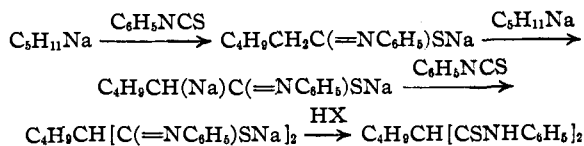


quence of reactions shown below. Small quantities of the expected products were indeed obtained.



A peculiarity in this preparation was the repeated failure to obtain isolable quantities of either product when a creased flask³ was used as the reaction vessel. In such cases the product was a material which has not yet been characterized. Increases in yield of 50 to 100% in preparation of certain Grignard reagents and improvement in other reactions have been noted in this Laboratory when the experiments have been carried out in flasks of this special design but this is the first instance where the products appeared different.

Reactions of amylsodium with carbon disulfide, sulfur dioxide, and sulfur trioxide in hope of obtaining various sulfur-containing acids gave mixtures which were not separated readily.

Experiments

Butyldithiomalon Dianilide.—Amylsodium was prepared from 37 g. of *n*-amyl chloride with 20 g. of fine sodium sand in petroleum ether at 0° in an ordinary 3-neck flask arranged in the conventional manner,⁴ described before. Phenyl isothiocyanate, 47 g. (0.35 mole) was added dropwise over a period of ten minutes. After stirring for thirty minutes, the mixture was decomposed with 30 ml. of alcohol followed by 150 ml. of water. The two latter were then separated. The aqueous layer was acidified and extracted with ethyl ether and with ethyl acetate. The combined extracts were evaporated to a dark brown non-crystalline solid, and the latter extracted with petroleum ether and with ligroin. Crystals melting from 67 to 68° were obtained.

The organic layer in turn yielded a heavy residual oil on evaporation which was distilled in a Hickman alembic at 5 to 10 microns. The fraction boiling from 100 to 135° slowly crystallized yielding a product identical with the crystals obtained by extraction of the aqueous layer. The combined product was recrystallized from alcohol giving 1.2 g. (2% based on the amyl chloride used) of material melting at 67–68°.

Anal. Calcd. for $\text{C}_{19}\text{H}_{22}\text{N}_2\text{S}_2$: S, 18.71; N, 8.19. Found: S, 18.3; N, 8.10.

Upon saponification with excess alkali aniline was evolved and the alkali consumed was equivalent to the above formula. Saponification equivalent calcd., 171; found, 169.

Phenyldithiomalon Dianilide.—Preparation of *n*-amylsodium was carried out as before. Toluene, 33 g., was

then added and the mixture refluxed for one hour, after which phenyl isothiocyanate, 47 g., was added dropwise. Decomposition and separation were carried out in the same manner as before. Crystals from the aqueous layer were identical with those obtained from the organic layer by distillation at 2 to 3 microns at 120 to 150°. The combined product was carefully sublimed at 60–80° and 2 microns giving a clean white material melting 66 to 67°; yield 1.5 g. or 2.4% based on the amyl chloride used.

Anal. Calcd. for $\text{C}_{21}\text{H}_{18}\text{N}_2\text{S}_2$: S, 17.68; N, 7.74. Found: S, 17.1; N, 7.70. Aniline was detected upon hydrolysis with alkali. Saponification equivalent. Calcd., 181; found, 184.

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RESEARCH LABORATORY OF ORGANIC CHEMISTRY
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
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The Identity of α - and β -Earleine with Betaine and Choline, Respectively

Y ARTHUR STEMPEL AND ROBERT C. ELDERFIELD

In a previous communication from this Laboratory¹ the isolation of two non-toxic, nitrogenous bases from *Astragalus earlei*, or Big Bend locoweed, was described. These were assigned the empirical formulas $(\text{C}_{16}\text{H}_{37}\text{N}_3\text{O}_7)_x$ and $(\text{C}_{16}\text{H}_{37}\text{N}_3\text{O}_4)_x$ on the basis of analytical data on their salts and were named α - and β -earleine, respectively. With the limited amount of material at hand it was not possible to characterize the bases further at that time. In the meantime we have secured additional amounts of the weed and have identified the bases as betaine and choline, respectively. A study of the thermal decomposition of " β -earleine" provided the clue for the correct interpretation of its nature, from which the identity of " α -earleine" with betaine was surmised. From the decomposition products of " β -earleine" we have identified trimethylamine and acetaldehyde. A sample of " β -earleine" produced a typical choline effect on white mice when tested in the Parke, Davis laboratories through the kind cooperation of Dr. Oliver Kamm. The names α - and β -earleine should, therefore, be stricken from the literature.

Experimental

Identification of Betaine.—The isolation was carried out as previously described.¹ The picrate melted at 184° and gave no depression in melting point when mixed with betaine picrate.

Anal. Calcd. for $\text{C}_6\text{H}_{11}\text{O}_2\text{N}\cdot\text{C}_6\text{H}_5\text{O}_7\text{N}_3$: C, 38.2; H, 4.1; N, 16.2. Found: C, 38.3; H, 4.1; N, 15.6.

The styphnate melted at 186–188° (dec.).

(3) Morton, *Ind. Eng. Chem., Anal. Ed.*, **11**, 170 (1939).

(4) Morton and Richardson, *THIS JOURNAL*, **62**, 123 (1940).

(1) Pease and Elderfield, *J. Org. Chem.*, **5**, 192 (1940).

Anal. Calcd. for $C_8H_{11}O_2N \cdot C_6H_5O_2N_3$: C, 36.5; H, 3.9; N, 15.5. Found: C, 36.8; H, 4.1; N, 15.0.

The hydrobromide melted at 225°.

Anal. Calcd. for $C_8H_{11}O_2N \cdot HBr$: C, 30.3; H, 6.1; N, 7.0; Br, 40.4. Found: C, 30.8; H, 6.2; N, 7.0; Br, 40.1.

Identification of Choline.—When "β-earleine" was decomposed by heating in a stream of nitrogen, trimethylamine was isolated from the products by means of its picrate, which melted at 228–229°, and gave no depression of melting point when mixed with a known sample. Acetaldehyde was also isolated as the 2,4-dinitrophenylhydrazone which melted at 164°.

Anal. Calcd. for $C_8H_9O_4N_4$: C, 42.9; H, 3.6; N, 25.0. Found: C, 43.1; H, 3.4; N, 24.8.

The picrate melted at 247° and no depression was observed when it was mixed with choline picrate.

Anal. Calcd. for $C_8H_{11}ON \cdot C_6H_5O_2N_3$: C, 39.8; H, 4.8; N, 16.9. Found: C, 39.8; H, 4.7; N, 16.9.

The picrate of the acetate, prepared with acetic anhy-

dride, melted at 111.5–112.5° and gave no depression in melting point with acetylcholine picrate.

Anal. Calcd. for $C_7H_{15}O_2N \cdot C_6H_5O_2N_3$: C, 41.7; H, 4.8; N, 15.0. Found: C, 42.0; H, 4.9; N, 15.1.

The micro analyses reported were performed by Mr. Saul Gottlieb of these laboratories.

We wish to acknowledge our appreciation for the kind coöperation of S. B. Penick and Co., of New York City, and of Parke, Davis and Co., of Detroit, Michigan, in carrying out preliminary extraction of the weed which was secured with the aid of Dr. Frank P. Mathews, of the Loco Weed Laboratory, Alpine, Texas. Our thanks are also due the American Academy of Arts and Sciences for a grant for technical help in this investigation.

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NEW BOOKS

The Chemical Composition of Foods. By R. A. McCANCE and E. M. WIDDOWSON, Department of Medicine, University of Cambridge. Chemical Publishing Co., Inc., 148 Lafayette St., New York, N. Y., 1940. 150 pp. 14.5 × 22.5 cm. Price, \$2.50.

The data recorded in this book were accumulated as the result of the chemical analyses of foods supported by the Medical Research Council (England) in the laboratory of the senior author. Accordingly, they do not represent a compilation of the data recorded in the literature. This, at least, implies that the analytical methods employed were uniform and that the results are strictly comparable. In the introduction reference is made to certain difficulties involved in the choice of analytical procedures, particularly in the instance of the fat determination, where the Soxhlet method gives much lower results than the Liebermann method with certain foods. The authors also discuss the doubtful propriety of recording phytin (or phytic acid) phosphorus as "non-available," and of classifying as "available" that portion of the iron which reacts with α,α'-dipyridyl, as has been done in two special tables in the closing portion of the book. The occasions for substantial gains in certain elements during the processing for consumption are also presented. Since particular attention is given to cooked dishes ready for the table, the recipes and methods of preparation are given in some detail, so that one section of the volume reads like a veritable cookbook. For the American reader this is desirable, since numerous of the dishes are not well known on this side of the Atlantic, particularly in the instance of the puddings and meat pies.

About four-fifths of the book is occupied by the tabulated

data of analyses. The first series of tables includes the proximate analysis (excluding crude fiber), Na, K, Ca, Mg, Fe, Cu, P, S and Cl content, calories per 100 g. and acid-base balance of 541 foods classified as (1) cereals and cereal products, (2) dairy products, (3) meat, poultry and game, (4) fish, (5) fruit, (6) nuts, (7) vegetables, (8) sugar, preserves and sweetmeats, (9) beverages, (10) beers, (11) condiments, (12) vegetable fats, (13) cakes and pastries, (14) puddings, (15) meat and fish dishes, (16) egg and cheese dishes, (17) sauces and soups. Beginning with (3) the method of cooking, if any, nature of the edible material, and grams of edible matter as eaten, from each 100 grams of purchased food are also recorded and this continues through (7). In the instance of (8), (13), (14), (15), (16) and (17) there are cross references to the recipes appearing earlier in the book.

In the next block of tables the same data are set over into terms of *grams or milligrams per ounce*, presumably for the convenience of dietitians and others who are more accustomed to dealing with portions scaled in the common or avoirdupois system. The one exception to the system is in the instance of beers, which is based upon the pint.

An advantage in the organization of these data lies in the fact that they include food as served. One cannot but wonder if the English culinary practices are so uniform as to permit of accepting these data as representative of each item, however; also whether or not the average of only two preparations (p. 11, line 23) is sufficient to compensate for the variability in raw materials and technical skills.

The reviewer has tried the volume out on several dietitians, who have reacted rather favorably, and it seems probable that they, rather than the food chemist or tech-