeration, which increased with increasing ultrasonic energy densities, and complete stability of the 60-day-storage activated samples, in contrast to the non-activated control samples. Extended analysis might better quantify ultrasonic effect, but combustion tests could directly identify performance benefits. ✓

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# FOREWORD

This final report is submitted by the Energy and Minerals Research Company, Exton, Pennsylvania. The work was conducted under Contract F33615-79-C-2090, covering the period September 1979 - September 1980. Program sponsorship and guidance were provided by the Aero Propulsion Laboratory (AFWAL/POSF) Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio under Project 3048, Task 304805 and Work Unit 30480505. Mr James R. McCoy was the Government project engineer.

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## I. INTRODUCTION

In the past decade, high energy-density liquid hydrocarbon fuels (e.g., JP-10) have been developed which have increased the volumetric heating value of missile fuels by as much as 36 percent over the conventional JP-4 and JP-5 fuels. Although other liquid hydrocarbons with increased energy content meet specific ramjet or turbine-powered missile requirements, very little additional range can be realized through the application of these fuels.

The development of a carbon slurry fuel appears to offer the potential of a further 15 percent improvement in available energy per unit volume. Boron slurry fuels offer even greater available energy but may be limited by technical and combustion problems.

In addition to complex rheology, slurry fuels may also pose the problem of settling and gelling. Surfactants, which can be used to break up or disperse the particle network, will only somewhat reduce viscosity and gelling.

Ultrasonic dispersion techniques provide additional mechanisms for energy absorption at preferential sites on the particle. The resulting particles are generally smaller and display a more uniform size distribution. Cavitation within the ultrasonic field may give more thorough and uniform wetting of the particle by the surfactant. It was the purpose of this effort to explore the particle dispersion benefits resulting from proper application of ultrasonic energy.

The investigation was pursued through a program in which selected laboratory-type ultrasonic dispersing arrays were assembled and applied to high density fuel materials of interest to the Air Force. Multiple

analytical techniques were used on the experimentally treated fuel samples to determine ultrasonic effect in comparison to non-activated control samples. The samples were analyzed for particle size distribution, viscosity under low and high shear rates, and settling rates by a number of standard and some less routine analytical techniques.

The program effort demonstrated ultrasonic power transmission to these high density slurries in arrays which would be capable of translation to production equipment, and the analyses performed within the limits of the scope of work showed a measure of ultrasonic beneficiation in regard to deagglomeration and storage stability. It is considered that extensive laboratory analysis might better quantify the effect, but that comparison of the combustion performance of ultrasonically treated and control samples would directly identify the practical advantages.

## II. EXPERIMENTAL WORK

### A. Fuel Preparation, Considerations and Procedures

The high density fuels selected for study were based on mixtures of carbon blacks and boron powders with JP-10 liquid fuel. The formulation primarily studied was a material designated Formula A (whose specific composition and method of preparation carry a Confidential classification), which was comprised of carbon black and a surfactant in JP-10. A boron, surfactant and JP-10 mixture of the same proportions is designated as Formula Z. Additional preparations using "Statex MT<sup>R</sup>" and "Sterling R<sup>R</sup>" carbon blacks, singly and in combination, with reduced surfactant in JP-10 were investigated at various solids loadings.

All formulations were prepared by premixing just prior to the treatment with ultrasonics. The liquid slurry-type mixtures, such as Formula A and Formula Z, were routinely premixed in a Waring blender for twenty minutes. (Formula A was also prepared in a ball mill, using an eight-hour run, but since this was less convenient and had no advantage in reproducibility of the mix, it was discarded.) A few of the mix variations which yielded paste-like consistencies were premixed by hand with a spatula until a visually uniform consistency was obtained.

The preparations were subjected to ultrasonic dispersion immediately after premixing, to ensure mix uniformity at the start of the ultrasonic processing. A portion of the premix was withdrawn for comparative measurements of the slurry properties prior to dispersion, with analogous measurements of the samples which received ultrasonic dispersion treatment.

The alternate mix formulations contained varying amounts of two carbon blacks, "Statex MT<sup>R</sup>" and "Sterling R<sup>R</sup>", and blends of the two, with reduced percentages of surfactant. The difference between these carbon blacks is in their particle size distribution; the median particle size

of "Sterling R<sup>R</sup>" is 75 millimicrons, compared to 300 millimicrons for "Statex MT<sup>R</sup>". The finer particle material, with its consequently greater surface area, offers the prospect of more rapid and complete combustion as a fuel component; however, it has the negative impact of increasing viscosity of the slurry. Ultrasonic treatment was applied to these mixes with the objective of altering surfactant distribution within the fine-particle slurries to reduce viscosity.

Fuel slurries with reduced surfactant levels were prepared and tested with similar objectives. The surfactant is reported by Exxon to have a possibly negative effect on fuel combustion; however, lower contents give an increase in viscosity. Again, a possible redistribution by ultrasonic energy application of surfactant within the slurry could alter viscosity and thus permit formulating the fuel with lower concentrations of surfactant.

With these considerations in mind, the carbon-black fuel slurry formulations tested include Formula A, a "Statex MT<sup>R</sup>" concentration of 69 percent, and "Sterling R<sup>R</sup>" concentrations of 48.6, 50 and 60 percent. Blends of "Sterling R<sup>R</sup>" and "Statex MT<sup>R</sup>" at 30/30 percent and 28.4/28.4 percent also were tested. Surfactant loadings reduced to 25 and 50 percent of those in Formula A were evaluated.

#### B. Ultrasonic Dispersion Arrays

Three different types of ultrasonic devices were assembled to compare activation techniques. A flow-through device for continuous processing and two batch treatment units were used to treat the high density fuel slurries.

The flow-through disperser (Figure 1) consists of an ultrasonically activated coupling arrangement in a tubular enclosure designed so that the slurry pumped into the unit is necessarily exposed to the region of

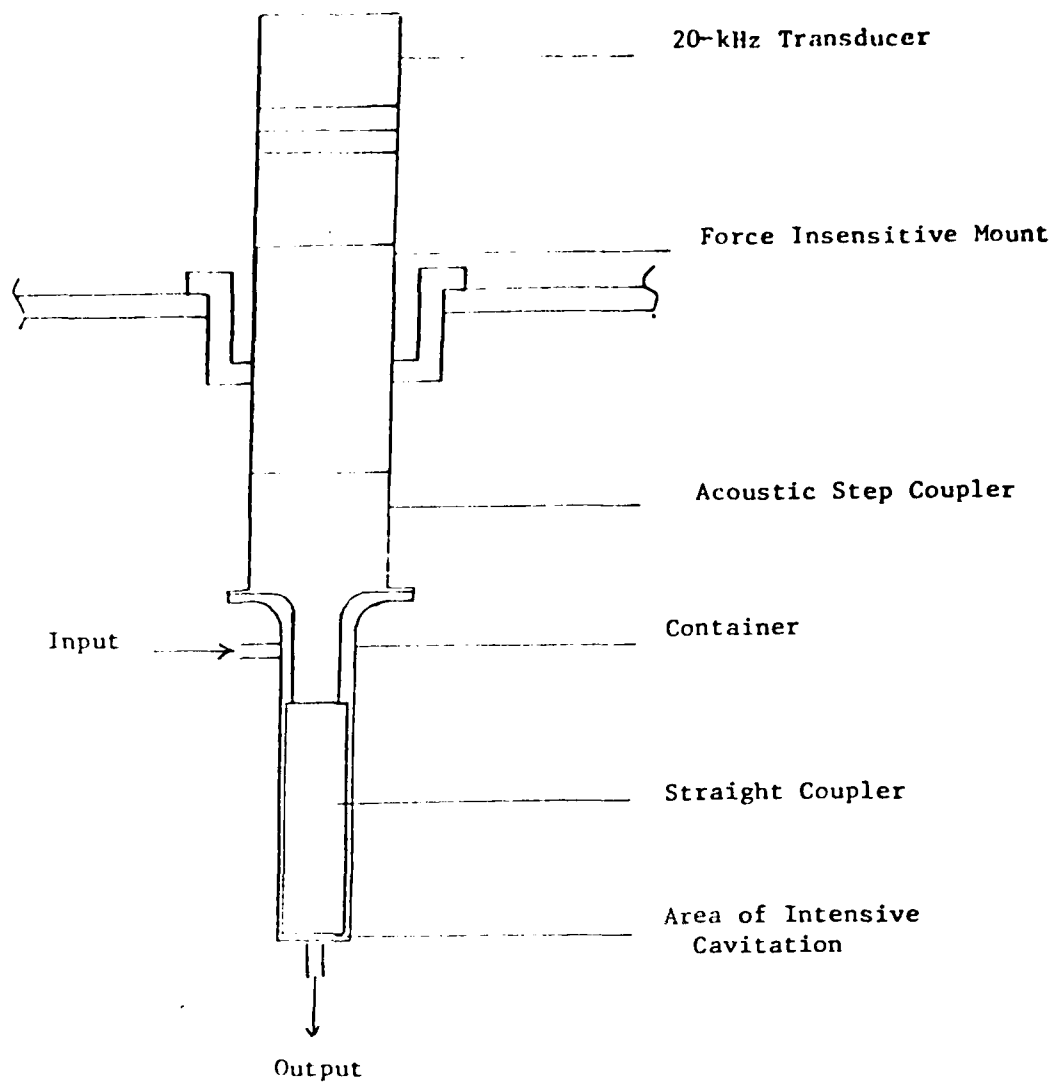


Figure 1

CONTINUOUS FLOW UNIT FOR ULTRASONIC DISPERSION

most intense ultrasonic energy, which is transmitted from the piezoelectric transducer to the slurry by a series of force-insensitive mounts and acoustic wave-guide couplers.

A stream of mixture is pumped onto the center of the ultrasonically activated face and directed through the narrow (1 mm) gap of this design to assure high power intensity per unit volume of mixture and per unit area of treatment surface. This approach provides a limited time exposure to a very intense ultrasonic field. Recirculation past the face can increase exposure time in this laboratory array.

The ultrasonic cup unit (Figure 2) consists of a metal vessel coupled to a force-insensitive mount and a piezoelectric transducer. The material to be treated is placed in the cup, which is designed for efficient transmission of ultrasonic energy. The extent of treatment in this unit can be controlled by ultrasonic power input and duration of exposure.

The array is designed to couple energy directly from the transducer into mixture contained in a cup attached to the driving end of the transducer. This approach directly couples energy, and while a batch treatment, can be adapted to a continuous processing mode. Stirring is used to move all elements of volume past the more active region at the bottom of the cup. Two designs of this configuration were tested, a low volume "shallow" cup and a higher volume "deep" cup.

The commercial batch-treatment unit, a Westinghouse "Cylsonic"<sup>R</sup> device, is shown in Figure 3. (It is normally used as part of a continuous processing array and therefore could be used for continuous treatment.) This unit is a right cylinder seven inches in length with a 2-7/8 inch inside diameter, with 60 magnetostrictive transducers mounted radially on the outside of the cylinder to focus ultrasonic energy on cylinder contents, with energy concentrated by the opposing transducer stacks. The unit

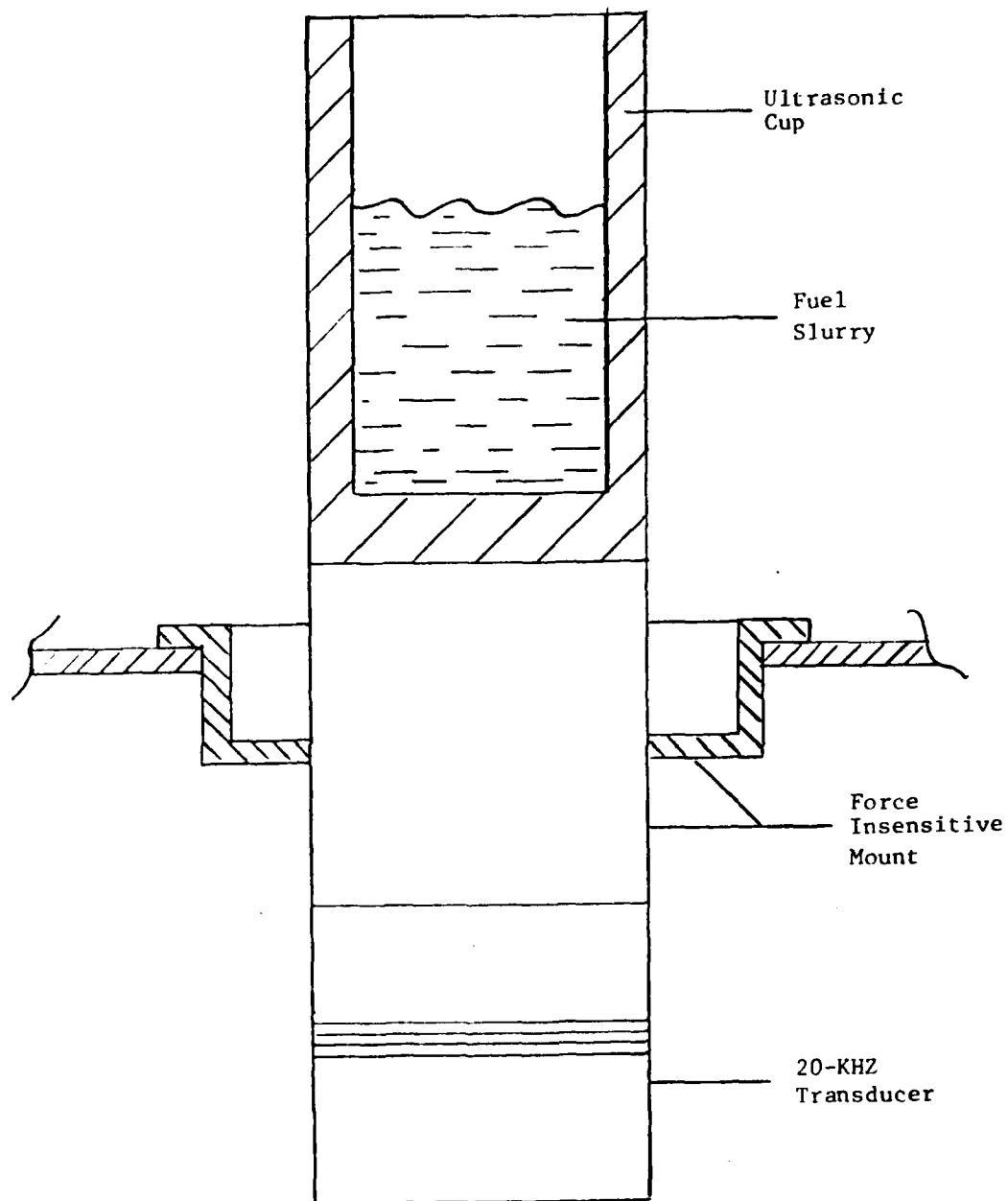


Figure 2

ULTRASONICALLY ACTIVATED CUP UNIT



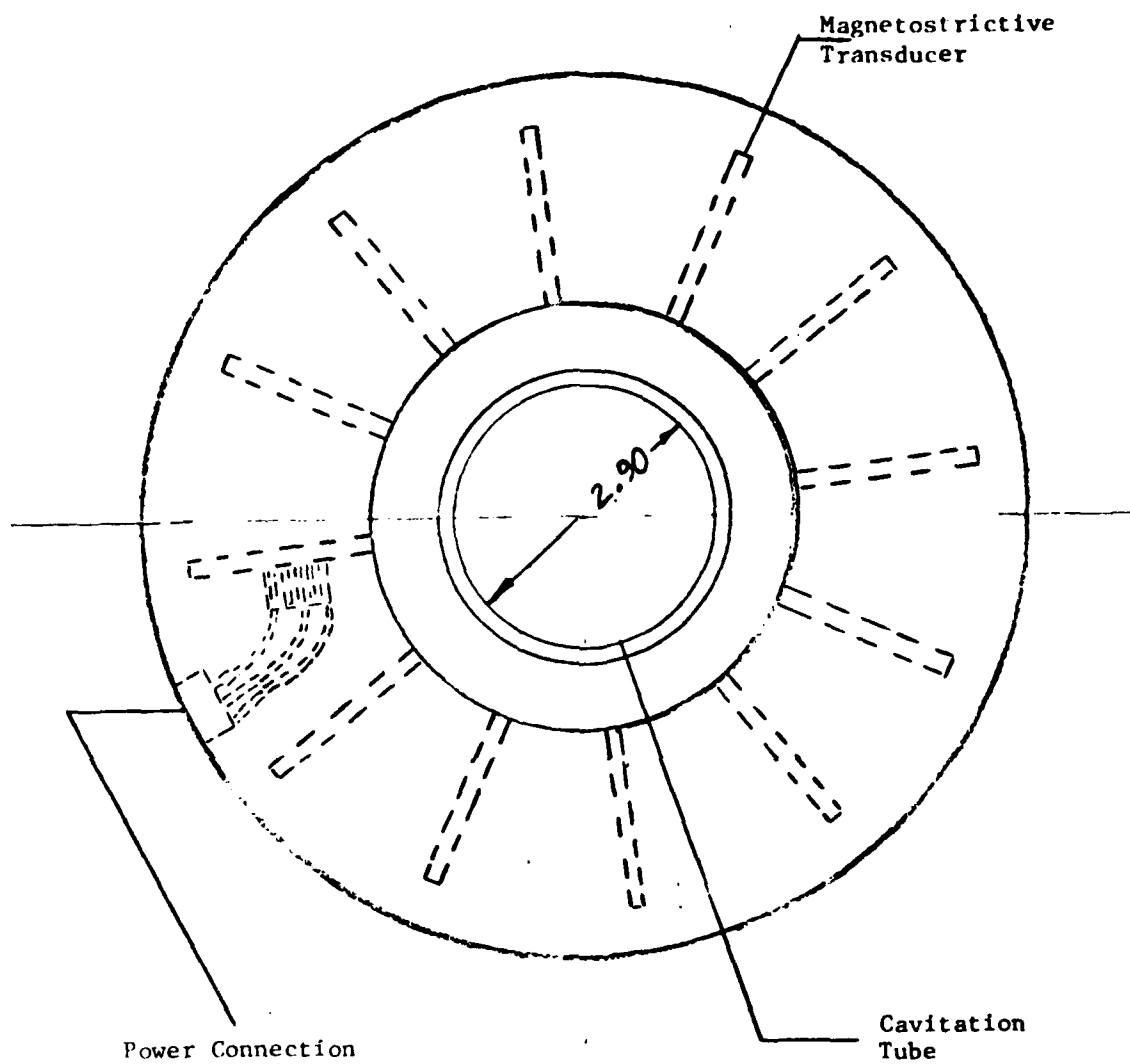


Figure 3

WESTINGHOUSE RADIAL UNIT

A. (Top View)

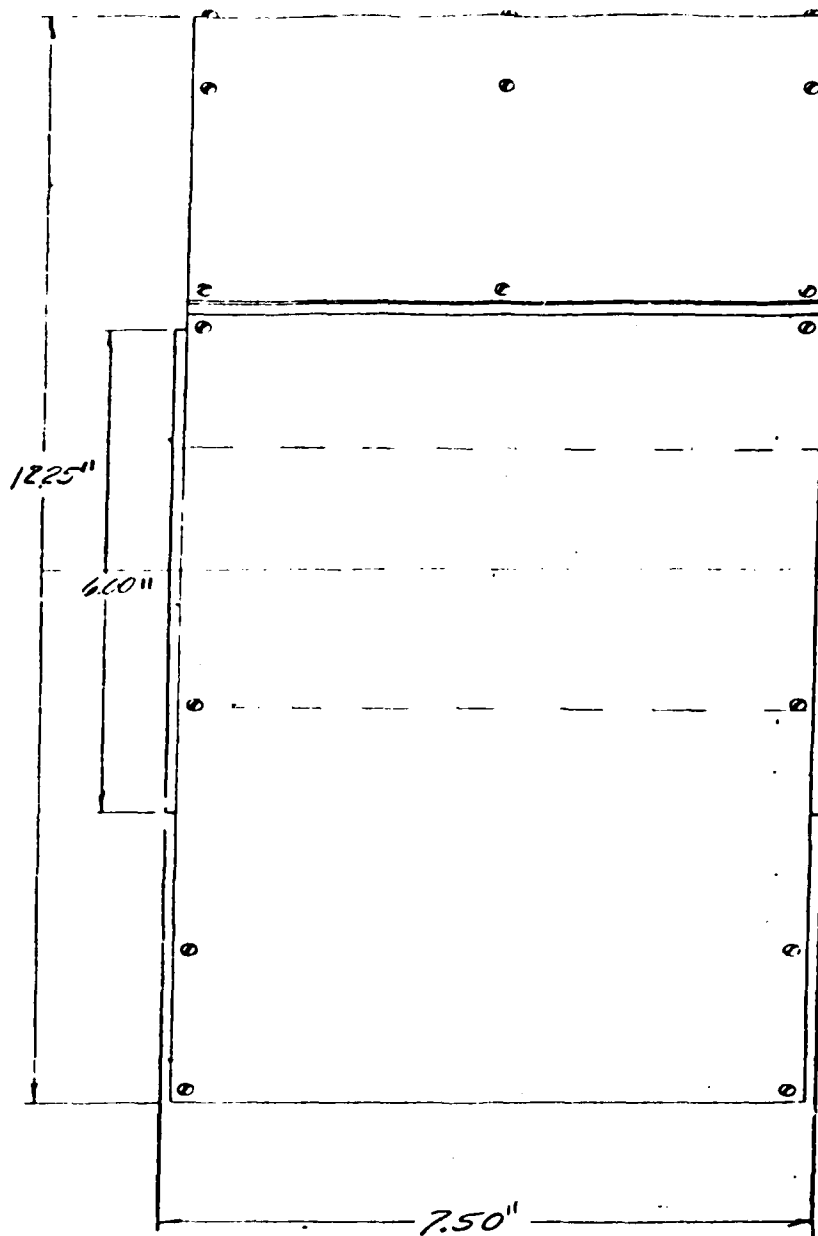


Figure 3 (Cont.)

WESTINGHOUSE UNIT

(B. Side View)

operates at a fixed input energy (1000 watts nominal\*) and treatment level is therefore exclusively controlled by exposure time.

The Westinghouse apparatus provides a uniform but lower intensity (limited by transducer power-handling capability) bulk excitation of the mixture. Neglecting end effects, the various transducers alternately compress and expand the entire contents of the cylinder. The apparatus is specifically designed for ultrasonic cleaning, for which it is reportedly very effective. While versatile in application mode, i.e., batch or flow-through, the device is somewhat power limited.

The fuel slurry mixes were treated in the various ultrasonic units at nominal power levels from 200 to 1100 watts. The continuous-flow unit was operated at levels from 200 watts to 1000 watts, primarily on the Formula A mix. The ultrasonic cup was operated with 200 to 1100 watts of power, with the unit used for dispersion with all of the fuel mixes investigated. The relatively simple construction of the cup permitted ultrasonic dispersion of thick paste-like premixes as well as Formula A. The Westinghouse unit, with the fixed power input of 1000 watts, was used to disperse Formula A.

Table I compares some of the power parameters of these three units, including input levels, area power density, and volume power density, which are functions of the geometry and size of the activating units.

Thus the continuous-flow unit provides the most intense treatment, the ultrasonic cup the next most intense, and the Westinghouse unit the lowest. The values used during the program are shown in box enclosures.

\*Magnetostrictive transducers are only about one-third as efficient as piezoelectric materials in electroacoustic conversion.

TABLE I

CALCULATED POWER INTENSITIES

| <u>Nominal<br/>Input<br/>Power</u> | <u>Ultrasonic<br/>Cup</u>          | <u>Westinghouse<br/>Radial<br/>Unit</u> | <u>Flow-Through<br/>Apparatus</u>      |
|------------------------------------|------------------------------------|-----------------------------------------|----------------------------------------|
| 200 watts                          | 9.8 w/cm <sup>2</sup><br>1.9 w/cm  |                                         | 39.5 w/cm <sup>2</sup><br>395.0 w/cm   |
| 300 watts                          | 14.8 w/cm <sup>2</sup><br>2.9 w/cm |                                         | 59.2 w/cm <sup>2</sup><br>592.0 w/cm   |
| 400 watts                          | 19.7 w/cm <sup>2</sup><br>3.9 w/cm |                                         | 79.0 w/cm <sup>2</sup><br>790.0 w/cm   |
| 1000 watts                         | 49.3 w/cm <sup>2</sup><br>9.7 w/cm | 0.8 w/cm <sup>2</sup> *<br>0.4 w/cm     | 197.5 w/cm <sup>2</sup><br>1975.0 w/cm |

\*Approximately calculated to correct for magnetostrictive transducers, which operate at about one-third the transmission efficiency of the electrostrictive types used in the other arrays, i.e., 1000 watts input yields only 350 watts applied to the mixture. The radial unit is a single non-adjustable power-level device. Theoretically, the highest calculated intensity is available with the flow-through apparatus, with the cup units about 1/3 to 1/4 as high. As noted, the Westinghouse unit is disappointingly low.

### C. Analytical Methods

The analytical methods employed on the high-density fuel mixes were directed at determining the degree of solid particle deagglomeration and mix stability. Conventional instrumentation for measuring particle size were not adaptable for use in this work because of the high concentration of particles in suspension and the very low median particle sizes (75-300 millimicrons) of the materials examined. Most of the methods used indicate particle size indirectly through the measurement of related properties of the slurries, with some assumptions of the relationship of particle size and the property being measured, and therefore a number of these methods were used to verify results. The analytical techniques included viscometry, drawdown gauging, conductance, accelerated sedimentation, micropore filtration, scanning electron microscopy, pycnometry, and static sedimentation. Specific procedures and data obtained are given in the following section of the report.

### III. ANALYTICAL TECHNIQUES AND RESULTS

#### A.. Viscometry

Viscosity measurement was one of the few which could be made directly on the fuel slurry. Theoretically, a change in the size of particles in a solid-liquid slurry will have an impact on viscosity, and additionally, viscosity is an important consideration in slurry pumping behavior. This prompted taking viscometry of all but the thickest mixes. Thus, using a Haake "Rotovisco<sup>R</sup>" viscometer, viscosity measurements were made on all Formula A mixes, all of the mixes with "Statex<sup>R</sup>" carbon and surfactant loading variations, all mix variations with "Sterling R<sup>R</sup>" carbon except loadings at 60 percent and above, and all Formula Z and other boron mixes. The viscosities of Formula A mixes both before and after ultrasonic treatment with the continuous-flow unit, the commercial unit and the ultrasonic cup are plotted in Figures 4 through 6. Viscometry readings on this instrument were similar both before and after ultrasonic dispersion. At the high shear rates, viscosity measured 100 to 110 centipoise; at low shear rates, it tended to increase and have wider variations. The graph of Figure 7 depicts typical measurement technique. Twelve separate readings were taken on the mix and were used to calculate a mean value and the standard deviation. The two sigma limits on the figure show the typical variation between low and high shear readings. The differences between ultrasonically treated and untreated mixes are within the expected variations in the measurement technique.

Viscosity measurements on mixes with carbon and surfactant loading variations are given in Tables II through IV. All of these mixes were treated in the ultrasonic cup. In Table II the increased loading of "Statex-MT<sup>R</sup>" carbon shows significantly increased viscosities compared to Formula A. Reduced surfactant levels show increasing viscosity at low shear rates ( $8.5$  to  $25 \text{ sec}^{-1}$ ) but readings similar to Formula A at

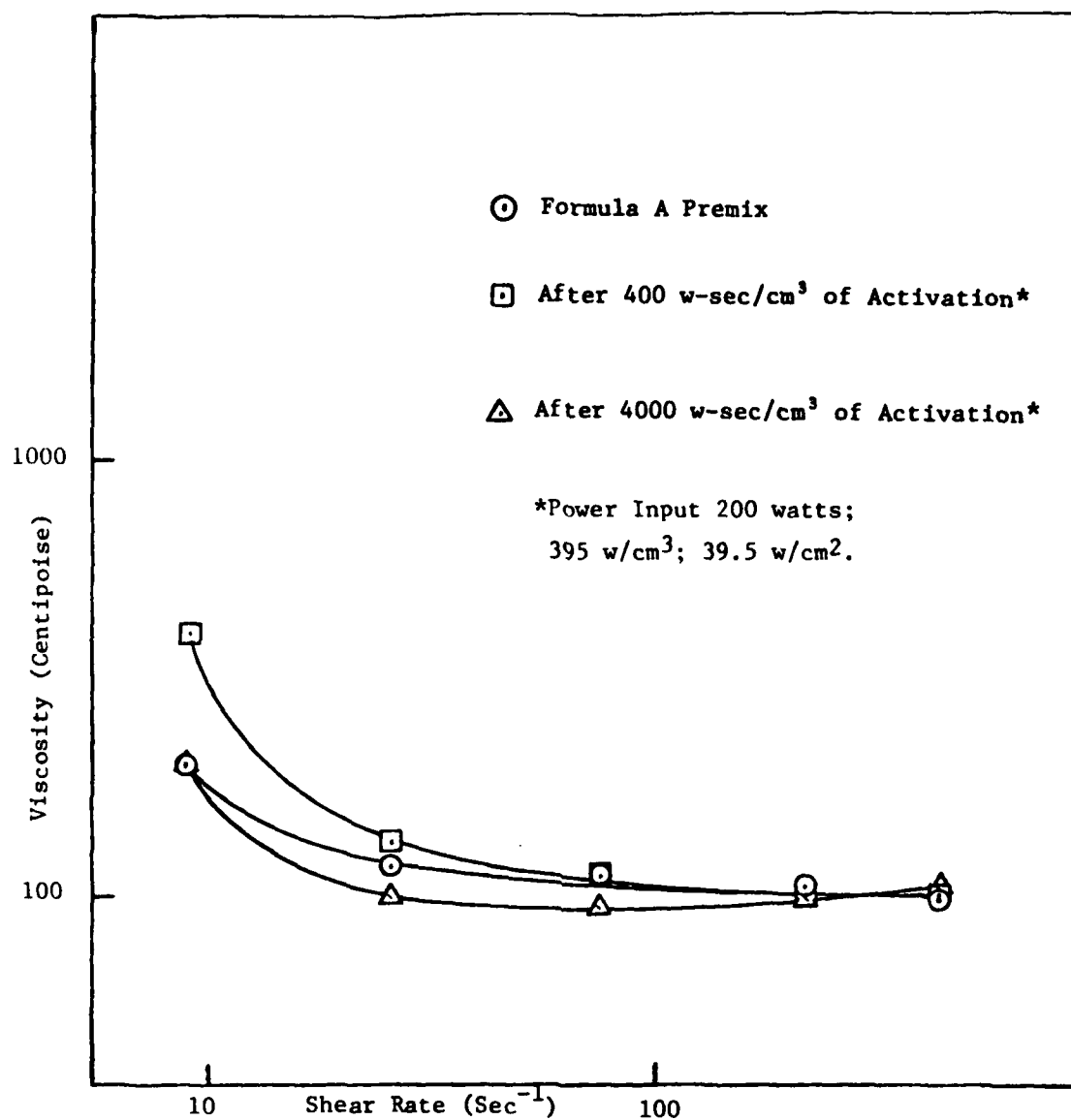


Figure 4

VISCOSITY EFFECT OF ULTRASONIC TREATMENT OF  
CARBON FUEL DISPERSION WITH THE CONTINUOUS FLOW UNIT

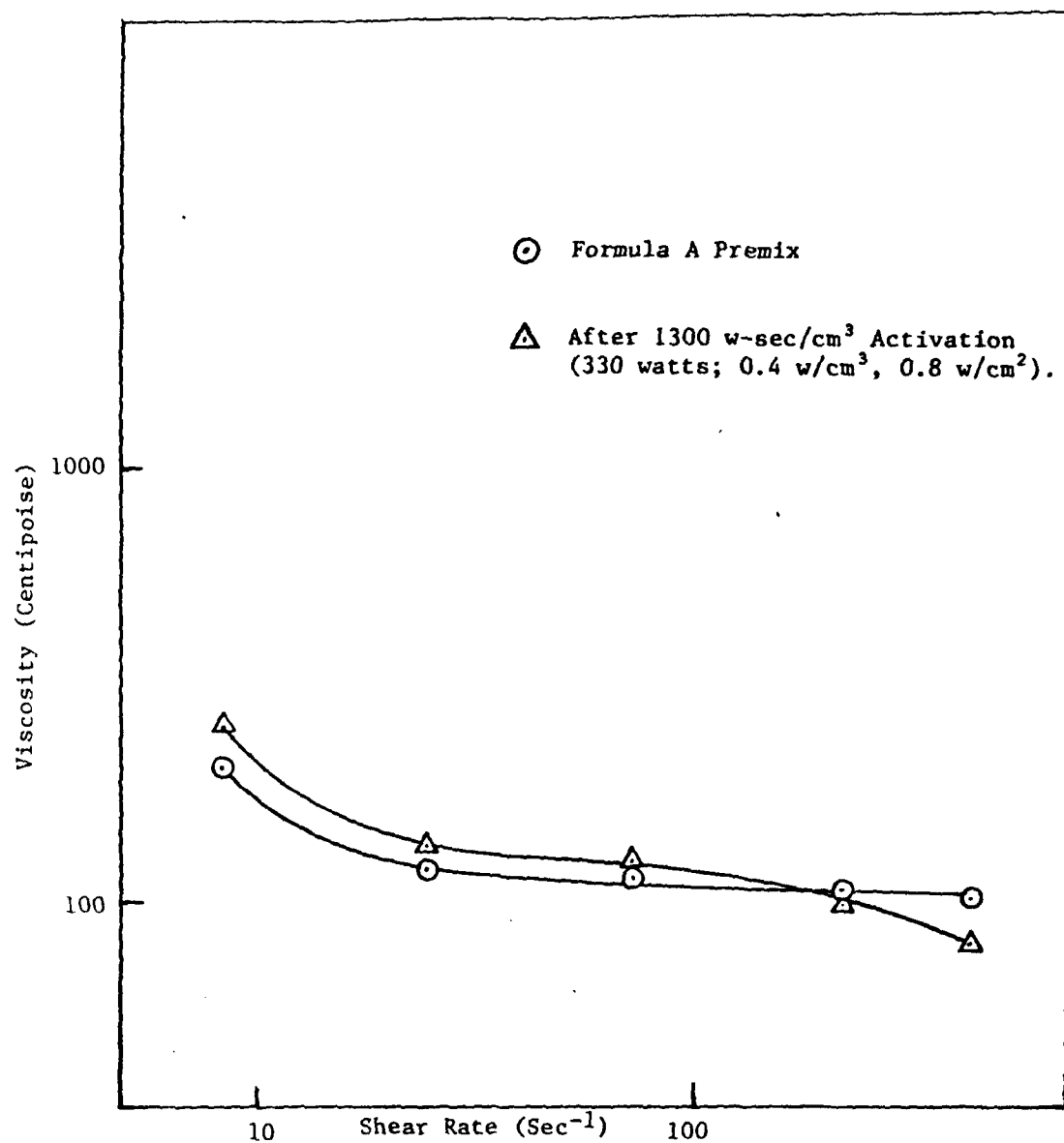


Figure 5  
EFFECT OF ULTRASONIC ACTIVATION WITH  
COMMERCIAL RADIAL UNIT ON THE VISCOSITY OF FORMULA A FUEL DISPERSION



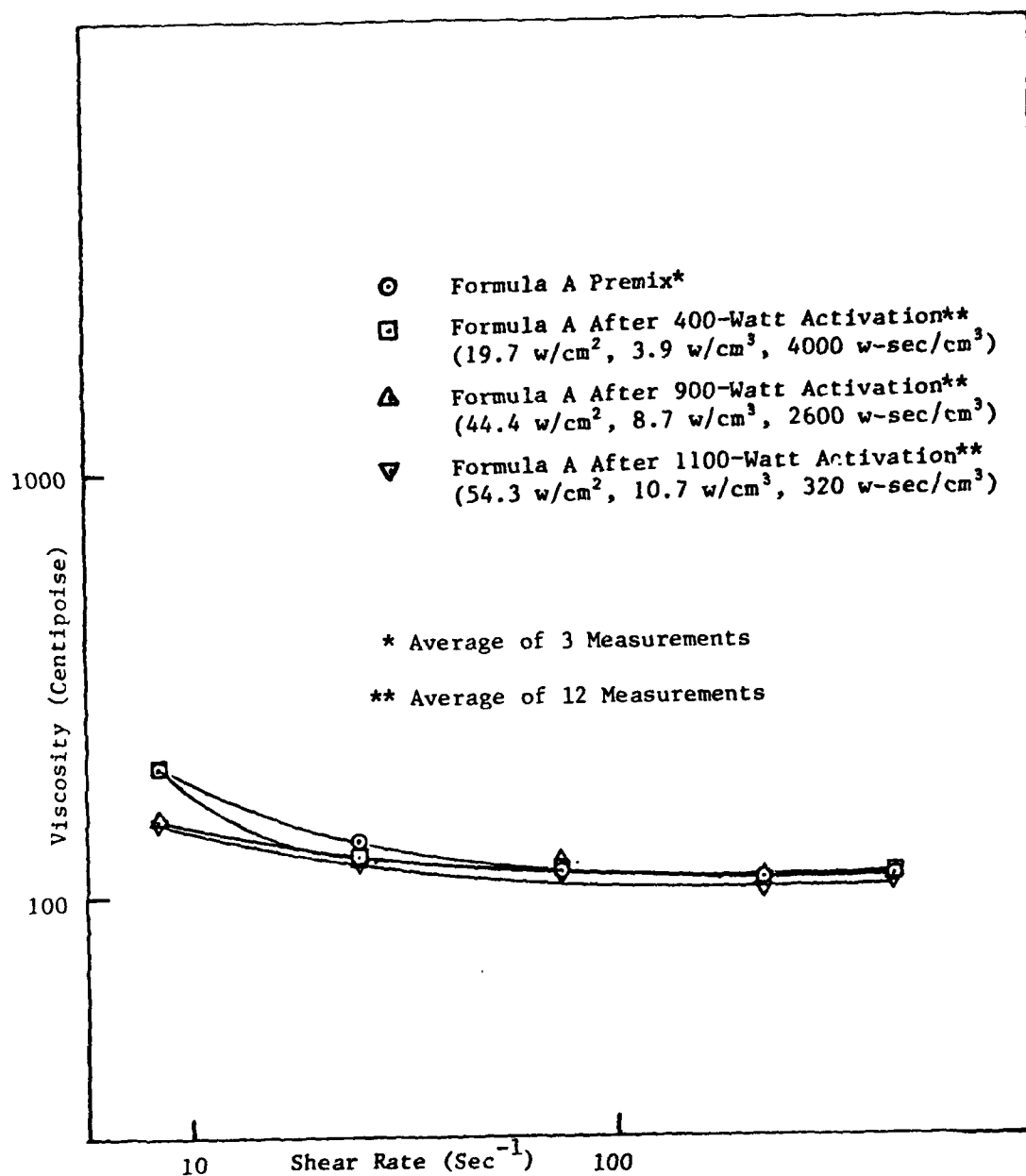


Figure 6

ACTIVATION OF FORMULA A IN THE ULTRASONIC CUP

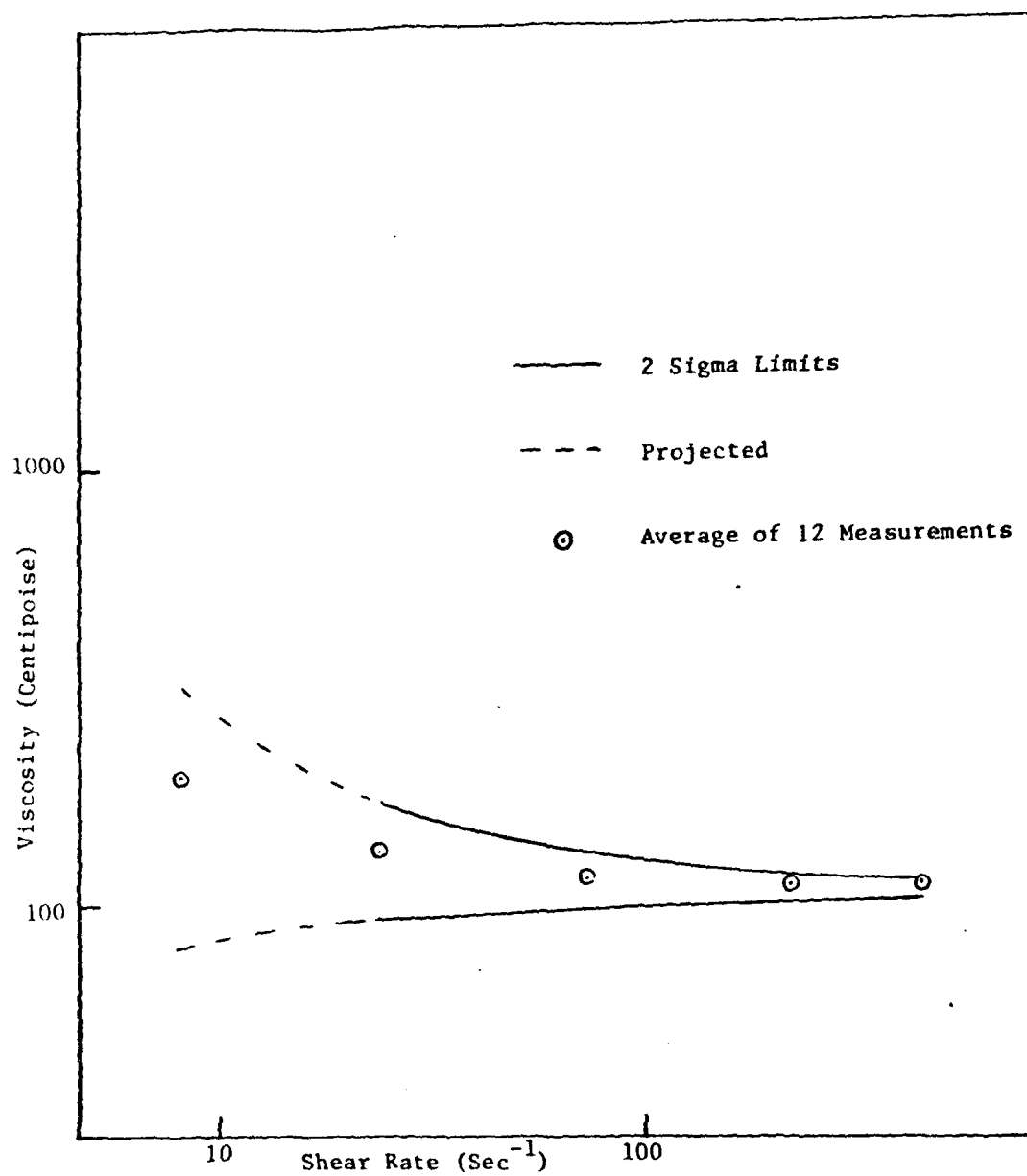


Figure 7

EXPERIMENTAL PRECISION OF VISCOSITY MEASUREMENTS OF  
FORMULA A PREMIX DISPERSION

Table II

EFFECT OF ULTRASONIC DISPERSION  
ON VARIOUS "STATEX MT"<sup>R</sup>-CONTAINING FUEL FORMULATIONS

| <u>Carbon Content</u> | <u>Shear Rate</u><br><u>(sec<sup>-1</sup>)</u> | <u>Viscosity (Centipoise)</u>      |                                   | <u>Apparent Effects</u>               |
|-----------------------|------------------------------------------------|------------------------------------|-----------------------------------|---------------------------------------|
|                       |                                                | <u>Before</u><br><u>Activation</u> | <u>After</u><br><u>Activation</u> |                                       |
| "Statex" <sup>R</sup> | 8.5                                            | 190                                | 238                               | Viscosity increased by<br>ultrasonics |
| Formula A             | 25                                             | 111                                | 159                               |                                       |
|                       | 76                                             | 106                                | 143                               |                                       |
|                       | 228                                            | 102                                | 120                               |                                       |
|                       | 456                                            | 100                                | 113                               |                                       |
| Increased             | 8.5                                            | 1618                               | 1904                              | Viscosity increased by<br>ultrasonics |
| "Statex" <sup>R</sup> | 25                                             | 1047                               | 1428                              |                                       |
| (69.3%)               | 76                                             | *                                  | *                                 |                                       |
|                       | 228                                            | *                                  | *                                 |                                       |
|                       | 456                                            | *                                  | *                                 |                                       |
| Reduced               | 8.5                                            | 286                                | 286                               | Viscosity reduced by<br>ultrasonics   |
| Surfactant            | 25                                             | 159                                | 174                               |                                       |
| (50% of               | 76                                             | 148                                | 137                               |                                       |
| Formula A)            | 228                                            | 123                                | 106                               |                                       |
|                       | 456                                            | 115                                | 84                                |                                       |
| Reduced               | 8.5                                            | 1190                               | 571                               | Viscosity reduced by<br>ultrasonics   |
| Surfactant            | 25                                             | 317                                | 317                               |                                       |
| (25% of               | 76                                             | 201                                | 212                               | Viscosity increased by<br>ultrasonics |
| Formula A)            | 228                                            | 74                                 | 127                               |                                       |
|                       | 456                                            | 63                                 | 108                               |                                       |

\*Not within instrument capability.

Table III

EFFECT OF ULTRASONIC DISPERSION ON SMALLER PARTICULATE  
CARBON ("STERLING R") FUELS

| <u>Carbon Content</u>         | <u>Shear Rate</u><br><u>(sec<sup>-1</sup>)</u> | <u>Viscosity (Centipoise)</u>     |                                  | <u>Apparent Effects</u>                                                                        |
|-------------------------------|------------------------------------------------|-----------------------------------|----------------------------------|------------------------------------------------------------------------------------------------|
|                               |                                                | <u>Before</u><br><u>Treatment</u> | <u>After</u><br><u>Treatment</u> |                                                                                                |
| "Statex" <sup>R</sup>         | 8.5                                            | 190                               | 239                              | Viscosity increased<br>by ultrasonics                                                          |
| Formula A                     | 25                                             | 111                               | 159                              |                                                                                                |
| (repeated for                 | 76                                             | 106                               | 143                              |                                                                                                |
| comparison)                   | 228                                            | 102                               | 120                              |                                                                                                |
|                               | 456                                            | 100                               | 113                              |                                                                                                |
| "Sterling" <sup>R</sup> 60%   | --                                             | --                                | --                               | Thick semi-dry paste -<br>No viscosity data<br>possible                                        |
| "Sterling" <sup>R</sup> 50%   | 8.5                                            | 17132                             | 9137                             | Viscosity reduced<br>by ultrasonic treatment<br>Viscosity increased<br>by ultrasonic treatment |
|                               | 25                                             | 3299                              | 5330                             |                                                                                                |
|                               | 76                                             | 1354                              | 1946                             |                                                                                                |
|                               | 228                                            | *                                 | *                                |                                                                                                |
|                               | 456                                            | *                                 | *                                |                                                                                                |
| "Sterling" <sup>R</sup> 48.6% | 8.5                                            | 2189                              | 1285                             | Viscosity reduced<br>by ultrasonic treatment                                                   |
|                               | 25                                             | 1110                              | 825                              |                                                                                                |
|                               | 76                                             | 666                               | 560                              |                                                                                                |
|                               | 228                                            | 338                               | 296                              |                                                                                                |
|                               | 456                                            | 254                               | 240                              |                                                                                                |

\*Not within instrument capability.

Table IV

EFFECT OF ULTRASONIC DISPERSION ON MULTIMODAL PARTICLE SIZE MIXES  
("STATEX MT" AND "STERLING R")

| <u>Carbon Content</u>                                         | <u>Shear Rate</u><br><u>(sec<sup>-1</sup>)</u> | <u>Viscosity (Centipoise)</u>     |                                  | <u>Apparent Effects</u>                        |
|---------------------------------------------------------------|------------------------------------------------|-----------------------------------|----------------------------------|------------------------------------------------|
|                                                               |                                                | <u>Before</u><br><u>Treatment</u> | <u>After</u><br><u>Treatment</u> |                                                |
| "Statex" <sup>R</sup> 30%/<br>"Sterling" <sup>R</sup> 30%     | 8.5                                            | 4283                              | 10,660                           | Viscosity increased by<br>ultrasonic treatment |
|                                                               | 25                                             | 1904                              | *                                |                                                |
|                                                               | 76                                             | *                                 | *                                |                                                |
|                                                               | 228                                            | *                                 | *                                |                                                |
|                                                               | 456                                            | *                                 | *                                |                                                |
| "Statex" <sup>R</sup> 28.4%/<br>"Sterling" <sup>R</sup> 28.4% | 8.5                                            | 2189                              | 1332                             | Viscosity decreased by<br>ultrasonic treatment |
|                                                               | 25                                             | 730                               | 730                              |                                                |
|                                                               | 76                                             | 560                               | 497                              |                                                |
|                                                               | 228                                            | 367                               | 353                              |                                                |
|                                                               | 456                                            | *                                 | *                                |                                                |

\*Not within instrument capability.

the moderate to high shear rates. These data are consistent with ultrasonic phenomena. Before ultrasonic treatment, the large agglomerates tend to form a high-viscosity gel structure at low shear rate. Higher shear rates break down the gel structure, but not the agglomerates, resulting in a low viscosity mix. Ultrasonic treatment reduces agglomerate size and the tendency to gel formation, which causes low-shear viscosity to be significantly reduced but high-shear viscosity to be increased to values comparable to those with the higher surfactant concentrations of Formula A.

Thus, ultrasonic treatment produced some changes in the viscosity of the suspensions which could be further investigated. The present data indicate that ultrasonic treatment may produce useful dispersions at lower surfactant concentrations.

Mix formulations with "Sterling R<sup>R</sup>" carbon show dramatically increased viscosities down to loading of 48.6 percent. This reflects the much smaller median particle size (75 millimicron vs. 300 millimicron for "Statex-MT<sup>R</sup>"). Again, there was some effect on viscosity due to ultrasonics.

Fifty-fifty blends of "Statex-MT<sup>R</sup>" and "Sterling R<sup>R</sup>" carbon also produced high viscosities, as shown in Table IV. Mixes with 60 percent loading and 56.8 percent loading demonstrate rheological properties well above those of Formula A. The small particle size and related high surface area of the "Sterling R<sup>R</sup>" apparently is the dominating influence in these results.

Viscosity measurements on boron dispersions in JP-10 fuel are shown in Figures 8 and 9. Formula 2, a fuel mixture with boron in the same proportion as Formula A, has viscosities of 100 to 120 centipoise at moderate to high shear rates, similarly to Formula A. This is surprising in view of the change in particle density and larger median particle size, 2 microns as reported by the supplier. Other properties of the boron apparently compensate for these factors.

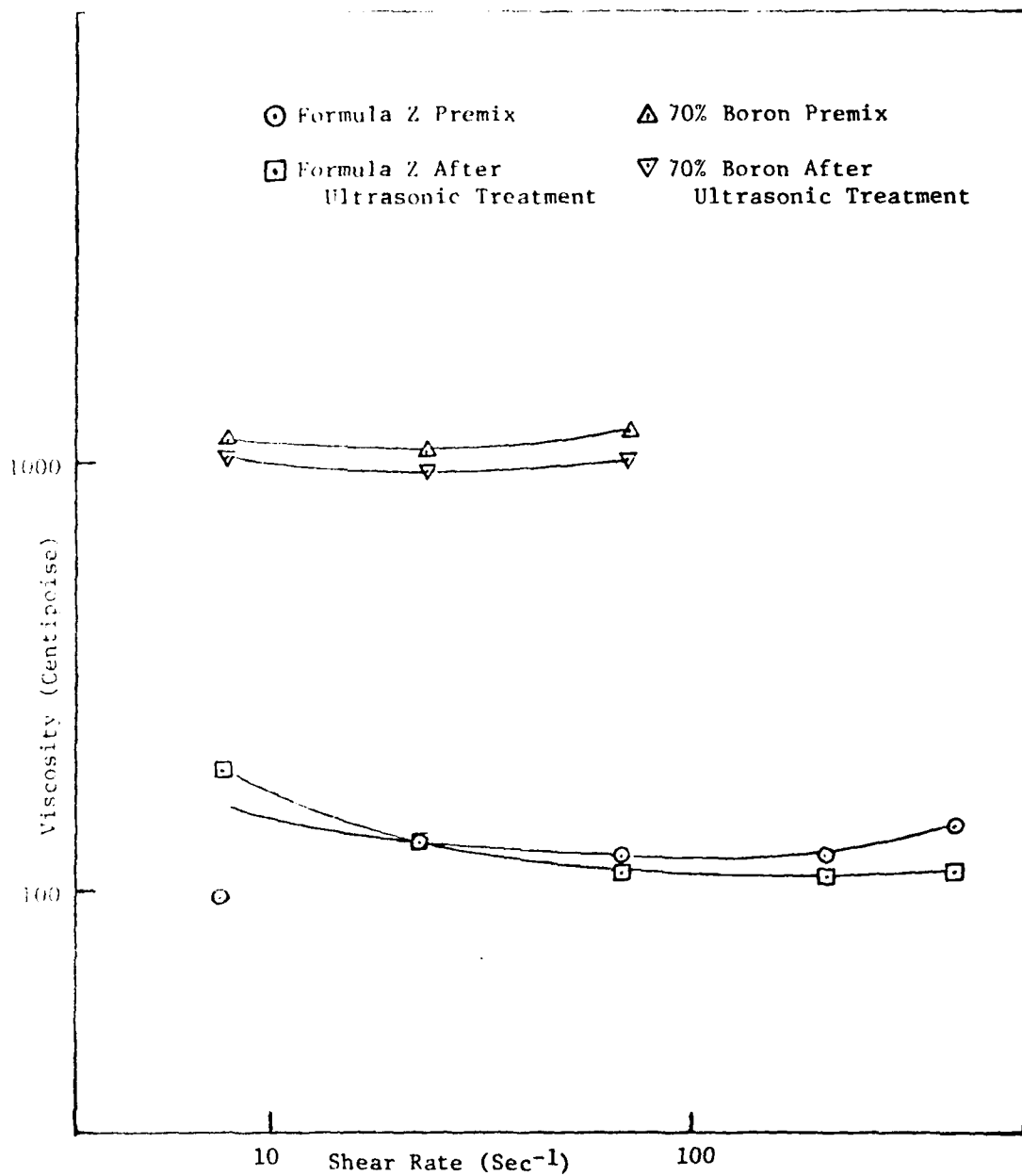


Figure 8

ULTRASONIC VS. NON-TREATED BORON FUEL DISPERSIONS

(Treated in Ultrasonic Cup Unit: 400 Watts, 19.7 w/cm<sup>3</sup>, 1400 w-sec/cm<sup>3</sup>)

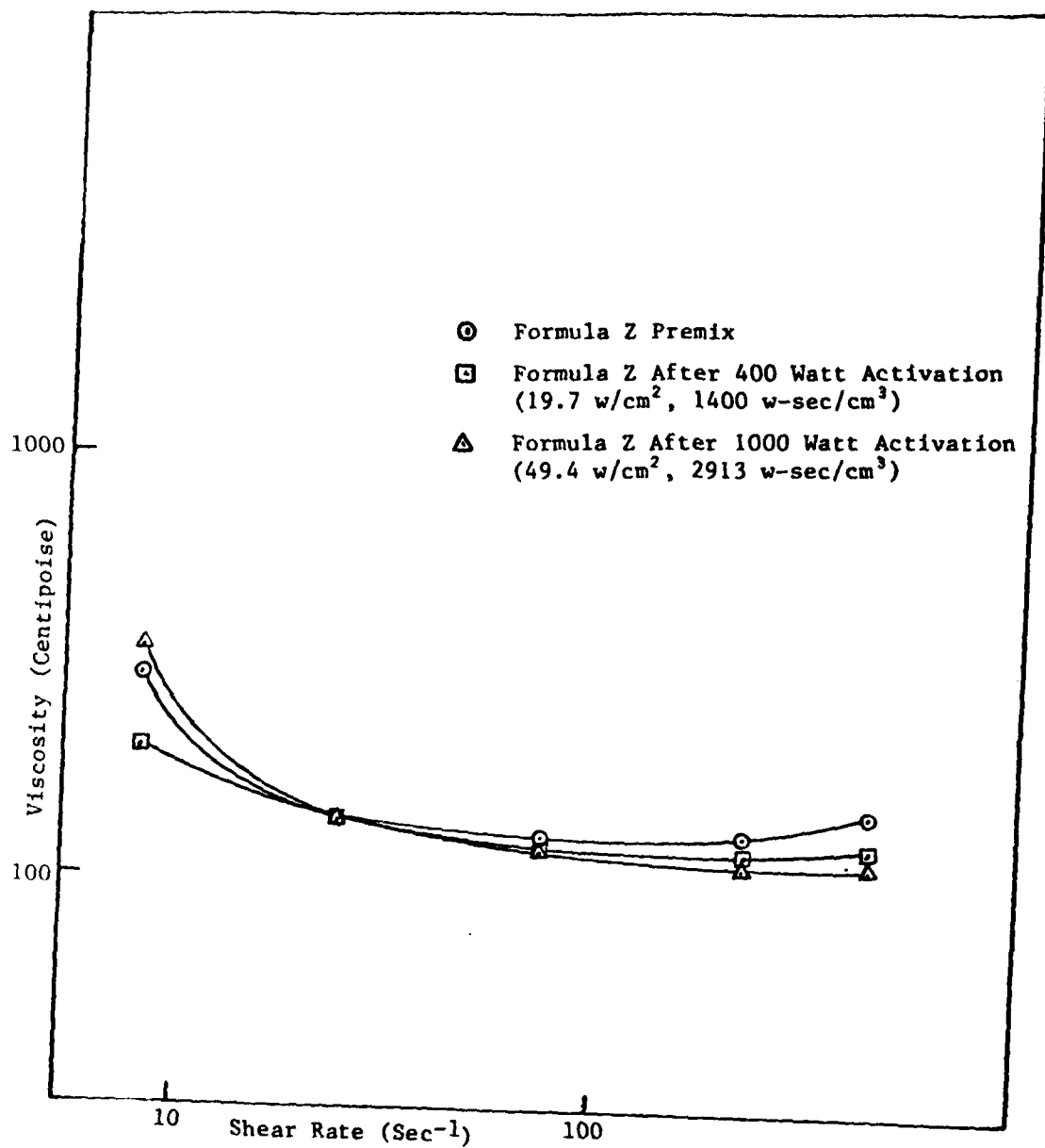


Figure 9

EFFECT OF ULTRASONIC CUP ACTIVATION ON VISCOSITY  
OF FORMULA Z DISPERSION



A mix with 70 percent boron loading, also shown on Figure 8, produced viscosities of 1000 centipoise both before and after ultrasonic treatment. This value is well above the desirable viscosity for the high density fuels. Figure 9 compares viscosities of Formula Z after 400 and 1000 watt ultrasonic treatment to a Formula Z premix. As in the case of Formula A, the rheological properties did not appear to change significantly after exposure to ultrasonics.

B. Hegman Gauge Drawdown

The drawdown technique over a calibrated groove in the Hegman Gauge also allows particle size measurement directly on the fuel mixes. Useful data, however, did not result, since qualitative data is dependent on resolution of the gauge to 5 microns at best, much larger than the 75 to 300 millimicron size range of the carbon blacks. The use of this instrument, consequently, was discontinued early in the program.

C. Conductance

It is possible that the conductance of carbon fuel mixes could change with degree of agglomeration. High degrees of agglomeration provide relatively long conduction paths through the liquid, and should thus provide a relatively high conductance. Well-dispersed carbon would have very short conducting paths and, therefore, low conductance. The measurements obtained indicate that there is some interference with this property. The surfactant in the mix may be coating the electrodes and generating a high-resistance film which overrides conductance and masks any change due to particle dispersion.

D. Accelerated Sedimentation

The sedimentation of solid particles in liquid follows Stokes' law and is a function of the particle and liquid densities, the acceleration

applied and the size of the particle. Previous work has demonstrated that this method can provide useful information on particle sizes below that generally analyzed by commercial instruments. The only negative aspect of the analysis is that the dispersion must be diluted to a 2 percent or less concentration, which raises a question as to the effect of this dilution on the dispersion. In order for the results to be valid, this factor must either have no effect or at least a uniform effect. Measurement can be also accomplished via gravitational acceleration, but inordinate time is required; by increasing acceleration through the use of a centrifuge, size separation to 0.5 micron can be obtained in 45 minutes. In using the accelerated sedimentation technique with the fuel slurries, the portion of sample containing the fine material was dried and the fraction of solids determined. As this method was refined over the course of the program, relatively consistent results were obtained.

The diluted samples of the fuel mixes were subjected to centrifugation followed by removal of the uppermost supernate containing particles of 0.5-micron diameter or smaller. This portion was dried and the percentage of fine particles in the slurry determined. Tables V through VIII and Figure 10 show the results of accelerated sedimentation analysis of Formula A before and after various ultrasonic treatments. The 0.5-micron and below particles were increased by 1.6 to 11.3 percent. Figure 10 demonstrates an upward trend in the fine particle content as ultrasonic power is increased. These measurements confirm that deagglomeration is being accomplished with ultrasonic treatment.

Table IX shows the accelerated sedimentation results for Formula Z boron fuel slurries. Due to the density difference between carbon black and boron, the determination is based on 0.4-micron particles rather than 0.5 micron. The data show little change in concentration of fine particles with this material, probably reflecting the relatively large median particle size (2 microns) of the boron powder, since even with deagglomeration, the particle size remains larger than 0.4 micron.

Table V

ACCELERATED SEDIMENTATION ANALYSIS  
Before and After Ultrasonic Dispersion  
In the Continuous-Flow Unit

|                                                              | <u>Percent Finer Than</u><br><u>0.5 Micron</u> |      |
|--------------------------------------------------------------|------------------------------------------------|------|
| Formula A                                                    | 42.7                                           | 41   |
| Before Activation                                            | 39.3                                           |      |
| Formula A                                                    | 49.6                                           | 52.3 |
| After Ultrasonic<br>Activation $3$<br>(400 w-sec/cm $^3$ )*  | 55.6                                           |      |
| Formula A                                                    | 57.0                                           | 49   |
| After Ultrasonic<br>Activation $3$<br>(4000 w-sec/cm $^3$ )* | 41.0                                           |      |

\*(Power input 200 watts; 395 w/cm $^3$ ; 39.5 w/cm $^2$ )

Table VI

ACCELERATED SEDIMENTATION ANALYSIS  
In the Ultrasonic Cup

|                          | <u>Percent Finer Than 0.5 Micron</u> |      |
|--------------------------|--------------------------------------|------|
| Formula A (in duplicate) | 33.8                                 | 36.4 |
| Before Activation        | 38.9                                 |      |
| Formula A (in duplicate) | 36.4                                 | 38.0 |
| After Activation         | 39.6                                 |      |

Table VII

ULTRASONIC TREATMENT OF FORMULA A IN THE ULTRASONIC CUP

| <u>Energy Input</u> | <u>Accelerated Sedimentation</u> |      |                   |      |
|---------------------|----------------------------------|------|-------------------|------|
|                     | <u>(% Finer Than 0.5 Micron)</u> |      |                   |      |
|                     | <u>Before</u>                    |      | <u>After</u>      |      |
|                     | <u>Activation</u>                |      | <u>Activation</u> |      |
| 400 Watts           | 33.8                             | 36.4 | 36.4              | 38.0 |
|                     | 38.9                             |      | 39.6              |      |
| 900 Watts           | 35.6                             | 37.5 | 38.9              | 40.8 |
|                     | 39.4                             |      | 42.6              |      |
| 1100 Watts          | 35.6                             | 37.5 | 42.5              | 42.1 |
|                     | 39.4                             |      | 41.7              |      |

Table VIII

ULTRASONIC TREATMENT OF FORMULA A IN CONTINUOUS-FLOW UNITS

| <u>Energy Input</u> | <u>Accelerated Sedimentation</u> |    |  |                             |      |      |
|---------------------|----------------------------------|----|--|-----------------------------|------|------|
|                     | <u>(% Finer Than 0.5 Micron)</u> |    |  |                             |      |      |
|                     | <u>Before</u>                    |    |  | <u>After</u>                |      |      |
|                     | <u>Activation</u>                |    |  | <u>Activation</u>           |      |      |
|                     |                                  |    |  | 400                         | 4000 |      |
|                     |                                  |    |  | <u>w-sec/cm<sup>3</sup></u> |      |      |
| 200 Watts           | 42.7                             | 41 |  | 49.6                        | 52.3 | 57.0 |
|                     | 39.3                             |    |  | 55.6                        | 41.0 | 49   |
| 1000 Watts          | 37.5                             |    |  | 40.5                        | 39.6 |      |
|                     |                                  |    |  | 38.6                        |      |      |

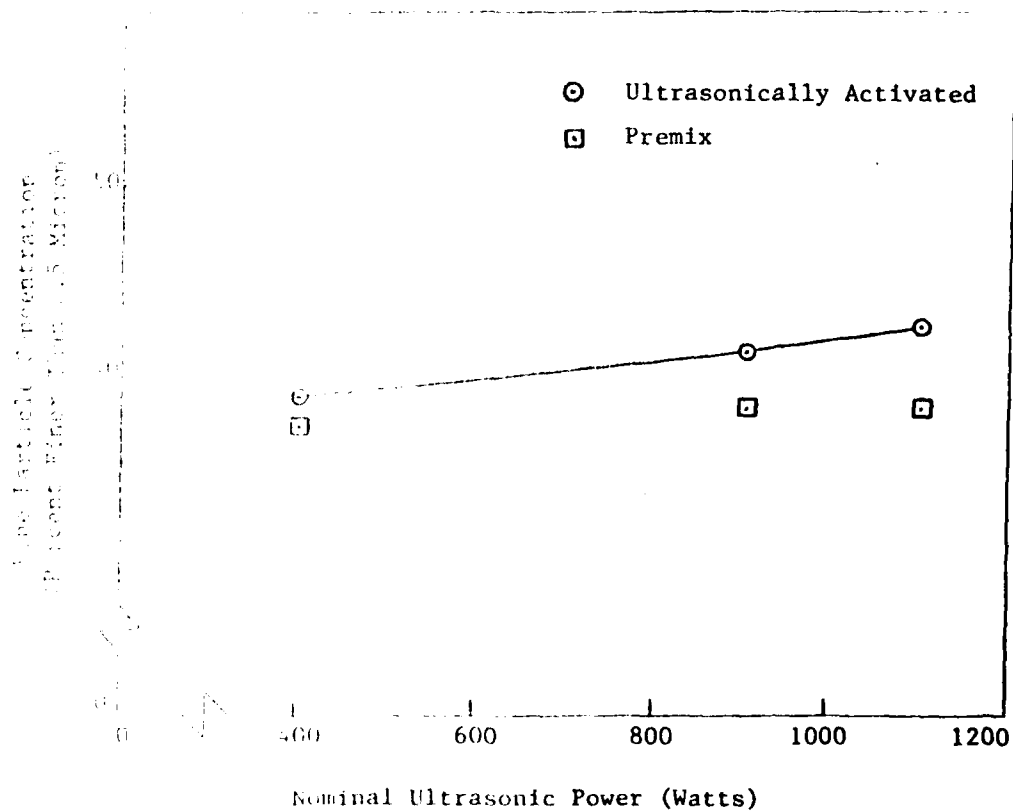


Figure 10  
 ULTRASONIC EFFECT (WITH THE ULTRASONIC CUP) ON  
 FINE PARTICLE CONCENTRATION IN FORMULA A

Table IX

ACCELERATED SEDIMENTATION - BORON FORMULA Z  
APPROXIMATE PERCENT FINER THAN 0.4 MICRON

|                                                                                    | Before Activation |      |                |      | After Ultrasonics<br>(%) |
|------------------------------------------------------------------------------------|-------------------|------|----------------|------|--------------------------|
|                                                                                    | Premix            |      | Premix         |      |                          |
|                                                                                    | Before Stirring   |      | After Stirring |      |                          |
|                                                                                    | (%)               |      | (%)            |      |                          |
| 400 Watts <sub>2</sub><br>(19.7 w/cm <sup>2</sup><br>1400 w-sec/cm <sup>3</sup> )  | 14.7              | 14.4 | 11.4           | 11.3 | 12.6                     |
|                                                                                    | 14.0              |      | 11.2           |      | 13.3                     |
|                                                                                    |                   |      |                |      | 10.6                     |
|                                                                                    |                   |      |                |      | 11.7                     |
| 1000 Watts <sub>2</sub><br>(49.4 w/cm <sup>2</sup><br>2913 w-sec/cm <sup>3</sup> ) | 14.9              |      |                |      | 14.5                     |
|                                                                                    | 12.8              | 13.2 |                |      | 13.8                     |
|                                                                                    | 12.1              |      |                |      | 13.4                     |
|                                                                                    | 12.8              |      |                |      | 12.6                     |



#### 12. Pycnometric Determination

Pycnometry, developed as an adjunct to accelerated sedimentation, is based on the change in slurry density as solids content changes. In the procedure, the product of accelerated sedimentation which is calculated to contain particles of 0.5 micron or finer is placed in a pycnometer, where the density is measured. The concentration of solids in the sample is then determined based on the known densities of the individual components.

Results from these tests showed positive correlation to those from the initial accelerated sedimentation technique. Table X shows typical pycnometric data for Formula A before and after ultrasonic treatment with the continuous flow unit. The averages of four determinations at each condition indicate a trend to increased fine particle content. Again, deagglomeration of the carbon particles by ultrasonics is indicated.

#### 13. Microprobe Filtration

With this method, samples of dispersion are diluted and forced through a filter of a known particle size; the percentage of particles passing the given pore diameter can then be determined.

The data obtained were inconsistent with that obtained from other methods, probably because samples of sufficient size to be meaningful produced blocking or plugging of the filter.

#### 14. Scanning Electron Microscopy

Scanning electron microscopy provides the capability of observing particles in the size ranges occurring in the fuel slurries. However, this tool was not able to give meaningful data on the degree of agglomeration, since direct measurements on the slurry could not be made. All of the

Table X

PYCNOMETRIC DETERMINATION  
Formula A Before and After Ultrasonic Activation

|                                                                                      | <u>% Finer Than 0.5 Micron</u> |      |
|--------------------------------------------------------------------------------------|--------------------------------|------|
| <u>Before Ultrasonic</u><br><u>Activation</u>                                        | 17.9                           |      |
|                                                                                      | 14.1                           | 16.2 |
|                                                                                      | 15.4                           |      |
|                                                                                      | 17.3                           |      |
| <u>After Ultrasonic</u><br><u>Activation</u><br>(1000 Watts in Continuous-Flow Unit) | 17.2                           |      |
|                                                                                      | 13.4                           | 16.6 |
|                                                                                      | 18.0                           |      |
|                                                                                      | 17.6                           |      |

variety of techniques developed for slurry dilution and slide preparation resulted in particle agglomeration.

#### H. Static Sedimentation

In order to determine the relative stability of the premix control samples and the ultrasonically dispersed slurries, samples of each were placed on shelf storage for up to three months. Periodically, portions of these sample slurries were taken from near the top of the container and analyzed for solids content. This value was compared to that of the original formula to determine the degree of settling.

The measurement made directly on undiluted samples of high density fuels provides the data on the stability of the slurries. Table XI reports the relative concentration of carbon suspended in non-treated and ultrasonically treated mixes after shelf stands of 30 and 60 days. The untreated samples show more carbon settling than those ultrasonically treated, indicating that ultrasonic exposure does increase fuel slurry stability.

Table XI

EFFECT OF ULTRASONICS ON UPPER LAYER SEDIMENTATION OF  
CARBON FROM FORMULA A FUEL DISPERSION

|                                                | <u>Ultrasonic</u><br><u>Treatment</u> | <u>Non-Ultrasonic Controls</u><br><u>(Percent of Ultrasonic Test)</u> |
|------------------------------------------------|---------------------------------------|-----------------------------------------------------------------------|
| After 30 Days                                  | Full Content                          | 98.2                                                                  |
| Resample @ 60 Days<br>(same samples as 30 day) | Full Content                          | 98.6                                                                  |
| New Sample @ 60 Days                           | Full Content                          | 99.4                                                                  |

#### IV. CONCLUSIONS AND RECOMMENDATIONS

##### A. Conclusions

1. Laboratory arrays amenable to modification and scale-up for production equipment were demonstrated as feasible to transmit ultrasonic energy into high density fuel slurries of interest to the Air Force.
2. Ultrasonic activation at the power levels employed had a detectable effect on compositional variations of the slurries, with an increase in fine particle content as ultrasonic power was increased.
3. Ultrasonic effect with varying compositions indicated the possibility of using lower loadings of surfactant.
4. Complete dispersion stability for the 60-day shelf period tested was demonstrated with the ultrasonically activated samples, in contrast to some breakdown of the non-activated control samples.

##### B. Recommendations

The nature of these high-density slurries is such that most analytical techniques are insufficiently sensitive to reveal what may be significant dispersion improvement as a result of ultrasonic activation. It would be possible to extend laboratory sample analysis to better identify the trends revealed, but the practical advantages of ultrasonic activation can best be determined by comparing the combustion performance of ultrasonically treated vs. non-activated sample formulations.

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