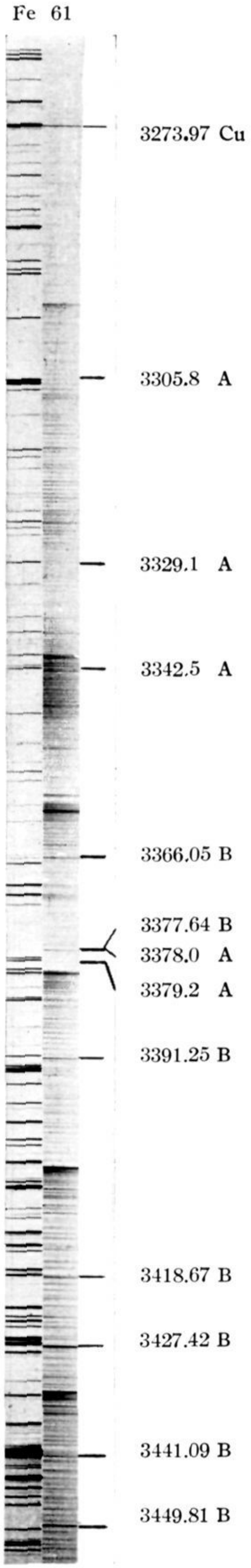

Fig. 1.—Arc emission spectrum of the 3260–3500 Å. region (sample 3). Spectra from the top down: Element 61, Fe. Wave lengths followed by A are those in Table A; wave lengths followed by B are those in Table B. Emission lines are black.

Note on the Arc Spectrum of Element 61[1]

BY CYRUS FELDMAN

An examination has been made of the arc emission spectra of three samples of the chloride of element 61 prepared by Paul Lantz. This material was separated from a mixture of uranium fission products by fractionation and concentration on a series of Amberlite IR-I and Dowex resin columns.[1a] Detailed accounts of the isolation of milligram amounts of element 61 are to be reported elsewhere by G. W. Parker and P. Lantz of this Laboratory. The amount of element 61 present in these samples was estimated on the basis of published values[1a] of the half-life and energy of the β-radiation of this element. The characterization of material which is radiochemically and spectrographically identical with these samples as element 61 has been confirmed by X-ray emission work.[2]

Once isolated, the material was prepared in the form of approximately 0.05 ml. of a hydrochloric acid solution A thin layer of Zapon lacquer was deposited on a flat-topped $^1/_4''$ diam. high purity graphite electrode; the sample solution was deposited atop this and dried under an infrared lamp. This electrode was made the anode of a 220-volt, 9-ampere d.c. arc. A $^1/_8''$ diameter high purity graphite rod served as cathode. The arc gap was 4 mm. The burning took place inside a chamber which permitted light to enter the spectrograph without allowing the vapor containing element 61, which is highly radioactive, to escape into the laboratory.

The light was focused on the slit of a 21-foot (6.5-meter) Jarrel-Ash spectrograph. Further details of dispersion, etc., are given in Table A.

When sample 1 was exposed, spectra of iron, neodymium, element 61, and samarium were obtained in juxtaposition in the order mentioned; in the other two cases, only the spectra of iron, and element 61 were photographed. The placement of the spectra on the plate was effected by means of a Hartmann diaphragm at the slit; the camera was not moved at any time during the exposure. The exposure conditions were essentially the same as those used by Harris, Yntema and Hopkins.[3]

A search was made for the five lines specifically mentioned by them as being common to the spectra of the neodymium and samarium fractions and being somewhat more intense in the fractions intermediate between them.[3,4] Wave lengths were located by interpolation between iron lines of known wave length; observations were made on an ARL–Dietert projection comparator, which gives an enlargement of twenty-one-fold.

In all, three samples were tested; the results are summarized in Table A.

TABLE A

Sample no.		1	2	3
Purity		Impure (see text)	Very pure	Very pure
61 content, in μg		50	50	100
Dispersion of spectrum in Å./mm.		5.0	2.5	2.5
Observations in connection with given lines	3305.8	Doubtful	Absent	Absent
	3329.1	Absent	Absent	Absent
	3342.5	Doubtful	Absent	Absent
	3378.0	Absent	Absent	(see text)
	3379.2	Absent	Absent	Absent

Sample 1 was received on February 20, 1948; its spectrum showed the presence of comparatively large amounts of calcium and magnesium, moderate amounts of iron, nickel, sodium and chromium, and small amounts of neodymium. Calcium was easily the major metallic constituent of this sample.

Although no definite line was seen in its spectrum at 3305.8, the proximity of iron 3305.98, which was present, might have masked a weak line at 3305.8. A line was observed at 3342.5, but this line may have been chromium 3342.586.

Two highly purified samples were received from the same source on March 7, 1949. The two samples contained 50 and 100 micrograms, respectively, of element 61. Arc spectra of these samples were taken under the same conditions as used for sample 1, except that higher dispersion was used. The spectrum of sample 3 is shown in Fig. 1.

The results are summarized in Table A. A weak line, not assignable to any known element, was found at 3377.64 ± 0.03 Å. in the high dispersion spectra, but in view of the fact that Yntema's observations were also made in the second order of a 6.5-meter spectrograph,[4] this line cannot be assumed to be identical with 3378.0. It was the weakest of several lines believed due to element 61 which were observed in the 3000–3450 Å. region of spectra 2 and 3. Seven such lines are listed in Table B; although these lines are fairly close to minor lines of various elements listed in the M. I. T. Wavelength Tables, qualitative examination of the spectrum showed that the only impurities present in samples 2 and 3 were faint traces of copper and calcium, and these did not interfere with the lines listed. A detailed study of the arc spectrum of element 61 is being made by W. F. Meggers and B. F. Scribner, Jr., of the National Bureau of Standards.

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[1] This document is based on work performed under Contract No.7405 eng. 26 for the Atomic Energy Project, and the information covered therein will appear in the National Nuclear Energy Series (Manhattan Project Technical Section) as part of the contribution of the Oak Ridge National Laboratory.

[1a] *Chem. Eng. News*, **26**, 205 (1948); J. A. Marinsky, L. E. Glendenin and C. D. Coryell, THIS JOURNAL, **69**, 2781 (1947); W. E. Cohn, E. R. Tompkins and J. X. Khym, *ibid.*, **69**, 2769 (1947); B. H. Ketelle and G. E. Boyd, *ibid.*, **69**, 2800 (1947).

[2] L. E. Burkhart, W. Peed and E. Spitzer, *Phys. Rev.*, **75**, 86 (1949).

[3] J. A. Harris, L. F. Yntema and B. S. Hopkins, THIS JOURNAL, **48**, 1585 (1926).

[4] L. F. Yntema, *ibid.*, **46**, 37 (1924).

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

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[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).