

**Oleum Rosmarini.**—Oils distilled from samples taken from 15 shipments of Rosmary leaves gave the following data:

|                  | Limits.         | Usual.      | U. S. P. X.    |
|------------------|-----------------|-------------|----------------|
| Yield            | 1.3% to 2.0%    | 1.7%        |                |
| Specific gravity | 0.911 to 0.932  | 0.920       | 0.894 to 0.912 |
| Optical rotation | +4.8° to +19.3° | Below +10°  | 0.0° to +15.0° |
| Refractive index | 1.464 to 1.472  | Below 1.470 | 1.466 to 1.472 |

**Oleum Santali.**—Oils distilled from samples taken from 30 shipments of Sandalwood gave the following data:

|                    |                  |              |                   |
|--------------------|------------------|--------------|-------------------|
| Moisture (in wood) | 4.0% to 8.8%     | 6.0%         |                   |
| Yield              | 0.8% to 8.0%     | 4.0% to 6.0% | N. F. 3.5%        |
| Specific gravity   | 0.964 to 0.979   | Over 0.970   | 0.965 to 0.980    |
| Optical rotation   | -14.0° to -21.8° | Below -16.0° | -15° to -20°      |
| Refractive index   | 1.502 to 1.505   | .....        | 1.504 to 1.508    |
| Santalol           | 89% to 96%       | Over 93%     | Not less than 90% |

**Oleum Sassafras.**—Oils distilled from samples taken from 7 lots of sassafras bark of domestic origin obtained from drug houses gave the following data:

|                  |                |              |                |
|------------------|----------------|--------------|----------------|
| Yield            | 1.0% to 7.0%   | Approx. 4.0% |                |
| Specific gravity | 1.060 to 1.080 | Over 1.070   | 1.065 to 1.077 |
| Optical rotation | +1.7° to +4.0° | Over +3°     | +3° to +4°     |
| Refractive index | 1.522 to 1.532 | Over 1.524   | 1.525 to 1.535 |

The author is continuing investigations on this class of products, and expects to publish further articles from time to time as material is accumulated. These will include interpretations of the data accumulated and it is believed will warrant recommendations for changes in the standards for judging the quality of such products. Sufficient information is already available, for example, to point to the conclusion that much of the practically worthless cinnamon bark now on the market, can be excluded from the country by the adoption of a suitable quality standard.

#### A NOTE ON THE EXTENT OF EMULSIFICATION OF ALKALOID-CONTAINING PREPARATIONS WITH IMMISCIBLE SOLVENTS AT DIFFERENT DEGREES OF $p_H$ .

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Sub-committee No. 6, Proximate Assays, has voted to use the general method of U. S. P. IX in outlining crude drug assay, that is, general directions on proximate assays in Part II with specific directions in each monograph. In Part II of U. S. P. IX under the general directions for alkaloidal assay, the following statement was questioned: "Emulsions are less apt to form in strongly acid or alkaline solutions than in those which are neutral....."

In the assay of alkaloidal-containing crude drugs and their preparations troublesome emulsions frequently occur. In order to determine whether the extent of emulsification would be affected by changes in the  $p_H$  as indicated by the above statement appearing in the U. S. P. IX, portions of five official alkaloid-

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containing preparations were treated, respectively, with varying amounts of *N/1* KOH and *N/1* HCl. These portions were then shaken with a definite volume of water and an immiscible solvent. The time that emulsification persisted was observed in each case. The preparations studied were Fluidextract of Belladonna Leaves, Fluidextract of Cinchona, Fluidextract of Hydrastis, Tincture of Nux Vomica and Tincture of Stramonium. The immiscible solvents used were chloroform, ether, amyl alcohol, benzene and petroleum benzin.

## EXPERIMENTAL.

Two hundred and fifty cc. of each of the five alkaloid-containing preparations were titrated to a  $p_H$  of exactly 7.0 using the quinhydrone electrode. Each of these was then divided into five series of 50 cc. each. The series were treated as follows:

## PH AS MEASURED WITH QUINHYDRONE ELECTRODE.

| Series. | <i>N/1</i> KOH added. | <i>N/1</i> HCl added. | Fldext. Bellad. Fol. | Fldext. Cinchon. | Fldext. Hydrast. | Tr. Nuc. Vom. | Tr. Stramon. |
|---------|-----------------------|-----------------------|----------------------|------------------|------------------|---------------|--------------|
| 1       | 10 cc.                |                       | 9+                   | 9+               | 9+               | 9+            | 9+           |
| 2       | 5 cc.                 |                       | 8.7                  | 9+               | 9+               | 9+            | 9+           |
| 3       |                       |                       | 7.0                  | 7.0              | 7.0              | 7.0           | 7.0          |
| 4       |                       | 5 cc.                 | 5.0                  | 1.8              | -1.0             | 1.3           | 1.7          |
| 5       |                       | 10 cc.                | 3.8                  | 1.4              | -1.0             | -1.0          | -1.0         |

The  $p_H$  of the resulting solutions was not uniform, probably due to the fact that a hydro-alcoholic solution was titrated which, with the quinhydrone electrode, is hardly dependable especially when different alcoholic concentrations are represented as was the case in these experiments.

Each of the alkaloid-containing preparations was shaken with the five immiscible solvents under each of the five  $p_H$  degrees—a total of 125 observations. The time that emulsification persisted in minutes is shown in the following table.

## TIME OF EMULSIFICATION IN MINUTES.

|                      | Series. | CHCl <sub>3</sub> .            | Ether.                        | AmOH.                         | Benzene.                       | Benzin.                       |
|----------------------|---------|--------------------------------|-------------------------------|-------------------------------|--------------------------------|-------------------------------|
| Fldext. Bellad. Fol. | 1       | 1 <sup>3</sup> / <sub>4</sub>  | 2 <sup>3</sup> / <sub>4</sub> | 6 <sup>3</sup> / <sub>4</sub> | 20.0                           | *30+                          |
|                      | 2       | 1 <sup>1</sup> / <sub>4</sub>  | 3                             | 8 <sup>3</sup> / <sub>4</sub> | 10 <sup>1</sup> / <sub>2</sub> | 30.0                          |
|                      | 3       | 2 <sup>1</sup> / <sub>2</sub>  | 3 <sup>1</sup> / <sub>4</sub> | 6 <sup>1</sup> / <sub>2</sub> | 1 <sup>1</sup> / <sub>4</sub>  | 4.0                           |
|                      | 4       | 3 <sup>1</sup> / <sub>2</sub>  | 24.0                          | 4.0                           | 13.0                           | 4 <sup>1</sup> / <sub>2</sub> |
|                      | 5       | 12 <sup>1</sup> / <sub>4</sub> | 24.0                          | 5 <sup>1</sup> / <sub>2</sub> | 13.0                           | 1 <sup>1</sup> / <sub>2</sub> |
| Fldext. Cinchon.     | 1       | 30+                            | 30+                           | 30+                           | 20.0                           | 30+                           |
|                      | 2       | 30+                            | 30+                           | 30+                           | 30+                            | 30+                           |
|                      | 3       | 5 <sup>1</sup> / <sub>4</sub>  | 30+                           | 30+                           | 30+                            | 20.0                          |
|                      | 4       | 2.0                            | 15.0                          | 2 <sup>1</sup> / <sub>4</sub> | 30.0                           | 30+                           |
|                      | 5       | 1 <sup>1</sup> / <sub>2</sub>  | 20.0                          | 2.0                           | 30+                            | 30+                           |
| Fldext. Hydrast.     | 1       | 2 <sup>1</sup> / <sub>2</sub>  | 2 <sup>1</sup> / <sub>2</sub> | 30+                           | 10.0                           | 30+                           |
|                      | 2       | 30+                            | 2 <sup>1</sup> / <sub>2</sub> | 30+                           | 30+                            | 30+                           |
|                      | 3       | 30+                            | 30+                           | 5.0                           | 30+                            | 30.0                          |
|                      | 4       | 30+                            | 30+                           | 30.0                          | 30+                            | 2 <sup>1</sup> / <sub>2</sub> |
|                      | 5       | 30+                            | 30+                           | 30.0                          | 30+                            | 4.0                           |
| Tr. Nuc. Vom.        | 1       | 30+                            | 14.0                          | 18.0                          | 20.0                           | 20.0                          |
|                      | 2       | 30+                            | 30+                           | 15.0                          | 20.0                           | 20.0                          |
|                      | 3       | 4.0                            | 30+                           | 30+                           | 9.0                            | 3.0                           |
|                      | 4       | 1 <sup>1</sup> / <sub>4</sub>  | 30+                           | 30+                           | 2 <sup>1</sup> / <sub>2</sub>  | 3 <sup>1</sup> / <sub>4</sub> |
|                      | 5       | 1 <sup>1</sup> / <sub>4</sub>  | 30+                           | 30+                           | 1 <sup>3</sup> / <sub>4</sub>  | 3 <sup>1</sup> / <sub>4</sub> |

|              |   |                 |      |      |     |     |
|--------------|---|-----------------|------|------|-----|-----|
| Tr. Stramon. | 1 | 9 $\frac{1}{2}$ | 30+  | 30+  | 30+ | 30+ |
|              | 2 | 10.0            | 30+  | 30+  | 30+ | 30+ |
|              | 3 | 25.0            | 30+  | 30+  | 30+ | 30+ |
|              | 4 | 4.0             | 7.0  | 11.0 | 3.0 | 3.0 |
|              | 5 | 4 $\frac{1}{2}$ | 11.0 | 12.0 | 3.0 | 3.0 |

\* Emulsification persisted more than 30 minutes.

#### CONCLUSION.

The results obtained indicated no general uniformity. Fldext. Bellad. Fol. with the various solvents showed in general least emulsification at the neutral point; Tr. Stramon showed least emulsification in acid solution; the remaining three showed no uniformity. Considering the solvents, petroleum benzin with one exception, Fldext. Cinchon. showed least emulsification in acid solution. The remaining solvents showed no uniformity. The results indicate that the statement "Emulsions are less apt to form in strongly acid or alkaline solutions than in those which are neutral" is true only in specific instances.



*Photo, Courtesy Monsanto Chemical Works*

#### RESTORED CHEMICAL LABORATORY AND PHARMACY OF CASTLE OF SAINT ANGELO.

The castle was built by Hadrian in 136; nearly destroyed in 1378. Restored by the Popes. The reproduction of the chemical laboratory and pharmacy was completed in the 17th century; it is located in the rooms on the ground floor.