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The Isolation of New Antioxidants from Vegetable Fats

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THE PREPARATION OF COLORIMETRIC STANDARDS

FERRIN B. MORELAND

In colorimetric determinations it is often found when the color is developed that the standard and unknown differ too much for accurate readings. Usually this is remedied by beginning the determination again and using a different amount of standard or unknown sample. It would be quicker to increase the color by the addition of more standard directly to the paler of the two colored solutions, if it were known that the increase in color intensity were proportional to the amount of added standard. The present investigation established the proportionality between increased color and additional standard added after color has been developed according to the usual procedure, as follows: for the determination of phosphate (Fiske and Subbarow), with an error of less than 2%; of creatinine (Folin), with an error of less than 1%; and of ammonia (performed or from digestion of nitrogenous material) by nesslerization (Koch and McMeekin) within 5%. Uric acid added after the other reagents in the Christman and Ravvich modification of the method of Benedict and Franke, produces no color, hence the colored solution cannot be fortified in this determination.

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THE ISOLATION OF NEW ANTIOXIDANTS FROM VEGETABLE FATS¹

CALVIN GOLUMBIC

Many vegetable oils owe their resistance to oxidative deterioration to the presence of natural inhibitors (inhibitols) such as the tocopherols. Equally important antioxygenic factors are the chroman-5, 6-quinones, the reduced forms of which have now been isolated from hydrogenated vegetable fat substrates by chromatographic adsorption on activated alumina. The adsorbed zone contained colorless antioxygenic substances, other than tocopherols, which lost their activity after acetylation but not after oxidation with ferric or gold chlorides. Concentrates obtained from the oxidized fractions, showed the qualitative chemical and spectral absorptive properties of the chroman-5, 6-quinones. The source of these highly colored o-quinones is thus their corresponding hydroquinones, 5-hydroxy tocols, and not tocopherols.

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The chroman-5, 6-quinones have been isolated as such from incipiently rancid vegetable fats by repeated chromatographic adsorption on permutit and silicic acid, followed by saponification in the presence of sodium hydrosulfite. In the absence of this reducing agent, they were rapidly destroyed by alkali. The o-quinones which appear during the autoxidation of hydrogenated vegetable fats are not increased in amount by previous addition of synthetic tocopherol and their presence was never detected in autoxidizing animal fats containing added tocopherol. These o-quinones, their precursors (as acetates) and the chroman-5, 6-quinone prepared from synthetic α -tocopherol by nitric acid oxidation are devoid of vitamin E activity.

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FORMATION AND PROPERTIES OF SOME URETEDIONES

HARLAN B. FREYERMUTH AND L. CHAS. RAIFORD

Hofmann (Ber., 4, 246. 1871.) found that triethylphosphine served as a catalyst to cause the polymerization of phenyl isocyanate to a dimer which he designated as diphenyl diisocyanate. Staudinger ("Die Ketene", Enke, Stuttgart, 1912, p. 126.) regarded this product as a four atomic ring derivative. It has now been found that triethylphosphine polymerizes a number of substituted phenylisocyanates, such as λ - and β -naphthyl isocyanates, and p-xenyl isocyanate. These products which have more recently been listed as derivatives of uretedione, react with aliphatic amines to produce substituted biurets.

In an attempt to prepare the isocyanate of 2-amino-4-methyl-6-bromophenyl p-tolysulfonate, it was found that phosgene reacted with the amine to produce the corresponding uretedione directly. This compound had been previously prepared by Raiford and Shelton (J. Org. Chem., 4, 207. 1939.) by the action of hot pyridine on 2-carbophenoxyamino-4-methyl-6-bromophenyl p-tolysulfonate. The latter method of formation has been confirmed in the present work.

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