chloride yielded the dihydrate (18% volatile found, calcd. 19.0%) which was further dried at 100°, 0.1 mm. to constant weight (6 hr.) for analysis.

The anhydrous compound has $[\alpha]^{25}_{D}$ +28° (*c*, 2 in 5% NaOH) $[\alpha]^{25}_{D}$ +59° (*c*,2 in 5% HCl). It decomposes without melting over a broad range, 240–290°, depending on rate of heating. Titration of an aqueous solution indicates a dibasic acid, pK_a 6.7, and 9.1, eq. wt. 72 and 156. (Calcd., eq. wt. 74 and 148.1.)

Anal. Calcd. for $C_4H_8N_2O_4$: C, 32.43; H, 5.44; N, 18.91. Found C, 32.42; H, 5.23; N, 18.89.

The ultraviolet absorption spectrum in water solution shows only end absorption, $\epsilon = 38$ at 220 mμ. The infrared absorption spectrum (KBr pellet) shows bonded OH and NH absorption at 3–3.5μ, and a very broad carbonyl absorption band at 6–6.2μ.

A quantitative analysis (paper chromatography–ninhydrin) of the broth indicates the presence of about 1–2 g. of (+)-2,3-diaminosuccinic acid per liter of the broth examined. Unfermented broths showed no diaminosuccinic acid.

Acid stability of diaminosuccinic acid. A solution containing 2% diaminosuccinic acid in 10% aqueous HCl was heated under reflux for 24 hr. Aliquots were withdrawn at 2, 6, and 24 hr. Neutralization to *p*H 3 yielded crystalline acid in each case.[5] Paper chromatographic examination of the samples indicated that slight decomposition to other ninhydrin positive substances had occurred at 6 hr.; definite decomposition was detectable at 24 hr. A sample of the soybean meal used in the fermentation was hydrolyzed in 10% hydrochloric acid at reflux for 24 hr. Paper chromatographic examination of the hydrolysates showed no diaminosuccinic acid.

N,N'-Dibenzoyl-(+)-2,3-diaminosuccinic acid. One-half g. of (+)-diaminosuccinic acid, was dissolved in 9 ml. of 5% sodium hydroxide, cooled to 0°, and 1 ml. of benzoyl chloride added to the stirred solution. After standing at room temperature overnight, the solution was acidified to *p*H 2 with HCl, and the resulting suspension was extracted twice with an equal volume of ether to yield 0.8 g. of crude dibenzoyldiaminosuccinic acid. This crude product was twice crystallized from 5 ml. ethanol by addition of water. The pure product, 0.35 g., melts at 164–166° with softening and darkening from 158°. It was dried for 2 hr. at 80°, 0.05 mm. for analysis. $[\alpha]^{25}_{D}$ +109° (*c*, 1 in MeOH).

Anal. Calcd. for $C_{18}H_{16}N_2O_6 \cdot H_2O$: C, 57.75; H, 4.85; N, 7.48. Found: C, 57.51; H, 4.89; N, 7.71.

N,N'-di-2-Napthalenesulfonyl-(+)-2,3-diaminosuccinic acid. Two hundred mg. of (+)-diaminosuccinic acid in 27 ml. 1*N* sodium hydroxide was stirred with 1.2 g. 2-naphthalenesulfonyl chloride in 5 ml. ether. Three 2.7-ml. portions of *N* NaOH were added at 1, 3, 4 hr. The aqueous phase was separated, acidified, and the crude product, 0.55 g. was crystallized twice from ethanol-water and dried at 26°, 0.01 mm. for analysis. The product obtained in this way melted at 190–195° with much prior decomposition. This m.p. is dependent on rate of heating. This substance did not analyze satisfactorily.

Anal. Calcd. for $C_{24}H_{20}N_2S_2O_8 \cdot {}^1/_2\, H_2O$. C, 53.70; H, 3.95; N, 5.21. Found: C, 53.78; H, 4.38; N, 5.90.

The corresponding *dl* derivative melts at 200°, the *meso* derivative at 234°.[1]

Examination of S. aureofaciens beers. Beers from the fermentations of two strains of *S. aureofaciens* in media containing corn steep liquor as the proteinacious ingredient were filtered and examined directly by chromatography on a methyl isobutyl ketone-acetic acid–water system. No (+)-2,3-diaminosuccinic acid could be detected. A standard containing 0.1% diaminosuccinic acid gave a strong positive test in the same run.

Acknowledgments. I am indebted to Dr. F. W. Tanner, Jr., for the fermentation broths, to Mr. M. J. Lynch for the paper chromatographic studies, and to Dr. R. L. Wagner and his associates for the physical measurements and analyses. I wish to thank Mr. A. Adriansen for his competent technical assistance. Dr. A. S. Yard kindly provided the *dl*-2,3-diaminosuccinic acid for comparison.

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[5] R. Kuhn and F. Zumstein, *Ber.*, **59**, 479 (1926), state that *dl*-diaminosuccinic acid is converted to the *meso* form after 3 hrs. reflux in 15% hydrochloric acid. A similar conversion may have occurred here, but was not evident from the paper chromatograms.

[CONTRIBUTION FROM THE ROCKEFELLER INSTITUTE]

Reaction of Ethylene Dibromide with Triethylamine and the Restoring Action of Some Alkanebis(triethylammonium) Ions upon Sodium-Deficient Nerve Fibers

FREDERICK VIDAL

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It is shown that the reaction of ethylene dibromide with triethylamine yields no ethylenebis(triethylammonium bromide-contrary to the reports in the literature. The only crystalline products obtained are triethylamine hydrobromide and 1) bromoethane-2-triethylammonium bromide.

The synthesis and properties of the ethylenebis(triethylammonium halides), prepared in the course of studies on neurophysiological problems, are reported. The bisquaternary ammonium salts were formed by quaternization of *N,N,N',N'*-tetraethylethylenediamine with ethyl halide. In addition to the bisquaternary ammonium compounds mixed tertiary-quaternary ammonium salts, ethane-1-diethylamino-2-triethylammonium halides, were also isolated, whose properties are described.

For many years this laboratory has been engaged[1] in testing the neurophysiological effects of different nitrogenous basic compounds on nerve fibers. Some of these onium ions, of which the tetraethylammonium ion may be regarded as one of the prototypes, restore the ability to conduct impulses in sodium-deficient nerve fibers.

In order to study the biological activities of the alkanebis(triethylammonium chlorides) in this system, we prepared the corresponding compounds containing polymethylene chain lengths of C_2, C_3, C_5, and C_{10}. The reaction of α,ω-alkene dibromides with triethylamine yielded the 3-, 5-, and 10-carbon members.[2,3] However, the 2-carbon member could not be obtained employing this method, despite a claim[4] that ethylenebis-(triethylammonium bromide) can be synthesized by the addition of ethylene dibromide to triethylamine.

The reaction of ethylene dibromide with triethylamine in ethanol, performed in a pressure bomb according to Lucius,[4] yielded in our laboratory two crystalline products. One of them was identified as triethylamine hydrobromide, which previously had been claimed to be ethylenebis-(triethylammonium bromide). This compound and its picrate showed no depression in melting points when mixed with the corresponding triethylamine salts and its bromine analysis checked with the theoretically required value. The second crystalline product of this synthesis was 1-bromoethane-2-triethylammonium bromide.[5] The nitrogen: bromine ratio of this salt was 1:2 and only one half of the total bromine was ionically bound. Furthermore, the reaction was accompanied by formation of a gas which presumably was vinyl bromide.

We also obtained the same crystalline reaction products either by heating under reflux in an open flask[2] or by simply allowing the starting materials to stand at room temperature for several weeks. In neither instance were we able to detect any ethylenebis(triethylammonium bromide).

Our findings are in agreement with these of Hofmann[6] who could not obtain the bisquaternary ammonium compound by reacting ethylene dibromide with triethylamine. It is also known that the expected bisquaternary ammonium salts are not always produced by the reaction of ethylene dibromide with tertiary amines.[7] Furthermore, it has recently been demonstrated that the reaction of ethylene dibromide with trimethylamine yielded a mixture of products. One of these was actually identified as tetramethylammonium bromide[8] and was not, as had been previously claimed, ethylenebis(trimethylammonium bromide).

Authentic ethylenebis(triethylammonium halides) were obtained by a two-step process. At first we prepared *N,N,N',N'*-tetraethylethylenediamine according to the procedure of Laasko and Reynolds.[9] Quaternization of this bistertiary amine was achieved by refluxing the base for several hours with ethyl bromide or ethyl iodide in ethanol, yielding ethylenebis(triethylammonium halide). In addition to this bisquaternary ammonium salt a mixed tertiary-quaternary ammonium compound, ethane-1-diethylamino-2-triethylammonium halide, was also isolated. On further reaction with ethyl halide the latter product yielded the desired bisquaternary ammonium halide. Ethylenebis(triethylammonium bromide) could be quantitatively converted to the corresponding dichloride by adsorbing on the cation exchange resin Dowex 50-WX8 and eluting with 3*N* hydrochloric acid.

The reason for the failure to obtain the bisquaternary ammonium compound in the reaction between ethylene dibromide and triethylamine in all probability is due to steric hindrance in the final product. An inspection of a Courtauld model of ethylenebis(triethylammonium) ion clearly shows almost complete restriction of C—C rotation as a result of the relatively bulky triethylammonium groups in close proximity with one another. It is therefore not surprising that the addition of one molecule of triethylamine to the dibromide with the subsequent formation of the isolated monoquaternary compound proceeds smoothly. On the other hand, the addition of the second molecule of triethylamine to this intermediate is greatly impeded resulting in a negligible yield of the diquaternary ammonium bromide. The isolation of triethylamine hydrobromide from the reaction mixture indicates that hydrogen bromide is also formed. It is split off from the unreacted ethylene dibromide under the conditions of the experiment with the simultaneous production of vinyl bromide.

The neurophysiological results of some of the onium ions tested are shown in Table I. It will be seen that with the exception of ethane-1-diethylamino-2-triethylammonium chloride whose activity was dubious, all the other compounds were able to restore conduction of impulses in sodium-deficient bullfrog splanchnic nerve fibers. However, the restoring ability of tetraethylammonium ion was greater than the activity of the alkanebis-(triethylammonium) ions. Furthermore, it can be seen that ethylenebis(triethylammonium chloride) restored the ability to conduct impulses to more fibers than triethylammonium chloride.

Full details of the neurophysiological experiments, the methods used, and the results obtained will be reported elsewhere.

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TABLE I

RELATIVE ABILITY OF ONIUM IONS TO RESTORE CONDUCTION OF IMPULSES IN SODIUM-DEFICIENT NERVE FIBERS

Compound	Degree of Restoring Ability
Ethane-1-diethylamino-2-triethylammonium chloride	X?
N,N,N',N'-tetraethylethylenediamine hydrochloride	X
Triethylammonium chloride	XX
Ethylenebis(triethylammonium) chloride	XXX
Pentamethylenebis(triethylammonium) chloride	XXXX
Decamethylenebis(triethylammonium) chloride	XXXX
Tetraethylammonium chloride	XXXXX

EXPERIMENTAL[10-12]

Reaction of triethylamine with ethylene dibromide.[4] (*a*) A mixture of 22.5 g. triethylamine, 45 g. ethylene dibromide, and 30 ml. absolute ethanol was heated for 6 hr. in a pressure bottle to 80–90°. During this period the pressure rose constantly. After cooling the solution to room temperature the crystalline material was removed by filtration and recrystallized from ethanol; 21 g. of white product, m.p. 247–249°,[13] were obtained. A mixed melting point with a sample of ethylenebis(triethylammonium bromide), prepared by a different method (see below), showed a marked depression. But it showed no depression in a mixed melting point with triethylamine hydrobromide.

Anal. Calcd. for $C_{14}H_{34}Br_2N_2$: Br, 40.96; for $C_6H_{16}BrN$: Br, 43.88. Found: Br, 43.99.

With saturated aqueous picric acid triethylamine picrate, m.p. 173–175°, was obtained. A mixed melting point with an authentic sample was not depressed.

Upon addition of ether to the alcoholic filtrate from triethylamine hydrobromide, a solid material precipitated which after recrystallization from propanol-ether yielded 3.1 g. 1-bromoethane-2-triethylammonium bromide, m.p. 216–217°, (reported[4] m.p. 241–242°).

Anal. Calcd. for $C_8H_{19}Br_2N$: C, 33.24; H, 6.63; Br^-, 27.65; Br (total), 55.29; N, 4.84. Found: C, 33.34; H, 6.67; Br^-, 27.82; Br (total), 54.68; N, 4.73.

This salt yielded with saturated aqueous picric acid 1-bromoethane-2-triethylammonium picrate as a slightly soluble crystalline salt, m.p. 141–143°.

Examination of the mother liquor from 1-bromoethane-2-triethylammonium bromide failed to reveal ethylenebis-(triethylammonium bromide) or any other substance.

(*b*) Triethylamine (125 g.), 63 g. ethylene dibromide, and 50 ml. ethanol were refluxed in an open flask[2] for 24 hr. The crystalline products were isolated as described above. 24 g. triethylamine hydrobromide and 3.9 g. β-bromoethyltriethylammonium bromide were thus obtained.

(*c*) A mixture of 20.5 g. triethylamine and 16.3 g. ethylene dibromide was allowed to stand at room temperature for 2 weeks in a stoppered container. On working it up as described in (*a*), 1.3 g. triethylamine hydrobromide and 2.9 g. 1-bromoethane-2-triethylammonium bromide were isolated. In both experiments (*b* and *c*) no ethylenebis(triethylammonium bromide) was found.

Preparation of 1-bromoethane-2-triethylammonium chloride. Six grams of 1-bromoethane-2-triethylammonium bromide was dissolved in 60 ml. water and stirred with 18 g. of silver chloride at room temperature. The slurry was filtered and the filtrate evaporated *in vacuo* to dryness. The residue was recrystallized twice from propanol-ether to yield 1-bromoethane-2-triethylammonium chloride, m.p. 224–226° (dec.).

Anal. Calcd. for $C_8H_{19}BrClN$: Br, 32.67; Cl, 14.50. Found: Br, 32.90; Cl, 14.51.

Preparation of ethylenebis(triethylammonium) iodide. *N,N,N',N'*-tetraethylethylenediamine[9] (17.2 g., 0.1 mole) in 15 ml. ethanol and 33 g. (0.21 mole) ethyl iodide were refluxed for 2 hr. Upon cooling to room temperature, the precipitate which formed was filtered and washed with ethanol, m.p. 228–230° (dec.). After several recrystallizations of the crude material from water, 17.4 g. (35.5%) of ethylenebis(triethylammonium iodide), m.p. 236–238° (dec.), were obtained. This salt is slightly soluble in cold water, soluble in hot water, and practically insoluble in methanol.

Anal. Calcd. for $C_{14}H_{34}I_2N_2$: C, 34.72; H, 7.07; I, 52.42; N, 5.78. Found: C, 34.55; H, 7.21; I, 52.57; N, 5.69.

A *picrate* was prepared in aqueous solution and recrystallized from methanol. Ethylenebis(triethylammonium picrate) melted at 244–245°.

Anal. Calcd. for $C_{26}H_{38}N_8O_{14}$: C, 45.47; H, 5.58; N, 16.32. Found: C, 45.64; H, 5.83; N, 16.12.

After concentrating the combined aqueous mother liquors of the ethylenebis(triethylammonium iodide) recrystallizations to a small volume and subsequent cooling, 0.2 g. (0.5%) ethane-1-diethylammonium-2-triethylammonium diiodide, m.p. 193–194° (dec.), was obtained.

Isolation of ethane-1-diethylamino-2-triethylammonium iodide. Ether was added to the alcoholic mother liquor which remained after filtration of the bisquaternary ammonium iodide until a white precipitate started to separate. The precipitate was filtered, washed with ether, and dried *in vacuo*, m.p. 89–92°. Recrystallization from acetone-ether gave 14.3 g. (43.5%) ethane-1-diethylamino-2-triethylammonium iodide, m.p. 90.5–92.5°. This product is very soluble in water, ethanol, and acetone, insoluble in ether.

Anal. Calcd. for $C_{12}H_{29}IN_2$: C, 43.90; H, 8.90; I, 38.66, N, 8.53. Found: C, 43.91; H, 8.87; I, 38.55; N, 8.57.

A *picrate* was prepared from this monoiodide. Ethane-1-diethylammonium-2-triethylammonium dipicrate was precipitated as a sparingly soluble crystalline salt. On recrystallization from methanol, its m.p. was 186–188°.

Anal. Calcd. for $C_{24}H_{34}N_8O_{14}$: C, 43.77; H, 5.20; N, 17.02. Found: C, 43.95; H, 5.29; N, 17.31.

Preparation of ethane-1-diethylammonium-2-triethylammonium diiodide. This salt was prepared by adding the theoretical amount of hydriodic acid to an aqueous solution of ethane-1-diethylamino-2-triethylammonium iodide. Removal of the solvent under reduced pressure and recrystallizing the residue twice from propanol, gave the desired product, m.p. 194–195° (dec.).

Anal. Calcd. for $C_{12}H_{30}I_2N_2$: I, 55.64. Found: I, 55.79.

This diiodide yielded with picric acid, ethane-1-diethylammonium-2-triethylammonium dipicrate, m.p. 186–188°.

Formation of ethylenebis(triethylammonium iodide) from ethane-1-diethylamino-2-triethylammonium iodide (I). A mixture of 6.5 g. (0.02 mole) (I), 11 g. (0.07 mole) ethyl iodide, and 15 ml. ethanol was refluxed for 8 hr. The precipitate which formed upon cooling was removed by filtration and washed with ethanol. Recrystallization from water yielded 5.1 g. (53%) ethylenebis(triethylammonium) iodide m.p. 236–238° (dec.).

After concentrating the ethanolic filtrate and subsequent cooling, a product precipitated. Removal of the solid by filtration and recrystallizing it from propanol, gave 2.0 g. (22%) of ethane-1-diethylammonium-2-triethylammonium diiodide, m.p. 193–195° (dec.). By adding ether to this last alcoholic mother liquor, 1.5 g. (23%) of material precipi-

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(10) All melting points are corrected. Determinations were made with the Kofler block.

(11) Microanalysis for C, H, and N were carried out by Mr. T. Bella of this Institute.

(12) The halogen of the quaternary halides was determined as ionic halid by the Volhard method. The nonionic bromine was determined by the Carius method.

(13) Lucius[4] reported m.p. 245–246° and a good analysis for ethylenebis(triethylammonium bromide).

tated. This was identified as unchanged starting material (ethane-1-diethylamino-2-triethylammonium iodide).

Preparation of ethylenebis(triethylammonium bromide). Eight and six-tenths grams (0.05 mole) *N, N, N', N'*-tetraethylethylenediamine,[9] 13 g. (0.12 mole) ethyl bromide, and 8 ml. ethanol were refluxed for 12 hr. The solid material which formed on cooling was filtered and the precipitate washed with ethanol and dried *in vacuo,* m.p. 228–235° (dec.). After several recrystallizations from ethanol, 2.2 g. (11.3%) ethylenebis(triethylammonium bromide) were obtained, m.p. 243–244° (dec.). A mixed melting point with the product obtained according to the procedure of Lucius[4] showed a depression, m.p. 225–230° (dec.).

Anal. Calcd. for $C_{14}H_{34}Br_2N_2$: C, 43.08; H, 8.78; Br, 40.95; N, 7.18. Found: C, 42.93; H, 8.89; Br, 40.82; N, 7.31.

After concentrating the alcoholic mother liquor of ethylenebis(triethylammonium bromide) and subsequent cooling, 0.5 g. (2.8%) ethane-1-diethylammonium-2-triethylammonium dibromide precipitated. This salt was purified by recrystallization from propanol-acetone and had a m.p. 204–205° (dec.).

Anal. Calcd. for $C_{12}H_{30}Br_2N_2$: Br, 44.13. Found: Br, 43.83.

Upon adding aqueous picric acid to this dibromide, ethane-1-diethylammonium-2-triethylammonium dipicrate, m.p. 186–188°, was formed. It was identified by a mixed melting point with the sample prepared above.

Ethane-1-diethylamino-2-triethylammonium bromide. All the solvent from the ethanolic mother liquor which remained after filtration of ethane-1-diethylammonium-2-triethylammonium dibromide was removed *in vacuo.* The oily residue did not solidify on cooling. Yield was 11.9 g. (84.5%). This oil was soluble in acetone, insoluble in ether and gave a positive Br^- test. A picrate was prepared in ethanol which was identified by a mixed melting point, as ethane-1-diethylammonium-2-triethylammonium dipicrate, m.p. 186–188°.

Preparation of ethane-1-diethylammonium-2-triethylammonium dibromide. By adding the theoretical amount of hydrobromic acid to an aqueous solution of this oily residue, evaporating the water *in vacuo,* and recrystallizing the residue from propanol-acetone, a white crystalline material was obtained, m.p. 205–206° (dec.). This product was shown to be ethane-1-diethylammonium-2-triethylammonium dibromide. The oil was therefore ethane-1-diethylamino-2-triethylammonium bromide.

Preparation of ethylenebis(triethylammonium chloride). A solution of ethylenebis(triethylammonium bromide) was chromatographed over the cation exchange resin DOWEX 50-WX8, 200–400 mesh, used in hydrogen form. The column was eluted with 3*N* hydrochloric acid, the effluent collected, the solvent evaporated *in vacuo,* and the residue crystallized from propanol-acetone. Ethylenebis(triethylammonium chloride) had a m.p. 278–279° (dec.).

Anal. Calcd. for $C_{14}H_{34}Cl_2N_2$: Cl, 23.53. Found: Cl, 23.42.

This dichloride gave with picric acid ethylenebis(triethylammonium picrate), m.p. 244–245°.

NEW YORK, N. Y.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, ANDHRA UNIVERSITY]

Synthesis of Chromones. II. Some Derivatives of 7-Hydroxy-2-methylchromone

CH. BHEEMASANKARA RAO, G. SUBRAMANYAM, AND V. VENKATESWARLU

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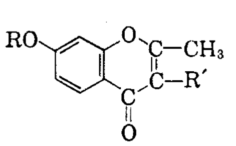
7-Hydroxy-2-methylchromone has been synthesized by an improved method and a number of its derivatives have been prepared.

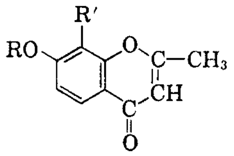
An earlier publication[1] which may be considered as Part I of this series deals with the chemistry of some naturally occurring chromones and reports the synthesis of 5-methoxy-, and 5,8-dimethoxy-2-methylchromones. The present communication deals with the synthesis of a number of derivatives of 7-hydroxy-2-methylchromone.

7-Hydroxy-2-methylchromone[2] (I) has been synthesized by an improved method using the Kostanecki reaction, with the intermediate, 7-acetoxy-3-acetyl-2-methylchromone (II), isolated in good yield and characterized by the ready formation of its 2,4-dinitrophenylhydrazone. Hydrolysis of II using aqueous sodium carbonate gave rise to I, which gave its 2,4-dinitrophenylhydrazone during twenty four hours.

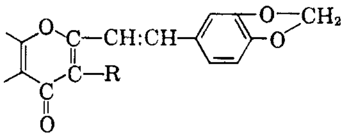
Methylation of I using excess diazomethane gave its methyl ether (III) which yielded its 2,4-dinitrophenylhydrazone during twenty-four hours.



I. R = R' = H
II. R = R' = $COCH_3$
III. R = CH_3; R' = H
VI. R = $COCH_3$; R' = H



IV. R = H; R' = CHO
V. R = $COCH_3$; R' = CHO
VII. R = H; R' = $COCH_3$



VIII. R = H or $COCH_3$

Condensation of I using hexamine in glacial acetic acid produced 8-formyl-7-hydroxy-2-methylchromone (IV), as pale yellow rectangular plates, characterized by an intense red ferric color and the ready formation of its 2,4-dinitrophenylhydrazone. Its acetate V was prepared, which was further characterized by the ready formation of its 2,4-dinitrophenylhydrazone. Acetylation of I using

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