TABLE B

STRONGEST ARC LINES OF ELEMENT 61 IN THE 3000–3450 Å. REGION

Wave length, Å.	Relative intensity
3366.05 ± 0.03	Weak
3377.64 ± .03	Weak
3391.25 ± .03	Medium
3418.67 ± .03	Weak
3427.42 ± .03	Strong
3441.09 ± .03	Weak
3449.81 ± .03	Medium

Unfortunately, it was impossible to compare these (arc) spectra with the (spark) spectrum observed by Timma.[5] His observations covered the 3630–4400 Å. region; aside from the fact that the methods of excitation used were different, his principal lines could not be looked for in the present arc spectrum with any hope of success because of interference by C_2 and CN bands. However, the spark lines he mentioned as due to element 61 were observed in the spectrum of aliquots of samples 1, 2 and 3 when excited by the copper spark technique.

[5] D. Timma, MonC-166 (U. S. Atomic Energy Commission).

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A Convenient Synthesis of Phenaceturic Acid[1]

BY JARED H. FORD

Phenaceturic acid was required as a starting material in numerous experiments directed toward the synthesis of benzylpenicillin.[2] The following method which employs methyl phenylacetate as the starting material was found to be more convenient than the literature method[3] in which phenylacetyl chloride was used.

Experimental

Glycine (75.1 g.) was added to a solution of 23 g. of sodium in 850 ml. of anhydrous methanol, and the mixture was boiled under reflux a few minutes to obtain a clear solution. One hundred fifty grams of methyl phenylacetate was then added and the solution was boiled under reflux for three days. The methanol was distilled off and the residue was dissolved in 400 ml. of cold water which contained 20 g. of sodium bicarbonate. The resulting solution was twice extracted with ether to remove unchanged methyl phenylacetate, and then acidified to *p*H 2 with concentrated hydrochloric acid. After standing overnight in a refrigerator, the product was filtered, washed with cold water and dried in a vacuum oven. The resulting white crystals melted at 139–141° (lit.,[3] 143°); yield, 122.6 g. (63.5%). One recrystallization from hot water (400 ml.) gave 114.2 g. of product which melted at 143–144°. From the ether extracts were obtained 42.1 g. (28%) of methyl phenylacetate; b. p. 96–98° (14 mm.).

A longer period of heating did not increase the yield and it appears likely that an equilibrium is involved

$$C_6H_5CH_2COOCH_3 + NH_2CH_2COONa \rightleftarrows C_6H_5CH_2CONHCH_2COONa + CH_3OH$$

ANTIBIOTICS RESEARCH DEPARTMENT
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[1] This work was done under contract between the Office of Scientific Research and Development and The Upjohn Company (Contract OSRD-cmr-399).

[2] Clarke, Johnson and Robinson, "The Chemistry of Penicillin." Princeton University Press, Princeton, N. J., 1949.

[3] Hotter, *J. prakt. Chem.*, [2] **38**, 98 (1888).

Alkyl Derivatives of Ethylenediamine

BY ALBERT E. FROST, JR., STANLEY CHABEREK, JR., AND ARTHUR E. MARTELL

In the course of other work a number of symmetrical dialkyl ethylenediamines have recently been prepared and characterized. A number of other amines, containing a smaller or larger number of alkyl groups, and a few dialkyl piperazines were obtained as by-products. The by-products not previously reported for this method and their properties are listed in Table I. The method of preparation, which involved the reaction of ethylene dichloride with an excess of primary amine, has been described by others.[1,2,3,4,5] The main products were characterized as the dipicrates and as the phenylureas, which are listed in Table II. Attempts to prepare the phenylurea from tribenzylethylenediamine were unsuccessful, and it was found that picrates could not be formed readily from dioctylpiperazine, tetraoctylethylenediamine, didodecylpiperazine and tribenzylethylenediamine. The picrate obtained with dibenzylethylenediamine was shown by analysis to be the monopicrate. Repeated recrystallization from various solvents did not change the melting point or the analysis.

TABLE I

BY-PRODUCTS

Amine	B. p., °C.	B. p., Mm.	M. p., °C.	N, % Calcd.	N, % Found
N,N′-Dioctylpiperazine	187–190	1	53–55	9.02	9.09
N,N,N′,N′-Tetraoctylethylenediamine	.....		130–133	5.51	5.29
N,N′-Didodecylpiperazine	265–275	8	54–56	6.63	6.34
N-Monobenzylethylenediamine	155–160	5	.....	18.65	18.72
N,N,N′-Tribenzylethylenediamine	225–235	4	99–100	8.48	8.64
Trioctyldiethylenetriamine[a]	213–215	2	.....	9.55	9.28

[a] *Anal.* Calcd. for $C_{28}H_{61}N_3$: C, 76.5; H, 14.0. Found: C, 76.4; H, 14.0.

For this method of preparation, it was found that the separation of the insoluble crystalline monohydrate obtained by treating the reaction product with a dilute aqueous solution of strong base provided the most convenient method of

[1] Sebrell and Clifford, U. S. Patent 1,948,317 (Feb. 20, 1934).

[2] Kyrides, U. S. Patent 2,126,560 (Aug. 9, 1938); U. S. Patent 2,267,685 (Dec. 23, 1941).

[3] Zienty and Thielke, THIS JOURNAL, **67**, 1040 (1945).

[4] Zienty, *ibid.*, **68**, 1388 (1946).

[5] Clifford, U. S. Patent 2,216,620 (Aug. 9, 1938).