

## SCIENTIFIC SECTION

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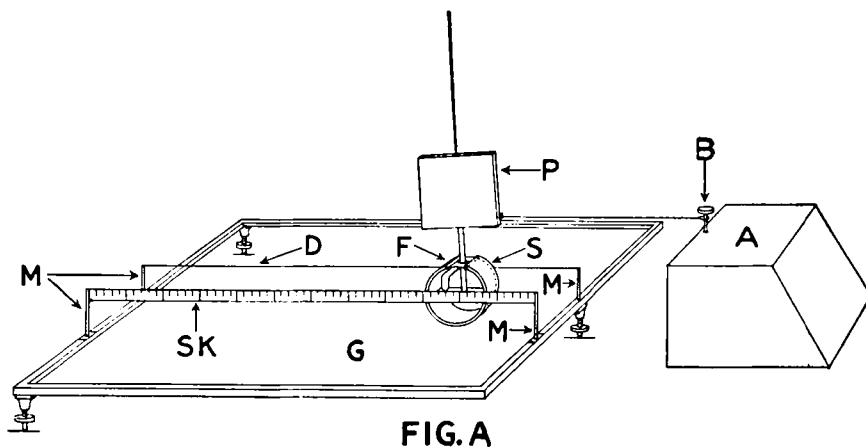
### A METHOD FOR THE MEASUREMENT OF CERTAIN MECHANICAL PROPERTIES OF PHARMACEUTICAL AND TECHNICAL CREAMS.\*

BY JOHN URI LLOYD, WOLFGANG OSTWALD AND HANS ERBRING.

#### INTRODUCTION.

It is a generally known fact that the mechanical properties of cosmetic skin creams are widely different. Olive oil, for example, lends itself well to massage, while with lanolin, massage is practically impossible, because in the latter case the film produced on the skin is sticky instead of being smooth. Still more complicated phenomena are observed, as the senior author has pointed out, with certain skin creams in which the base consists, for example, of plant mucilages. If a small quantity of such an aqueous mucilage is put upon the back of the hand and rubbed down with the other hand, one notes at first only the sensation of wetting with water. Upon continued rubbing, however, this sensation suddenly changes to that of stickiness, and the skin, *i. e.*, the film upon it, appears pronouncedly rough. When rubbing is continued, there is again a rather sudden change; the rough skin suddenly becomes smooth again, and one has the same sensation as in rubbing soft kid leather.

This interesting observation of the complicated change which the mechanical properties of a cosmetic film undergo solely in consequence of a continuous rubbing down, formed the starting point in investigations now to be described.



First of all the question arose, whether this change of the mechanical properties was merely of a subjective, or perhaps physiological nature, dependent, *e. g.*, on resorption by the living skin, or whether there are not changes in the film itself

\* Translated from the German by Dr. Sigmund Waldbott.

that might be demonstrated objectively, independent of the living skin. Be it stated as one outstanding result, that the aforementioned complicated changes in the mechanical condition of such cosmetic films may in fact be demonstrated objectively, and that they are not a function of the living skin as a substrate.

#### DESCRIPTION OF THE APPARATUS.

The apparatus used in the subsequent experiments is illustrated in Figs. *A* and *B*.

A glass plate (Fig. *A*, *G*) is within a metal frame supported by 3 short legs which are provided with set-screws for horizontal leveling by means of a round box level. To the frame are attached at the right and the left, in each case, 2 vertical metal rods (*M*) each about 5 cm. long. One set of opposite rods is united by a metal bridge, on the front of which there is a millimeter scale (*SK*) and the other set by a very thin metal wire (*D*) drawn taut. These two metal connections serve as a guide for a sled (*S*) which can be moved to and fro between them.

The sled itself (Fig. *B*, *S*) is a short, hollow, horizontal cylinder of brass, tightly fitting into a slightly wider glass cylinder of the same length, which surrounds it except on top, where a strip of the glass is removed. An index (*Z*) at the front top edge of the sled measures its prevailing location by means of the scale.

In the lower half of the brass cylinder there is a horizontal brass plate with a round hole in the center, exactly below another hole in the top of the brass cylinder. Through these openings passes a metal rod, *i. e.*, the end of a pendulum (*P*) about 2 m. long, having a driving weight of 10 Kg. By this arrangement, the metal rod has no rigid connection with any part of the sled, and the amount of friction of the sled against the guide connections is negligible.

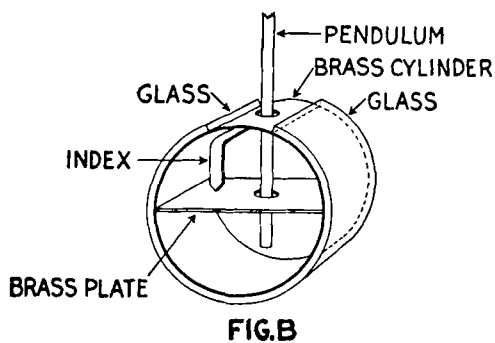
When the pendulum swings, the sled is moved along, and from the character of the vibration and the character of the diminution curves (*Dämpfungskurven*), conclusions may be drawn as to the lubricating and sliding ability of the medium put between the sled and the glass plate.

In order to obtain a constant altitude of fall for the pendulum, *i. e.*, a constant driving force with which to move the sled on its base, an arresting device (*A*) for the pendulum is installed. The pendulum is set swinging by removing a steel pin (*B*) in order that the altitude and the number of vibrations upon swinging *freely* be always the same.

With the aid of this arrangement, pure liquids and plant mucilages, emulsions and creams were examined, by observing their behavior upon friction between glass surfaces.

#### EXPERIMENTAL RESULTS.

*I. Pure Liquids.*—As mentioned at the beginning of this article, different plant mucilages present the phenomenon of having their gliding ability altered



with length of time of trituration. This effect, no doubt of complex nature, rendered it advisable for the purpose of seeking an explanation, first to use *pure* and *homogeneous* liquids and semi-fluid substances.

The experiments were conducted as follows:

A small portion of the substance to be examined was put upon the glass surface, and spread over the total surface involved in the rubbing. Then the rod of the pendulum was put through the holes of the sled, and the pendulum was "fixed" in place by the arresting device. Upon removing the little steel bolt, the pendulum began swinging, moving the sled with it back and forth on the glass plate. The number of vibrations, as well as the "rate of diminution" ("Dämpfung") then becomes a measure of the "gliding allowance" of the thin lubricating or sliding film between sled and glass surface.

When this sliding film has formed, we should expect that for pure systems in which mechanical manipulations cause no changes whatsoever, the "sliding ability" would be independent of the *number* and the *duration* of the sled movements, provided that the film does not evaporate.

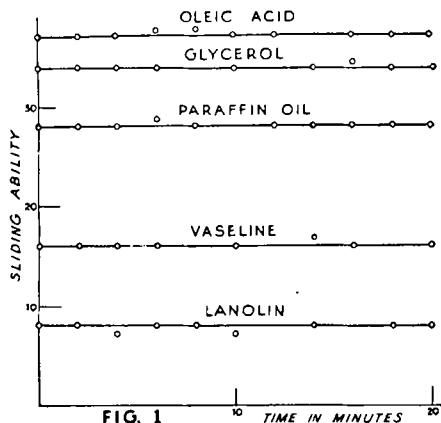


FIG. 1

This phenomenon has indeed been clearly verified with all systems examined. The results are shown in Fig. 1.

The abscissæ represent times in minutes, during which the sliding ability was continuously observed. The ordinates show the (relative) sliding ability, *i. e.*, number of vibrations within a given time.

For example, with *oleic acid*, 10 consecutive experiments were carried out; the number of vibrations proved to be the same in each experiment. Similar graphic results are obtained for the other

substances (systems) examined. In each case the result is a straight line (each at a different ordinate) which indicates the fact that sliding ability is a specific *constant* of the substance in question, and independent of the duration of the mechanical trituration. The figures for the "slide value" or "slide degree" of each system are given in Table I. The order in which the systems range themselves by the results of these experiments (oleic acid, glycerol, paraffin oil, vaseline, lanolin) is in accordance with experience.

TABLE I.

Substance.	Relative Slide Degree.
Oleic acid	37
Glycerol	34
Paraffin oil	28
Vaseline	16
Lanolin	8

Parallel measurements with *viscosities* of these substances show that the apparatus we use measures a distinct *sliding effect*, which does not simulate viscosities.

Thus the glycerol used gave a result in viscosity about 20% higher than the paraffin oil examined, yet the sliding effect of glycerol is considerably greater. A large number of other systems examined, demonstrated similar facts.

II. *Creams of the Type "Water in Oil."*—An essentially different picture is obtained when no longer pure systems, but *compound systems*, are investigated.

We have examined a large number of commercial pharmaceutical and cosmetic creams for their frictional or sliding capacities. First, we examined so-called "fatty creams," which represent emulsions of the type "water in oil." The graphic picture of all samples investigated, which differ but slightly one from the other in their behavior, is represented for 1 sample in Fig. 2.

A similar graph we obtained with emulsions prepared by ourselves with varying water content in vaseline or paraffin oil. We do not here need to give the different curves and figures.

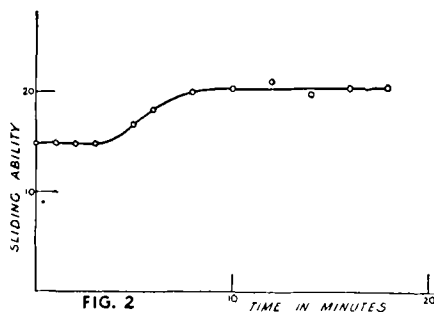


FIG. 2

At the beginning of the measurement we note at first that in each single experiment the numbers of sled movements at the same driving power until the pendulum stops swinging, remains the same. Then, however, the curve becomes more or less steep upward, that is, the sliding ability of the cream increases. When the experiments are further continued, there will again be a constant end value.

Different causes may be held responsible for this interesting increase in the sliding, lubricating or massage ability of such "water in oil" creams. First, the thought suggests itself that on account of the continued mechanical manipulation of the "emulsion film" produced, a further dispersion effect is obtained. An emulsion cream of higher dispersion perhaps has a better sliding ability than one more coarsely dispersed. However, just as well an opposite process might take place, caused by mechanical, temperature or electrical influence, *i. e.*, destruction of the emulsion in the film and a sliding of the sled finally upon the pure "fatty" surface.

A more detailed examination of the effect has not yet been made; our present purpose was to find and work out a method for the examination and technical development of such creams.

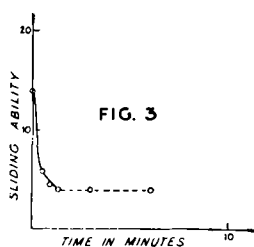


FIG. 3

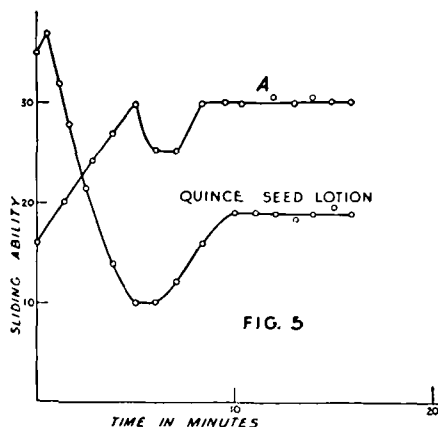
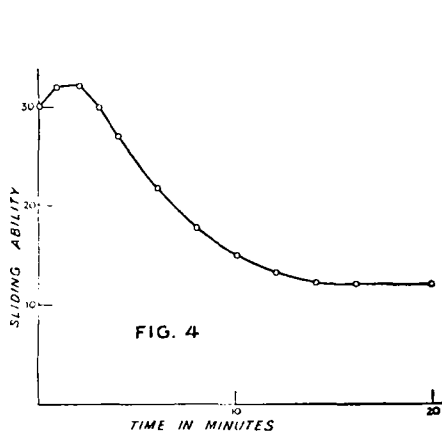
III. *Creams of the Type "Oil in Water."*—Another group of systems examined by us is Creams which one might designate as "oil in water" emulsions. They are the so-called "day creams," "face creams" or "matt creams." Since in these creams the coherent (dispersing)

phase is water, in which, for example, finely divided stearic acid particles are distributed in very highly dispersed form, one may expect from the start a different appearance of the sliding curves. When rubbed upon the hand, these creams show very rapidly a certain resistance, *i. e.*, the sliding ability decreases more and more during rubbing. Indeed, the same effect is obtained with our apparatus. Figure 3 shows the nature of the curves obtained with these samples. With continuous rubbing we note a steep decrease, and the manifestation of a certain end value

at a certain low sliding ability (or better, degree of "roughness"), of the stearic film produced. We suppose that in this end condition, the aqueous dispersing phase has to a large extent evaporated.

IV. *Vegetable (and Gelatin) Lotions with Glycerol.*—Undoubtedly the most interesting phenomena are obtained with by far the largest number of plant jellies. With these systems we find the complex behavior mentioned in the beginning: An intermediate stage of stickiness which makes the skin rough, and upon further rubbing the change to smoothness, whereby the skin feels like kid leather. Our apparatus illustrates objectively these different conditions which would well recommend the apparatus for examination of the mechanical properties of creams, rubbing materials, sliding materials, etc.

A commercial gelatine-glycerol jelly, showed a curve represented in Fig. 4, *i. e.*, of a nature very similar to that obtained by rubbing on the skin. The slight rise of sliding ability at the beginning of the rubbing, indicates only a *gradually*



intimate contact at first of the sled with the sliding material. This initial rise is especially pronounced in the following examples:

Two samples were examined: (1), a Quince seed lotion, and (2), a plant product of similar composition in American commerce. In both samples we distinctly observe the intermediate stage of "stickiness" (see Fig. 5). But we furthermore recognize that the end value of sliding ability, *i. e.*, the condition of "smoothness" in the commercial preparation, is considerably higher, *i. e.*, better, than in the Quince seed lotion.

A further plant product, a mucilage-jelly that had been obtained from flax seed was examined. Figure 6, I-III, informs us about the degrees of mechanical usefulness of this product.

After the real slide film has been formed, sliding ability decreases. The polished layer becomes rough and dull, evidently on account of decreasing water content, as in general one may perhaps be justified in associating the degree of stickiness with a definite quantity of water that happens to exist in the system. Experiments to avoid, or rather to improve this rough and dull terminal step in the gliding experiment, so undesirable in mechanical, and more so in cosmetic respect,

led to improvement of this preparation by addition of glycerol. We observe the steady improvement of the final stage; the curve moves to higher and higher degrees of sliding ability.

#### GENERAL SURVEY.

The experiments herein described show that the apparatus used, gives in an excellent manner information on certain mechanical properties of Creams, Emulsions, lubricating materials, etc. No doubt this may be of some importance in

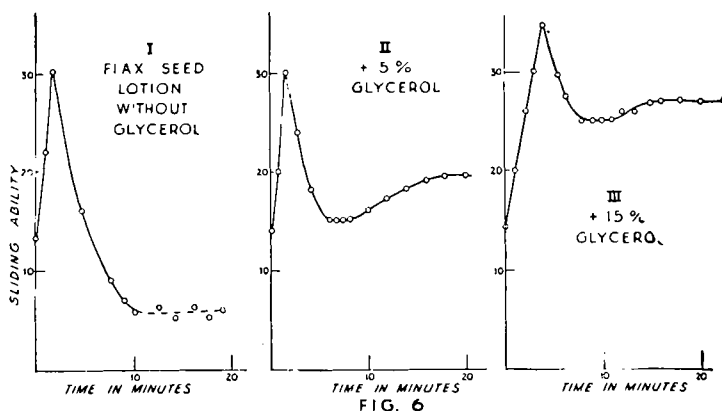


FIG. 6

technology, especially as to cosmetics. By means of it, it will be possible to get information within a few minutes concerning important mechanical properties of creams (*e. g.*, for massage purposes), objectively and reinforced by figures. Of course, besides the mechanical qualities, the specific, pharmacological and physiological effects upon the skin are of no less importance to the question of quality and adaptability of a cosmetic cream.

#### SUMMARY.

A method is described making it possible to characterize, objectively and numerically, the "sliding ability" of skin creams, lubricating and anti-friction materials, etc., and their very manifold and characteristic changes which take place upon continuous trituration.

A few examples of interest cosmetically are represented by curves.

### DRUG EXTRACTION. IX. THE EFFICIENCY OF REPERCOLATION FOR BELLADONNA ROOT AND NUX VOMICA.\*<sup>1</sup>

BY WILLIAM J. HUSA<sup>2</sup> AND C. L. HUYCK.

Although the process of repercolation has been known for many years there is practically no information in the literature giving definite data as to the efficiency of the U. S. P. X process of repercolation. The pioneer work of Squibb was con-

\* Scientific Section, A. P. H. A., Portland meeting, 1935.

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