The paper calls attention to the haziness throughout the liquid, which Mr. Ėwe finds to be "an almost colloidal suspension of a trace of the aqueous liquids from which the alkaloids are extracted." In this connection, which is one of the problems I have met in studying the meniscus between liquids, I have referred to as a characteristic of semi-physical relationships. This is touched upon by Professor Ostwald and Mr. Walter Haller, as follows:

"A phenomenon observed years ago by the senior author (John Uri Lloyd), with acetone-glycerol and other pairs of liquids, namely, the lack of sharpness of the menisci, is very remarkable."

It will be noted that this lack of sharpness of the menisci does not exactly parallel the haziness described by Mr. Ėwe as follows:

"The chloroformic extractions, instead of being brightly clear, are often hazy. This haziness, if not due to the presence of solid matter, is due to an almost colloidal suspension of a trace of the aqueous liquid from which the alkaloids are extracted."

My opinion was that the haziness shown about the menisci of acetone and glycerin resultant, was by me unexplainable, nor did I attempt to do more than record the fact of observation.

Mr. Ėwe has, in an independent direction, referred to a haziness, not alone a meniscus complication, but of the chloroformic liquid as a whole, indicating, as he expresses it, a disturbance of alkaloidal assaying exactness. Let me venture to hope that he will continue his research in the direction of colloidal disturbances.

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NOTES ON CASCARA SAGRADA.*

BY MILFORD HARRIS AND EDWARD D. DAVY.

The varying results reported on Cascara Sagrada, the lack of positive evidence as to the active constituents, the effect of alkalies and oxidation, led us to try fractionation of the active constituents by varying solvents only.

All of the common immiscible solvents were tried either in the extraction of the drug, or in subsequent efforts to fractionate the active material from aqueous and alcoholic extracts. Water, Ether and Ether-Alcohol mixtures were found more satisfactory than the others.

The references listed indicate the wide interest in this subject and we have consulted them freely, using such help as might apply to any particular case.

The cascara used for this work was approximately six years old and solvents used for the initial extractions were water and alcohol.

(*A*) *Water Extract.*—A preliminary extraction of twenty-five grams of drug was made in a Soxhlet with water, using an air condenser, so that the water reaching the drug was just short of boiling. When the extraction was complete the drug was dried and reëxtracted with alcohol. The alcoholic extract yielded considerable emodin, a small amount of resin, but no glucoside.

Five pounds of drug were then extracted with boiling water, and after reducing to extract by evaporating in an open container, alcohol was added to precipitate the alcohol-insoluble extractive. This was filtered and the precipitate washed thoroughly with alcohol, the filtrate and washings combined and reduced to an extract, the extract then diluted to fluidextract strength with water. A portion

* Scientific Section, A. PH. A., Rapid City meeting, 1929.

of this water-alcohol soluble extract was completely extracted with ether. (Completeness of extraction determined by absence of crystals from last extraction.) The ethereal solution on spontaneous evaporation yielded the typical rosette crystal forms referred to by others, and after several crystallizations from ether, needles were obtained having a melting point 243° to 249° C. The lowering and lack of a sharp melting point is undoubtedly due to the presence of a small amount of resin which is eliminated when crystallized from glacial acetic acid, when the melting point of emodin is reported at 250° C.

(*B*) *Alcoholic Extract.*—Soxhlet extraction made with alcohol instead of water as in *A* with water condenser. The drug was dried and reëxtracted with water. The water extract yielded no emodin, and was devoid of bitter principle, but it showed considerable reduction of Fehling's, undoubtedly due to reducing sugar not removed by alcohol.

A one-pound lot of drug was then digested in alcohol at water bath temperature, but otherwise treated as in *A*, using water to precipitate water-insoluble material. The aqueous filtrate was evaporated to fluidextract strength and a portion shaken out with ether. This again yielded the typical crystalline material (emodin) referred to in *A*, together with resin. Undoubtedly a small amount of glucoside is extracted with ether, since the aqueous wash liquid from these crystals reduces Fehling's solution.

(*C*) *Dialysis.*—The diffusate from aqueous solutions *A* and *B* when concentrated and extracted with ether, resulted in the same crystalline material admixed with resin as in *A* and *B*, and since this offered no special advantage, it was not studied further.

(*D*) *Absence of Crystalline Material from Aromatic Fluidextract of Cascara U. S. P. IX.*—One pint of fluidextract was evaporated and treated as in *A*, the alcoholic filtrate reduced and the extract made to original volume with water. Ether extractions before and after acidifying with hydrochloric acid failed to yield any crystalline material.

(*E*) *Effect of Magnesium Hydroxide on Ether Extractions A and B.*—A portion of the ether-soluble crystalline materials *A* and *B* was dissolved in alcohol, and magnesium hydroxide added in excess. The filtrates gave a negative test for emodin. The precipitate was suspended in water, acidified with hydrochloric acid and extracted with ether. Spontaneous evaporation of the ether resulted in the recovery of the crystalline emodin.

(*F*) *Glucoside.*—A small amount of sugar-bearing material was isolated by washing the crystalline material from ether extractions with water, filtering and extracting the filtrate with ether. On evaporation of the ether, colorless plates were obtained. These were again dissolved in water and it was noted that a small amount of water-insoluble resinous material appeared. This was filtered, yielding a colorless solution, and on extraction with ether the colorless plates were again obtained, but on exposure to air for twenty-four hours they became water insoluble, but reduced Fehling's solution. The fact that they did not oxidize on the first crystallization can be accounted for only by the protection afforded by the small, amber-colored resinous coating. The emodin reaction was negative, indicating perhaps a fraction, resulting from the hydrolysis of the glucoside if such a combination did exist.

Absolute alcohol as a solvent for the glucoside in a powdered extract yields a large amount of extractive, rich in sugar (glucoside) as shown by its reduction of copper. An equal volume of ether added to the alcoholic solution above precipitates a gray amorphous mass, which on exposure to air for a few hours has the appearance of tar, becoming almost insoluble in alcohol, but is completely water soluble. That portion held in solution in the alcohol-ether reduced Fehling's as did the precipitate. The fraction precipitated by the ether-alcohol mixture gave a very faint reaction for emodin, while the soluble portion gave a very positive test. Attempts to crystallize from water or alcohol failed.

(*G*) *Effect of Alkali and Oxidation on the Bitter Principle.*—Three test-tubes containing the aqueous extract which was very bitter were stoppered and set aside. Additions were made as follows and results noted after seventy-two hours:

1. Hydrogen Peroxide. Bitterness appeared to be intensified.
2. Sodium Hydroxide. Bitterness somewhat decreased.
3. Sodium Hydroxide and Peroxide. Entirely debitterized.

(*H*) *Activity of the Various Fractions.*—Because of the willingness of some of our students in helping to rate the activity of the various fractions we were able to make certain eliminations.

From five to eight individuals were given doses proportional to activity established in preliminary tests. Those which in advance tests showed no activity were given in doses when diluted to fluidextract strength from three to sixteen times the U. S. P. dosage.

1. Fluidextract made according to U. S. P. Active. No griping.
2. Same fluidextract but extracted with ether. Active. Very griping.
3. Drug extracted as in *B* and extracted with ether. Active. Very slightly griping.
4. Alcohol-insoluble material from *A*. Inactive.
5. Water-insoluble material from *B*. Inactive.
6. Ether extractives from *A* and *B*. Inactive.

The fact that No. 3 shows practically no griping and No. 2 extremely griping can be accounted for only by the fact that the alcoholic solution was reduced by distillation, thereby obviating oxidation, while the aqueous extraction *A* was reduced in an open container and at a higher temperature.

This procedure was repeated three times and always with the same result. This suggests that emodin either free or combined probably acts as an anti-griping agent, and we shall make such additions later to fraction *H*(*2*) in an effort to verify it.

CONCLUSIONS.

1. Aromatic Fluidextract of Cascara is less active than the Fluidextract because of the formation of a water-insoluble compound of the active agent, presumably the glucoside.
2. The total extractive is no criterion of activity.
3. That both acids and alkalies hydrolize the glucoside at boiling temperature.

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"TURKISH" RHUBARB.*

BY R. A. KONNERTH AND R. E. SCHOETZOW.

Our interest was recently aroused by a sample of Rhubarb offered on the market under the title of "Turkish Rhubarb."

On superficial examination, this lot appeared exceptionally good in that it was well peeled, of good bright color and free from dark centers. The odor was much less smoky than that of the Chinese Drug.

Prior to 1910 "Turkish" Rhubarb was the name given to a Chinese Rhubarb exported to Russia and from there to Turkey. The apparent revival of this custom appeared doubtful.

One of the outstanding differences from the Chinese drug was the complete

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