a d^{20}_4 of 1.030. A sample of this was distilled in a molecular still at bath temperature of 55° (13 μ) and the distillate analyzed; d^{20}_4 1.037; $n^{20}D$ 1.5010.

Anal. Calcd. for $C_{15}H_{16}O_3$: C, 69.20; H, 7.74; (O), 7.68. Found: C, 69.1, 69.0; H, 7.79, 7.54; (O), 7.44, 7.51.

Di-*t*-amyl Peroxide.—*t*-Amyl hydrogen sulfate was prepared by adding at 5° with stirring 176 g. (2 moles) of *t*-amyl alcohol to 140 g. (1 mole) of 70% sulfuric acid. To this mixture was then added simultaneously in the course of four hours with vigorous stirring, keeping the temperature at 5°, 126 g. (1 mole) of 27% hydrogen peroxide and 341 g. of concentrated sulfuric. The reaction mixture separated into two layers; the organic layer was removed, washed with water, dried, and distilled through a Vigreux column under reduced pressure and the fraction (43 g.) boiling at 44° (10 mm.) collected and analyzed; d^{20}_4 0.821; $n^{20}D$ 1.4095.

Anal. Calcd. for $C_{10}H_{22}O_2$: C, 68.91; H, 12.72. Found: C, 68.8, 69.1; H, 12.4, 12.5.

Pyrolysis of Di-*t*-amyl Peroxide.—Di-*t*-amyl peroxide (20 g.) was pyrolyzed at 250° using the apparatus described in a previous paper.[2] The pyrolysis products were as follows: (1) A liquid (12.4 g.) collected in an ice trap, 11.5 g. of which was acetone, b. p. 55–56°; 2,4-dinitrophenylhydrazone; m. p. 125.5–126.5°. From the residue (approx. 1 g.) was obtained a 2,4-dinitrophenylhydrazone, m. p. 115.5–117° which agrees with the melting point of the same derivative of methyl ethyl ketone. (2) A liquid condensate (4.1 g.) in the carbon dioxide ice trap having a b. p. of −1 to 0° and by analysis proved to be *n*-butane.

Anal. Calcd. for C_4H_{10}: O_2/C_4H_{10}, 6.5; CO_2/C_4H_{10} 4.0. Found: O_2/gas, 6.9; CO_2/gas, 3.9.

(3) A gas (360 cc.) which had an analysis corresponding to about 80% ethane and 20% propane.

Acknowledgment.—The authors wish to thank Mr. C. O. Ewing for assistance in some of the experiments and the Union Bay State Chemical Company for financial aid in carrying out this investigation.

Summary

1. *t*-Amyl hydroperoxide and di-*t*-amyl peroxide have been synthesized and some of their properties determined.

2. *t*-Amyl perbenzoate has been prepared from *t*-amyl hydroperoxide.

3. The thermal decomposition of di-*t*-amyl peroxide has been found to lead mainly to the formation of acetone and *n*-butane. These results are best accounted for by the formation of intermediate free radicals.

CAMBRIDGE, MASSACHUSETTS
RECEIVED DECEMBER 31, 1945

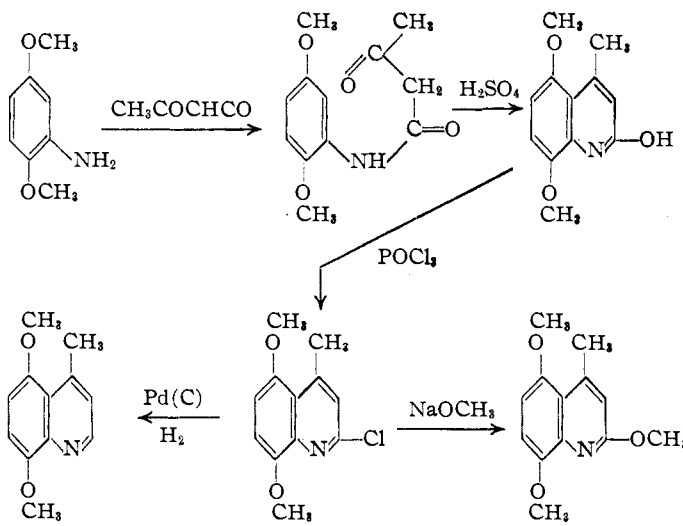
[CONTRIBUTION FROM THE CHEMISTRY LABORATORY OF INDIANA UNIVERSITY]

Substituted Lepidines[1]

BY C. E. KASLOW AND N. B. SOMMER[2,3]

A useful method for the synthesis of substituted lepidines has been developed. As an illustration of the reactions described in the present paper, the following equations may be cited which show the synthesis of 5,8-dimethoxy- and 2,5,8-trimethoxylepidine.

The first step in this synthesis involves the treatment of a primary aromatic amine with ketene dimer which formed the substituted acetoacetanilides in 78–98% yields. The yields by this method were much higher than those obtainable by the action of an aryl amine on ethyl acetoacetate which reaction sometimes leads to the formation of β-arylaminocrotonates.[4] The other steps in the syntheses are well-known reactions and proceed smoothly so that the substituted lepidines are obtained from the primary aromatic amines in over-all yields which range from 64–80%. These yields are an improvement over previous direct methods of synthesis.[5]



In the experimental part of this paper, there are described the preparation of six substituted lepidines and two benzoquinolines by use of the above synthetic scheme. The intermediate substituted acetoacetanilides, carbostyrils and 2-chlorolepidines were also isolated and characterized.

(1) Presented in part before the Division of Organic Chemistry, American Chemical Society, New York, N. Y., September 13, 1944.

(2) Abstracted from a thesis submitted to the Faculty of the Graduate School in partial fulfillment of the requirements for the degree, Doctor of Philosophy, in the Department of Chemistry, Indiana University.

(3) Present address: American Cyanamid Company, Stamford, Connecticut.

(4) Krahler and Burger, THIS JOURNAL, **64**, 2417 (1942).

(5) Campbell and Schaffner, *ibid.*, **67**, 86 (1945).

TABLE I

N-ARYLACETOACETAMIDES, $ArNHCOCH_2COCH_3$

Ar =	M. p., °C.	Yield, %	2,4-Dinitrophenylhydrazone: M. p., °C.	Empirical formula	N Analyses, % Calcd.	Found
2-$CH_3C_6H_4$—	107-108[a]	86	213-214	$C_{17}H_{17}N_5O_5$	18.87	18.71
4-$CH_3C_6H_4$—	94-95[b]	93	210-211	$C_{17}H_{17}N_5O_5$	18.87	18.47
2,5-$(CH_3)_2C_6H_3$—	98-99[c]	94	228-230	$C_{18}H_{19}N_5O_5$	18.11	17.90
2,4-$(CH_3)_2C_6H_3$—	88-89[d]	78	208-209	$C_{18}H_{19}N_5O_5$	18.11	18.10
4-$CH_3OC_6H_4$—	115-116[e]	93	209-210	$C_{17}H_{17}N_5O_6$	18.09	17.90
2,5-$(CH_3O)_2C_6H_3$—	67-68	94	143-144	$C_{18}H_{19}N_5O_7$	16.79	17.23
α-$C_{10}H_7$—	108-109[f]	86	241-242	$C_{20}H_{17}N_5O_5$	17.20	16.90
β-$C_{10}H_7$—	91-92[g]	98	220-221	$C_{20}H_{17}N_5O_5$	17.20	16.80

[a] Ewins and King, *J. Chem. Soc.*, **103**, 104 (1913), reported the m. p. 107-108°. [b] Ewins and King, *ibid.*, reported the m. p. 94-95°. [c] Thomson and Wilson, *ibid.*, 111 (1935), reported the m. p. 98-99°. [d] Fierz-David and Ziegler, *Helv. Chim. Acta*, **11**, 776 (1928), reported the m. p. 88-89°. [e] Bayer and Co., German Patent 268,318, **11**, 454 (1912), reported the m. p. 115-116°. [f] Gibson, Hariharan, Menon and Simonsen, *J. Chem. Soc.*, 2247 (1926), reported the m. p. 107°. [g] Knorr, *Ber.*, **17**, 540 (1884), reported the m. p. 92°.

Experimental[6]

2,5-Dimethoxyacetoacetanilide.—To a stirred solution of 45.9 g. (0.3 mole) of 2,5-dimethoxyaniline in 100 ml. of warm benzene contained in a 500-ml., round-bottomed, three-necked flask fitted with a reflux condenser, was added dropwise 27 g. (0.32 mole) of ketene dimer. After addition of the ketene dimer, the reaction mixture was refluxed for thirty to forty minutes, then cooled in ice water. The 2,5-dimethoxyacetoacetanilide was removed by filtration, washed with two 50-ml. portions of a benzene–ligroin (2:1) solution and dried in air. The yield of the white crystalline substance was 66.8 g. (94%); m. p. 63-66°. An additional 3.2 g. of less pure material (m. p. 58-62°) was obtained by concentration of the combined mother liquor and filtrate. The substance was purified by crystallization either from a benzene–ligroin solution or from 50% ethyl alcohol; m. p. 67-68°.

Anal. Calcd. for $C_{12}H_{15}NO_4$: N, 5.90. Found: N, 5.94.

The 2,4-dinitrophenylhydrazone was prepared according to the directions of Shriner and Fuson[7] and was recrystallized from 95% ethyl alcohol; m. p. 143-144°.

Anal. Calcd. for $C_{18}H_{19}N_5O_7$: N, 16.79. Found: N, 17.23.

The other N-arylacetoacetamides were prepared in a manner similar to the above procedure and are summarized in Table I. No advantage was gained by purification of the N-arylacetoacetamide before proceeding to the next step.

5,8-Dimethoxy-4-methylcarbostyril.—Sixty-nine grams (0.29 mole) of 2,5-dimethoxyacetoacetanilide was added in 2-3 g. portions to 100 ml. of warm concentrated sulfuric acid at a rate sufficient to maintain the temperature at about 85°, and finally heated in a boiling water-bath for fifteen minutes. After cooling to about 60°, the reaction mixture was poured into 500 ml. of ice and water and, after adding more ice, concentrated sodium hydroxide solution was added until there was no further precipitation. After filtration, the white solid was washed thoroughly with successive 100-ml. portions of water, 2% sodium bicarbonate solution and distilled water. After drying at 105-110°, the yield was 53.4 g. (84%); m. p. 178-179.5°. The substance crystallized as a white granular solid from hot 50% ethyl alcohol; m. p. 180-181°.

Anal. Calcd. for $C_{12}H_{13}NO_3$: N, 6.39. Found: N, 6.01.

The other substituted 4-methylcarbostyrils were prepared in a similar manner; the results are summarized in Table II. Analyses are given in the table for new compounds; those recorded previously in the literature were characterized by melting point only.

It was not necessary to purify the carbostyril before conversion of it to the chloro derivative.

2-Chloro-5,8-dimethoxylepidine.—A mixture of 51 g. (0.233 mole) of 5,8-dimethoxy-4-methylcarbostyril and 77 g. (0.5 mole) of freshly distilled phosphoryl trichloride in a 125-ml. flask, to which was attached an air condenser, was heated in a boiling water-bath until most of the material had liquefied; then it was warmed gently with a flame to complete the reaction. The warm reaction mixture was poured into 500 ml. of ice and water, the solution was filtered, then ice added and concentrated sodium hydroxide solution added until no further precipitate was formed. The solid was removed by filtration, washed thoroughly with successive 100-ml. portions of water, 1% sodium bicarbonate solution and distilled water and then dried at 80-85°. The yield was 52.8 g. (95.5%); m. p. 117-117.5°. The substance crystallized from ethyl alcohol as fine white needles; m. p. 118-118.5°.

Anal. Calcd. for $C_{12}H_{12}ClNO_2$: N, 5.89. Found: N, 5.97.

The other 2-chlorolepidines were prepared in a similar manner. The results are summarized in Table II. In most cases, a very viscous oily substance separated when the reaction mixture was poured into water. A flocculent solid was formed as the metaphosphoric acid dissolved slowly thus making it necessary to allow the mixture to stand for twelve to twenty-four hours before addition of sodium hydroxide and filtration of the solid. The chlorolepidines were purified by recrystallization before catalytic reduction was attempted.

5,8-Dimethoxylepidine.—The procedure used was essentially that described by Ainley and King.[8] Ten grams (0.042 mole) of pure 2-chloro-5,8-dimethoxylepidine was dissolved in 200 ml. of glacial acetic acid, 3 g. of anhydrous sodium acetate and 1.5 g. of palladized charcoal[9] was added, the mixture heated to 70° and reduced in a low pressure hydrogenation apparatus at forty-five pounds per square inch. After removal of the catalyst, most of the acetic acid was removed by vacuum distillation, the residue diluted with 200 ml. of water and neutralized with sodium hydroxide. The crude 5,8-dimethoxylepidine was removed by filtration and recrystallized from 50% ethyl alcohol solution; yield 7.6 g. (90%), m. p. 91-93°. After a second recrystallization, the substance was obtained as a fluffy white solid which melted at 94-95°.

Anal. Calcd. for $C_{12}H_{13}NO_2$: N, 6.89. Found: N, 6.92.

The picrate was prepared according to the directions of Shriner and Fuson[10] and recrystallized from ethyl alcohol; m. p. 190-191°.

(6) All melting points are corrected.

(7) Shriner and Fuson, "Identification of Organic Compounds," 2nd ed., John Wiley and Sons, Inc., New York, N. Y., 1940, p. 143.

(8) Ainley and King, *Proc. Roy. Soc.* (London), **B125**, 60 (1938); *C. A.*, **32**, 4219 (1938).

(9) Shriner, "Quantitative Analysis of Organic Compounds," 2nd ed., Edwards Brothers, Inc., Ann Arbor, Michigan, 1944, p. 54.

(10) Shriner and Fuson, ref. 7, p. 149.

TABLE II

SUBSTITUTED LEPIDINES

R	Substituted carbostyril M. p., °C.	Yield, %	2-Chloroquinoline M. p., °C.	Yield, %	Quinoline M. p., °C.	Yield, %	Over-all yield, %[t]	2-Methoxyquinoline M. p., °C.	Yield, %
4,8-$(CH_3)_2$—	217–218[a]	99	64.5–65.5	98[h]	53–54[m]	90	75	35–36[u]	76
4,6-$(CH_3)_2$—	249–250[b]	99	95–96	94[i]	[n]	87	75	41–42[v]	82
4,5,8-$(CH_3)_2$—	234–235	90[c]	76–77	95[j]	[o]	86	70	73–74[w]	70
4,6,8-$(CH_3)_3$—	252–253	92[d]	130–131	99[k]	55–56[p]	91	64	76.2–76.6[x]	75
4-CH_3-6-CH_3O—	271–272[e]	75	144–145[l]	97	32–35[q]	96	65	55–56[y]	99
4-CH_3-5,8-$(CH_3O)_2$—	180–181	84	118–118.5	95	94–95	90	67	134–134.6	99

SUBSTITUTED BENZOQUINOLINES

	Hydroxybenzoquinoline		Chlorobenzoquinoline		Benzoquinoline			Methoxybenzoquinoline	
4-CH_3-Benzo(h)—	291–292[f]	99	134–135[bb]	99	71–72[r]	85	71	68.5–69.2[z]	75
1-CH_3—Benzo(f)—	285–286[g]	99	153–154[cc]	92	94–95[s]	90	80	88.5–89[aa]	94

[a] Ewins and King, *J. Chem. Soc.*, **103**, 104 (1913), reported the m. p. 217–218°. [b] Knorr, *Ber.*, **17**, 540 (1884), reported the m. p. 245°. [c] *Anal.* Calcd. for $C_{12}H_{13}NO$: N, 7.49. Found: N, 7.30. [d] *Anal.* Calcd. for $C_{12}H_{13}NO$: N, 7.49. Found: N, 7.24. [e] Bacheberg and Kermack, *J. Chem. Soc.*, 377 (1934), reported the m. p. 272°. [f] Conrad and Limpach, *Ber.*, **21**, 523 (1888), reported that the substance did not melt below 300°. [g] Knorr, *Ber.*, **17**, 540 (1884), reported the m. p. 286°. [h] *Anal.* Calcd. for $C_{11}H_{10}ClN$: N, 7.31. Found: N, 7.14. [i] *Anal.* Calcd. for $C_{11}H_{10}ClN$: N, 7.31. Found: N, 7.18. [j] *Anal.* Calcd. for $C_{12}H_{12}ClN$: N, 6.81. Found: N, 6.50. [k] *Anal.* Calcd. for $C_{12}H_{12}ClN$: N, 6.81. Found: N, 6.83. [l] Rabe, Huntenburg, Schultze and Volger, *Ber.*, **64**, 2487 (1931), reported the m. p. 145°. [m] Manske, Marion and Leger, *Can. J. Research*, **20B**, 133 (1942), reported 58°. [n] B. p. 137–138 (12 mm.); Knorr, *Ann.*, **245**, 357 (1888), reported 280° at 754 mm. [o] B. p. 155 (13 mm.). *Anal.* Calcd. for $C_{12}H_{13}N$: N, 8.18. Found: N, 8.04. Picrate, m. p. 186–187°. *Anal.* Calcd. for $C_{18}H_{16}N_4O_7$: N, 14.00. Found: N, 13.93. [p] *Anal.* Calcd. for $C_{12}H_{13}N$: N, 8.18. Found: 7.86. Picrate, m. p. 208–209°. *Anal.* Calcd. for $C_{18}H_{16}N_4O_7$: N, 14.00. Found: N, 14.24. [q] Koenigs, *Ber.*, **23**, 2669 (1890), reported the hydrate to melt at 50–52°. [r] *Anal.* Calcd. for $C_{14}H_{11}N$: N, 7.25. Found: N, 7.21. Picrate, m. p. 218–219°. *Anal.* Calcd. for $C_{20}H_{14}N_4O_7$: N, 13.27. Found: N, 13.41. [s] Knorr, *Ber.*, **17**, 540 (1884), reported the m. p. 91–92°. [t] Calculated from the primary aromatic amine used as the starting material. [u] *Anal.* Calcd. for $C_{12}H_{13}NO$: N, 7.49. Found: N, 7.31. [v] *Anal.* Calcd. for $C_{12}H_{13}NO$: N, 7.49. Found: N, 7.45. [w] *Anal.* Calcd. for $C_{13}H_{15}NO$: N, 6.96. Found: N, 7.40. [x] *Anal.* Calcd. for $C_{13}H_{15}NO$: N, 6.96. Found: N, 7.30. [y] Kermack and Muir, *J. Chem. Soc.*, 300 (1933), reported the m. p. 56°. [z] *Anal.* Calcd. for $C_{15}H_{13}NO$: N, 6.28. Found: N, 6.75. [aa] *Anal.* Calcd. for $C_{15}H_{13}NO$: N, 6.28. Found: N, 6.49. [bb] Gibson, Hariharan, Menon and Simonsen, *J. Chem. Soc.*, 2247 (1926), reported 134–135°. [cc] Ref. bb, reported the m. p. 153–154°.

Anal. Calcd. for $C_{18}H_{16}N_4O_9$: N, 12.96. Found: N, 12.67.

The hydrochloride of 5,8-dimethoxylepidine was prepared by bubbling dry hydrogen chloride into an ether solution of the lepidine. The hydrochloride was obtained as a yellow colored solid; m. p. 219–220 (dec.).

Anal. Calcd. for $C_{12}H_{14}ClNO_2$: Cl, 14.82. Found: Cl, 14.48.

The reductions of the other chlorolepidines were carried out in a similar way but in case the lepidine was a liquid the crude substance was extracted with ether and distilled under reduced pressure.

2,5,8-Trimethoxylepidine.—A solution of 4.6 g. (0.2 mole) of sodium in 50 ml. of methyl alcohol was refluxed with 11.9 g. (0.05 mole) of 2-chloro-5,8-dimethoxylepidine for five hours. The reaction mixture was diluted with 200 ml. of water, the solid removed by filtration, washed thoroughly with distilled water and dried at 80–85°. The yield of 2,5,8-trimethoxylepidine was 11.5 g. (99%); m. p. 132–134°. The substance was crystallized from ethyl alcohol; m. p. 134–134.6°.

Anal. Calcd. for $C_{13}H_{15}NO_3$: N, 6.01. Found: N, 6.25.

The length of time required for refluxing varied with the individual chlorolepidine; it is summarized in the following list: 2-chloro-8-methyllepidine (ten hours); 2-chloro-6-methyllepidine (five hours); 2-chloro-5,8-dimethyllepidine (five hours); 2-chloro-6,8-dimethyllepidine (eighteen hours); 2-chloro-6-methoxylepidine (six hours); 2-chloro-4-methylbenzo(h)quinoline (seventy-two hours) and 3-chloro-1-methylbenzo(f)quinoline (fifteen hours). If the crude 2-methoxy derivative gave a positive Beilstein test for halogen, it was extracted with 1–2% hydrochloric acid, filtered and the methoxy derivative precipitated from the filtrate by the addition of dilute alkali. The results are summarized in Table II.

The pure hydrochlorides of the 2-methoxy derivatives may be obtained only with difficulty; even by the addition of the theoretical amount of hydrogen chloride, the precipitated hydrochloride may contain an excess of 5–10% chloride ion, leaving some of the free base in the ether solution. Without doubt some of the hydrogen chloride may be held as an oxonium salt. The hydrochloride of 2,5,8-trimethoxylepidine as well as its water solution is a bright yellow color. The hydrochlorides decompose to the corresponding 2-hydroxy compound when heated to determine the melting point. The decomposition temperature depends upon the rate of heating and will sometimes occur without a visible change and melt either at or near the melting point of the corresponding carbostyril.

Summary

The preparation of 6-methyl- and 2-methoxy-6-methyllepidine; 8-methyl- and 2-methoxy-8-methyllepidine; 5,8-dimethyl- and 5,8-dimethyl-2-methoxylepidine; 6,8-dimethyl- and 6,8-dimethyl-2-methoxylepidine; 5,8-dimethoxy- and 2,5,8-trimethoxylepidine; 4-methyl- and 2-methoxy-4-methylbenzo(h)quinoline; 1-methyl-, and 3-methoxy-1-methylbenzo(f)quinoline from ketene dimer and the appropriate primary aromatic amine as starting materials has been described. The over-all yields of the substituted lepidines and benzoquinolines based on the primary aromatic amines were 64–80 per cent.

The following new compounds were also prepared as intermediates in the above syntheses: 2,5-dimethoxyacetoacetanilide, 4,5,8-trimethyl-

carbostyril, 4,6,8-trimethylcarbostyril, 5,8-dimethoxy-4-methylcarbostyril, 2-chloro-8-methyllepidine, 2-chloro-6-methyllepidine, 2-chloro-5,8-dimethyllepidine, 2-chloro-6,8-dimethyllepidine and 2-chloro-5,8-dimethoxylepidine.

BLOOMINGTON, INDIANA RECEIVED DECEMBER 5, 1945

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE UNIVERSITY OF COLORADO AND COLORADO A. AND M. COLLEGE]

The Glyoxalines. V. The Bromination of 2-Phenyl-4-benzal-5-glyoxalidone

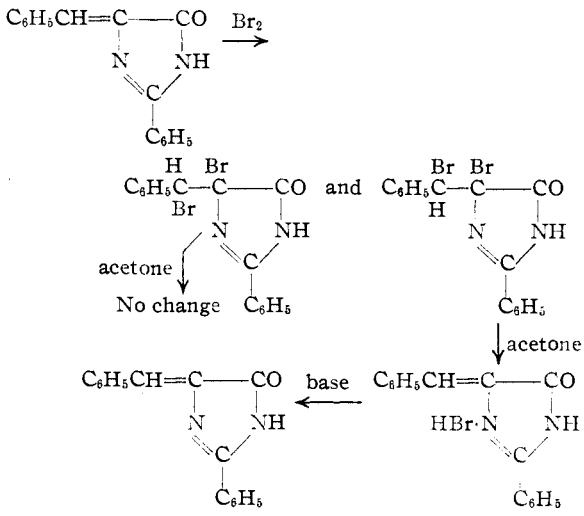
BY DAVID LLOYD WILLIAMS[1] AND ANTHONY R. RONZIO[2]

The synthesis of 2-phenyl-4-benzal-5-glyoxalidone by a number of methods gives varying melting points.[3] The theory has been proposed that this variation of melting points may be due to the *cis–trans* isomerism introduced into the compound by the double bond between the benzal group and the glyoxaline ring.

A study of the bromination of the compound was undertaken with a view of studying this structure and is here reported.

Bromination of the compound in glacial acetic acid by the method of Minovici,[4] yielded a finely divided, crystalline orange precipitate. Attempts at recrystallization from the common organic solvents brought about decomposition of the compound with the formation of a red solution. However, upon placing the product in acetone, partial solution took place leaving a small amount of an insoluble, pale yellow compound. After quickly filtering off the precipitate, the filtrate began to deposit small yellow needles and the acetone acquired the strong lachrymatory power of bromoacetone. The soluble fraction spontaneously lost bromine. Analyses confirmed this observation. The bromination of the glyoxalidone thus leads to two products. The bromination product of one form is unstable and loses bromine. The acetone used as solvent behaves as acceptor for the bromine forming bromoacetone and hydrogen bromide. The hydrogen bromide then, in turn, adds to the debrominated glyoxalidone which precipitates as the insoluble hydrobromide. These reactions are shown in Equation 1.

That a hydrobromide of 2-phenyl-4-benzal-5-glyoxalidone had formed was easily shown by removing the hydrobromic acid with dilute base. The melting point of the compound thus obtained was 280°. The pale yellow compound insoluble in acetone was found by analysis to be a dibromo glyoxalidone. The designation of α-2-phenyl-4-(α - bromobenzyl) - 4 - bromo - 5 - glyoxalidone has been assigned to the compound. The mixture of the two forms of the dibromo glyoxalidone has been designated as ω-2-phenyl-4-(α-bromobenzyl)-4-bromo-5-glyoxalidone.



Equation 1

Two possible explanations can be offered for this unusual phenomenon. Either the benzal glyoxalidone exists as a mixture of *cis* and *trans* forms, thus leading to two dibromo derivatives, having different properties; or, the loading of the carbon atom between the benzene and the glyoxaline ring brought about by the bromination leads to two forms of dibromo derivatives because of steric hindrance. When the bromine atoms are adjacent (*cis*) to one another they are easily removed. When they are opposite from each other (*trans*) they are stable.

The amount of bromine used in the bromination exerted an important influence upon the amount of brominated derivative crystallizing out of the acetic acid solution. When an excess of bromine over the calculated amount was used, no precipitate formed. Upon allowing the solution to stand twenty-four to forty-eight hours, however, glistening orange crystals separated. Analyses indicated that the tribromo derivative of the glyoxalidone had formed. This compound dissolved in acetone completely, then reprecipitated slowly as yellow crystals in a manner analogous to the dibromo derivative. The lachrymatory action of bromoacetone was again noticed. The yellow compound, a hydrobromide, was then treated with dilute sodium hydroxide. Analyses indicated that the free base thus formed was 2-

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(3) Williams, Symonds, Ekeley and Ronzio, THIS JOURNAL, **67**, 1157 (1945).

(4) Minovici, *Ber.*, **32**, 2206 (1899).