

is allowed to stand over night. The mass is then washed with a saturated solution of sodium bicarbonate and crystallized from 50% alcohol. The dried product melts with decomposition at 164° C. Baeyer¹ has prepared this compound in the same manner.

The dialkylamino derivatives were prepared by reacting the appropriate dialkylamine in excess (5%) with the bromoacetyl urea. Upon slight warming the reaction starts and soon melts, then solidifies. The mass is finally heated for two to three hours on a boiling water-bath to complete the reaction. The hard mass is then broken up in a mortar and extracted with ether followed by thorough washing with a saturated sodium bicarbonate solution. The product is then crystallized from a hot solution of 30% alcohol. The compounds were obtained in yields varying from 40% to 78%.

	Nitrogen Found.	Nitrogen Theoretical.	M. p. ° C.
1. Diethylamino acetyl urea	24.1%	24.27	102
2. Di- <i>n</i> -propylamino acetyl urea	20.7	20.89	123
3. Di- <i>n</i> -butylamino acetyl urea	18.3	18.34	122
4. Di-iso-butylamino acetyl urea	18.3	18.34	68
5. Di- <i>n</i> -amylamino acetyl urea	16.2	16.34	97
6. Di-iso-amylamino acetyl urea	16.2	16.34	99

The compounds were tested in the pharmacological laboratory of the Medical School and found to possess no hypnotic properties.

A study of the solubilities showed the compounds hydrolyze comparatively readily even at room temperature. It was therefore not possible to study their distribution. This hydrolysis may possibly account for their pharmacological inactivity.

SUMMARY.

The following new compounds have been prepared and studied: diethylamino, di-*n*-propylamino, di-*n*-butylamino, di-iso-butylamino, di-*n*-amylamino and di-iso-amylamino, acetyl ureas.

ACKNOWLEDGMENT.

The writer is grateful to Dr. C. D. Leake of the Medical School for the pharmacological testing of these compounds.

THE DETERMINATION OF FREE ALKALI IN SOFT SOAP.*

BY ROBERT M. LINGLE.

While attempting to accurately determine the amount of free alkali in soft soap the author has constantly observed wide discrepancies in following the U. S. P. method and by employing a direct titration method in which the use of a filter is omitted. The alkalinity figure in the direct titration procedure in the presence of neutral ethyl alcohol is invariably higher than is obtained according to the U. S. P. method. It has also been observed that when phenolphthalein indicator is added to the alcoholic solution, the resulting alkaline color either becomes fainter or disappears when passed through a filter.

These observations prompted a search of the literature to determine if the method which is official in the U. S. P. is the best procedure that has been devised,

¹ Baeyer, A., 130, 156 (1864).

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and to learn if an excessive amount of free alkali is being permitted by the official standard.

A reference by Simmons (1) was found to suggest that all soaps except the lowest grade household soaps should be practically free from caustic soda or potash. Oculus (2) states that a content of 0.08 per cent sodium hydroxide may cause irritation when applied to a sensitive skin.

In an effort to trace the origin of the official procedure for determining free alkali in soft soap three basic methods have been described in the literature. Briefly, these methods are indicated as follows:

1. Barium chloride precipitation method.
2. Salting-out method.
3. Alcohol solubility method.

The first method, in which barium chloride is used to separate the carbonates, stearates, oleates, etc., was devised by Herrmann (3) and (7). It is given official recognition in the British Pharmacopœia. The writer attempted to apply this procedure to several samples of soft soap and found difficulty in making a satisfactory separation either by filtering, decanting or by the use of the centrifuge.

Poethke (4), Kling, Genin and Florentin (5) and Bosshard and Huggenberg (6) and (8) investigated the barium chloride precipitation method and found many objections to its use.

The salting out method was the object of considerable study by Dietrich (9), Newington (10), Davidsohn (11) and (12), Bergell (13), Knigge (14), Schuttig (15) and Banninger (16) with the result that many sources of error were found.

Hope (17) devised the third basic method, known as the alcoholic solubility method. This has been investigated very thoroughly by Bosshard and Huggenberg (6), Izmailski (18), Newington (10), Simmons (19), G. Knigge (20), Schutte (21), Divine (22), Dudley and Pease (23), Lewkowitsch (24), Allen (25), U. S. Bureau of Standards (26) and Hurst (27). Most of these investigators found sources of errors in using this method.

At the present time, therefore, no method appears to have been devised which, on close scrutiny, does not yield some source of error. The alcohol method of the U. S. P. is in slight error for the reason that a small amount of atmospheric carbon dioxide is absorbed during the manipulation, and traces of alkali carbonates and silicates are dissolved in the alcohol. However, it seems that the degree of deviation from the true result is so slight as to be almost negligible when the nature of the product is considered.

There is also the problem as to whether some of the free alkali existing in the soap at the time of manufacture is gradually neutralized by the fatty acids, so that after several months a soap which originally contained 0.25 per cent free alkali becomes practically neutral. Experiments are now being conducted in order to obtain some information concerning this problem.

The author is grateful to E. J. Hughes for his many helpful and valuable suggestions.

SUMMARY.

1. A number of references are cited to show that the present U. S. P. allows more free alkali in soft soap than is desirable in a soap which is intended for medicinal use.

2. An examination of the literature has been made for the purpose of studying the various methods that are described for the determination of free alkalinity in soft soap.

3. Objections have been found to all of the available methods, although the procedure of the U. S. P. is more rapid, more convenient to use, and gives results which are sufficiently accurate for the nature of the product.

4. Experiments are being conducted to determine whether some of the free alkali is gradually neutralized by the fatty acids in the soap.

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A NEW GLUCOSIDE FROM BLUEBERRY LEAF.*

BY N. KENNETH EDGARS.

The use of blueberry leaves or extracts made therefrom as a remedy for diabetes is not new to medicine. Wagner (1) in 1925 and Allen (2) in 1927 both experimented with extracts and concentrates made from these leaves and proved that at least one hypoglycemic, or blood sugar lowering, substance exists and can be extracted in a stable form. Their product, which was named Myrtilin erroneously, is an example of such an extract. It was not pure enough to give uniform results, but its discovery marked an important step in the history of the pharmacology of this plant. The name Myrtilin properly belongs to a galactoside which exists in the fruit of this genus and discovered by Willstätter in 1915 (3).

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