Experimental

The vitamin E fraction from soybean oil was obtained by distillation in a centrifugal molecular still and further concentrated by redistillation. The resulting fraction (42.1% tocopherol by Emmerie-Engel assay) was cooled successively at −35 and −70° in ethyl formate solution (10%) to remove sterols. The recovered oil (55% tocopherol) was purified by chromatography and converted to the palmitate in the manner previously described.[2] The γ-tocopherol palmitate, after three crystallizations from acetone, melted at 44–45°. This is the same value previously reported.[2] The ester was saponified and the free tocopherol distilled in a small pot still at 0.15–0.2 mm. pressure. Its extinction coefficient ($E^{1\%}_{1cm.}$ (298 mμ)) = 94.5 and that of the palmitate ($E^{1\%}_{1cm.}$ (286 mμ)) = 40.0 were slightly higher than those previously reported.

The γ-tocopherol so prepared was assayed in the Biological Department of this Laboratory, using the technique previously described.[3] Its potency was only one-hundredth that of *d*,α-tocopherol, supporting the aforementioned hypothesis that pure γ-tocopherol has negligible activity in curing resorptive sterility.

Recently the vitamin A sparing activity of a specimen of natural γ-tocopherol from cottonseed oil was found to be fully equal to that of α- and β-tocopherols.[5] It appears unlikely that the sparing activity of the more highly purified preparation just described will be significantly different but the point is being investigated.

(5) Hickman, Kaley and Harris, *J. Biol. Chem.*, **152**, 327 (1944).

CONTRIBUTION NO. 70 FROM THE
LABORATORIES OF DISTILLATION PRODUCTS, INC.
755 RIDGE ROAD WEST
ROCHESTER 13, NEW YORK

RECEIVED APRIL 12, 1945

NEW COMPOUNDS

2-Phenyl-4-(4-quinolal)-5-oxazolone and 2-Phenyl-4-(4-quinolal)-5-glyoxalidone

In 1904 Erlenmeyer, Jr.,[1] suggested the general nature of the reaction between hippuric acid and benzaldehyde and the production of a glyoxalidone on treatment of the condensation product with alcoholic ammonia. Quinoline-4-aldehyde hydrate and hippuric acid condense in an analogous manner to yield 2-phenyl-4-(4-quinolal)-oxazolone-5. On treatment with ammonium hydroxide, the oxazolone yields 2-phenyl-4-(4-quinolal)-glyoxalidone-5.

2-Phenyl-4-(4-quinolal)-oxazolone-5.—A mixture of 5 g. (0.029 mole) of quinoline-4-aldehyde hydrate, 2.6 g. (0.29 mole) of fused potassium acetate, and 5.15 g. (0.29 mole) of hippuric acid was ground in a mortar and transferred to an Erlenmeyer flask. Twelve grams (0.18 mole) of acetic anhydride was added and the mixture allowed to stand for one hour. The temperature rose slowly to 70° when solution became almost complete. The mixture was cooled to room temperature, chilled in ice and filtered. The small amount of tarry matter was removed by washing with acetone. The portion of the product which dissolved in the acetone was recovered by evaporation under reduced pressure and again washed with acetone; total yield, 6.8 g. The product was purified by washing with hot water, drying and crystallization from 25 cc. of *s*-amyl alcohol. The yield consisted of fine greenish yellow needles, m. p. 171–172°. The compound is soluble in hot *n*-amyl and *s*-amyl alcohol, insoluble in dilute hydrochloric acid and dilute sodium hydroxide. It is slightly soluble in cold acetone, methyl alcohol and ethyl alcohol.

(1) Erlenmeyer, *Ann.*, **337**, 265 (1904).

Anal. Calcd. for $C_{19}H_{12}O_2N_2$: C, 75.98; H, 4.03; N, 9.33; mol. wt., 300. Found: C, 75.56, 75.86; H, 4.26, 4.07; N, 9.25, 9.38; mol. wt., 308.

2-Phenyl-4-(quinolal)-glyoxalidone-5.—A 2-g. portion of the oxazolone was suspended in 20 cc. of 95% alcohol in a 100-cc. flask. The mixture was heated to boiling under a reflux and treated with 10 cc. of concd. ammonium hydroxide. When the yellow compound dissolved 1 g. of potassium carbonate dissolved in a little water was added and the heating continued for one hour. Small amounts of ammonium hydroxide were added from time to time. Ten cc. of 25% sodium hydroxide solution was added to the orange colored solution and the heating continued for one-half hour. The solution was cooled and made slightly acid with acetic acid. The yellow precipitate was filtered, washed with water, dried, and crystallized from *s*-amyl alcohol, washed with ether and dried; yield 1.6 g., m. p. 304–305° (dec.).

Anal. Calcd. for $C_{19}H_{13}ON_3$: C, 76.24; H, 4.38; N, 14.04. Found: C, 76.07, 76.44; H, 4.01, 4.08; N, 14.20, 14.10. Melting point of picrate was 275–277° (dec.).

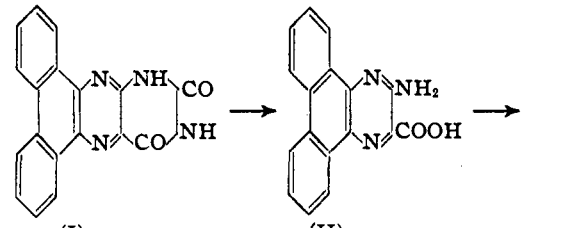
The glyoxalidone treated with hydrochloric acid yielded a brilliant red compound, m. p. 308–309° (dec.).

CHEMISTRY LABORATORY
UNIVERSITY OF COLORADO
BOULDER, COLORADO
DALE M. GRIFFIN
PAUL M. DEAN

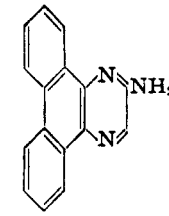
RECEIVED APRIL 30, 1945

2-Amino-dibenzo[f,h]quinoxaline-3-carboxylic Acid, 2-Amino-dibenzo[f,h]quinoxaline and 2-Sulfanilamido-dibenzo[f,h]quinoxaline

By the hydrolysis of 9′,10′-phenanthrolumazine, (dibenzo[f,h]pyrimido[4,5-b]quinoxaline-11,13(10,12)-dione)[1] (I), the amino acid, 2-amino-dibenzo[f,h]quinoxaline-3-carboxylic acid (II) was obtained which could be decarboxylated readily to 2-amino-dibenzo[f,h]quinoxaline (III). The parent substance, phenanthrapyrazine, has been reported by Mazon.[2]



(I) (II)



(III)

2-Amino-dibenzo[f,h]quinoxaline-3-carboxylic Acid.—To a solution of 9 g. of sodium hydroxide in 75 cc. of water was added 9 g. of 9′,10′-phenanthrolumazine, and the mixture was held in a steel bomb at 225–235° for twenty hours. The reaction mixture was dissolved in 1000 cc. of boiling

(1) Kuhn and Cook, *Ber.*, **70**, 761 (1937).

(2) Mazon, *ibid.*, **19**, 112 (1886); *ibid.*, **20**, 268 (1887); *J. Chem. Soc.*, **55**, 97 (1889).