in which case considerable difficulty was encountered in making correct interpretations. It will be noted that Lehmann's method yields results that are uniformly higher than those obtained by the other methods, except for sodium cacodylate, and that this method appears to be best adapted for the assay of arsphenamine and neoarsphenamine. On the other hand, the Ewins method, the perchloric acid method and the modified Ewins method all appear to give results that agree more closely with the theoretical amounts which would indicate that the higher figures obtained by Lehmann's method may be due to the nature of the method itself rather than to any loss of arsenic through volatilization in the three other methods.

In conclusion, our experience would indicate that the Ewins method is definitely superior for the assay of arsanilic acid, sodium cacodylate and carbarsone. The substitution of an arsenic-free cigarette paper in place of starch in the digestion mixture makes the Ewins method considerably more rapid, without apparently affecting its accuracy.

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## DIALKYLAMINO ACETYL UREAS.*

BY T. C. DANIELS.[1]

There are two general types of ureas that have found relatively wide application as hypnotics, the brominated acyl ureas (Bromural, Adalin) and the cyclic ureids of the barbital type.

An examination of the literature revealed that the dialkylamino acetyl ureids have not been examined and it was decided to prepare the following substances for study: Diethyl, di-*n*-propyl, di-*n*-butyl, di-iso-butyl, di-*n*-amyl, and di-iso-amyl-amino acetyl ureas.

### EXPERIMENTAL.

Bromacetyl urea was prepared by reacting 75 Gm. of urea (xs) with 224 Gm. of bromacetyl bromide. The acyl halide was added slowly with stirring to the urea. The reaction product first melts, then solidifies, and the reaction mass after heating on a water-bath for forty-five minutes

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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. Baeyer[1] 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-*n*-propylamino acetyl urea | 20.7 | 20.89 | 123 |
| 3. | Di-*n*-butylamino acetyl urea | 18.3 | 18.34 | 122 |
| 4. | Di-iso-butylamino acetyl urea | 18.3 | 18.34 | 68 |
| 5. | Di-*n*-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.

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## 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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