dissolved in *t*-butyl alcohol (15 ml.) and refluxed with a solution of potassium (1.0 g.) in *t*-butyl alcohol (60 ml.). After about 1 hour a crystalline precipitate (potassium *p*-toluenesulfonate) began to separate. The reflux was continued for 15 hours and then the *t*-butyl alcohol was removed *in vacuo*. The residue was partitioned between water and ether, the ether layer washed well with water and then extracted with 2 *N* hydrochloric acid. Basification of the acid solution gave a colorless oil which boiled at 80° (0.1 mm.) (1.7 g.).

Anal. Calcd. for $C_{18}H_{25}N$: C, 84.7; H, 9.7. Found: C, 84.7; H, 9.8.

The hydrochloride melted at 248–249° dec.

Anal. Calcd. for $C_{18}H_{26}NCl$: C, 74.1; H, 8.9. Found: C, 74.4; H, 9.1.

The same compound was obtained when the tosylate was heated with 2,6-lutidine for 48 hours.[16]

Catalytic reduction of the hydrochloride with Adams catalyst gave the hydrochloride of α-N-piperidinobenzylcyclohexane identical with the previously described sample.

α-N'-Methyl-N-piperazinobenzylcyclohex-2-ene was prepared in an exactly similar manner from the corresponding tosylate both with potassium *t*-butoxide and with 2,6-lutidine. After distillation (b.p. 65–70° (2×10^{-3} mm.)) it solidified and melted at 85° after recrystallization from pentane.

Anal. Calcd. for $C_{18}H_{28}N_2$: C, 80.0; H, 9.6. Found: C, 80.1; H, 9.6.

The ethiodide melted at 180–181° dec.

Anal. Calcd. for $C_{20}H_{31}N_2I$: C, 56.3; H, 7.3. Found: C, 56.2; H, 7.2.

The isopropyl iodide melted at 217–220° dec.

Anal. Calcd. for $C_{21}H_{33}N_2I$: C, 57.3; H, 7.5. Found: C, 57.2; H, 7.7.

The ethyl *p*-toluenesulfonate melted at 107–108°.

Anal. Calcd. for $C_{27}H_{38}O_3N_2S$: C, 68.9; H, 8.1. Found: C, 68.8; H, 8.3.

α-N'-Methyl-N-piperazinobenzyl- and α-N'-Ethyl-N-piperazinobenzylcyclohept-2-ene were prepared by heating the corresponding tosylates with 2,6-lutidine. The former compound is described above. The latter (prepared from crude tosylate–unsaturated compound mixture) boiled at 85–90° (0.1 mm.).

Anal. Calcd. for $C_{20}H_{30}N_2$: C, 80.5; H, 10.1. Found: C, 80.3; H, 10.0.

The ethiodide melted at 200° dec.

Anal. Calcd. for $C_{22}H_{35}N_2I$: C, 58.1; H, 7.7. Found: C, 58.2; H, 7.9.

Hydrogenation of the above base gave α-N'-ethyl-N-piperazinobenzylcycloheptane, b.p. 85° (0.1 mm.).

Anal. Calcd. for $C_{20}H_{32}N_2$: C, 80.0; H, 10.7. Found: C, 79.6; H, 10.5.

The ethiodide melted at 160–161°.

Anal. Calcd. for $C_{22}H_{37}N_2I$: C, 57.9; H, 8.1. Found: C, 58.1; H, 8.4.

The authors wish to thank Mr. S. W. Blackman for the microanalyses reported here.

TUCKAHOE 7, N. Y.

(16) In these hindered systems the use of a bulky base for the eliminations may not be necessary. In model experiments with cyclohexyl *p*-toluenesulfonate the superiority of *t*-butoxide to methoxide was marked. Diethylaniline and benzyldimethylamine in the absence of solvents did not react cleanly.

One-tenth mole portions of cyclohexyl *p*-toluenesulfonate were refluxed three hours with 0.3 mole of methanolic potassium hydroxide and sodium methoxide and with sodium *t*-butoxide in *t*-butyl alcohol. The reaction mixtures were steam distilled until a boiling point of 100° was reached and the distillates were made up to volume with methanol. Cyclohexene was then estimated by catalytic reduction of aliquots. In the reaction with *t*-butoxide 95% of the calculated quantity of cyclohexene was formed while in the other two cases the yield was 57%.

[CONTRIBUTION FROM THE STERLING-WINTHROP RESEARCH INSTITUTE]

The Preparation of N-Benzyl-3-morpholones and N-Benzyl-3-homomorpholones from N-(Hydroxyalkyl)-chloroacetamides

BY ALEXANDER R. SURREY, STANLEY O. WINTHROP, MARCIA K. RUKWID AND BENJAMIN F. TULLAR

RECEIVED AUGUST 27, 1954

The preparation and cyclization of some N-benzyl-N-(2-hydroxyethyl)-chloroacetamides to give N-benzyl-3-morpholone derivatives is described. A one-step process for the formation of the latter from N-benzylethanolamines also is reported. The homologous N-benzyl-3-homomorpholones have been prepared by cyclization of N-benzyl-N-(3-hydroxyalkyl)-chloroacetamides.

As part of our search for amebacidal agents[1] we prepared a series of N-benzyl-N-(2-hydroxyethyl)-chloroacetamides (I) which are listed in

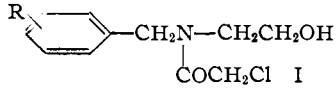
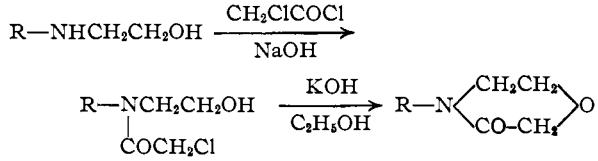


Table I. These compounds were prepared from the appropriate N-benzylethanolamines by acylation with chloroacetyl chloride in the presence of an equivalent amount of dilute sodium hydroxide solution. When tested in hamsters[2] the monochloroacetamides were found to be active but considerably less active than the corresponding dichloroacetamides reported previously.[1] The same was found true for the N-(2,4- and 3,4-dichlorobenzyl)-N-(2-hydroxypropyl)-chloroacetamides.

(1) A. R. Surrey, THIS JOURNAL, **76**, 2214 (1954).

(2) We are indebted to Dr. D. A. Berberian for the amebacidal screening.

Since these N-(2-hydroxyalkyl)-chloroacetamides were available, it seemed of interest to see if they could be cyclized to yield 3-morpholones to make them available for biological investigation. It was found that ring-closure could indeed be effected by treatment with alcoholic potassium hydroxide at room temperature. The N-benzyl-3-morpholones prepared by this procedure are reported

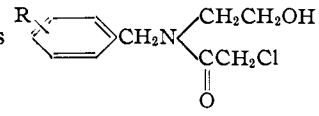


in Table II, and the N-(2,4- and 3,4-dichlorobenzyl)-6-methyl-3-morpholones are described in the Experimental part.

Attempts to effect cyclization using potassium carbonate, sodium acetate or triethylamine in

TABLE I

N-BENZYL-N-(2-HYDROXYETHYL)-α-CHLOROACETAMIDES

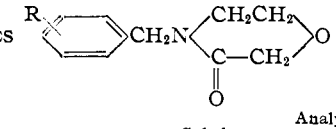


R	Yield, %	M.p., °C.	Re-crystn. solv.	Formula	Analyses, % Calcd. C	Calcd. H	Calcd. Cl	Found C	Found H	Cl[a]
H	48	82–84	[b]	$C_{11}H_{14}ClNO_2$	58.02	6.19	15.57	58.30	6.11	15.77
4-Cl	71	75–76	[c]	$C_{11}H_{13}Cl_2NO_2$	50.41	5.00	13.53	50.52	5.38	13.67
2-Cl	59	89–91	[c]	$C_{11}H_{13}Cl_2NO_2$	...	..	27.05	...	..	27.12[d]
2,4-Cl_2	78	101–102	[b]	$C_{11}H_{12}Cl_3NO_2$	44.54	4.07	11.96	44.75	4.34	12 02
3,4-Cl_2	22	85–86	[c]	$C_{11}H_{12}Cl_3NO_2$	...	..	35.87	...	..	35.95[d]
2,6-Cl_2	88	133–135	[e]	$C_{11}H_{12}Cl_3NO_2$	...	..	35.87	...	..	36.50[d]
3,4-CH_2O_2	72	93–95	[f]	$C_{12}H_{14}ClNO_4$	...	5.15[g]	13.07	...	5.10[g]	13.05
4-C_2H_5O	84	66–67	[c]	$C_{13}H_{18}ClNO_3$	57.44	6.67	13.05	57.54	6.62	13.33
4-NO_2	36	117–119	[f]	$C_{11}H_{13}ClN_2O_4$	48.44	4.80	13.00	48.47	4.90	13.56
4-*i*-C_3H_7	82	85–86	[c]	$C_{14}H_{20}ClNO_2$	62.34	7.47	13.14	62.21	7.51	13.16

[a] Aliphatic chlorine by potassium hydroxide wet digestion. [b] Ethylene dichloride. [c] Benzene–Skellysolve A. [d] Total chlorine by dry combustion. [e] Ethanol. [f] Benzene. [g] Nitrogen analysis.

TABLE II

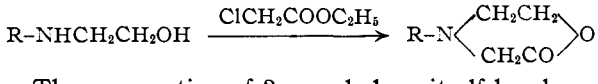
N-BENZYL-3-MORPHOLONES



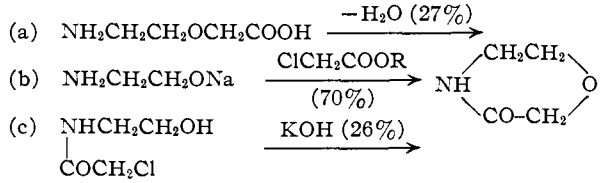
R	Yield, %	M.p., °C., or b.p. (mm.)	Re-crystn. solv.	Formula	Analyses, % Calcd. C	Calcd. H	Calcd. N	Found C	Found H	Found N
H	56	125–126[a] (0.07)	..	$C_{11}H_{13}NO_2$	69.10	6.85	7.33	68.80	6.93	7.16
4-Cl	77	45–46	[b]	$C_{11}H_{12}ClNO_2$	58.54	5.36	Cl, 15.71	58.50	5.10	Cl, 16.00
2-Cl	27	140–142[a] (0.4)	..	$C_{11}H_{12}ClNO_2$	58.54	5.36	15.71[f]	58.70	5.60	16.02[f]
2,4-Cl_2	88	95–96	[c]	$C_{11}H_{11}Cl_2NO_2$	50.80	4.27	27.26[f]	51.13	4.24	27.22[f]
3,4-Cl_2	38	69–70	[c]	$C_{11}H_{11}Cl_2NO_2$	50.80	4.27	27.26[f]	50.66	4.50	27.30[f]
2,6-Cl_2	53	109–110	[c]	$C_{11}H_{11}Cl_2NO_2$	50.80	4.27	5.38	50.70	4.49	5.39
3,4-CH_2O_2	62	88–90	[c]	$C_{12}H_{13}NO_4$	61.25	5.57	5.96	61.50	5.72	5.87
4-C_2H_5O	40	67–69	[d]	$C_{13}H_{17}NO_3$	66.34	7.28	5.95	66.25	6.95	5.91
4-NO_2	80	137–139	[c]	$C_{11}H_{12}N_2O_4$	55.92	5.12	5.90	55.82	5.17	5.89
4-*i*-C_3H_7	26	156–157[a] (0.8)	..	$C_{14}H_{19}NO_2$	72.02	8.20	6.00	72.16	8.47	6.10
4-NH_2	89	111–112	[e]	$C_{11}H_{14}N_2O_2$	64.04	6.84	13.53	63.91	6.90	13.35
4-C_4H_9O	33	55–57	[e]	$C_{15}H_{21}NO_3$	68.40	8.04	5.32	68.20	8.23	5.30

[a] Titration of basic nitration. [b] Skellysolve B. [c] Isopropyl alcohol. [d] Benzene–Skellysolve A. [e] Skellysolve A [f] Cl^- analyses.

ethanol were unsuccessful. Similarly ineffective were efforts to prepare a N-phenyl-3-morpholone. For example, treatment of N-(3-chlorophenyl)-N-(2-hydroxyethyl)-chloroacetamide with alcoholic potassium hydroxide gave only N-(3-chlorophenyl)-ethanolamine. The reaction of N-substituted ethanolamines with ethyl chloroacetate gave only the 2-morpholone derivatives.



The preparation of 3-morpholone itself has been described previously by (a) dehydration of 2-aminoethoxyacetic acid[3] and more recently by Vieles and Seguin[4] by (b) the reaction of the sodio derivative of ethanolamine with ethyl chloroacetate. They also reported the preparation of 2-methyl-3-morpholone and benzomorpholone by this procedure. 3-Morpholone was prepared (c) according to our method in 26% yield. When the method of Vieles and Sequin was tried with N-(3,4-dichlorobenzyl)-ethanolamine only a dark oil was obtained which could not be induced to solidify.



The main difficulty appeared to be in getting all of the sodium to react with the ethanolamine derivative.

Inasmuch as cyclization of N-benzyl-N-(2-hydroxyethyl)-chloroacetamides readily yielded 3-morpholones, it was decided to investigate the homologous 3-hydroxypropyl derivatives. The N-benzyl-3-hydroxypropylamines employed in this study (Table III) were prepared from the appropriate benzyl chloride and 3-aminopropanol or by reductive alkylation of the latter with a substituted benzaldehyde. With the exception of N-(2,4-dichlorobenzyl)-N-(3-hydroxypropyl)-chloroacetamide the amides in this series were obtained as viscous oils. The latter were cyclized directly

(3) R. Leimu and J. I. Jauson, *Suomen Kemistilehti*, **18B**, 3 (1945); *C. A.*, **41**, 769 (1947).

(4) N. N. P. Vieles and J. Seguin, *Compt. rend.*, **234**, 1980 (1952); *Bull. soc. chim. France*, 287 (1953).

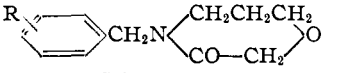
TABLE III

N-BENZYL-3-HYDROXYPROPYLAMINES 

R	Yield, %	B.p., °C.	Mm.	$n^{25}D$	Formula	Nitrogen, % Calcd.	Found
H	57	110–115	0.7	1.5370	$C_{10}H_{15}NO$	8.50	8.27
4-*i*-C_3H_7	58	135–138	.65	1.5230	$C_{13}H_{21}NO$	6.76	6.56
4-OCH_3	51	135–145	.8	1.5390	$C_{11}H_{17}NO_2$	7.19	6.52
4-OC_2H_5	46	145	.75[a]		$C_{12}H_{19}NO_2$	6.70	6.60
3,4-O_2CH_2	59	147–151	.75	1.5500	$C_{11}H_{15}NO_3$	6.70	6.63
2-Cl	52	130–135	.8	1.5445	$C_{10}H_{14}ClNO$	7.01	7.03
4-Cl	52	127–132	.8	1.5429	$C_{10}H_{14}ClNO$	7.01	7.16

[a] M.p. 43–47° after recrystallization from Skellysolve B.

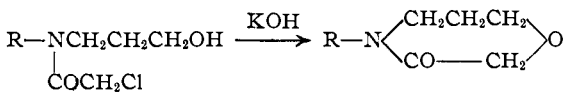
TABLE IV

N-BENZYL-3-HOMOMORPHOLONES 

R	Yield, %	M.p., °C.	Formula	Calcd. C	Calcd. H	Calcd. N	Found C	Found H	Found N
H	17	66.9–70.4[a]	$C_{12}H_{15}NO$	70.23	7.36	6.82	70.57	7.38	6.82
4-OCH_3	15	85.8–88.1[a]	$C_{13}H_{17}NO_3$	66.36	7.28	5.95	66.17	7.14	5.93
3,4-O_2CH_2	22	80.0–82.2[b]	$C_{13}H_{15}NO_4$	62.63	6.06	5.61	62.60	5.91	5.60
4-Cl	16	83.4–85.8[c]	$C_{12}H_{14}ClNO_2$	60.12	5.88	5.84	60.27	5.26	5.90
2,4-Cl_2	30[d]	90.1–91.8[e]	$C_{12}H_{13}Cl_2NO_2$	52.56	4.78	5.11	52.28	4.45	5.15
3,4-Cl_2	20	80.6–82.8[c]	$C_{12}H_{13}Cl_2NO_2$	52.56	4.78	25.87[f]	52.53	4.97	26.13[f]

[a] From ether–Skellysolve A. [b] From isopropyl alcohol. [c] From ether. [d] Based on purified chloroacetamide. [e] From benzene–Skellysolve A. [f] Cl analyses.

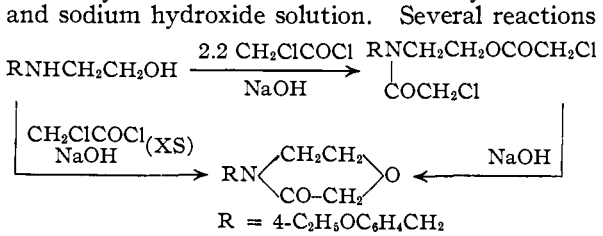
with potassium hydroxide to give the 3-homomorpholone derivatives. The over-all yields from



the N-benzyl-3-hydroxypropylamines were approximately 20–30%, considerably less than that obtained with the morpholones.

Several of the N-benzyl-3-morpholones were found to have interesting mild analgesic, antipyretic and anti-inflammatory activities. The most active compounds are the N-(2,4-dichlorobenzyl) and N-(4-ethoxybenzyl) derivatives.

In an effort to improve the yield (40%) of the latter compound the cyclization of N-(4-ethoxybenzyl)-N-(2-hydroxyethyl)-chloroacetamide in aqueous sodium hydroxide was investigated. This study was undertaken as a result of an observation that the O,N-bis-chloroacetyl derivative of N-(4-ethoxybenzyl)-ethanolamine gave an 80% yield of the desired 3-morpholone when treated with excess sodium hydroxide solution. The formation of N-(4-ethoxybenzyl)-3-morpholone was achieved finally in 80% yield in one step. This was accomplished by treating a 50% ethyl alcohol solution of N-(4-ethoxybenzyl)-ethanolamine simultaneously with an excess of chloroacetyl chloride and sodium hydroxide solution. Several reactions



probably are involved in this procedure. These may include N-acylation, O,N-diacylation, O- and N-deacylation, O → N acyl migration and finally, ring closure of the N-acyl derivative. This one-step procedure for the preparation of 3-morpholones also was applied to N-(2,4-dichlorobenzyl)-ethanolamine with equal success.

Experimental[5]

N-(3,4-Dichlorobenzyl)-N-(2-hydroxypropyl)-chloroacetamide.—The following example illustrates the preparation of the N-benzyl-N-(2-hydroxyethyl)-chloroacetamides listed in Table I.

Chloroacetyl chloride (14 g.) was added dropwise with stirring over a period of 60 minutes to a mixture of N-(3,4-dichlorobenzyl)-2-hydroxypropylamine (24 g.) and sodium hydroxide (5 g.) in 200 ml. of water and 100 ml. of ethylene dichloride. The temperature was held around 0° by means of an ice–salt-bath during the addition and the mixture was stirred for an additional two hours at room temperature. The organic layer was washed with dilute sodium hydroxide, dilute hydrochloric acid and then water, dried over calcium chloride and the ethylene dichloride was removed *in vacuo*. After two recrystallizations from benzene–Skellysolve A, the product, 19 g. (60%), melted at 84–86°.

Anal. Calcd. for $C_{12}H_{14}Cl_3NO_2$: C, 46.40; H, 4.54; Cl, 34.25. Found: C, 46.29; H, 4.59; Cl, 34.56.

N-(2,4-Dichlorobenzyl)-N-(2-hydroxypropyl)-chloroacetamide.—Prepared as above in 75% yield, m.p. 82–84°. *Anal.* Calcd. for $C_{12}H_{14}Cl_3NO_2$: Cl, 34.25. Found: Cl, 34.44.

N-(2,4-Dichlorobenzyl)-N-(3-hydroxypropyl)-chloroacetamide.—Prepared as above in 75% yield. A sample recrystallized from benzene melted at 84–86°. *Anal.* Calcd. for $C_{12}H_{14}Cl_3NO_2$: Cl_{KOH}, 11.30. Found: Cl_{KOH}, 11.42.

N-(3,4-Dichlorobenzyl)-6-methyl-3-morpholone.—The following example illustrates the general method employed for the preparation of the 3-morpholones listed in Table II.

A mixture of 11 g. of N-(3,4-dichlorobenzyl)-N-(2-hydroxypropyl)-chloroacetamide and 2 g. of powdered potassium hydroxide in 100 ml. of absolute alcohol was stirred for five hours at room temperature. Approximately 3.5 g. of potassium chloride was removed by filtration and the filtrate was evaporated *in vacuo* leaving a solid residue. The product, 6.0 g. (62%), was recrystallized from benzene–Skellysolve A, m.p. 78–80°.

(5) All melting points and boiling points are uncorrected.

Anal. Calcd. for $C_{12}H_{13}Cl_2NO_2$: C, 52.56; H, 4.78; Cl, 25.86. Found: C, 52.30; H, 4.81; Cl, 26.45.

N-(2,4-Dichlorobenzyl)-6-methyl-3-morpholone.—Prepared as above in 41% yield, m.p. 68–70°. *Anal.* Calcd. for $C_{12}H_{13}Cl_2NO_2$: C, 52.56; H, 4.78; Cl, 25.86. Found: C, 52.70; H, 4.88; Cl, 25.74.

N-(4-Aminobenzyl)-3-morpholone.—4-(4-Nitrobenzyl)-3-morpholone (9.0 g.) in 500 ml. of absolute ethanol was reduced catalytically with palladium-on-charcoal at an initial hydrogen pressure of 45 pounds per square inch. The reduction was complete after five hours. The catalyst was filtered off and the filtrate was distilled *in vacuo* leaving a solid residue. The product, 7.0 g. (89%), was recrystallized first from benzene and then from isopropyl alcohol, m.p. 111–112°.

Anal. Calcd. for $C_{11}H_{14}N_2O_2$: C, 64.04; H, 6.84; N, 13.53. Found: C, 63.91; H, 6.90; N, 13.35.

N-Chloroacetoxyethyl-4-ethoxybenzylamine Hydrochloride.—A solution of 42 g. of N-(4-ethoxybenzyl)-ethanolamine hydrochloride in boiling ethylene dichloride was treated with 21 g. of chloroacetyl chloride with stirring. Refluxing was continued for about 30 minutes until the evolution of hydrogen chloride ceased. During this time some of the hydrochloride of the N-chloroacetoxyethyl-4-ethoxybenzylamine crystallized. Crystallization was completed by cooling to 20°. The product was collected, washed with cold ethylene dichloride and petroleum ether and dried at 70°; yield 47 g. (85%), m.p. 168–170°.

Anal. Calcd. for $C_{13}H_{19}Cl_2NO_3$: N, 4.54. Found: N, 4.66.

When the mother liquor was evaporated an oil remained which could not be induced to crystallize. This oil appeared to be the O,N-bis-(chloroacetyl)-4-ethoxybenzylethanolamine since treatment of it in 50% alcohol with excess sodium hydroxide solution gave an 80% yield of N-(4-ethoxybenzyl)-3-morpholone.

N-(4-Ethoxybenzyl)-3-morpholone.—To a solution of 195 g. (1 mole) of N-(4-ethoxybenzyl)-ethanolamine in 2 l. of 50% ethyl alcohol was added 100 ml. of 35% sodium hydroxide solution and then while keeping the temperature between 15° and 20° by means of an ice-bath there was added simultaneously with strong stirring 350 ml. of 35% sodium hydroxide solution and 290 g. (2.5 moles) of chloroacetyl chloride during about 1 hour. Stirring was continued for another 30 minutes at about 20° whereupon the solution was made acid to litmus with hydrochloric acid, concentrated *in vacuo* to a volume of about 2 l. and diluted with two liters of ice-water. The crystalline precipitate was collected, washed with cold water and dried *in vacuo* yielding 191 g. (81%) of N-(4-ethoxybenzyl)-3-morpholone, m.p. 69–72°, which after recrystallization from isopropyl alcohol melted at 70–72.5°.

N-(2,4-Dichlorobenzyl)-3-morpholone.—A solution of 11 g. (0.05 mole) of N-(2,4-dichlorobenzyl)-ethanolamine in 100 ml. of 50% ethanol was treated with 12.5 ml. of 35% sodium hydroxide solution and then at 15–20° with strong stirring 12.5 ml. of 35% sodium hydroxide and 15 g. of chloroacetyl chloride were added simultaneously. After another half-hour at 20° the solution was worked up as described above yielding 10.5 g. (80%) of N-(2,4-dichlorobenzyl)-3-morpholone, m.p. 92.5–96°, which after recrystallization from isopropyl alcohol melted at 94–96°.

The same over-all yields of morpholones were obtained when the substituted ethanolamines were first converted to the O,N-bis-(chloroacetyl) derivatives by treating with 2.2 equivalents of chloroacetyl chloride in ethylene dichloride solution (finishing with a one-hour reflux) and the total oil residue from this acylation was added to excess alkali in 50% ethanol solution.

N-Benzyl-3-hydroxypropylamines (Table III).—The following two procedures were employed for the preparation of the N-benzyl-3-hydroxypropylamines. A. This method was used with the chloro- and dichlorobenzyl chlorides. One mole of the latter was added dropwise with stirring to 3 moles of 3-aminopropanol over a period of about 45 minutes. After stirring for two hours longer the mixture was treated with an excess of 35% sodium hydroxide and the product was extracted with ethylene dichloride and distilled. B. Equimolar quantities of the benzaldehyde and 3-aminopropanol were heated *in vacuo* on a steam-bath for one hour. The product was then dissolved in alcohol and reduced catalytically with palladium-on-charcoal.

N-Benzyl-3-homomorpholones (Table IV).—The procedure described above for the preparation of the 3-morpholones was employed for the preparation of these compounds. In most cases the crude N-benzyl-N-(3-hydroxypropyl)-chloroacetamides were dissolved in absolute ethanol and treated directly with powdered potassium hydroxide.

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A New Method for the Preparation of 3-Substituted-2-oxazolidones

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It has been found that N-benzyl-2- and 3-hydroxyalkylamines react with methyl or ethyl trichloroacetate to yield N-benzyl-2-oxazolidones and N-benzyl-2-pentoxazolidones, respectively. With N-benzyl-4-hydroxybutylamine and methyl trichloroacetate an N-substituted pyrrolidine was obtained. Possible mechanisms for these reactions are presented.

In our investigation of potential amebacidal agents we had planned to prepare some N-benzyl-N-(2-hydroxyalkyl)-monochloroacetamides (R = CH_2Cl), -dichloroacetamides (R = $CHCl_2$) and -trichloroacetamides (R = CCl_3). The first two types presented no difficulties. The monochloro-

$$R'—C_6H_4CH_2N(—CO—R)—CH_2CH_2OH$$

acetamides were formed in the usual manner from the reaction of N-benzylethanolamines with chloroacetyl chloride in the presence of aqueous sodium hydroxide.[2] For the dichloroacetamides it was found that acylation of the N-benzylethanolamines could be brought about with methyl or ethyl dichloroacetate as well as with dichloroacetyl chloride.[3] It seemed reasonable, therefore, to expect similar acylations with methyl or ethyl trichloroacetate, or with trichloroacetyl chloride. However, this did not prove to be the case.

When N-(2,4-dichlorobenzyl)-ethanolamine and ethyl trichloroacetate were warmed at 50–60° for two hours, a product was formed whose analysis indicated a loss of a mole of chloroform as well as a mole of ethanol. In a repetition of this experi-

(1) (a) This paper is constructed from part of a dissertation to be presented to the Rensselaer Polytechnic Institute by George Y. Lesher in partial fulfillment of the requirement for the degree of Doctor of Philosphy. (b) Adjunct Professor, Rensselaer Polytechnic Institute.

(2) A. R. Surrey, S. O. Winthrop, M. K. Rukwid and B. F. Tullar, THIS JOURNAL, **77**, 633 (1955).

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