This invention relates to the preparation of dispersions, and is particularly concerned with improvements in method and apparatus for preparing finely dispersed emulsions and suspensions.

Colloid mills and other high speed rotary impeller type mixers and disintegrators, such as have been heretofore generally used commercially for continuous formation of fine dispersions, operate on the principle of developing intense centrifugal forces and hydraulic shear effects within a zone of restricted cross-section through which the dispersion constituents are forced. Because of the high stresses which are developed this type of apparatus is essentially complicated and expensive, and any process based on the indicated operating principle is limited as to the rate at which dispersions are formed, has a low power utilization efficiency, and has the additional disadvantage of a substantial frictional heating effect on the dispersion constituents.

A more specific object of the present invention is to provide improved method and apparatus which is adapted for continuous formation of fine dispersions economically at a comparatively high rate; and to provide improved method and means for forming fine dispersions without the disadvantages above indicated as resulting from an operation depending on high centrifugal forces and hydraulic shear effects within a dispersion-forming zone of restricted cross-section.

In order to insure formation of dispersions at a commercially satisfactory rate the invention contemplates the use of a metal diaphragm of substantial area as the medium by which vibration energy is transmitted directly to the dispersion constituents. To insure intimate contact between the dispersion constituents and the diaphragm, and to increase the vibration tension of the diaphragm, the invention in its preferred form further contemplates the use of high superatmospheric pressure (at least five pounds per square inch, and in some cases upwards of 200 pounds per square inch) on the dispersion constituents within the dispersion-forming zone. In order to insure that the pressure thus obtaining in the dispersion-forming zone shall have no vibration-damping effect and no distorting effect on the diaphragm, the invention further features a diaphragm mounting including a spring support for the diaphragm under a tension which is adjustable to counterbalance such pressure. The combined use of superatmospheric pressure and a countervibrating spring support at opposite sides of the diaphragm serves the important function of amplifying the natural vibration period of the diaphragm by increasing the stress tension thereof without at the same time damping the amplitude of the vibrations developed.

Thus an important feature of the invention resides in the use of the metal diaphragm both as a vibration energy transmitting element and as a vibration frequency amplifying element adapted for amplifying the vibration frequencies within the sonic and supersonic range. In order that the diaphragm may function as a frequently amplifier it is an important feature of the invention to mount the diaphragm in such a way that it is free to develop independent vibration frequencies and so that it is physically independent from the power hammer or other energy source by means of which vibration energy is applied to the diaphragm for transmission thereby to the dispersion constituents.

When the solid or liquid to be dispersed has a higher specific gravity than the liquid dispersion medium, the metal diaphragm is preferably mounted as the charge supporting base of the chamber in which the treatment is carried out. With this construction the natural interface between the heavier material to be dispersed and the dispersing medium is located by gravity pull at a point adjacent the diaphragm base, where the greatest vibration effect is developed. It has been observed that the vibration energy developed adjacent the diaphragm is sufficiently intense to insure disintegration of any solid material (including lead shot) into very fine particle size. When the liquid or solid to be dispersed is of lighter gravity than the liquid dispersing medium, the diaphragm is located as the top of the treating chamber where gravity classification will be operative to establish the normal interface between the constituents in the zone adjacent the diaphragm.

Since one of the primary factors which determines the speed at which a suspension is formed is the area of diaphragm surface in physical contact with the dispersion constituents, an advantageous feature of the preferred apparatus design is the use of at least two physically connected spaced diaphragms, one of which may...
form the liquid sealed base for the treating chamber while the other diaphragm is connected by a central stem to the base of the diaphragm and has its peripheral edge free to develop independent vibration frequencies within the treating chamber.

With the above and other objects and features in view, the invention consists in the improved method of and apparatus for preparing fine dispersions which is hereinafter described, and particularly defined by the accompanying claims.

In the following description of the invention, reference will be had to the accompanying drawing, in which:

Fig. 1 illustrates diagrammatically, partly in longitudinal section, apparatus embodying the preferred features of the invention; and

Fig. 2 illustrates a modified form of apparatus having a more restricted field of application.

Referring to Fig. 1, the preferred form of apparatus therein illustrated comprises a chamber 10 within which fine dispersions of a finely divided solid or liquid in a second liquid dispersion medium are produced as a result of subjecting the materials to the action of a vibrating metal diaphragm 12. In the particular arrangement illustrated in Fig. 1, chamber 10 is shown as formed within an upright cylinder 14 having an integral dome 16. Diaphragm 12 is detachably mounted to form the charge-supporting base of chamber 10. The peripheral rim of the diaphragm is clamped between ground bearing surfaces of a flange joint having as elements an annular detachable flange ring 18 secured to the lower end of cylinder 14, as a second clamping flange ring 20. A spacing ring 22 may be inserted between clamping ring 22 and diaphragm 12. The two flange rings 18 and 20 are clamped together by bolts 24 which are provided with extensions serving as spacing bolts whereby to adjustably space and align external vibration energy source and balancing spring elements with the center of diaphragm 12.

The liquid or finely divided solid substance which is to be dispersed, and the liquid dispersing medium, may be introduced successively or simultaneously into chamber 10 through an inlet pipe 26. An outlet pipe 28 having a regulating valve 29 is provided for removal of the dispersion medium and the mouth of pipe 28 can be adjusted vertically within chamber 10 by adjusting the length of pipe 28 in a packing gland 30 in the dome 16. An important feature of the present invention is that of holding the constituents of dispersion under controlled superatmospheric pressure within the dispersion-forming zone. Pressure may be developed within the chamber 10 by the use of pumps 31, 32 and connections whereby to supply a preformed coarse dispersion of the liquid-liquid or liquid and solid materials to the chamber. An alternative method for developing controlled pressure within the chamber consists in supplying a gas such as nitrogen to the dome of the chamber under controlled superatmospheric pressure by means of a compressor 33 and a pipe connection 34.

Diaphragm 12 may be supplied by an electromagnetc or solenoid vibrator 35 which is illustrated diagrammatically as mounted in adjustably spaced and aligned relation to the center of diaphragm 12 by means of a collar 37 and extensions of bolts 24. Vibrator 35 is provided with a magnetized reciprocating core rod hammer 38 which is aligned with the center of diaphragm 12 and the vibrations of which are transmitted to the center of the diaphragm through the medium of a plunger 40. The upper end of plunger 40 is held in striking contact with a metal target 42 which is rigidly affixed to the center of diaphragm 12. A coil spring 44 is mounted between a collar 45 engaging a shoulder 46 below the head of plunger 40 and a spring support bracket 47 which is adjustably mounted on bolts 24 by spring compression adjustment nuts 48. The spring 44 thus is disposed to counterbalance any distorting and vibration damping effect of pressure obtaining in chamber 10 on the diaphragm, so that the hammer 38, acting through plunger 40, imparts upward impacts to the center of diaphragm 12 at a frequency which may be 60 per second when the energizing current for the solenoid is a 60-cycle alternating current.

To further insure against undue distortion of diaphragm 12 outwardly as a result of superatmospheric pressure in chamber 10, and the vibration harmonics of the diaphragm, the central aperture in flange ring 20 is threaded to demountably engage a nut 49. A lock nut 50 is mounted on sleeve 49 below ring 20 and is used to lock the sleeve in predetermined suitably stop position below the diaphragm.

To increase the area of vibration transmitting surface in contact with the liquid dispersing medium in chamber 10, a second metal diaphragm disc 52 is mounted within chamber 10 in parallel spaced relation to diaphragm 12. Diaphragm 52 is circular and of smaller diameter than the internal diameter of the chamber. The center of diaphragm 52 is rigidly connected to a stem extension 43 of target plug 42, while the peripheral rim of the diaphragm 52 is free to develop its own independent vibration frequencies within the dispersion-forming zone. The forward end of stem 43 is provided with an axial bore 54 and a plurality of connecting radial bores 55 whereby to disperse materials circularly between a point in chamber 10 above diaphragm 52 and the space between the two vibrating diaphragms.

According to the preferred apparatus design the dome 16 is elevated a considerable height above the diaphragms 12 and 52. Also the inlet end of the discharge pipe 28 is adjustable vertically within the chamber 10. As previously indicated, the apparatus design illustrated is adapted for preparing dispersions of a material of high specific gravity in a liquid dispersion medium of lighter gravity; and the apparatus may be operated so as to partly fill the chamber 10 with the dispersion constituents, while trapping gas under pressure above the surface of the dispersion liquid. On the other hand it may be advantageous to completely fill chamber 10 with the dispersion constituents and to elevate pipe 28 so that its inlet is adjacent the top of the dome. By this arrangement of disintegration of the solid or liquid disperse material to fine particle size takes place at or adjacent the surfaces of the diaphragms, and vibration-gravity classification of the dispersion thus formed takes place in the upper portion of chamber 10, so that only the finest dispersion leaves the chamber through the offtake pipe 28. The vibration to which the entire body of liquid in chamber 10 is subjected hastens the speed with which the coarser par-
articles of disperse material are classified and returned by gravity settling to the bottom disintegration zone of the chamber.

In that modification of the apparatus which has been illustrated in Fig. 2, certain advantageous features of the preferred design have been sacrificed in order to meet a particular problem which is sometimes encountered when, for example, it is desirable to carry out the dispersion-forming operation at a relatively high temperature and to simultaneously effect removal of liberated gases or vapors of volatile liquid from the zone of dispersion formation. The change in design of apparatus to meet problems of this type includes connection 55 whereby to maintain a partial vacuum within the chamber 16; and other modifications made necessary by the use of such vacuum for limiting the effects thereof on the vibration transmitting efficiency of the diaphragm 12. Thus, in this modification, the plug stem 45 which connects the two diaphragms 12 and 52 is provided with a long threaded extension rod 56 which is encircled by a collar 52 on which there is in turn supported the lower end of a coil spring 64. The upper end of the spring 64 is held in place by the top of a cylindrical sleeve 55. Sleeve 56 has external threads which engage matching threads in the bore of flange ring 20. The position of collar 52 is adjustable by means of an adjusting nut 68 on rod 56, and the lower end of extension rod 56 is provided with a target cap 70 which receives and transmits to the diaphragms the impacts of a hammer rod 38 which is vibrated at the frequency developed by an electro-magnetic vibrator 36. Thus it will be noted that in this construction the spring 64 is set up to resist and counterbalance the pull of vacuum in tank 10 tending to distort diaphragm 12.

The preferred apparatus is adapted for continuous throughput of dispersion constituents in sufficient volume to insure production of fine dispersion at a commercially practical rate. The rate at which fine disintegration and dispersion is effected is primarily a function of the diaphragm area and tension, and the diaphragm tension is in turn a function of the pressure which is maintained in the treating chamber 10 and of the counterbalancing pressure of spring 44. The diaphragm 12 is preferably constructed of a high tensile steel having sufficient strength to stand up under the operating conditions and having sufficient hardness to possess a high natural period of vibration. Diaphragm 12 should also have a high diameter to thickness ratio (at least 100 to 1) in order to provide a large vibration surface in combination with sufficient flexibility as to be adopted for developing and transmitting vibrations of substantial amplitude. Another important contributing factor is that of having the vibration energy source, i.e., the hammer, by which vibration impacts are imparted to the diaphragm, physically independent of the diaphragm, so that the diaphragm is free to vibrate and to develop its natural vibration frequencies independently of the hammer. By providing a second diaphragm physically attached to the main diaphragm by a central stem and having an unsupported rim free to develop its own independent vibration frequencies within the treating zone, the area of vibrating surface is multiplied, the zone of maximum vibration is also enlarged, and the range of vibration periods is multiplied.

As evidence of the intensity of vibration energy which is developed and transmitted to the dispersion constituents, substantial cavitation has been observed while preparing dispersions in apparatus of the general form described, but differing therefrom by employing a single plane diaphragm disk 12 having a central plug target 42. The cavitation thus developed was sufficiently intense to rapidly release metal from the central portion of the hard steel diaphragm face where the greatest cavitation is developed. It was because of this noted erosion effect of cavitation on the diaphragm surface that the bored stem 54 and additional diaphragm 52 were added to more widely distribute the area of high vibration intensity, and to provide passages around the second diaphragm and through the bores of the stem whereby to utilize the forces evidenced by cavitation to promote circulation of the dispersion constituents in the zone between the parallel spaced diaphragms.

The dispersion constituents are preferably delivered to the dispersion-forming zone in the form of a preformed coarse dispersion which is preferably proportioned with a large excess of dispersion liquid. The solid or liquid which is to form the dispersion phase is naturally drawn by gravity pull to the immediate vicinity of the vibrating diaphragm surface where it is subjected to the intense vibration turbulence which develops throughout the liquid, and particularly in the zone adjacent the diaphragm surfaces. The metal diaphragms are under sufficient tension so that the sharp high frequency hammer blows impart thereto by the hammers 38 and 40 develop within the diaphragms supplemental vibrations having frequencies within the sonic and supersonic range. The supplemental vibrations thus developed in the diaphragms are entirely out of phase with the vibrations imparted thereto by the hammer, so that interfering vibration waves are transmitted through the liquid in the treating zone and develop therein vibration waves of such intensity as to insure thorough and rapid disintegration and dispersion effects.

The process of the present invention has been found to be particularly adapted for the preparation of finely dispersed suspensions and emulsions wherein a small amount of disperse material is distributed in a large excess of dispersion liquid. The process of the present invention has been used for producing a fine dispersion or emulsion of water in gasoline. In fact, more stable water-and-oil emulsions generally can be produced by this method than by any other method known to applicants. The invention has also been applied with success to the production of suspensions of graphite or sulphur in lubricating oil. The present process is also adapted for effecting dispersions of lead suboxide or other chemical treating agents in gasoline. Also the process is suitable for securing uniform dispersions of anti-knock agents such as triphenylguanidine in gasoline. The dispersions developed are of such frequency and amplitude as to thoroughly break down flocculent adherence of solid particles and promote disintegration and dispersion of the solid throughout the liquid dispersing medium in apparently ultimate fine particle size.

Another field of application for the invention is in the field of cosmetics, such as, for example, in the preparation of finely dispersed stable suspensions or emulsions of greatly improved stability as compared to present day hand lotions and the like.
The invention having been thus described, what is claimed as new is:

1. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared, said chamber having as one wall thereof a metal diaphragm of relatively large area having its rim anchored and its center free to vibrate, means for supplying liquid to be treated to the chamber in contact with said diaphragm, means outside said chamber for vibrating said diaphragm including a reciprocating hammer movable independent of the diaphragm and mounted in a position to impart a succession of sharp impacts at high frequency to the central portion of said diaphragm, and means for transmitting the hammer impacts to the central portion of the diaphragm.

2. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared, said chamber having as one wall thereof a diaphragm of relatively large area, means for vibrating said diaphragm, means for supplying liquid to be treated to the chamber in contact with said diaphragm, a conduit for removing formed dispersion from the chamber, and means for adjusting the spacing of the inlet of said conduit from the diaphragm a distance sufficient to permit vibration-gravity classification of the formed dispersion prior to its removal from the chamber.

3. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared, said chamber having as one wall thereof a diaphragm of relatively large area, means for supplying liquid to be treated to the chamber, a second diaphragm mounted within the chamber in spaced parallel relation with respect to the first diaphragm and having its center attached by a stem to the center of the first diaphragm for vibration therewith, an aperture in said stem whereby liquid may circulate between the zones on opposite sides of said diaphragm, and means for vibrating said diaphragms.

4. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared having as one wall thereof a metal diaphragm of relatively large area, means for supplying liquid to be treated to the chamber in contact with said diaphragm, a second thin metal diaphragm secured to the center of the first diaphragm in spaced parallel relation thereto within the chamber for free vibrational movement therewith, a communication channel from the space between the diaphragms to the space on the opposite side of the second diaphragm, and means for vibrating said diaphragms.

5. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared, said chamber having as one wall thereof a diaphragm of relatively large area, means for vibrating said diaphragm, means for supplying liquid to be treated to the chamber in contact with the diaphragm, and a counter-balancing spring mounted on the diaphragm outside the chamber and arranged to counter a balance the diaphragm against the pressure maintained in the chamber, thereby preventing damping of the diaphragm.

6. An apparatus for preparing dispersions in liquids, comprising a chamber in which the dispersions are prepared, said chamber having as one wall thereof a metal diaphragm of relatively large area having its rim anchored and its center free to vibrate, a second metal diaphragm secured to the central portion of the first diaphragm in spaced parallel relation thereto within the chamber having its rim spaced slightly from the chamber wall and free to vibrate, means for supplying liquid to be treated to the chamber in contact with said diaphragms, and means for vibrating said diaphragms.

7. An apparatus for preparing a fine dispersion of a fluid or solid with a liquid, comprising a transmitter of vibration in the form of a metal diaphragm of relatively large area having its rim anchored and its center free to vibrate, a dispersion-forming chamber having the diaphragm as one wall thereof, means for maintaining a suitably proportioned mixture of the fluid or solid and liquid under superatmospheric pressure in contact with the chamber side of the diaphragm, a metal target attached to the center of the diaphragm and a source of vibration energy mounted at the other side of the diaphragm having a hammer in position to impart successive sharp impacts at high frequency to the target to develop vibration of the diaphragm.

8. The dispersion apparatus defined in claim 7 in which a second diaphragm is supported by the target and spaced from the metal diaphragm and a circulation channel is formed in the target to permit circulation of fluid from above the target into the space between the diaphragms.

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