organic salts of those metals whose sulphates are stable; in the case of salts of K and Na, persulphates are likely to be formed, giving an excessive weight, but this can be prevented by treatment with ammonia or ammonium carbonate solution, evaporation to dryness and re-ignition.

4. Ignition with $(NH_4)_2SO_4$ as above; this does not form persulphates.

5. In the case of organic salts, the acid of which when liberated may be removed by immiscible solvents, as ether, chloroform, etc., a measured excess of an Acid V. S. may be added, the organic acid extracted with the solvent and the excess of Acid V. S. titrated with an Alkali V. S., (in the aqueous portion), methyl orange indicator.

6. Extraction of the acid as in (5), but allowing the volatile solvent to evaporate at ordinary temperature and weighing the residue of free organic acid.

The following modification of (5) and (6) gave satisfactory results:

0.200 to 0.500 gm. of the salt, placed in a separator and dissolved in 20 to 30 cc. water, add 2 to 4 cc. N/1 H_2SO_4 (or HCl) (note); a mixture of ether 1 vol. and chloroform 2 vols. is used for extraction, of which three portions of 15-20 cc. each are usually sufficient for complete extraction; to the mixed ether-chloroform extractions in a flask, add a little water (10cc.) and titrate with N/10 NaOH V. S., phenolphthalein indicator; or, the first portion separated may be titrated, then the second added, titration continued and so on until no more Alkali V. S. is required to give a permanent pink color to the aqueous layer after thorough agitation.

The following results were obtained:

Sodium Salicylate	Ammonium Salicylate	Strontium Salicylate	Sodium Benzoate	Lithium Benzoate
98.088%	98.28%	98.38%	99.063%	99.059%
99.32%	98.147%	98.38%	97.41%	
99.72%				
100.039%				
100.12%				
98.82%				

Note.—The amount of acid required here is slightly more than the calculated amount to decompose all the salt (e. g. each 0.100 gm. sodium salicylate requires a little over 0.6 cc. N/1 V. S. for an excess).

Chloroform is the most convenient solvent, but more of it is required than of ether; the latter is less convenient to separate, but a heavy mixture of the two has been found quite satisfactory.

CHEMICAL LABORATORY OF THE PHILADELPHIA COLLEGE OF PHARMACY.

THE QUALITY OF DRUGS.*

W. A. PEARSON, PHILADELPHIA, PA.

Asafetida.—Asafetida is a drug that cannot be powdered without considerable loss of the active volatile constituents present. Of five lots of the pow-

* Notes made from the files in the Analytical Department of Smith, Kline and French Co. upon the drugs examined from June 1, 1911, to June 1, 1912.

dered drug examined the ash amounted to 34.3%, 38.9%, 37.7%, 35.8% and 26.4%, and the alcohol soluble portions respectively were 29.5%, 31.1%, 27.6%, 33.1% and 37.9%.

There was an improvement in the quality of the samples of crude drug examined. One trial sample contained 60% of alcohol soluble material and only 6% of ash. However when the consignment arrived and two samples from the cases examined the alcohol soluble material amounted to 38.7% and 37.6% and the ash to 17.1% and 22.4% and two samples of the best lumps in the cases contained 57.8% and 58.3% of material soluble in alcohol and 6.8% and 15.6% respectively of ash.

Acetic Acid.—Two samples were rejected because they were dark in color.

Antimony and Potassium Tartrate.—A trial sample was examined which contained calcium, chlorides, heavy metals, and assayed only 94%.

Tannic Acid.—One sample was examined which contained 1% of ash. This drug continues to be of different colors and quality, no doubt due to its source and method of preparation.

Apiol Green.—The physical appearance and solubility of commercial samples of this drug vary considerably. The color varies from light green to dark brownish green and some samples will mix clear with an equal part of alcohol and some will not. All but one of the four samples examined when mixed with ten times their volume of alcohol formed considerable deposits and only one sample mixed clear with 4 volumes of olive oil.

Alkanet Root—One consignment of this drug was rejected because it was inferior in appearance and was deficient in the amount of coloring matter present. About half of the material was in the form of a gray powder and only 2.2% of Anchusin was present. After rejecting this lot, a consignment was supplied which contained 9.7% of Anchusin.

Burgundy Pitch.—The analytical results on two samples of this drug were as follows:

	Sample No. 1	Sample No. 2
Acid Number	135.0	142.8
Ester Number	32.2	22.0
Saponification Number	167.2	164.8
Iodine Number	131.2	132.1

After the determination of the saponification number the solution was acidified and the alcohol evaporated. This treatment left a clear, dark brown and brittle residue from No. 2, while the residue from No. 1 after similar treatment was a dark brown, sticky, viscid substance with a turpentine odor.

Precipitated Calcium Phosphate.—It is difficult to obtain lots of this product that will comply strictly with U. S. P. specifications. Most of the samples examined failed to respond to test for absence of acid calcium phosphate, excess of chlorides, heavy metals or magnesium. One sample contained 7.5% of magnesium.

Calomel.—This salt becomes dark in color on standing even when protected from the light. It may take a year or more for the color to change perceptibly.

Cannabis Indica.—A Mexican and an American grown sample were found which were much below standard.

Celery Seed.—Microscopical sections were made of the carpels of medicinal

celery seed and compared in detail with the structure of the domestic celery seed. The medicinal celery seed usually contains more than six oil ducts in each carpel while the domestic was never found to have more than six.

Chloroform.—Four samples were rejected on account of the presence of chlorinated products.

Copper Carbonate.—An analysis of a sample of commercial Copper Carbonate was made with the following results:

Solution in hydrochloric acid	Complete
Sulphates	Considerable present
Strength computed as metallic copper	50.2%
Strength computed as copper oxide (CuO)	62.83%

The theoretical percent of metallic copper in the normal copper carbonate ($CuCO_3$) is 51.4% and in the basic Copper Carbonate ($Cu_2(OH)_2CO_3$) is 57.5%.

Cacao Butter Substitute.—This product, known as Vegetable Cacao Butter, has recently been quoted. It is labeled as hard palm kernel fat with cocoa flavor.

	Grade No. 1	Grade No. 2	Grade No. 3
Physical Appearance	Yellowish white	Yellowish white	White
Melting Point	30.5°C	31.5°C	32.5°C
Specific Gravity at 25° C.	0.8976	0.890	0.915
Acid Number	4.9	1.08	1.2
Saponification Number	256.0	253.0	258.3
Iodine Value	8.995	7.95	5.21

By comparing the above data with the specifications for Cacao Butter given by the U. S. P. it may be seen that all of the samples above have higher saponification numbers and lower specific gravities and iodine values than U. S. P. permits.

The U. S. P. requires the saponification value to be between 188 and 195, the specific gravity at 25° C. from 0.970 to 0.976 and the iodine value not less than 33 nor more than 38.

The odors of grades number one and number two are very similar to genuine cacao butter, but grade number three has practically no odor.

The taste of all samples resembles that of cacao butter but is somewhat different. It would probably be difficult to detect grades number one and number two without a chemical analysis. Undoubtedly this material could be used as an adulterant of cacao butter, but owing to the considerable variation in specific gravity, saponification value and iodine value a small admixture could be detected.

Disinfectants.—The scientific standardization of disinfectants has now become a possibility through the use of the Rideal-Walker Method, and not only can the relative merits of disinfectants be accurately stated, but disinfectants may be standardized under a variety of conditions and the best product for a specific purpose ascertained.

Digitalis Leaves.—One sample was tested which was not sufficiently active physiologically. 0.23 gm. of the drug was required to kill a 250 gm. guinea pig.

Ergot.—One solid extract, three powdered extracts, two fluid extracts, a sample of Ergotin Bonjean and an active extract of this drug were found which would not raise the blood pressure of a dog nor darken the comb of a rooster.

Glycerin.—Two samples were rejected on account of their yellow color and excess of butyric acid.

Goose Grease.—One sample was rejected on account of its rancid odor, objectionable deposit and an excessive (7.14) acid number.

Gambir Cubes.—The U. S. P. states that Gambir should contain at least 70% of material soluble in alcohol and that when incinerated it should leave not more than 5% of ash. The five samples examined contained the following proportions of alcohol soluble matter and ash:

	Soluble in Alcohol	Ash
Sample No. 1	78.5%	5.2%
Sample No. 2	82.8%	4.6%
Sample No. 3	80.2%	5.2%
Sample No. 4	82.0%	3.7%
Sample No. 5	77.6%	4.9%

Honey.—One sample was found which was evidently adulterated with cane sugar.

Indian Gum.—This gum is no doubt used even at the present time as an adulterant of tragacanth and has to some extent replaced tragacanth. Nine samples of Indian gum were compared and the mucilages made from them were found to vary considerably both in color and consistency. A good sample of Indian gum will form a stiff jelly with 50 parts of water, and the jelly is not very much darker than a similar jelly made with tragacanth.

Ferrous Lactate.—Two samples were compared. Both samples contained excessive amounts of sulphates, were not completely soluble in forty parts of water and left excessive residue on ignition.

Japanese Hemp Seed.—The sample examined was somewhat smaller and lighter in color than the ordinary variety.

Vienna Lime.—One sample was analyzed with the following results:

Physical Appearance	Yellowish white powder
Silica (SiO_2)	0.75%
Iron and Aluminum Oxides (Fe_2O_3, Al_2O_3)	0.75%
Calcium Oxide (CaO)	61.47%
Magnesium Oxide (MgO)	36.75%

Leptandrin.—Two samples were examined with the following results:

	Alcohol Soluble	Ash
Sample No. 1	79.95%	3.1%
Sample No. 2	94.13%	1.175%

Mercuric Chloride.—One sample was found which was dark in appearance.

Magnesium Sulphate.—The U. S. P. states that this salt must be 99.7% pure, but gives no tests for limit of chlorides. When the chlorides are estimated in many samples of commercial magnesium sulphate the amount of chlorides present computed as magnesium chloride amount to over 0.3%, hence these products cannot be of U. S. P. quality.

Peppermint.—A sample of peppermint leaves and one of peppermint herb were examined which contained spearmint leaves and spearmint herb respectively.

Olive Oil.—When the government permitted olive oil to be imported duty free, after being denatured with 8 ounces of oil of rosemary to each barrel, the quality of the second grade of olive oil, which had previously been used for lubricating purposes, was denatured, and the quality of the lubricating oils became lower. This may have resulted from various causes, such as the fact that 8 ounces of oil of rosemary in each barrel could scarcely be detected and the denatured olive oil could no doubt be used in many cases in place of the

duty paid oil. At any rate the acid numbers of the commercial lubricating olive oils were higher than before. Previously the acid numbers were approximately 10 to 15, but samples were found during the past year which had acid numbers as follows: 31.2, 31.0, 27.7, 27.7, 31.0 and 40.1. Lots were obtained however after some difficulty which were equal in quality to those obtained in former years.

Oil of Citronella. The geraniol content of three lots was estimated with the following results: 49.2%, 52.7% and 56%.

Oil of Cajuput.—Traces of copper were found in both samples examined, and one sample had an optical rotation of -3° 21′, which is over one degree greater than the U. S. P. allows. It is stated that authentic samples have an optical rotation up to -3° 40′.

Cottonseed Oil.—One sample had a saponification number of 188 and an iodine number of 109.

Oil of Turpentine.—The quality of this commodity has greatly improved during the past year but many samples are not colorless as the Pharmacopœia demands. Until the price is dependant upon the amount of color present we cannot expect to uniformily obtain commercially a colorless product.

Oil of Wintergreen.—One lot was rejected on account of the probable addition of synthetic methyl salicylate.

Oil of Sweet Almond.—One sample was examined which had the following characteristics:

Specific Gravity at 25°C	0.9161
Acid Number	1.98
Saponification Number	194.8
Iodine Number	110.27
Bromine Number	63.0
U S. P. Nitric Acid Test	Abnormal
Olive, Arachis, Cottonseed, Sesame and other fixed oils	Negative
Bielier's Test	Abnormal
Nitric Acid Test (A. J. P. 1886, page 408)	Abnormal
Congealing Point of fatty acids	0°C
Melting Point of mixed fatty acids	+3°C

This oil did not conform to U. S. P. specifications in several respects and we considered that it contained apricot kernel oil.

Oil of Pennyroyal.—Six samples were examined with the following results:

	Specific Gravity at 25°C	Optical Rotation at 25°C	Solubility in 70% Alcohol
Sample No. 1	0.9197	+30° 14′	In 0.7 parts
Sample No 2	0.9242	+27° 44′	In 0.7 parts
Sample No. 3	0.9196	+29° 40′	In 0.7 parts
Sample No. 4	0.9218	+28° 29′	In 0.8 parts
Sample No. 5	0.9222	+25°	In 0.8 parts
Sample No. 6	0.9195	+24° 58′	In 0.8 parts

Poppy Seed Oil.—One sample was examined which would not dissolve in 100 volumes of cold alcohol. Although Lewkowitsch states that poppy seed oil should be soluble in 25 volumes of cold alcohol and six volumes of boiling alcohol, we have never examined an oil that would conform with these specifications.

Lard Oil.—One sample was found which had an iodine value of 81.67. The U. S. P. states that lard oil should have an iodine value of not less than 56 nor more than 74.

Rennin.—One sample, labeled "Free from salt," was examined for chlorides. A considerable quantity was found.

Resorcinol.—This product frequently has a slight pink cast which may gradually become darker. One lot was rejected on account of its inferior appearance.

Resin of Jalap.—One sample was examined which contained 3.8% of moisture and did not strictly fulfill the U. S. P. specifications for limit of saponifiable substances.

RosinOil.—Two samples were examined with the following results:

	Sample A	Sample B
Color	Dark reddish brown	Reddish brown
Specific Gravity at 25°C	0.989	0.9043
Acid Number	56.37	41.37
Saponification Number	69.85	56.94
Ester Number	13.48	15.57
Iodine Number	84.3	41.6
Unsaponifiable matter	59.8%	73.9%

We considered that Sample B was adulterated with mineral oil.

Resin of Scammony.—Four samples were examined with the following results:

	No. 1	No. 2	No. 3	No. 4
Proportion soluble in alcohol	98.85%	98.65%	99.25%	99.1%
Proportion soluble in ether	99.55%	98.25%	55.54%	55.41%
Proportion soluble in turpentine	Insoluble	Insoluble	Insoluble	Insoluble
Appearance of NaOH solution	Cloudy	Cloudy	Cloudy	Cloudy
Appearance of NaOH solution after adding acid	Cloudy	Cloudy	Cloudy	Cloudy
Acid Number	17.7	18.5		
Ester Number	224.1	209.0		
Saponification Number	241.8	227.5		
Sulphuric Acid Test (A. J. P. April 6-1912)	Normal	Normal	Abnormal	Abnormal

We considered samples No. 3 and No. 4 of inferior quality probably prepared from Mexican Scammony.

Fluidextract of Sanguinaria.—One sample was assayed which contained only 1.8 gm. of alkaloids in 100 cc.

Spigelia.—One lot was examined which contained roots of seven different plants, however most of the sample consisted of spigelia marilandica.

Sodium Bromide.—One sample was rejected on account of an excessive amount of bromate present.

Saffron.—Several samples were examined which would not conform to 1890 U. S. P. test for absence of coal tar colors.

Purified Talc.—One sample was examined which lost 6.1% on ignition. The loss was due mainly to the presence of carbonates.

Venice Turpentine, Artificial.—Three samples were examined with the following results:

	No. 1	No. 2	No. 3
Acid Number	106.8	118.0	127.7
Ester Number	21.2	16.0	18.1
Saponification Number	128.0	134.0	145.8

Wild Cherry Bark.—Two lots were rejected because the bark failed to conform with the U. S. P. description. Most of the barks on the market are too thick and old.

I desire to express my indebtedness to my co-workers, J. G. Roberts, H. M. Sechler, M. Becker and R. I. Grantham for much of the analytical work connected with the above contribution.—ANALYTICAL DEPARTMENT, SMITH, KLINE & FRENCH CO., JUNE 12, 1912.