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RESEARCH MEMORANDUM

STABILIZATION OF 50-PERCENT MAGNESIUM - JP-4 SLURRIES WITH SOME ALUMINUM SOAPS OF C_8 ACIDS

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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SUMMARY

An exploratory investigation with three aluminum disoaps of C_8 acids was conducted to determine the gelling properties and stabilizing ability in slurries of 50 percent magnesium powder and JP-4 fuel. Each of the soaps was found to gel the slurries satisfactorily and to exhibit adequate reproducibility of gel properties. The effect of soap concentration on the useful life (time during which gel structure is adequate to prevent undue settling of the metal powder) was found to be different and specific for each soap. In the range of soap concentration investigated in slurries made on a laboratory scale, one soap produced a maximum useful life at 0.6 percent (weight); the second soap showed no effect of concentration on the useful life; and the third soap, considered to be the best of the three, exhibited an increased useful life with increasing soap concentration. The best soap produced slurries which had a useful life of 42 days with 0.6 percent concentration and in excess of 153 days with 4.0 percent.

After partial or complete breakdown of gel structure in the slurries prepared with the third soap, the slurries were regelled by stirring in an additional 0.6 percent soap. The useful life of the regelled slurries was found to be independent of the original soap concentration and to average 78 days. The combined useful life obtained by first gelling with 0.6 percent soap and later regelling the same slurry with an additional 0.6 percent soap was 120 days, which is a greater useful life for the 1.2 percent total soap involved than the 110 days obtained with 2.0 percent soap originally. The third soap was also used to stabilize slurries made in larger batches up to 100 pounds in size. While small samples removed from the larger batches (0.6 percent soap) exhibited properties which agreed with those of the small laboratory-scale slurries, some larger-scale batches which had been kept sealed at temperatures ranging from 0° to 100° F were still usable after $5\frac{1}{2}$ months.

[redacted]

The physical properties measured were apparent viscosity at low rate of shear and, in a few cases, apparent viscosity at high rates of shear. All slurries investigated at high rates of shear showed fluidity estimated to be adequate to permit pumping and handling. In addition, a technique was demonstrated whereby a slurry with high apparent viscosity at low shear could be temporarily rendered more fluid by subjecting it to high shear for a few minutes.

INTRODUCTION

Analytical and experimental studies of aluminum, boron, and magnesium as fuels for ram-jet engines have indicated advantages for these fuels over petroleum fuels. For example, as shown in references 1 and 2, all three can give increased thrust over petroleum fuels, and, in addition, magnesium permits greater operating limits and boron permits greater range. A promising technique for handling these solid fuels is the use of slurries composed of finely divided powder dispersed and suspended in a hydrocarbon carrier (refs. 3 and 4). However, to date there has been no completely satisfactory method developed for preparing slurries that retain ideally the three selected basic requisites: (1) adequate fluidity for pumping and handling in the more or less conventional aircraft fuel systems, (2) adequate storage stability, and (3) adequate reproducibility of slurry properties.

Previous investigations (ref. 5) at the Lewis laboratory have shown that a suspension of metal powder in slurries could on occasion be very effectively maintained for several weeks or more by the use of aluminum octoate soaps which act as gelling or thickening agents in the hydrocarbons. Unfortunately, the gelling properties of the aluminum octoate soaps available at that time were completely erratic. The same conditions and materials which would on occasion produce a nearly ideal gel slurry would often produce slurries of unpredictable characteristics in both laboratory and larger batches. In addition, after the original gel structure had almost disappeared during storage, regelation of the slurry as a means of extending the useful life had previously been unsuccessful.

The work reported herein was an exploratory investigation of the gelling properties of three aluminum soaps of C_8 acids to determine whether any of the soaps exhibited desirable properties for magnesium-slurry fuel application. This investigation, which was prompted by the availability of these new soaps, had the following objectives: (1) to develop a method for preparing slurries (small batch size) of magnesium and JP-4 fuel which have properties that approach the foregoing selected requisites of a ram-jet fuel; (2) to prepare these slurries in larger batches adequate for use in experimental combustors.

To investigate the properties of these soaps, slurries of atomized magnesium of about 15-micron average particle size were prepared in dry JP-4 fuel. The concentration of the three soaps was varied in small steps from 0.4 to 0.7 percent or more, in laboratory-size samples of 50 percent magnesium slurries; in the case of one soap, the range of concentration was extended to 4.0 percent. The gelled slurries were prepared from all three soaps at room temperature, 75° to 82° F, and from one of the soaps at about 110° F. The feasibility of regelling broken slurries was investigated with only one of the soaps. (Specialized terms used herein are defined in the appendix.) Gelled JP-4 fuel samples containing no magnesium were prepared with one of the soaps for the purpose of comparing the viscosity of gelled JP-4 fuel with the viscosity of the 50 percent magnesium slurries. Apparent viscosity at a low rate of shear was obtained for all samples of slurries and gelled JP-4 fuel, and apparent viscosity at high shear rates was obtained for a limited but representative number of the samples. Information obtained in the early part of the investigation was applied in the preparation of larger-scale slurries. Limited data were also obtained on the recovery of apparent viscosity of a slurry which had been subjected to successively increased periods of shear in a colloid mill.

MATERIALS AND APPARATUS

Materials

Magnesium. - The magnesium powders used were obtained from Golwynne Chemicals Corp. The powders were produced by atomization and were predominantly spherical in shape. The chemical and particle size analyses of the two lots of magnesium powder used are as follows:

Lot number	Particle size distribution[a], weight percent					Average particle size[b], micron	Assay[c], weight percent pure Mg
	8.5 μ	8.5-17 μ	17-26 μ	26-40 μ	40 μ		
1	13.5	21.9	23.5	40.0	1.1	13.0	94.5
2	9.2	25.0	33.6	31.8	.4	17.5	92.6

[a]Obtained by means of a Roller Air Analyzer.

[b]Obtained by means of a Fisher Sub-Sieve Sizer.

[c]Gravimetric method was used. The remainder was assumed to be magnesium oxide.

[redacted]

Magnesium powder from lot number 1 was used throughout for the laboratory-scale slurries of 300-gram size, and the magnesium powder from lot number 2 was used for the slurries of larger size.

Gelling agents. - The three gelling agents used in the investigation, hereinafter designated soaps I, II, and III, were aluminum soaps made from aliphatic C_8 acids. The soaps, as indicated by the analyses presented in table I, were found to correspond approximately to dioctoates. The acids from which soap I was made were reported to be a mixture of monocarboxylic C_8 acids. Soaps II and III were both reported to 3079
be made from 2-ethylhexoic acid under the same specifications. In addition, these last two soaps were reported to have improved gelling characteristics as compared with previous aluminum di(2-ethylhexoate) soaps. Soaps II and III were manufactured by precipitation with aluminum chloride, whereas aluminum sulfate was used to precipitate the soaps discussed in reference 5. Soaps II and III were very fine, light powders, whereas soap I was quite granular and had the general size and appearance of a medium white sand.

Hydrocarbon fuel. - The hydrocarbon base used in the preparation of all slurries was from a single batch of fuel, specification MIL-F-5624A, grade JP-4, which had been dried with activated alumina. The procedure used for drying the JP-4 fuel was to allow the liquid fuel to percolate slowly through a drying tower, 4 inches in diameter by 10 feet in length, filled with activated alumina 8 to 14 mesh.

The data obtained from analyzing a sample of the dry fuel are given in table II.

Apparatus

Osterizer. - The Osterizer, which is a small industrial mixer or food blender, has a four-blade impeller that operates at about 20,000 rpm. Its unique whipping action blended the soap (particularly soap I) into the JP-4 fuel more uniformly than the other more conventional laboratory stirrers tried.

Constant-speed stirrer. - For the dispersion of magnesium powder into the soap - JP-4 mixtures, an adjustable-speed electric laboratory stirrer (fig. 1) was operated with impeller a at a speed of 300±2 rpm. Operation of the stirrer was held constant for the purpose of assured similar treatment in the preparation of each slurry. The stirrer assembly and the threaded jar-retaining ring, which were mounted in a fixed position on a stand, allowed the impeller to be immersed to the same position when each pint sample jar was attached from beneath. The removable lid was used to minimize evaporation loss of the JP-4 fuel and to provide access to the jar through the wide opening so that the magnesium powder could be added without interruption of stirring.

The stirrer assembly was modified slightly to incorporate the additional soap required in the regelation of broken slurries; stirrer b was used at a speed of 1800±10 rpm.

Rollers. - The roller used in the preparation of small batches of slurries was adjustable in speed and was enclosed by a box equipped with a transparent lid. The speed of the roller was adjusted to rotate the pint jars at 46±3 rpm. Facilities were provided inside the box for thermostatically controlled electric heating and for forced-air circulation. Where elevated temperature for the preparation of a slurry is mentioned in the text and tables, the indicated temperature is that in the box and not actually that of the slurry.

A conventional drum roller which could handle drums as large as 50 gallons was used in the preparation of the 100-pound slurries.

Brookfield viscometer. - For obtaining apparent viscosity data at a low shear rate, a Brookfield Synchro-lectric Viscometer, model LVF, illustrated in reference 6, was operated with spindle number 4 at 6 rpm without a guard. All measurements were made at room temperature (75° to 80° F) by immersing the spindle directly into the pint sample jars of slurry. The readings were taken on the outer scale to the nearest 100 centipoises at 0.5 minute (3 revolutions). Newtonian liquids of known viscosity were used to check the accuracy of the instrument under these conditions and the results are as follows:

Viscosity of standard, centipoises	Brookfield reading, centipoises
1,000	900
12,500	12,500
60,000	59,000

The accuracy of the instrument should not change from the measurement of a Newtonian liquid to that of a non-Newtonian, but a variation in apparent viscosity may actually occur due to the non-Newtonian characteristics (thixotropy) of the slurry. In addition, the slurry may channel or it may clump and cling around the spindle, which would cause, respectively, a lower or a higher reading. Omission of the guard around the viscometer spindle helped to minimize the effects of channeling and clumping.

Mobilometer. - The consistency of a slurry was measured with the mobilometer (see ref. 7) in accordance with the procedure and plunger specifications set forth by the National Military Establishment for incendiary oils (ref. 8). In brief, the procedure involves the determination of total load in grams required to force a perforated plunger a marked distance through the fluid contained in a tube in 100 seconds as determined by linear interpolation of two successive loads that straddled the desired time of fall.

Data obtained at room temperature from the mobilometer and the Brookfield viscometer on a few magnesium slurries and gelled JP-4 fuel samples are plotted in figure 2 and show fair correlation. The Brookfield apparent viscosity of each sample was obtained prior to the determination of consistency with the mobilometer. The time base of 100 seconds, as specified for the mobilometer in reference 8, was too short to allow measurements on slurries with a viscosity less than about 10,000 centipoises. 3079

The mobilometer was used to a very limited extent but it was found to be less suited and less versatile for measuring the consistency of the slurries, considering the large number of samples and the frequency of determinations required.

Severs rheometer. - The Severs rheometer and the operating procedure used are well described in references 6 and 9. All determinations were made at room temperature with an orifice having a 0.2011-centimeter inside diameter and a 5.00-centimeter length. The data were converted as described in reference 6 to the reciprocal of the rate of shear and to apparent viscosity.

PROCEDURE

The preparation of small-scale slurries for the exploratory investigation of the gelling properties of the three soaps is described in a generalized procedure. Other details pertinent to the preparation of each slurry are presented in table III. All slurries were made to a total weight of 300 grams and contained 50 percent by weight of magnesium powder, while the concentration of the soaps was varied from 0.4 percent to 0.7 percent or more. With soap III, slurries were prepared with a soap concentration up to 4.0 percent. The remainder of each slurry was dry JP-4 fuel. All percentages throughout the report are expressed as percent by weight of total sample. Duplicate slurries of each composition were independently prepared.

Preparation of small-scale slurries. - The desired amount of JP-4 fuel was first weighed to the nearest 0.1 gram in a round pint glass jar and then transferred to the Osterizer, to which in turn was added the desired amount of soap weighed to the nearest 0.005 gram. Immediately with the introduction of soap, the Osterizer was turned on and a stop watch was started (time zero). At 2.0 minutes, the Osterizer was stopped and the contents were transferred back to the glass jar, which was promptly (average delay, 0.5 min) fitted to the constant-speed stirrer.

The stirrer was started immediately and the magnesium powder, 150±0.1 grams was added within 3.0 minutes from time zero. Stirring of the slurries was continued until the powder was well mixed and dispersed. If gelation at room temperature occurred within 1 hour, preparation of the slurries was completed on the constant-speed stirrer. (The few exceptions which received longer stirring are indicated in table III.) Gelation time for each of the slurries was taken as the interval from time zero to the time when the vortex surrounding the stirrer shaft (shown
3079 in fig. 1) disappeared as a result of the increase of viscosity and formation of gel structure.

If gelation required more than 1 hour or was carried out above room temperature, preparation of the slurry was completed on the roller. (Note exceptions in table III.) This type of agitation allowed a sample jar to be sealed with its cap to prevent excessive loss of JP-4 fuel by evaporation. The slurries which were to be gelled at an elevated temperature were transferred from the constant-speed stirrer to the roller as soon as the powder appeared to have been well wetted and dispersed, whereas slurries which were to be gelled on the roller at room temperature were transferred at convenience from the constant-speed stirrer (note table III).

Gelation time for those slurries which required rolling was determined by a combination of observations. With but little experience, an operator can distinguish, upon shaking a container of slurry, the sharper sound and "feel" of the slurry before gelation from the dull, sluggish slap - similar to that of paint or syrup - of a gelled slurry. Furthermore, depending upon the concentration of the soap in the slurries, soon after this initial thickening has occurred, the viscosity rapidly increases to a point such that the slurry begins to slip from (fails to wet) the glass wall of the jar. As constant attendance was not possible, either condition was used to demarcate gelation time and to indicate when agitation of the slurries could be stopped, although agitation in some cases was allowed to continue beyond gelation time (see table III).

Regelation of broken slurries with soap III. - Limited tests were conducted with soap III to determine the feasibility of extending the useful life of slurries by regelation. Slurries (seven samples in all) from soap III, which were in various stages of breaking and which originally contained from 0.3 to 0.6 percent soap, were regelled at about 110° F by the addition of a quantity (0.60 percent) of the soap. The glass jar which contained the broken slurry was attached to the constant-speed stirrer and stirred at 1800±10 rpm until the magnesium was uniformly redispersed. The weighed quantity of additional soap was added and after the mixture was stirred 3.0 to 5.0 minutes, the jar was capped and transferred to the roller. Regelation time was determined as described previously, but in all cases slippage of the slurry from the glass wall of the jar was used to demarcate the final time. Specific details pertinent to regelation of each of the slurries are presented in table IV.

Preparation of gelled JP-4 fuel samples. - The method of preparation of gelled JP-4 fuel samples was identical to that used for slurries gelled with soap III at elevated temperature. The total sample weight was held to 200.0 grams and samples were made in duplicate for the soap concentrations 0.8, 1.2, 2.0, and 4.0 percent by weight. Gelation of the JP-4 fuel caused a viscosity increase which was manifested by the formation of gelatinous sheets of liquid perpendicular to the axis of the rotating jar, hanging continuously from the inside upper surface. The details in the preparation of these clear gel samples are given in table V.

Preparation of larger batches of slurries. - Several larger batches 3079
of 50 percent magnesium slurries were prepared with soap III (0.6 percent) and the preparations for three samples are as follows:

1. The weighed quantities of soap, magnesium powder, and JP-4 fuel, which totaled 4.2 pounds, were placed in a gallon round paint can. The mixture was rotated on the small roller at 30 rpm at a temperature of about 110° F. Gelation, which was determined by inspection, required $3\frac{1}{2}$ hours.

2. An equal quantity (2.25 lb) of magnesium powder was added to gelled JP-4 fuel (with 1.2 percent soap) which had been gelled 47 days previously at about 110° F. The can was rolled at room temperature for 2 hours to disperse the magnesium.

3. A slurry which totaled 100 pounds was made in a 20-gallon drum in a manner identical to slurry 1 except that the temperature was 90° F and the drum was rotated at 25 rpm. Gelation time was about 4 hours.

Exhaustive shearing of a slurry in a colloid mill. - An attempt to use a colloid mill to blend and mix the ingredients in the preparation of larger batches of slurries was unsuccessful. In an effort to account for the failure to obtain gelation, 9.0 pounds of previously gelled slurry, which contained 0.6 percent soap III, was transferred to a laboratory colloid mill. The Brookfield apparent viscosity of the slurry prior to milling was 26,000 centipoises. The slurry was continuously recycled through the mill for 40 minutes and samples were removed at successive intervals throughout this period. The time of removal of each sample was recorded and the Brookfield apparent viscosity of each sample was obtained at intervals thereafter until the viscosity after milling was equal to the viscosity of the unmilled slurry. When the apparent viscosity was 500 centipoises or less, the slurries were stirred with a spatula to redisperse the magnesium immediately before the measurement on the viscometer; above 500 centipoises the magnesium settled less rapidly and stirring was unnecessary.

RESULTS AND DISCUSSION

Each soap investigated demonstrated a characteristic behavior when it was used as a gelling agent for stabilizing 50 percent magnesium - JP-4 fuel slurries. The resultant slurries differed in appearance, in useful life, and in viscosity-age characteristics with each soap.

For each of the three soaps the reproducibility of data from pairs of samples with identical composition was considered adequate. In figure 3(a) the data for apparent viscosity against age for the duplicate pairs of slurries which contain 0.6 percent of soaps I, II, and III are presented. Examination of this figure shows that although the daily viscosity changes for the duplicate samples are not identical, the trend of the data does not differ greatly between the two samples. The agreement in useful life of duplicate samples was quite satisfactory, and the reproducibility of gelation time was generally satisfactory (see table III).

Part of the difference between duplicate samples in the daily viscosity changes apparently results from a tendency of the viscosity of a slurry to decrease irregularly with age. Figure 3(b), in which data are presented for duplicate slurries with 0.7 percent soap III gelled at 110° F, shows strong unmistakable deflections in the curves that coincide in age to produce a minimum and maximum before declining further. Examination of the curves in figure 3 reveals a number of lesser deflections which are found to occur with each of the soaps but not always to coincide with respect to age of the slurry. Attempts to relate the fluctuations to the variation of ambient temperature or humidity in storage of the slurries were unsuccessful. The fluctuations appear to be merely a characteristic that each of the three soaps could show on occasion.

The use of each soap as a stabilizing agent will be presented separately and then soaps I, II, and III will be compared.

Gelation with soap I. - Slurries of 50 percent magnesium powder in JP-4 fuel were gelled by soap I dependably and very rapidly at room temperature. For example, a slurry with 0.6 percent soap I (table III), gelled within 12 minutes. The Brookfield apparent viscosities are plotted against the age of the slurries in figure 4 from the data in table VI. A pronounced concentration effect for 0.6 percent soap is readily apparent in the figure. An undesirable characteristic of slurries gelled with soap I is indicated by the extrapolated dashed lines extending from the solid curves to the day the slurries were observed to be broken at 500 centipoises. The gel structure appeared to break soon after the apparent viscosity of the slurries came within the range of 2000 to 4000 centipoises, and the magnesium powder settled out in a hard cake leaving clear JP-4 fuel above. Another characteristic of soap I was that the slurries were not completely homogeneous even with the use of the Osterizer. It appeared that the grains of soap swelled into granules and that the wetted magnesium powder was supported between the granules. No difficulties due to this granular structure were encountered, however, in fuel injectors during combustion studies (ref. 10).

Gelation with soap II. - Slurries of 50 percent magnesium powder in JP-4 fuel were gelled dependably with soap II and appeared to be completely homogeneous. The slurry was jelly-like in appearance and free of granulation. Gelation time for soap II was relatively long; for a slurry containing 0.6 percent soap, as shown in table III, the time required for gelation was about 5 hours at room temperature. The Brookfield apparent viscosity - age profiles for the slurries prepared with soap II are summarized in figure 5 with smooth curves only, and the data from which the summary curves were obtained are presented in table VII. As shown in figure 5, the variation of soap concentration caused very little difference in the viscosity-age profiles or in the useful life of the slurries. Each of the slurries began to show evidence of settling at a viscosity of about 5000 centipoises. The metal portion of the slurry would settle lower as the viscosity decreased but without hard packing. The metal powder could be redispersed easily and quickly by stirring the settled slurry with a spatula. The supernatant liquid was viscous and stringy, which indicated that gel structure was still present. This supernatant layer was black, probably because the smallest particles of magnesium remained suspended while the larger particles settled. Stringiness disappeared from the slurry when the viscosity became 500 centipoises or lower.

Gelation with soap III at room temperature. - Soap III was found to gel the 50 percent magnesium powder - JP-4 fuel slurries dependably, although at room temperature the time required for gelation was relatively long. As shown in table III, with a soap concentration of 0.6 percent, gelation time for a slurry averaged about $6\frac{1}{2}$ hours. A slurry gelled with soap III was completely homogeneous, free of granulation, and jelly-like in appearance. The Brookfield apparent viscosities are plotted against age of the slurries in figure 6(a) from the data in table VIII(a). In this figure, the useful life of the slurries made with soap III is found to increase progressively with an increase of soap concentration in the range of 0.4 to 0.6 percent soap.

Slurries prepared with soap III at 110° F. - Gelation times for slurries prepared with soap III at 105° to 112° F were considerably reduced as shown in table III. A slurry with 0.6 percent soap had an average gelation time of about $2\frac{1}{2}$ hours at this temperature. The data for Brookfield apparent viscosity against age for slurries made with soap III gelled at higher temperature (presented in table VIII(b)) are plotted in figure 6(b). The data for slurries with 4.0 percent soap III are included in table VIII(b) to facilitate comparison, even though they gelled at room temperature before heat could be applied; the Brookfield apparent viscosity of these slurries remained above 100,000 centipoises for more than 153 days. In the summary plot (fig. 6(b)), it may be seen that a progression of viscosity-age profiles was obtained in which the useful life increased with an increase of soap concentration up to 2.0 percent.

In figure 6(c), the viscosity-age profiles of the slurries with 0.6 percent soap III gelled at room temperature and 110° F are reproduced on a common abscissa scale for comparison. The curve obtained at the higher temperature differs a little at the beginning and the end, but in general the profiles are similar.

Comparison of the three soaps. - Figure 7 is a summary cross plot of the soap concentration against the average useful life of the slurries which shows how the three soaps differed in this respect. The plot for soap I shows that there was a general increase of useful life with increased concentration of gelling agent, but that the response was not uniform. In the range of concentrations examined, the optimum useful life was obtained with 0.6 percent soap I. Soap II slurries produced a curve which showed that the response of useful life in general was almost insensitive to increased concentration of the soap in the range 0.4 to 0.9 percent. In the curve obtained from slurries with soap III gelled at room and elevated temperatures, the increase of useful life was almost directly proportional to the soap concentration between 0.4 and 1.0 percent. The inset curve for soap III, which extends the range of concentration to 2.0 percent, shows that above about 1.0 percent soap the response of useful life to concentration has begun to diminish.

Soap I gelled the slurries at room temperature much more quickly than the other soaps did, and more quickly even than soap III when the latter was used at 110° F. One serious shortcoming found with soap I was, however, the rapid disappearance of gel structure when the viscosity of the slurries had decreased to about 5000 centipoises. Soaps II and III produced slurries which were almost identical in appearance; gelation times for the two soaps at room temperature were somewhat similar.

Regelation of broken slurries from soap III. - By the incorporation of 0.6 percent additional soap, regelation was accomplished quite successfully and very rapidly. For example in table IV, regelation time for seven slurries averaged about 30 minutes (as compared to the original gelation time of about $2\frac{1}{2}$ hours for a slurry with 0.6 percent soap to gel at the same temperature). In figure 8, four representative curves which were selected from the seven regelled slurries are presented in a summary plot of Brookfield apparent viscosity against age from the day of regelation, designated zero. The data for viscosity against age for the seven slurries which were regelled are presented in table IX. Also included in figure 8 are the viscosity-age profiles for the four original slurries, each of which had different initial soap concentrations as indicated. Distance from zero on the abscissa in a minus direction to the lower ends of the curves of the original slurries indicates the number of days each original slurry was broken before regelation was carried out. Reproducibility of results of the regelled slurries is comparable to that obtained from the original slurries. Regelation with a soap concentration of 0.6 percent produced an average useful life (78 days) almost double that obtained from the original slurry (42 days) of the same soap concentration. The useful life of the regelled slurries appeared to be independent of the soap concentration in the original slurries. The tendency for the viscosity of a slurry to decrease irregularly with age, mentioned previously, is strongly demonstrated by the regelled slurries.

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A greater total useful life was obtained from gelation of the original slurry plus regelation of the broken slurry than the useful life that was obtained by gelation alone with the same total soap concentration. For example in figure 8, the total useful life of a slurry obtained by gelation with 0.6 percent soap (42 days) and regelation with 0.6 percent soap (average 78 days) was 120 days for 1.2 percent total soap. This total useful life of 120 days exceeded by 28 days the useful life interpolated for 1.2 percent soap concentration in figure 7 (inset) and exceeded by 10 days even the useful life obtained by gelation with 2.0 percent soap.

Comparison between clear gels and slurries. - In figure 9, Brookfield apparent viscosity data, which are presented in table X for samples of JP-4 fuel gelled with 0.8, 1.2, 2.0, and 4.0 percent soap III, are plotted against age of the samples in days. Superimposed as dashed lines in each of the figures are curves from gelled slurries containing the same ratio of JP-4 fuel to soap. For example, a 50 percent magnesium powder - JP-4 slurry with 0.4 percent soap has a soap concentration of 0.8 percent with respect to the JP-4 fuel. The Brookfield apparent viscosity of the different samples of gelled JP-4 fuel was found to rise to a maximum, then decrease to a viscosity slightly higher than the original, and finally, in most cases, to remain constant for a considerable length of time beyond the useful life of the corresponding slurry. The difference in behavior between the slurries and the gelled JP-4 fuel indicates that in the presence of the magnesium powder some reaction of the soap occurred which resulted in the more rapid deterioration of the gel structure. Some products of this hypothetical reaction might be responsible for the greatly reduced regelation time and the modified viscosity-age profiles of the regelled slurries.

Viscosity behavior of gelled JP-4 fuel and slurries at high rates of shear. - The data obtained by means of the Severs rheometer from a few selected gelled JP-4 fuel samples and magnesium slurries are plotted as reciprocal of rate of shear in seconds against apparent viscosity in centipoises in figure 10. The respective samples are identified in the legend by the soap concentration for each of the respective samples. Brookfield apparent viscosities which were taken prior to Severs rheometer determinations are presented for comparison. The residual viscosities were determined by drawing a straight line through the data obtained from each of the samples and extrapolating to the abscissa (ref. 6) where the rate of shear is infinite. The residual viscosities all fall within a narrow range of 23 to 49 centipoises. It can be seen that there is no relation in this range of shear rate between the initial apparent viscosity (Brookfield) and the rate of decline of the apparent viscosity with increasing rate of shear. Thus it can be seen that in fuel systems exerting high rates of shear, these viscous materials can become quite fluid despite the presence of 50 percent metal content and the gel structure. In fact, the residual viscosities begin to approach the viscosity of conventional liquid hydrocarbon fuels.

Effect of batch size on the viscosity-age characteristics of slurries with soap III. - The data for Brookfield apparent viscosity against age which were obtained from small samples removed from the larger-scale slurries are presented in table XI and are plotted in figure 11. The viscosity-age profile for laboratory-scale slurries is included in the figure for comparison. The curve for samples of the large-scale slurries falls below, but is similar to, the curve for the laboratory-scale slurries. The useful life was 45 days compared to 47 days for the laboratory-scale slurries of like composition. However, two larger batches of slurries, which had been kept sealed in their original containers with ambient temperature ranging from 0 to 100° F, were examined at $5\frac{1}{2}$ months and found to be still usable; in fact, one had a Brookfield apparent viscosity of 25,000 centipoises and had settled very little. This increased useful life, which had been observed on other larger batches too, was not detected soon enough to obtain data.

Effect of exhaustive shearing on a gelled magnesium slurry. - The data obtained from samples of the gelled slurry which had been sheared in a colloid mill for successively increased periods of time are presented in figure 12. The Brookfield apparent viscosity in centipoises is plotted against time after shear. The scale of the abscissa is extended to show that the viscosity is recovered completely if sufficient time is allowed. A family of curves was obtained which demonstrate that the viscosity decreased markedly with increased time of milling. The recovery of the apparent viscosity with time was slower as milling time was increased. At the end of 6 hours all the samples had regained 50 percent or more of the original viscosity and by 105 hours had reached an average apparent viscosity of

27,000 centipoises, which is slightly higher than the original viscosity, 26,000 centipoises. These data show that a slurry with an initially high apparent viscosity may be altered temporarily to a fluid of quite low apparent viscosity by the application of high rates of shear.

SUMMARY OF RESULTS

An exploratory investigation was carried out to determine the gelling properties and stabilizing ability of three aluminum disoaps (designated I, II, and III) in slurries of 50 percent magnesium (average particle size, 15 microns) and MIL-5624A, grade JP-4 fuel. Soap I was reported to be made from a mixture of monocarboxylic C_8 acids while soaps II and III, which were supposed to be alike, were reported to be made from 2-ethylhexoic acid. The results obtained in the investigation are summarized as follows:

1. Formation of gel structure was always consistent for duplicate samples. The viscosity-age profiles and the useful life of the slurries were fairly reproducible.

2. With each of the three soaps investigated, slurries were obtained which had a useful life in excess of 20 days. With soap III a useful life of 110 days was achieved using a soap concentration of 2 percent.

3. Soap I produced a maximum useful life at 0.6 percent soap concentration; soap II showed no concentration effect in the range 0.4 to 0.9 percent soap; and soap III exhibited progressively longer useful life with increased soap concentration over the range 0.3 to 4.0 percent soap.

4. Although soaps II and III were supposed to be alike in composition and preparation and showed similar inspection data, they exhibited different behavior with regard to concentration and useful life in this investigation.

5. The fluidity of all slurries investigated at high rates of shear was estimated to be adequate to permit pumping and handling.

6. After partial or complete break of gel structure in several slurries stabilized with soap III, the slurries were regelled by adding 0.6 percent more soap III to the redispersed slurry. The regelled slurries exhibited a useful life (average of 78 days) which was nearly double that of the original slurry with the same soap concentration. Reproducibility of properties with regelled slurries was comparable to that for the original slurries, and the useful life of the regelled slurries was independent of the original soap concentration in the broken slurries.

7. By gelation of a slurry and subsequent regelation when the original slurry broke, it was demonstrated that greater total useful life could be obtained over gelation alone with an equal total concentration of soap III.

8. Small samples removed from larger batches of slurry were similar to the laboratory samples with regard to physical appearance and viscosity-age profile, but the useful life of larger batches which were kept sealed appeared to be greater than the smaller samples indicated.

9. A technique was demonstrated whereby slurries with high apparent viscosity could be temporarily rendered more fluid by subjecting the slurry to high shear for a few minutes.

CONCLUDING REMARKS

An investigation of the gelling properties and stabilizing ability of three aluminum disoaps of C_8 acids in slurries of 50 percent magnesium powder - JP-4 fuel showed that these soaps are much improved over previous soaps of this type. Presumably the improvement results from the precipitation of the soaps from aluminum chloride instead of from aluminum sulfate. The improved soaps offer considerable promise where the magnesium to be used in slurries of JP-4 fuel has an average particle size that requires a thickener for stabilization. Slurries stabilized with these gelling agents have several desirable characteristics:

1. Preparation and equipment required, even for larger-scale slurries, are straightforward and simple.

2. Properties such as the apparent viscosity - age profile and the useful life of duplicate batches are similar.

3. Useful life and viscosity-age profile can be predicted within limits and controlled by the selection of soap concentration. By means of regelation the useful life of gel-stabilized slurries can be extended several months.

4. Regelling agents and equipment required for regelation are the same as for the preparation of the original slurry.

5. Viscosities at the high shear rates, believed to be similar to those encountered in a fuel system, are such that pumping and handling may not present great problems.

Two undesirable characteristics observed were: (1) the rather high apparent viscosity that exists for the first few weeks in the life of a stable slurry, and (2) the eventual breaking of the gel structure. It was demonstrated that (1) could be reduced by the application of high rates of shear to a slurry and (2) could be alleviated by regelation.

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National Advisory Committee for Aeronautics
Cleveland, Ohio, March 12, 1954 3079

APPENDIX - GLOSSARY OF TERMS

In presenting data and discussing the results of the present investigation, certain terms or phrases are used frequently. These expressions are defined below as they are used herein to describe the behavior of the JP-4 fuel or the magnesium slurries in JP-4 which were gelled by means of three soaps of C_8 acids.

Gelation time. - Time elapsed from the addition of soap to JP-4 fuel or slurry until thickening of the mixture was observed. For the slurries, the degree of thickening was required to be adequate to support the magnesium powder.

Thixotropic. - A thixotropic substance is one whose apparent viscosity decreases under the influence of shear and requires a measurable time to return to its normal level when shear is removed.

Gel structure. - The submicroscopic structure formed by the soap particles throughout the hydrocarbon. Visually, this was observed by the presence of stringiness and increased viscosity.

Useful life. - Age of a slurry after gelation at which the viscosity has decreased to 1000 centipoises, Brookfield apparent viscosity. (This viscosity, although arbitrarily selected for comparison of the gel structure longevity of the soaps in slurries, is capable of holding the magnesium powder fairly well suspended for a few hours. Thus, such a value has some practical significance for fuel use in experimental combustion work.)

Breaking of gel. - Loss of stringiness in a gelled slurry with accompanying loss of ability to support magnesium particles more than a few minutes.

Gel-break time. - The age at which breaking of the gel in a slurry occurs.

Viscosity-age profile of slurry. - Smooth curve of Brookfield apparent viscosity against age of a slurry throughout the useful life of a slurry.

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TABLE I. - SOURCE AND ANALYSIS OF SOAPS

Soaps	Ash, weight percent			Acid, weight percent				Moisture, weight percent
	Total ash		Water-soluble salts	Total acid		Acetone-soluble[b]	Isooctane-soluble[b]	
	Theory[a]	Found[b]		Theory[a]	Found[b]			
I	15.4	17.6	0.2	87.0	76.0	4.3	0.1	1.47
II	15.4	16.1	.1	87.0	80.8	.7	.6	1.63
III	15.4	16.7	.2	87.0	81.8	.5	.1	1.56

[a]Based on a formula of aluminum hydroxy dioctoate.

[b]Values reported are based on the soaps as received and not on dry-weight basis.

TABLE II. - FUEL ANALYSIS

Fuel properties	JP-4 fuel
Distillation, °F	
Initial boiling point	152
Percentage evaporated	
5	199
10	221
20	245
30	266
40	283
50	301
60	322
70	345
80	379
90	429
95	465
Final boiling point	488
Residue, percent	1.3
Aromatics (by silica gel), percent by volume	10.50
Reid vapor pressure, lb/in.^{2}	2.3
Specific gravity, 60°/60° F	0.768
Hydrogen-carbon ratio	0.170
Aniline point, °F	137.5
Bromine number	0.15

TABLE III. - PREPARATION OF SMALL-SCALE SLURRIES

Concentration of gelling agent, weight percent	Gelation temperature, °F	Stirring interval[a], min	Rolling interval, min	Gelation time, min
Soap I				
0.30	76-78	23	1350	No gel
		164	0	No gel
.40	76-78	52		52
		159		159
.50	76-78	27		27
		35		35
.60	76-78	12		12
		9		9
.70	76-78	11		11
	76-78	11		11
	80-82	8		8
	80-82	8		8
	80-82	8		8
	80-82	8		8
.80	76-78	7		7
		9.5		9.5
Soap II				
0.40	73-78	13	1245	1258
		14	1207	1221
.50	73-78	49	112	161
		16	974	(b)
.60	73-78	31	296	327
		32	265	297
.70	73-78	31	None	31
		139	None	[c]107
.90	73-78	9	None	9

[a]Stirring interval is total time from admixture of soap and JP-4 fuel until the slurry is removed from the constant-speed stirrer; stirrer speed was 300 rpm.

[b]Gelation time was not observed.

[c]Stirring was allowed to continue beyond the observed gelation time.

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TABLE III. - Concluded. PREPARATION OF SMALL-SCALE SLURRIES

Concentration of gelling agent, weight percent	Gelation temperature, °F	Stirring interval[a], min	Rolling interval, min	Gelation time, min
Soap III				
0.40	78	326 11	1036 1195	1362 1206
.50	78	7 10	1398 1384	1405 1394
.60	78	11 11	385 395	396 406
.30	110-112	9 8.5	1099 1105	1108 1113
.40	110-112	10 11	166 121	176 132
.50	110-112	7 7	179 171	186 178
.60	110-112	6.5 7	166 130	172 137
.70	110-112	11 11	149 121	160 132
1.00	104-106	10 10	42 42	52 52
2.00	105-107	10 10	28 21	38 31
4.00	80-82	7 7	None None	[d]1.0 [d]0.75

[a]Stirring interval is total time from admixture of soap and JP-4 fuel until the slurry is removed from the constant-speed stirrer; stirrer speed was 300 rpm.

[d]JP-4 fuel was gelled stiff before removal from the Osterizer.

[redacted]

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TABLE IV. - REGELATION OF SLURRIES WITH SOAP III

[Temperature, 110° to 112° F.]

Status of original slurry, days broken, (or apparent viscosity, centipoises)	Soap III			Stirring interval[a], min	Rolling interval, min	Gelation time, min
	Original concentration, weight percent	Additional concentration for regelation, weight percent	Total concentration, weight percent			
24 days	0.30	0.60	0.90	5.0	25.5	30
24 days	.30	.60	.90	4.5	26.0	30
10 days	.40	.60	1.00	3.5	30.0	33
13 days	.40	.60	1.00	3.5	25.0	28
900 centipoises	.50	.60	1.10	3.0	19.5	22
6 days	.50	.60	1.10	3.0	26.5	29
1300 centipoises	.60	.60	1.20	3.5	23.0	26

[a]Stirrer speed was 1800 rpm.

TABLE V. - PREPARATION OF CLEAR GELS WITH JP-4 FUEL AND SOAP III

Soap III, weight percent	Gelation temperature, °F	Stirring interval[a], min	Rolling interval, min	Gelation time, min
0.8	106	8.0	150	158
	106	8.0	142	150
1.2	106	8.0	125	133
	106	8.0	113	121
2.0	75	113		[b]113
	106	9.0	54	63
4.0	106	[c]2.5	10.5	13
	106	[c]2.5	8.5	11

[a]Stirring interval is total time from admixture of soap and JP-4 fuel until sample was removed from the constant-speed stirrer; stirrer speed, 1800 rpm.

[b]Gelation time was demarcated by the disappearance of the vortex on the stirrer shaft.

[c]Sample was stirred only on the Osterizer and transferred directly to the rollers.

TABLE VI. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR 50-PERCENT MAGNESIUM SLURRIES STABILIZED WITH DIFFERENT CONCENTRATIONS OF SOAP I

[Gelation at room temperature.]

Age of slurries, days	Brookfield apparent viscosity, centipoises													
	Concentration of soap in samples, percent													
	0.4		0.5		0.6		0.7						0.8	
	A	B	A	B	A	B	A	B	C	D	E	F	A	B
1	5500	(a,b)	25,800	38,500	47,800	52,800	27,400	31,100	30,700	21,800	14,000	20,000	59,200	47,100
2			17,200	15,700	46,500	33,800	18,700	18,300	35,000	18,200	24,000	26,000	40,000	29,100
3			6,800	5,300	55,300	53,300			23,000	12,000	12,000	11,800		
4					46,000	53,800								
5							7,000	14,300					23,600	20,800
6			2,700	3,400			9,800	5,500	9,400	3,900	4,500	4,500	18,100	14,500
7			(b)	(b)	27,800	47,300	7,400	9,000					26,900	11,400
8					26,400	31,800	6,800	9,000	3.900	2,000	2,500	3,000	19,600	14,000
9					31,500	33,500	4,400	7,900					20,700	14,500
10					29,000	31,000								
11					23,000	30,000								
13							(b)	(b)	(b)	(b)	(b)	(b)	8,500	5,400
15					11,300	20,000							12,900	3,800
16														4,700
17						15,500								
18					8,700	14,000								
19													8,200	8,300
20													6,500	(b)
21					10,900	10,000							5,600	
22						8,200							3,500	
23					3,800	12,200							3,300	
24					5,200	9,500								
25					8,100	8,200								
26													(b)	
28					4,200	7,300								
31					1,700	4,400								
32					(b)	2,500								
35						(b)								

[a]Gelation occurred when the slurry was prepared but slurry had "broken" before 1 day.

[b]Gel structure in slurry was observed to be broken on the day indicated.

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TABLE VII. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR 50-PERCENT MAGNESIUM SLURRIES STABILIZED WITH DIFFERENT CONCENTRATIONS OF SOAP II

[Gelation at room temperature.]

Age of slurries, days	Brookfield apparent viscosity, centipoises								
	Concentration of soap in samples, percent								
	0.4		0.5		0.6		0.7		0.9
	A	B	A	B	A	B	A	B	A
1	48,700	23,000	79,700	>100,000	>100,000	>100,000	>100,000	>100,000	>100,000
2	>100,000	95,000		>100,000	>100,000	>100,000		>100,000	
3	>100,000	89,000		>100,000	>100,000	>100,000	100,000	>100,000	
4	97,500	100,000	97,500	>100,000			>100,000		82,700
5			80,000				80,800		96,500
6			69,000				99,000		>100,000
7			55,500		93,000	89,500	54,500	>100,000	94,500
8	46,100	76,800	65,000	73,300			86,700	86,000	88,600
9					88,900	58,500		75,700	
10	30,000	49,300		47,000	71,800	40,700		47,000	
11	22,800	28,500		49,700					
12			12,700				25,500		37,200
13			23,500		46,300	28,000	17,200	33,300	
14	14,500	20,300	17,500	20,800	55,500	34,800	12,700	31,500	16,600
15		18,000	12,000	18,100	21,600	19,000	4,900	18,400	
16	10,400	12,500		11,200	22,000	13,000		14,000	8,500
17	8,600	11,300		14,000	11,900	5,100		8,200	
18	4,800	6,400	9,300	5,700			3,100		4,600
19							1,700		3,000
20			4,800		1,500	1,200	1,200	1,600	2,200
21	1,000	1,200	2,600	1,500		(a)	800		2,000
22	(a)		1,800	(a)			800		1,500
23					1,200			1,200	
24		600			700			700	
25		700	1,000				800		1,000
26					500		(a)		
27					500				
28		500	500					500	1,000
29									600
32									500

[a]Sample was destroyed in other tests.

[redacted]

TABLE VIII. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR 50-PERCENT MAGNESIUM SLURRIES STABILIZED WITH DIFFERENT CONCENTRATIONS OF SOAP III

(a) Gelation at room temperature

Age of slurries, days	Brookfield apparent viscosity, centipoises					
	Concentration of soap in samples, percent					
	0.4		0.5		0.6	
	A	B	A	B	A	B
1	29,700	8,900	29,500	44,400	38,600	29,300
4	28,800	34,500	>100,000	86,300	>100,000	>100,000
5	31,300	31,900	76,500	88,000		
6	34,800	26,500	88,200	>100,000		
7	33,000	47,000	64,000	84,000		
8	27,100	36,000	83,600	69,000	>100,000	>100,000
11	20,500	32,800	35,200	46,500	82,300	79,300
14	19,000		31,500	39,500	75,500	66,000
15	16,000	23,000	30,500	33,300	58,800	62,000
18	11,500	23,000	24,000	33,200	48,500	62,300
20	6,200	17,900	12,800	23,100	61,800	47,100
25	2,000	9,900	7,100	9,800	26,000	15,000
27	600	2,500	6,200	12,800	16,600	13,800
29	500	1,200	3,600	6,400	16,000	13,600
32		500	3,200	9,500	13,800	10,500
34			900	3,800	9,900	13,300
36			500	1,300	6,900	4,700
38				1,200		
39					3,000	2,800
40				900		
41			(a)	(a)	1,400	1,300
42	(a)	(a)				(a)
43					1,000	
46					800	
49					600	
53					700	
55					600	
57					600	
60					600	
63					500	

[a]Slurry was regelled on day indicated. (See table IX.)

TABLE VIII. - Continued. BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR 50-PERCENT MAGNESIUM SLURRIES STABILIZED WITH DIFFERENT CONCENTRATIONS OF SOAP III

(b) Gelation temperature, about 110° F

Age of slurries, days	Brookfield apparent viscosity, centipoises									
	Concentration of soap in samples, percent									
	0.3		0.4		0.5		0.6		0.7	
	A	B	A	B	A	B	A	B	A	B
1	1000	1000			50,000	54,000	79,000	81,000	86,000	84,000
2	600	600			41,200	45,800	77,000	77,500	83,400	86,800
3			14,500	14,000						
5	500	500	10,300	9,500	43,500	39,800	86,700	80,800	88,600	89,500
7					35,000	41,000	87,200	81,400	88,000	87,000
10			5,000	4,000						
12			7,000	4,600	28,100	27,900	78,000	72,300	85,300	77,400
14			8,400	6,000	22,700	23,000	65,700	59,400	70,000	59,500
16					21,000	21,500	60,000	59,000	66,000	62,500
17			5,500	1,800						
19			3,100	1,000	17,500	21,300	50,400	46,200	63,800	62,000
21			2,000	900	11,500	30,000	44,200	45,800	50,500	51,500
23					20,200	18,000	36,900	38,300	48,700	46,500
24	(a)	(a)	1,300	900						
26			1,000	800	11,000	15,400	31,000	34,800	44,000	44,000
28			700	600	8,800	8,200	28,000	29,200	42,000	36,300
30					13,000	8,700	(b)	24,700	37,200	32,500
31			800	800						
33					6,000	5,200		18,600	36,500	26,800
34			800	700						
36					1,800	1,800		14,100	27,300	19,800
38			600	600						
40			600	600	800	700		8,000	21,000	12,000
42			600	600	700	700		6,800	16,800	8,300
44					700	700		2,100	11,000	2,100
45			500	500						
47					700	600		1,200	4,200	1,500
50					500	500		900	2,300	1,000
54								800	1,400	800
57								800	1,700	900
61								800	3,200	1,200
64								900	2,100	1,500
68								800	1,600	900
72								800	1,100	1,000
75								500	500	500

[a]Slurry was regelled on day indicated. (See table IX.)

[b]Sample was destroyed in other tests.

[redacted]

TABLE VIII. - Concluded. BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR 50-PERCENT MAGNESIUM SLURRIES STABILIZED WITH DIFFERENT CONCENTRATIONS OF SOAP III

(b) Gelation temperature, about 110° F

Age of slurries, days	Brookfield apparent viscosity, centipoises					
	Concentration of soap in samples, percent					
	1.0		2.0		[a]4.0	
	A	B	A	B	A	B
2	>100,000	>100,000	>100,000	>100,000	>100,000	>100,000
10	↓	↓	↓	↓	↓	↓
17	↓	↓	↓	↓	↓	↓
23	↓	↓	↓	↓	↓	↓
30	↓	↓	↓	↓	↓	↓
37	>100,000	>100,000	↓	↓	↓	↓
48	38,000	19,500	↓	↓	↓	↓
51	38,000	19,500	↓	↓	↓	↓
55	34,000	20,000	↓	↓	↓	↓
59	26,400	14,100	↓	↓	↓	↓
62	16,500	10,700	↓	↓	↓	↓
66	30,000	11,000	↓	↓	↓	↓
69	10,800	7,700	↓	↓	↓	↓
73	7,700	2,600	↓	↓	↓	↓
76	2,000	1,400	14,700	23,000	↓	↓
80	1,500	1,000	25,000	30,000	↓	↓
84	1,000	1,000	25,000	24,000	↓	↓
87	700	700	16,000	18,000	↓	↓
92	900	1,200	3,700	3,500	↓	↓
94	500	500	2,200	2,500	↓	↓
98			1,700	1,700	↓	↓
101			1,500	1,500	↓	↓
105			1,200	1,100	↓	↓
111			800	900	↓	↓
118			600	700	↓	↓
120			500	500	↓	↓
153					>100,000	>100,000

[a]Slurries were gelled at room temperature.

TABLE IX. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR SLURRIES REGELLED WITH 0.6 PERCENT ADDITIONAL SOAP III

[Regelation temperature about 110° F.]

Age of slurries, days	Brookfield apparent viscosity, centipoises						
	Original concentration of soap in samples, percent						
	0.3		0.4		0.5		0.6
	A[a]	B	A[a]	B	A[a]	B	A[a]
1	27,000	27,000	11,800	9,000	7600	12,000	19,100
4	22,500	30,500	18,700	13,500	8300	18,300	19,400
7	15,000	23,500	11,700	9,500	8300	14,000	20,000
11	10,000	20,000	7,000	5,400	3500	8,000	8,000
13	6,400	14,500	4,000	4,000	3400	5,700	8,000
15	3,000	5,000	2,600	2,500	2900	3,600	4,800
18	2,000	4,400	2,500	1,800	1800	3,300	4,000
21	1,400	2,300	1,800	1,500	1400	2,100	2,800
25	1,200	1,700	1,500	1,200	1300	1,600	2,300
28	1,300	1,900	1,600	1,200	1200	1,500	2,000
32	1,900	4,300	2,000	1,000	1100	1,500	2,200
35	5,800	11,800	7,400	1,400	1900	4,200	8,700
39	5,500	9,000	4,500	1,700	2500	5,500	9,000
43	6,600	8,500	5,500	4,600	5500	10,200	12,500
46	3,900	8,400	3,100	3,900	3200	8,400	9,000
50	2,100	4,300	2,000	4,100	3200	8,400	9,700
53	1,300	2,000	1,400	2,100	1800	5,100	6,200
57	1,000	1,400	1,400	1,500	1200	2,400	2,800
60	1,200	1,400	1,000	1,100	1100	1,500	1,700
64	1,700	1,300	1,700	1,100	1100	1,500	1,800
68	1,800	1,500	1,500	1,100	1500	1,500	1,600
71	1,200	1,300	1,400	1,300	1700	1,300	1,200
76	1,300	1,600	1,200	1,500	1500	1,600	1,700
78	700	800	700	1,000	700	900	1,000
82	500	600	600	900	500	900	800
85		500	500	500		700	600
89						500	600
91							500

[a]Data for sample are plotted in figure 8.

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TABLE X. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR GELLED JP-4 SAMPLES PREPARED WITH DIFFERENT CONCENTRATIONS OF SOAP III

[Gelation temperature, about 110° F.]

Age of samples, days	Brookfield apparent viscosity, centipoises							
	Concentration of soap in samples, percent							
	0.8		1.2		2.0		4.0	
	A	B	A	B	A	B	A	B
3				900				
5	600	600	1600		3700	5,200	54,400	57,400
6				1000				
7	700	800	2000		5000	9,000	48,800	62,500
9	800	900	1900		9500	12,500	80,500	85,000
10				1800				
11				1700				
12	800	1000	2500	2100	8100	13,600	49,800	64,000
14	1200		2600			14,500	50,000	
16	1000	1000	2900			16,000	80,000	84,000
17				2800				
19	1200		3200			9,100	>100,000	
22	1100	1300	2600			8,700	75,000	
26	1000	1100	3800			15,000	78,000	
28	1000		2500			14,200	>100,000	
30				1900				
33	1000	1200	6500			6,200	75,000	
34				3800				
36	800	1000	4500			10,000	85,000	>100,000
40	800	900	2400			8,300	70,000	
41				5200				
47	900	800	2200			6,000	60,000	
49				9300				
51			2000					
55	1000	800	(b)	9200		29,000	>100,000	>100,000
64				4800				
65	700	700				8,500	>100,000	
75							35,000	
76	700	700						
77						10,700		
81				3800				
82						13,300	33,000	
83	700	700						
99						4,400	36,000	>100,000
100	No gel	No gel		2000				
114				1400				
118						4,800	35,000	
126				1000				
138						5,400	38,000	

[a]Most of the sample was destroyed in other tests and that remaining was observed up to 55 days and appeared to be the same as the duplicate sample in all respects.

[b]Sample was destroyed in other tests.

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TABLE XI. - BROOKFIELD APPARENT VISCOSITY - AGE DATA FOR LARGER SCALE SLURRIES STABILIZED WITH 0.6 PERCENT SOAP III.

[Gelation at room temperature.]

Age of slurries, days	Brookfield apparent viscosity, centipoises		
	Size of samples, pounds		
	4.6	4.2	100
	A	B	A
1			50,000
2		55,000	
3	48,000		
5			54,500
6		53,000	
7	53,000		56,250
10	45,700		
13		44,700	
14	19,300		38,000
16		40,000	
17	19,400		
20		25,000	
21	24,000		23,000
24		21,500	
25	20,000		
27		19,000	
28	14,000		16,000
31		18,500	
32	15,000		
33		21,500	
35	10,000		10,500
37		7,200	
39	4,800		
40		2,500	
42	1,200		3,000
44		1,200	
46	700		
47			1,000
48		800	
50	700		
51		500	
53	500		

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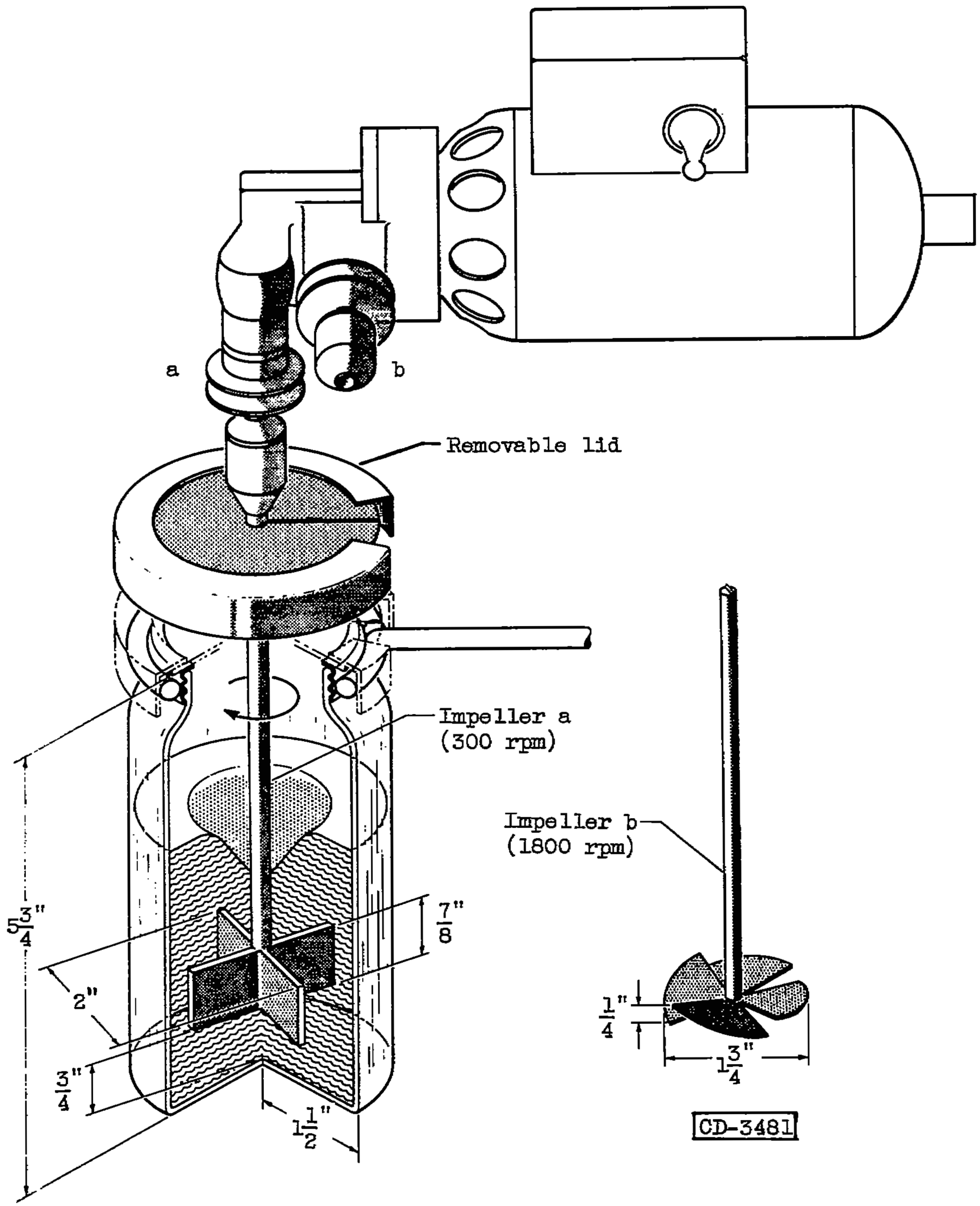

Figure 1. - Constant-speed stirrer assembly.

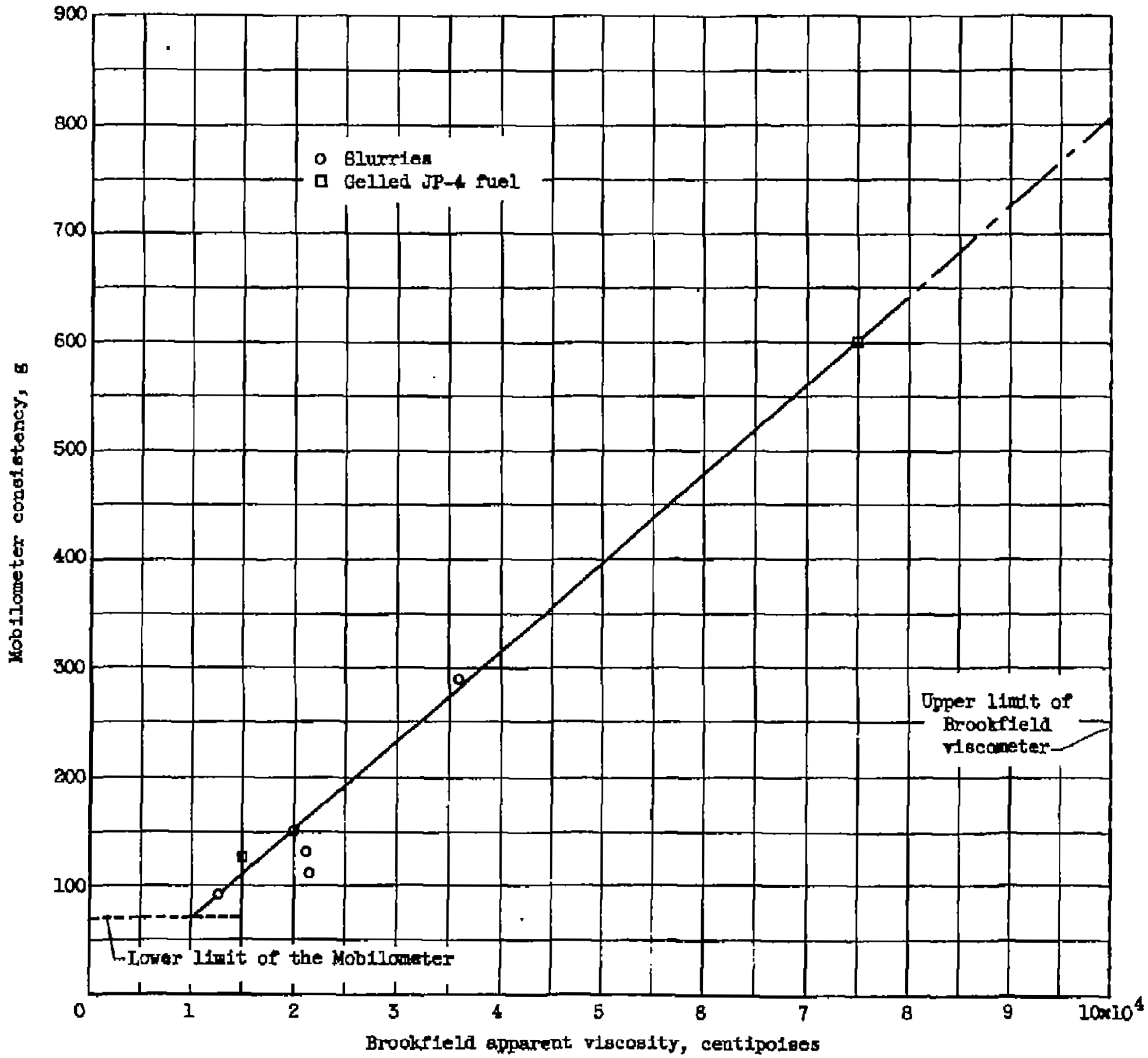

Figure 2. - Relation between data from Gardner Mobilometer and Brookfield viscometer.

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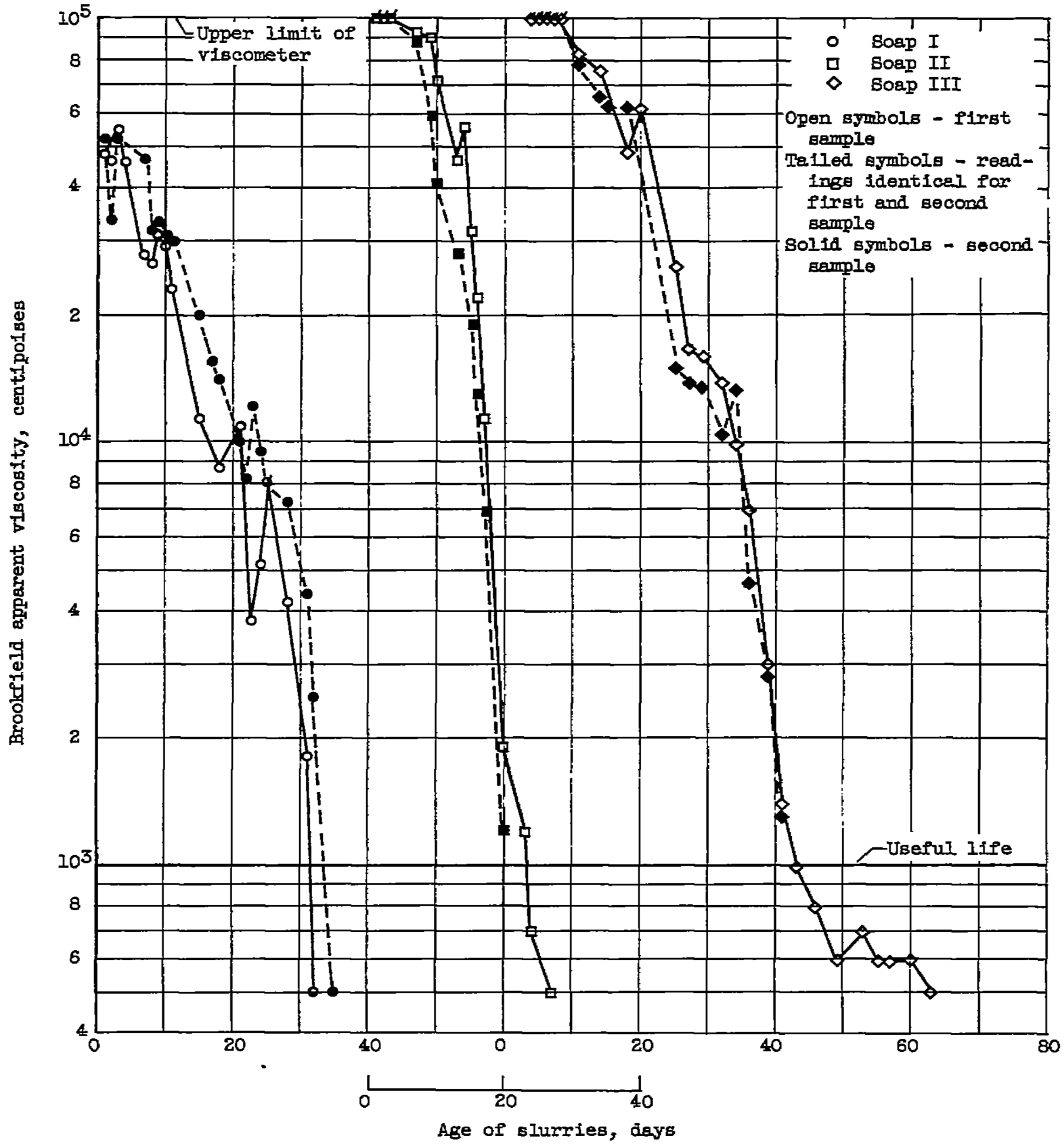
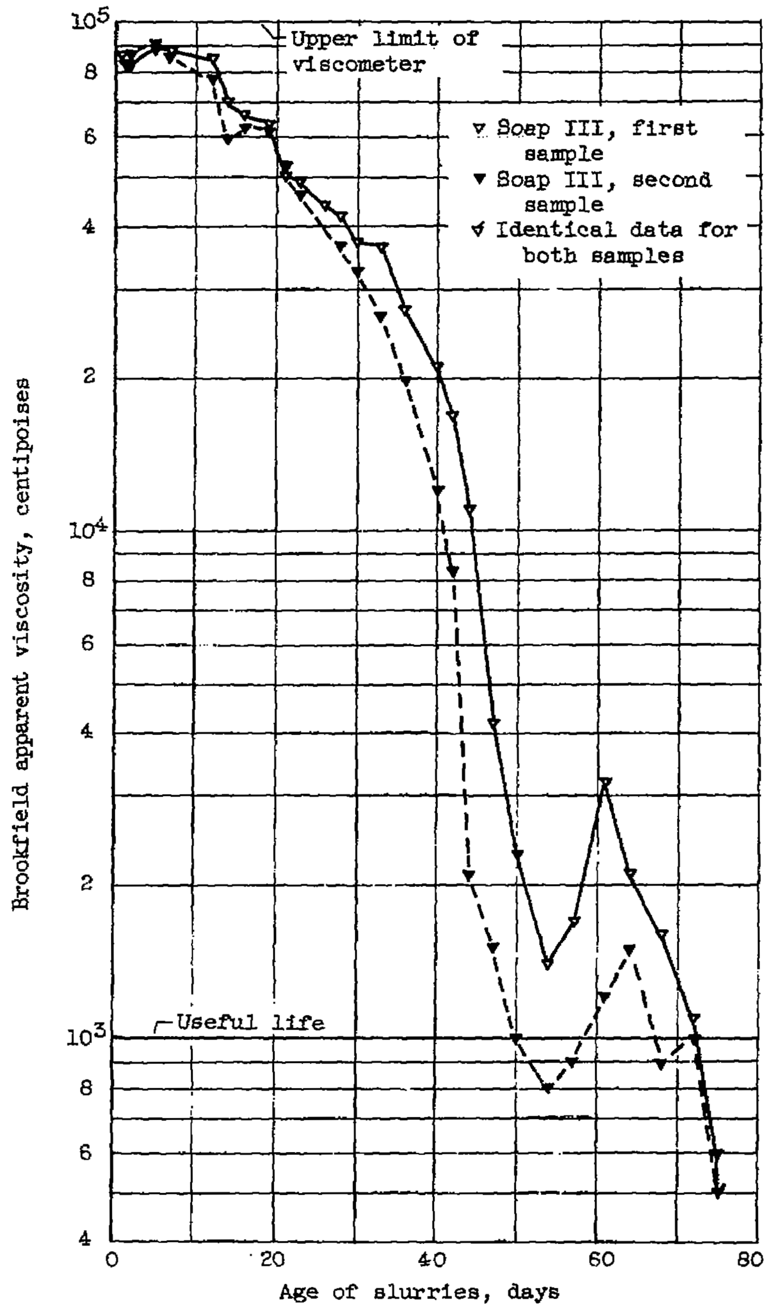
CS-5



(a) Stabilized with 0.6 percent of soap I, II, or III. Gelation at room temperature.

Figure 3. - Typical reproducibility of the variation of apparent viscosity with age for duplicate samples of 50 percent magnesium - JP-4 slurries.



(b) Stabilized with 0.7 percent soap III.
Gelation temperature, about 110° F.

Figure 3. - Concluded. Typical reproducibility of the variation of apparent viscosity with age for duplicate samples of 50 percent magnesium - JP-4 slurries.

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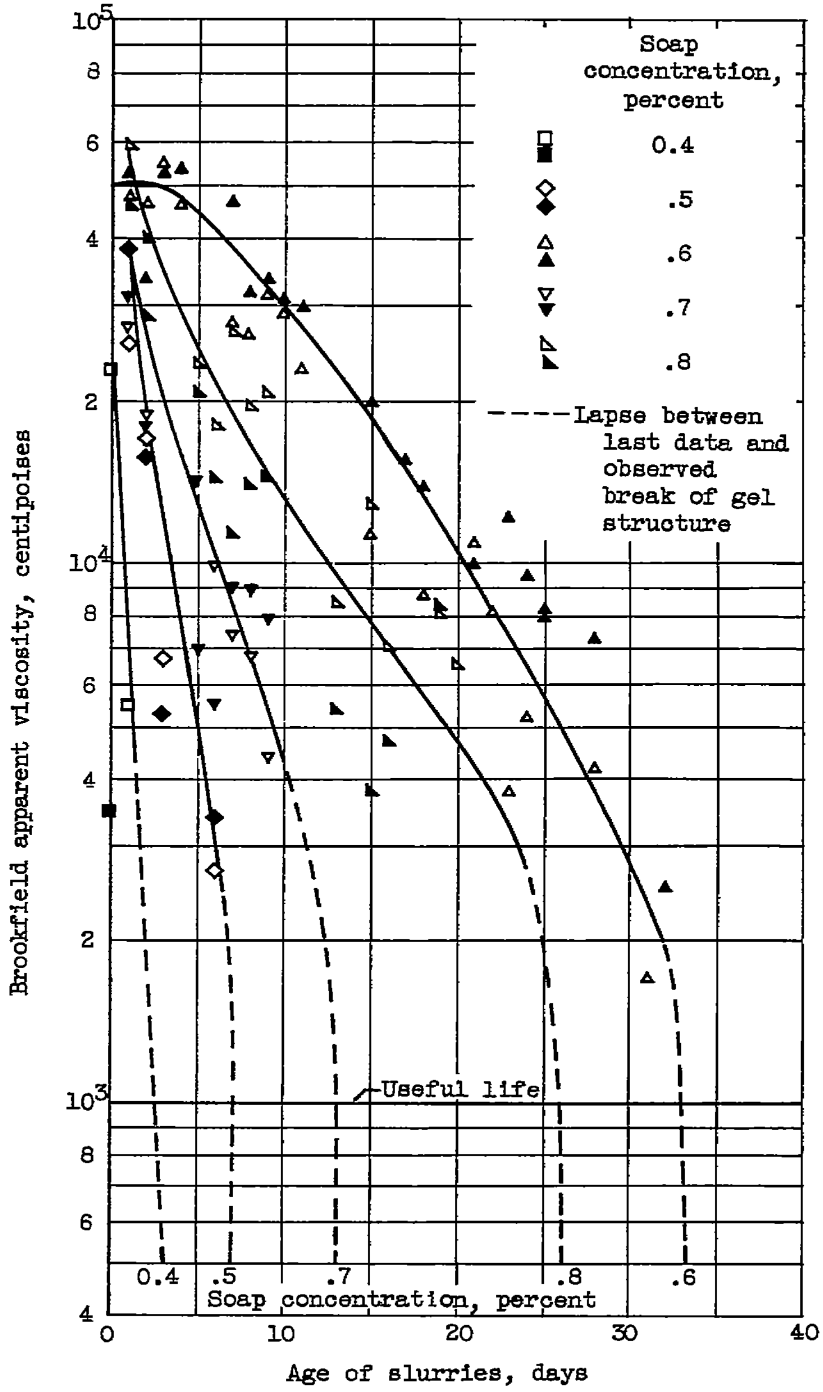

Figure 4. - Viscosity-age profiles of slurries stabilized with different concentrations of soap I. Gelation at room temperature.

[redacted]

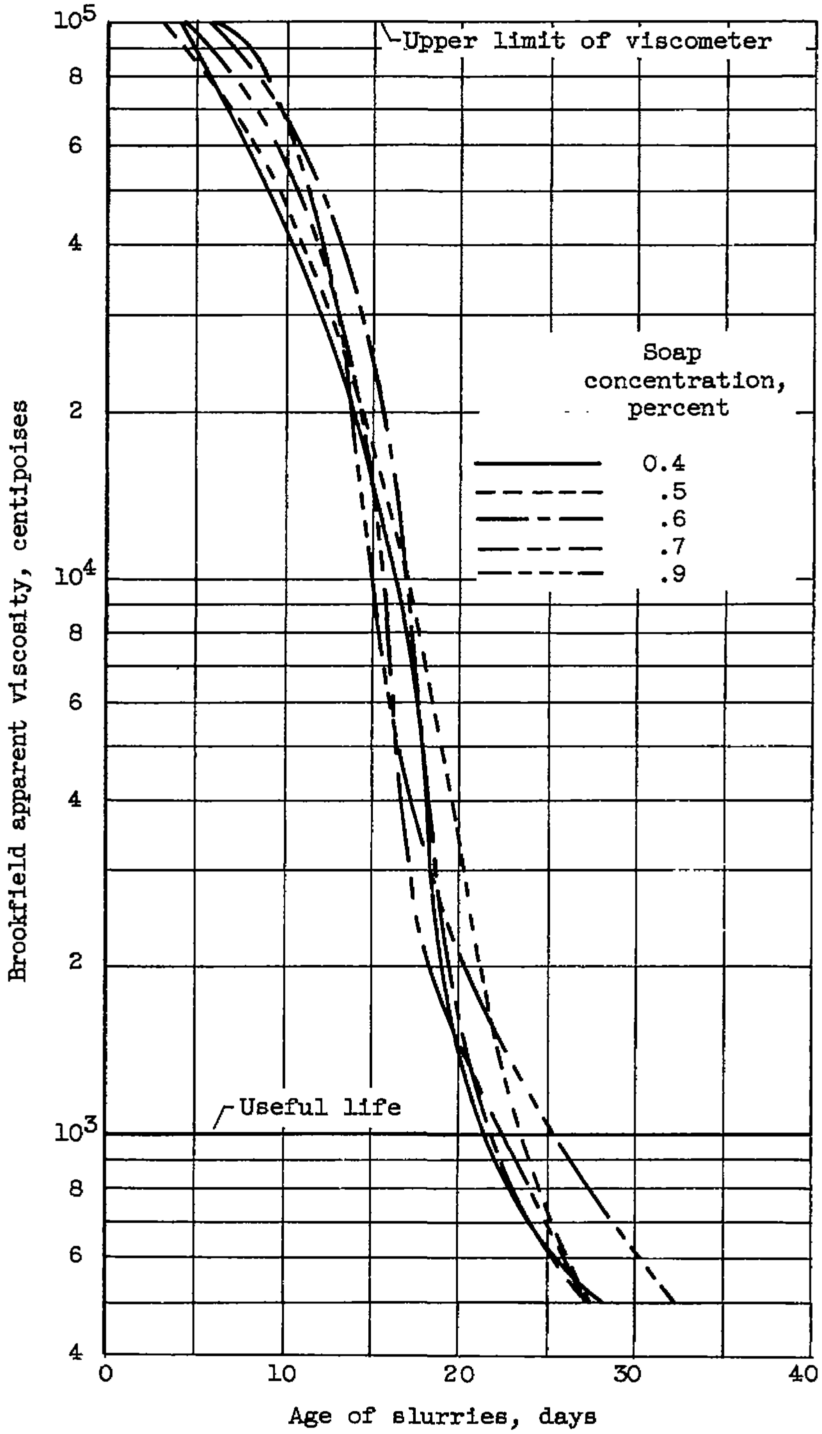

Figure 5. - Viscosity-age profiles of slurries stabilized with different concentrations of soap II. Gelation at room temperature.

[redacted]

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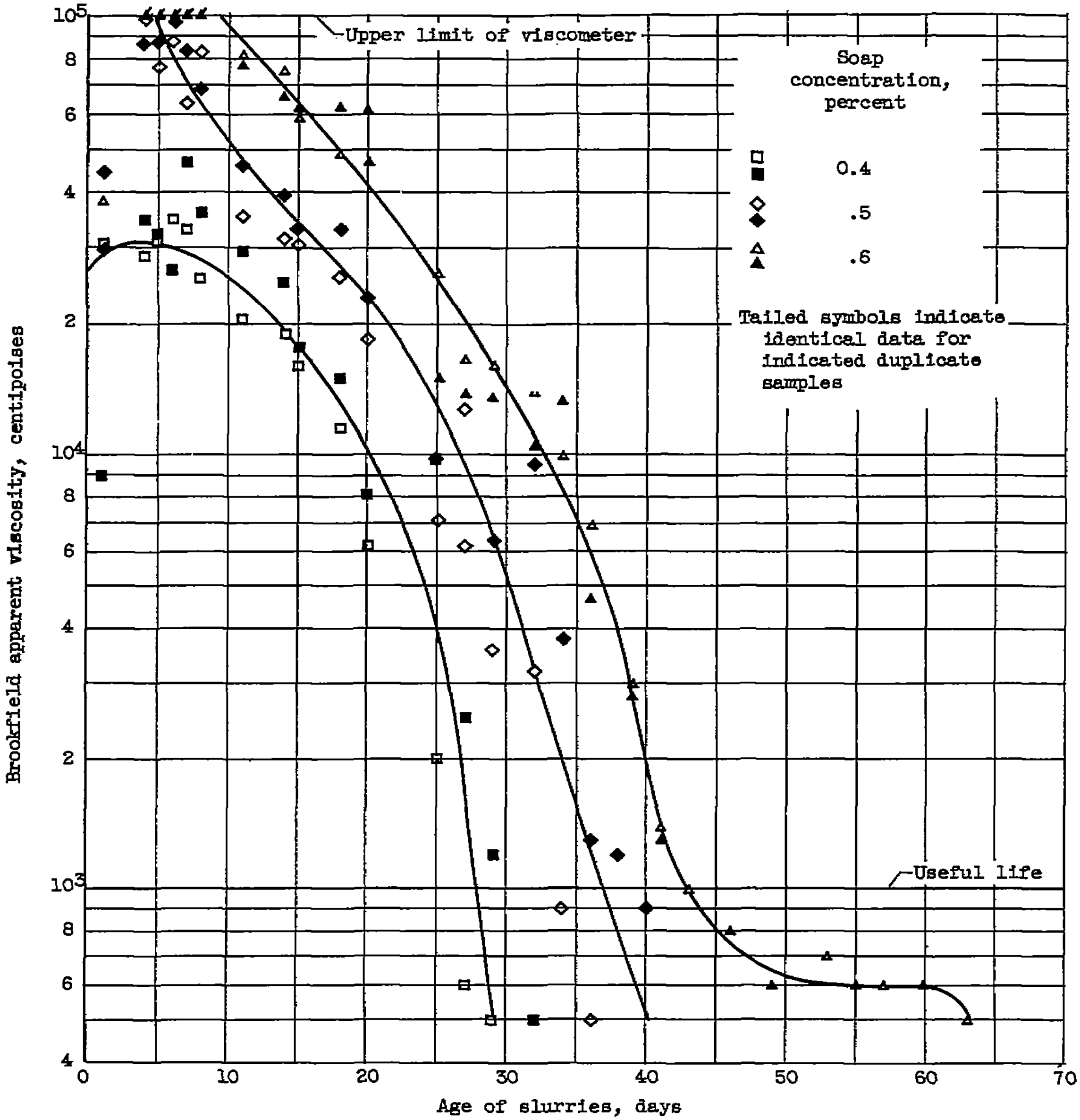

(a) Gelation at room temperature.

Figure 6. - Viscosity-age profiles of slurries stabilized with soap III.

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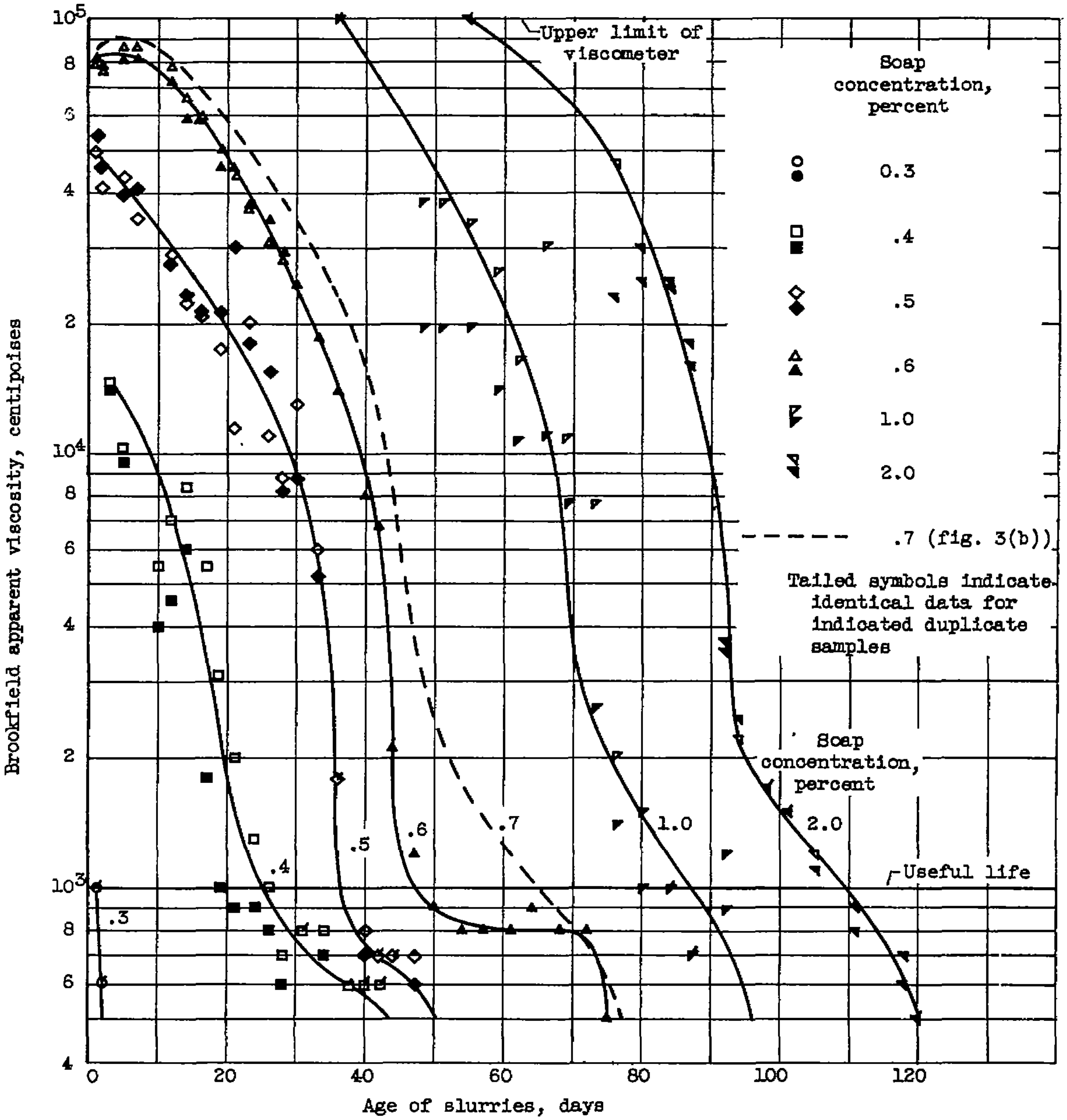

(b) Gelation temperature, about 110° F.

Figure 6. - Continued. Viscosity-age profiles of slurries stabilized with soap III.

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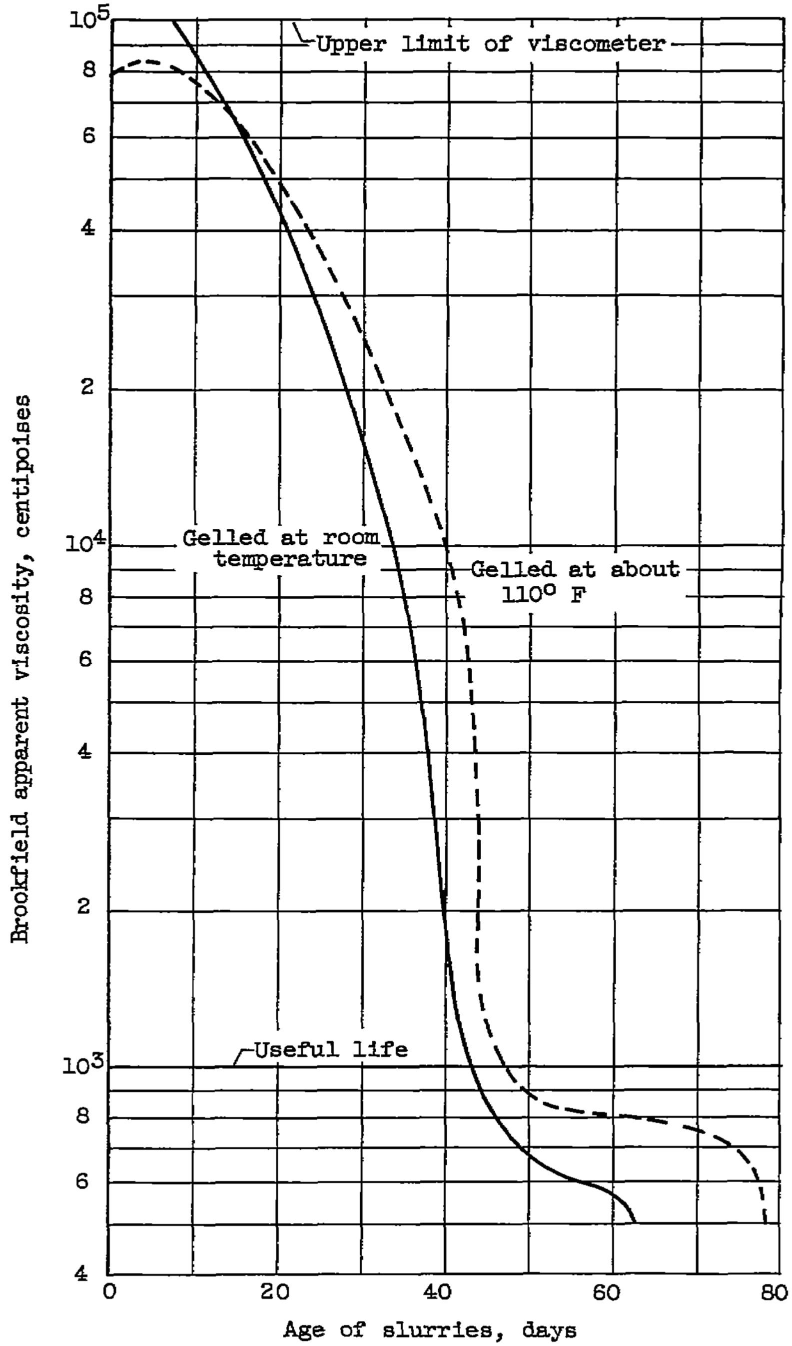

(c) Gelation with 0.6 percent soap at room temperature and about 110° F.

Figure 6. - Concluded. Viscosity-age profiles of slurries stabilized with soap III.

[redacted]

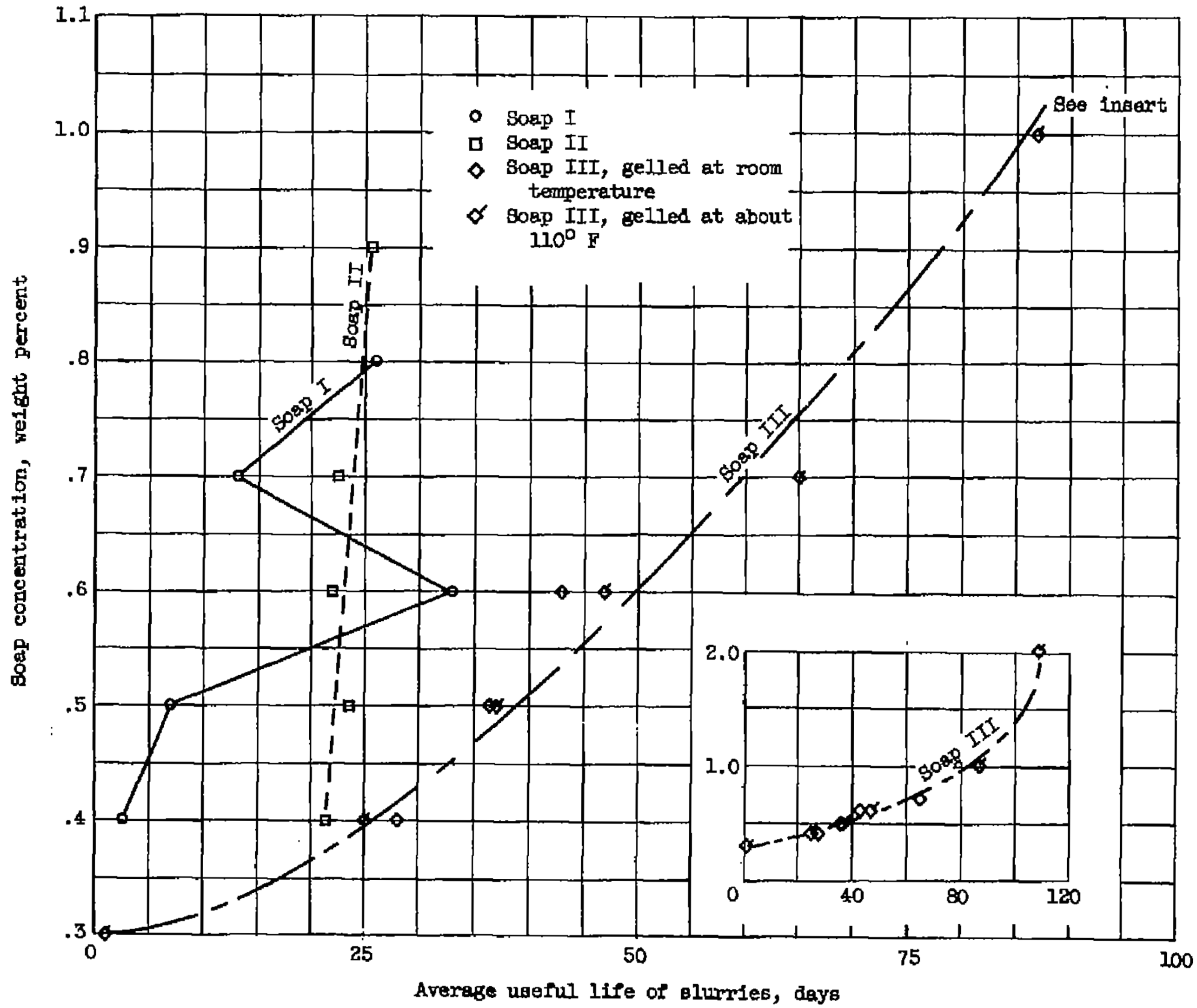

Figure 7. - Effect of soap concentration on the useful life of slurries stabilized with three different soaps.

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CS-6

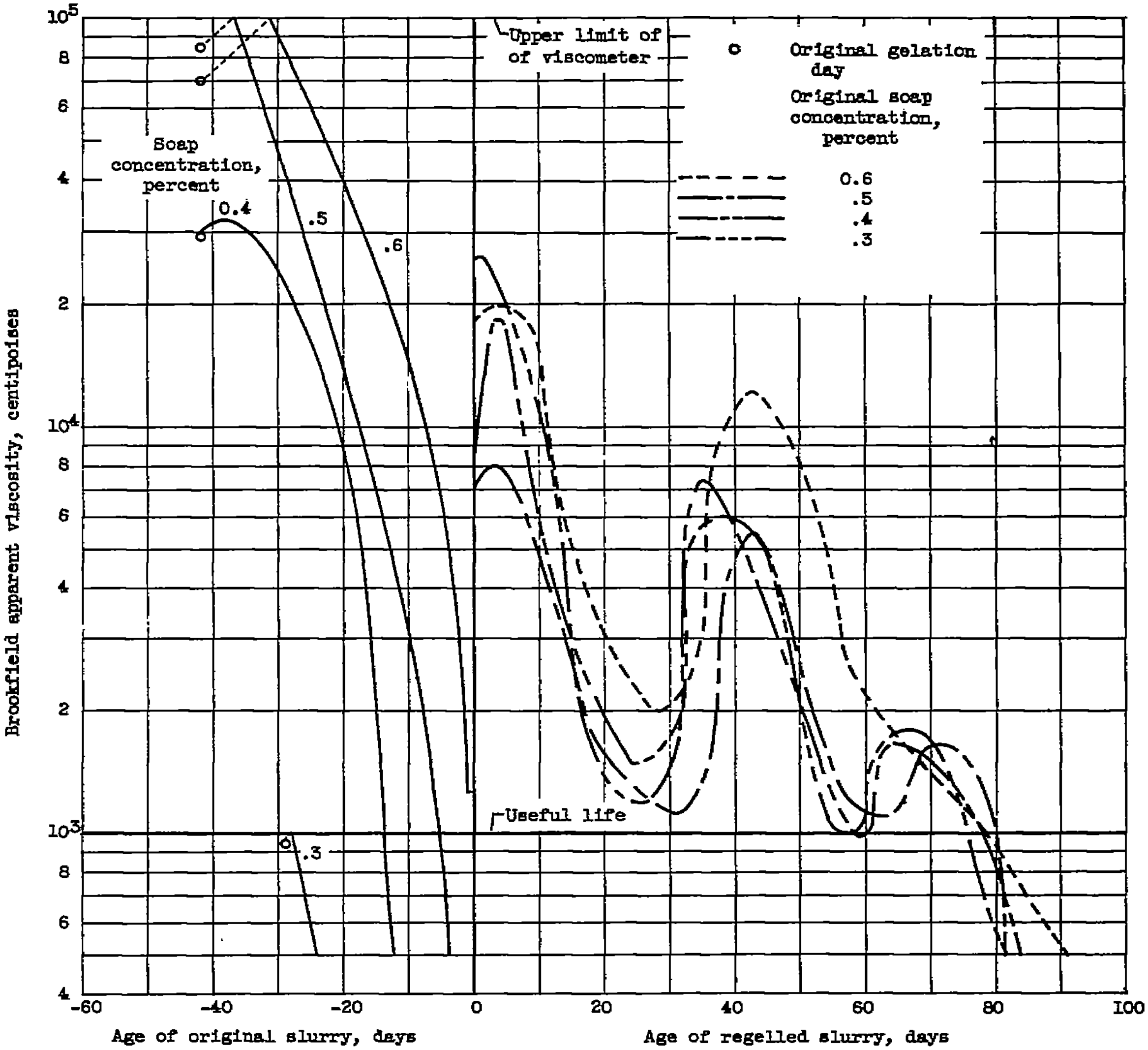

Figure 8. - Comparison of viscosity-age profiles of four regelled slurries with those of the original slurries. Gelation at room temperature (except for slurry with 0.3 percent soap); regelation temperature, about 110° F; soap III.

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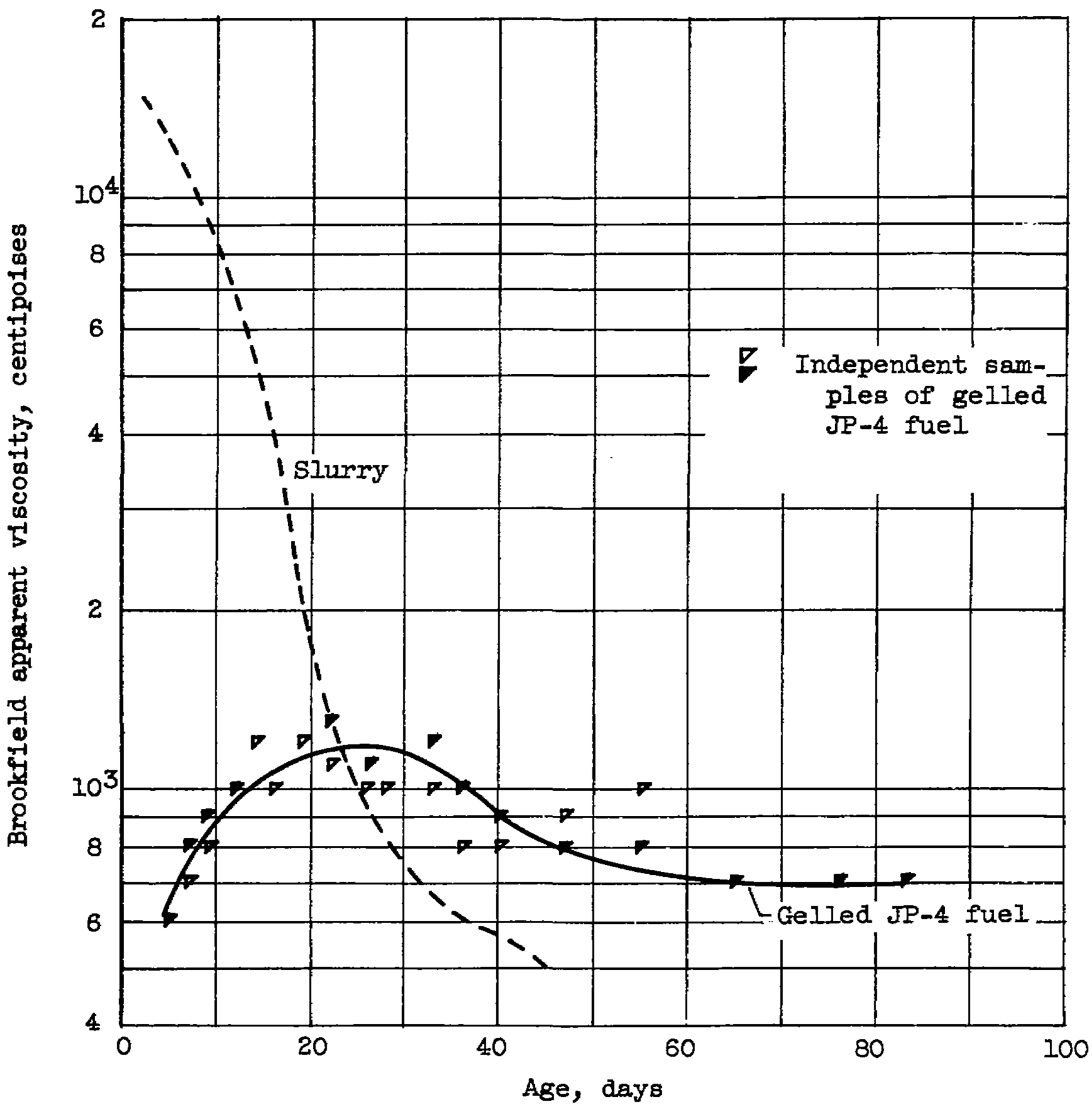

(a) Soap concentration, 0.8 percent based on JP-4.

Figure 9. - Comparison of viscosity-age profiles of gelled JP-4 and a slurry, both prepared with soap III at about 110° F.

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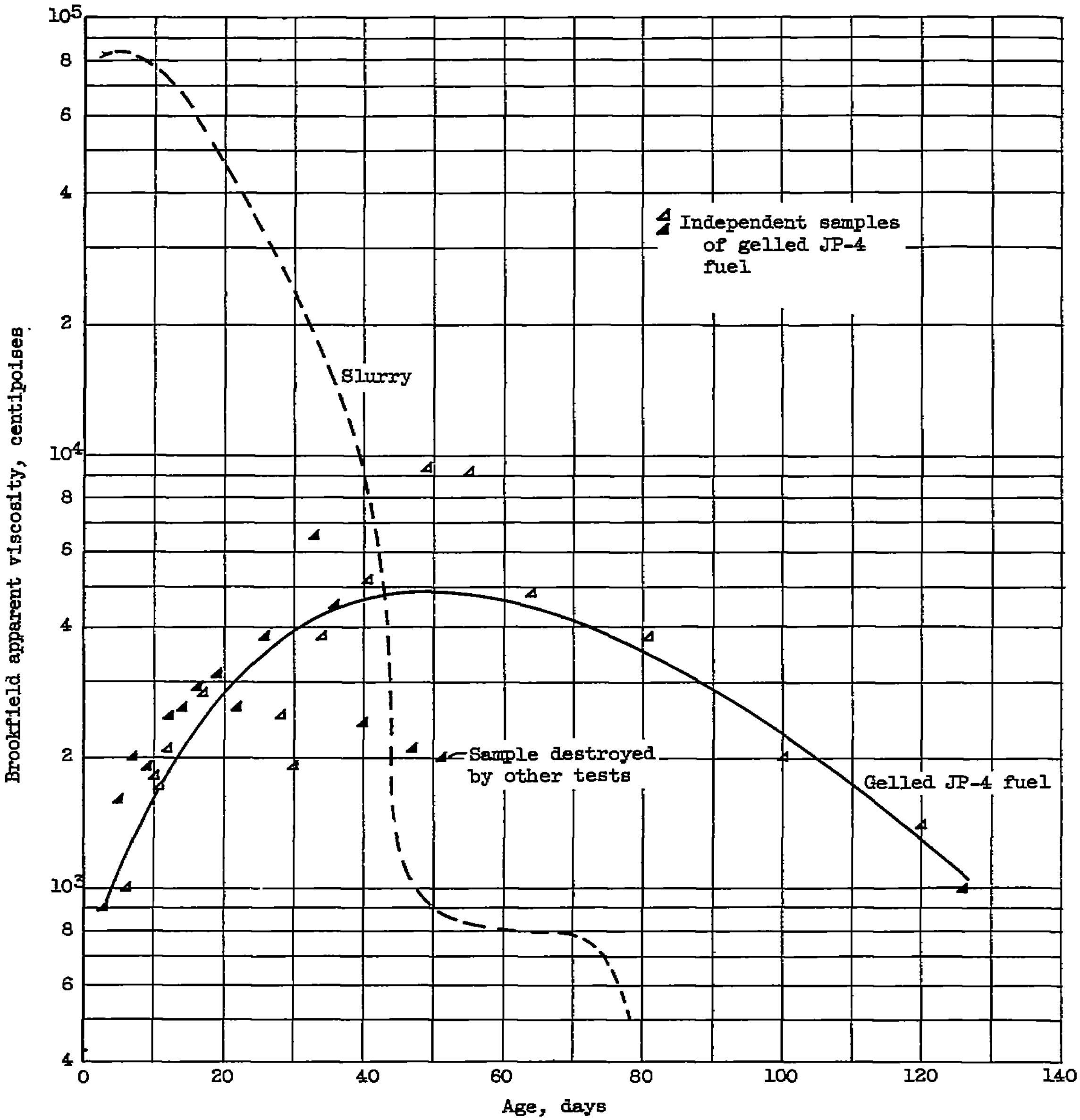
CS-6 back



(b) Soap concentration, 1.2 percent based on JP-4.

Figure 9. - Continued. Comparison of viscosity-age profiles of gelled JP-4 and a slurry, both prepared with soap III at about 110° F.

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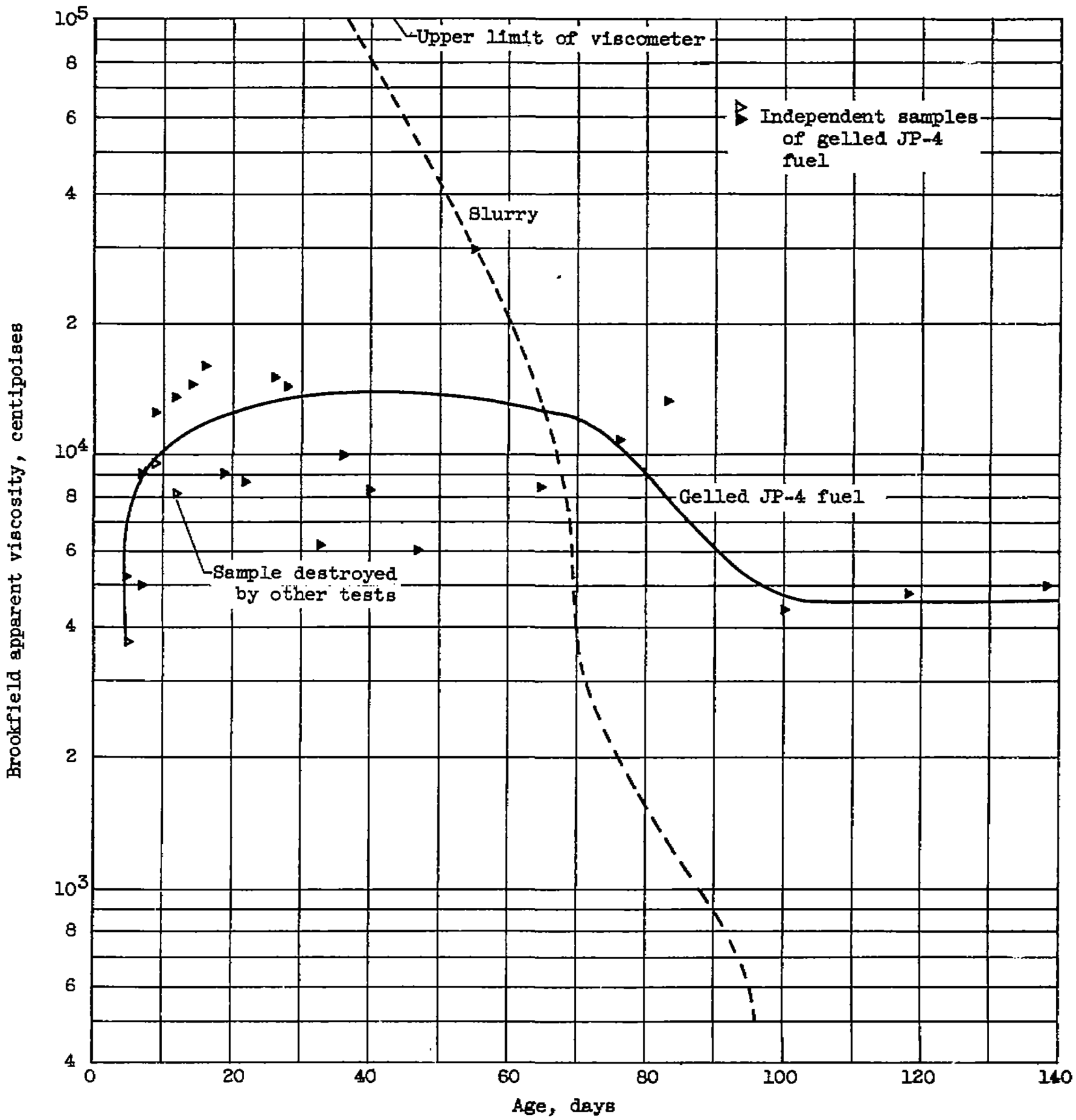

(c) Soap concentration, 2.0 percent based on JP-4.

Figure 9. - Continued. Comparison of viscosity-age profiles of gelled JP-4 and a slurry, both prepared with soap III at about 110° F.

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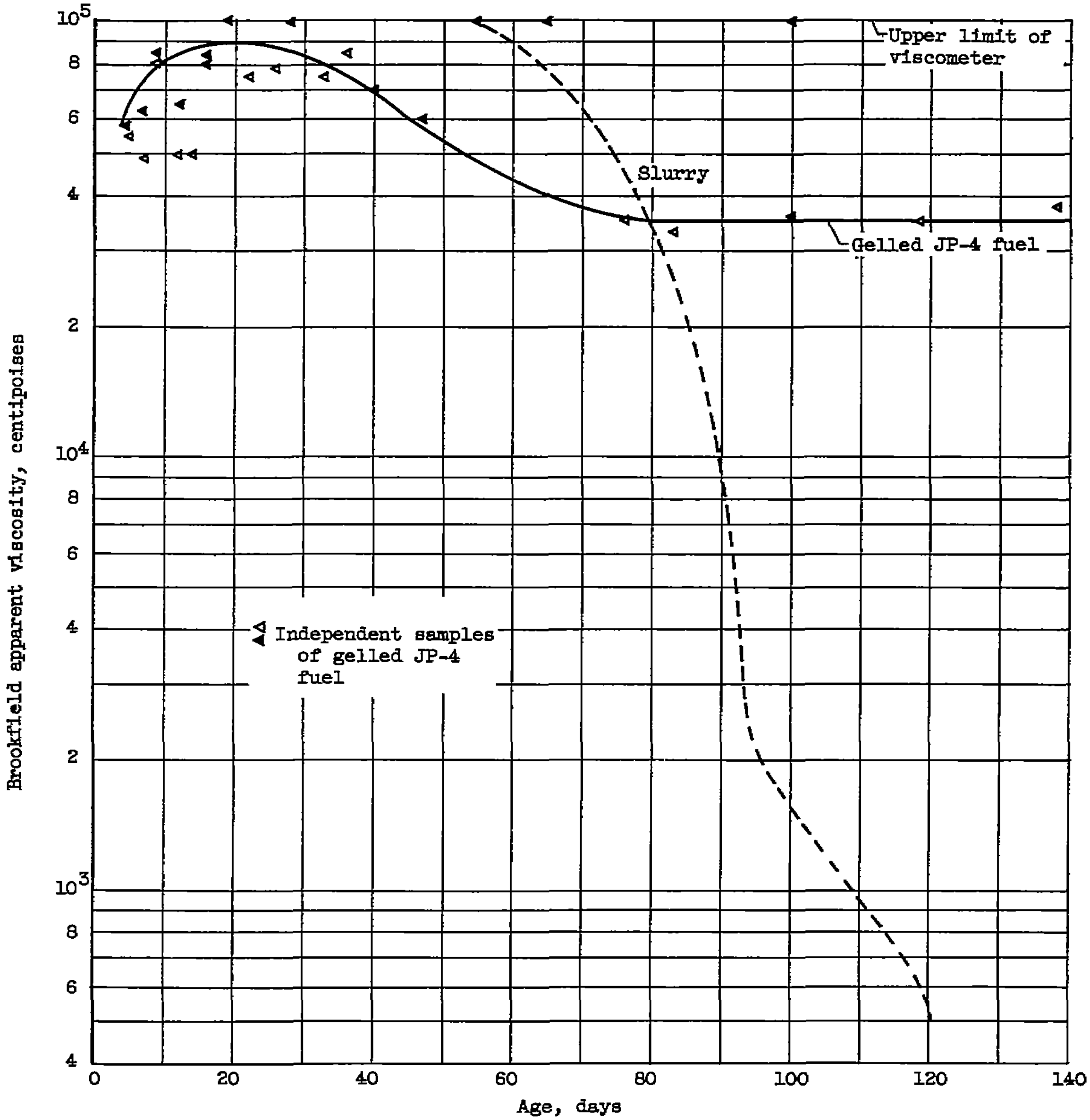

(d) Soap concentration, 4.0 percent based on JP-4.

Figure 9. - Concluded. Comparison of viscosity-age profiles of gelled JP-4 and a slurry, both prepared with soap III at about 110° F.

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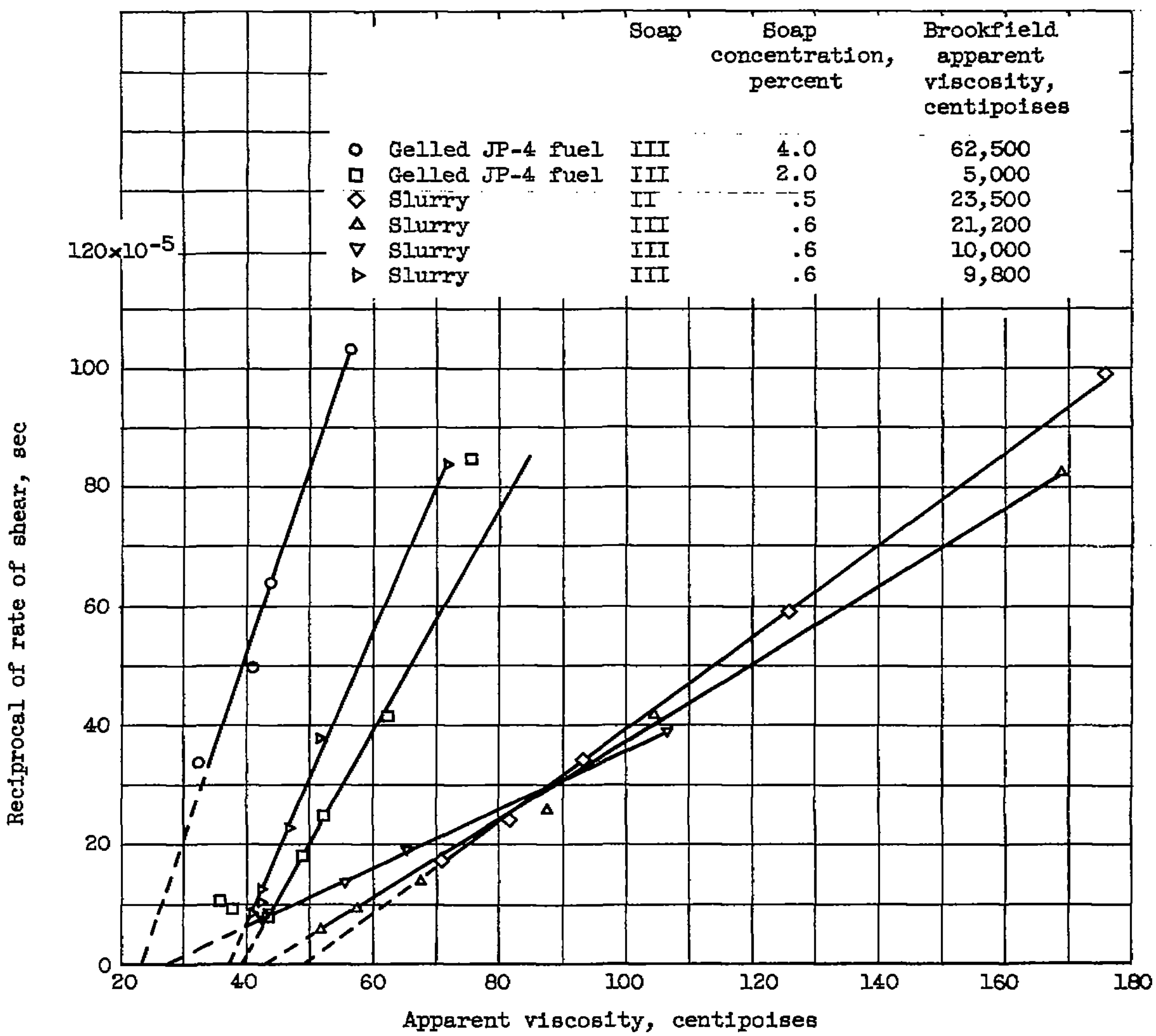

Figure 10. - Effect of rate of shear on apparent viscosity of slurries and gelled JP-4. Data obtained by means of the Severs Rheometer.

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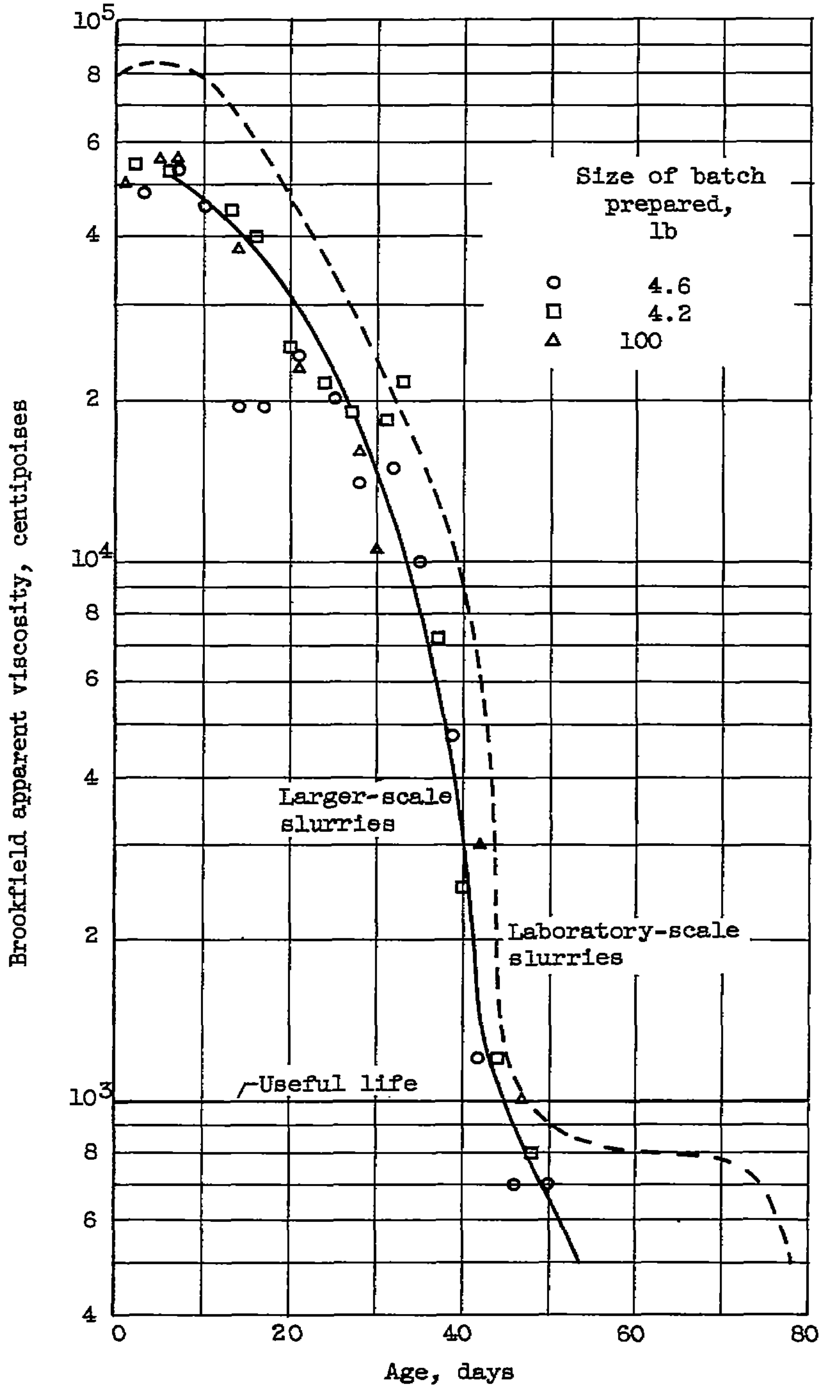

Figure 11. - Comparison of the viscosity-age profile of larger-scale slurries with that of the laboratory-scale slurries. Concentration of soap III, 0.6 percent.

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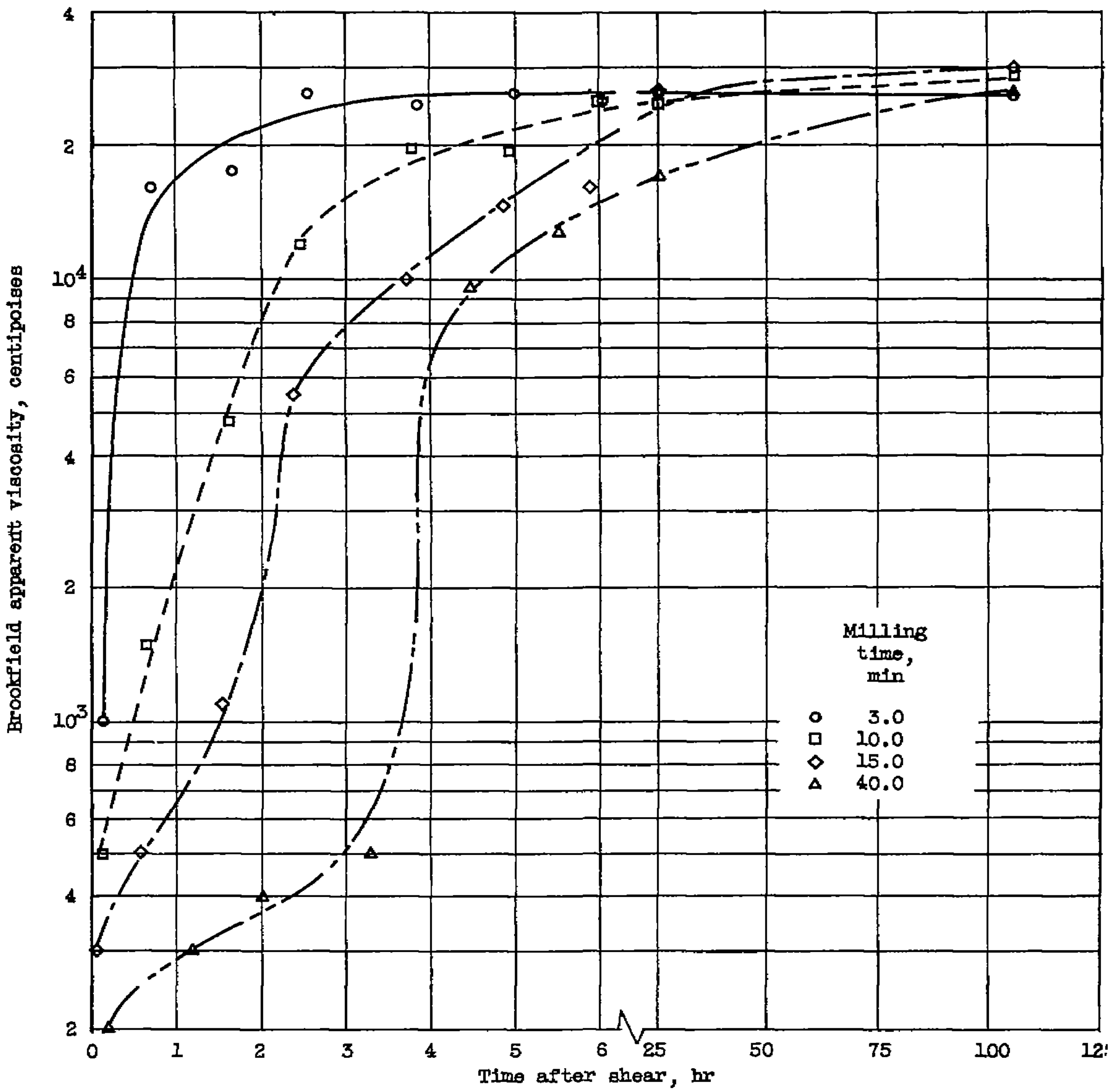

Figure 12. - Recovery of viscosity of a slurry after being sheared in a colloid mill for various intervals. Initial Brookfield viscosity, 26,000 centipoises.

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