triarylbismuth derivative in dry ether. A bright yellow powder rapidly separated out. It melted indefinitely when heated and probably contained some dichloride.

Anal. Calcd. for $C_{24}H_{18}BiCl$: Cl, 6.5. Found: Cl, 7.0.

Tri-biphenylbismuth Dinitrate.—Alcoholic silver nitrate solution was added to an equivalent amount of tri-biphenylbismuth dichloride suspended in a large volume of acetone. On concentrating the filtrate after removal of insoluble material, small plates were obtained that decomposed at approximately 162°, but deflagrated if heated quickly.

Anal. Calcd. for $C_{36}H_{27}BiO_6N_3$: Bi, 26.4. Found: Bi, 26.6.

PEARSON MEMORIAL LABORATORY
TUFTS COLLEGE, MASS.
RECEIVED JULY 7, 1936

Chile Seed

BY W. A. BUSH

The seeds of the Chile pepper (sometimes called pimento) obtained from the pods of the Southern Pacific Coast cultural variety of *Capsicum Annuum* have received little mention in the literature from the point of view of their composition.

A representative sample of a quantity of sixteen tons of dried seeds accumulated during a year from pods mostly of Californian origin gave results differing somewhat from those obtained by Ebert and Bailey[1] from Southeastern seed.

Yields from the seed by drying and extraction were: oil, 26.10%; moisture, 6.25%; dried extracted meal, 67.65%. Analysis of the meal, free from oil and moisture, shows: protein (N × 6.25), 28.92%; fiber, 29.10%; ash, 5.61%; N-free extract (carbohydrates), 36.37%. The constants of the oil, obtained by hot pressing (100°) and then filtering are

Specific gravity, 24.5°/25°	0.918
Refractive index at 25°	1.4738
Color (2.54 cm. column, Lovibond)	100 yellow—46 red
Acid number	2.18
Iodine number, Hanus	133.5
Acetyl number	7.0
Saponification number	192.0
Unsaponifiable matter	1.7%
Melting pt. of separated fatty acids	21.2°

The resemblance to tomato seed oil is striking.[2] However, the presence of colored veins on the outer edges of the seeds imparts to the oil the deep color noted above and a characteristic burning taste and paprika-like aroma.

LOS ANGELES, CALIF.
RECEIVED JULY 6, 1936

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[1] H. C. Ebert and H. S. Bailey, *Cotton Oil Press*, **7**, 35 (1924).

[2] Felice Sorgès, *Chim. Ind. Agr. Biol.*, **5**, 232 (1929); N. Barskii, *Masloboino-Zhirovoe Delo*, No. 6, 56 (1932).

COMMUNICATIONS TO THE EDITOR

MUREXIDE AND LEUCOMUREXIDE

Sir:

The recent publication of Kuhn and Lyman [*Ber.*, **69**, 1547 (1936)] concerning the oxidation-reduction potential of murexide prompts this report of results obtained in this field.

Preparation of Murexide.—The uncertainty in the preparation of murexide emphasized by previous writers including Kuhn and Lyman (who obtained only a 2% yield) has been removed by employing glacial acetic acid as the reaction medium. Furthermore, recrystallization has been effected without heating, by salting out at room temperature. Thus, 16.1 g. of finely powdered alloxantine suspended in 1600 cc. of boiling glacial acetic acid and treated with 80 g. of ammonium acetate with continued boiling for 1–2 minutes gave, on cooling, filtering, washing with acetic acid, alcohol and ether, and drying at 100°, 14.0 g. (calcd. 14.2 g.) of crude murexide. One gram of this dissolved in 900 cc. of water at 30° gave, on salting out the filtered solution with 60 g. of ammonium chloride, filtering, washing with 60% methanol and then with absolute methanol, and drying at 110°, about 75% of beautiful, reddish-brown crystals possessing a green reflex, which, on titration with sodium hydrosulfite, were found to be pure murexide.

Reduction of Murexide.—Leucomurexide has not been described previously, although postulated by Labes and Freisburger [*Arch. exptl. Path. Pharmakol.*, **156,** 226 (1930)] as well as by Kuhn and Lyman. One gram of pure murexide dissolved in 900 cc. of water at 30° was treated with a filtered solution of one g. of sodium hydrosulfite in a mixture of 20 cc. of water and 10 cc. of ammonia. In a few moments complete decolorization occurred. Salting out with 60 g. of ammonium chloride gave on filtering, washing with 60% methanol and finally with absolute methanol, and drying at 110°, 0.98 g. (98%) of a buff-colored microcrystalline powder which turned pink in the air. It contained the theoretical percentage of nitrogen for ammonium dihydropurpurate. Since its composition is identical with uramil (its molecular formula is simply twice that of uramil), a distinguishing test was sought. This was found in the action of potassium ferricyanide, which immediately converts leucomurexide to murexide, while uramil yields colorless oxidation products. Aeration of a dilute suspension of leucomurexide in water quickly regenerates murexide, 70% (by titration) having been obtained in one experiment after thirty minutes of aeration.

BROOKLYN COLLEGE DAVID DAVIDSON
BROOKLYN, N. Y.

RECEIVED JULY 24, 1936

RHEOPEXY IN BENTONITE

Sir:

Recently during the course of an investigation in the thixotropic properties of the clay mineral bentonite, gels showing pronounced rheopectic properties were observed. These gels are of particular interest inasmuch as Freundlich and Juliusburger in the original article describing rheopexy [*Trans. Faraday Soc.*, **31,** 920 (1935)] state that they were unable to find any evidence of rheopexy in bentonite.

The discovery of rheopexy in bentonite may be attributed to a new technique of fractionation which the authors have developed and described at the thirteenth Colloid Symposium at St. Louis, Mo., in June of 1936. Natural bentonite is dispersed in distilled water and then run through a high speed continuously fed centrifuge of the Sharples type. At various distances up the centrifuge bowl different particle size fractions of bentonite are removed and then refractionated. The finest fractions are a beautiful perfectly transparent golden-yellow color and completely amicronic while the coarser fractions are more opaque. There is a large percentage of amicronic particles present in the Wyoming Bentonite being used. By such a method of preparation the authors are establishing the influence of particle size upon thixotropy and rheopexy in bentonite.

Specifically 10 cc. of a very fine fraction (1.3% bentonite) was placed in a test-tube 1 cm. in diameter and 1 drop of saturated potassium chloride added. The setting time at 23° when the tube was kept perfectly still was twenty-five minutes, whereas slight tapping of the tube on a pad of rubber reduced the setting time to fifteen seconds. Additional potassium chloride makes it so stiff that it cannot be liquefied. Such a gel is perfectly transparent. In addition these fine bentonite fractions show pronounced streaking upon being stirred. This streaking phenomenon is greatly increased after electrodialysis. Altogether the bentonite fractions prepared in this manner act very similarly to the vanadium pentoxide system described by Juliusburger and Pirquet [*Trans. Faraday Soc.*, **32,** 445 (1936)].

From a consideration of the properties of a 1.3% bentonite gel in comparison to those gels of 10–20% concentration described by Freundlich, Schmidt and Lindau [*Kolloidchem. Beih.*, **36,** 43 (1932)], one is forced to the conclusion that particle size is of paramount influence in this case of thixotropy. In fact we have found evidences of gel structure at concentrations as low as 0.01% by wt. in the low colloidal range. It may be stated, however, that if hydrogen bentonite produced by electrodialysis is ever allowed to dry, it fails to exhibit thixotropy in low concentrations. This property can still be observed providing the material is never allowed to dry.

DEPARTMENT OF CHEMICAL ENGINEERING E. A. HAUSER
MASSACHUSETTS INSTITUTE OF TECHNOLOGY C. E. REED
CAMBRIDGE, MASS.

RECEIVED JULY 31, 1936

EXCHANGE BETWEEN SODIUM IODIDE AND ETHYL IODIDE*

Sir:

It was reported a few months ago in these columns [Hull, Schiflett and Lind, THIS JOURNAL,

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* This work was supported in part by the Fluid Research Fund of the Graduate School.

58, 535 (1936)] that while there is no exchange between radioactive iodine and ethyl iodide when heated together for fifteen minutes at 87°, as determined by the subsequent measurement of the activities of the two substances, yet in alcoholic solution at 100° a ready exchange takes place between sodium iodide and ethyl iodide. Further experiments on this latter reaction have shown the existence of a marked temperature effect.

The general procedure in these experiments has been as follows: A solution of 50 micromoles of iodine in a liter of iodobenzene is irradiated with the neutrons from a Rn–Be source for a period of one and one-half hours or longer. After removal of the neutron source, the iodine is extracted and precipitated as silver iodide, which is then transformed into dry sodium radioiodide. This is dissolved in 1–2 cc. of ethyl alcohol and mixed with 100 micro-moles of inactive ethyl iodide. This mixture is heated to the desired temperature for a stated interval of time and then quickly cooled. (In the one experiment carried out below room temperature both solutions were cooled before being mixed.) The two iodides are separated by shaking the solution with excess ether and water, and are transformed into silver iodide for measurement. The activity of each precipitate is measured by means of a tube counter. The results obtained in a series of experiments at different temperatures are given in tabular form.

Temp., °C.	Time, min.	Exchange
100	5	Complete
100	1	Complete
80	5	Complete
29	5	Partial
29	1	None
24	5	None
19	5	None

The authors are grateful to Dr. K. W. Stenstrom of the University Hospital for the gift of 150 millicuries of radon, and to Dr. J. H. Williams of the Department of Physics for the use of a low resistance amplifier [of the type described by Neher and Harper, *Phys. Rev.*, **49**, 940 (1936)], without which it would not have been possible to carry on this work during the recent hot weather.

SCHOOL OF CHEMISTRY
UNIVERSITY OF MINNESOTA
MINNEAPOLIS, MINN.
D. E. HULL**
C. H. SCHIFLETT
S. C. LIND
** National Research Fellow.

RECEIVED AUGUST 22, 1936

THE PHOTOCHEMICAL DECOMPOSITION OF METHANE

Sir:

Methane at atmospheric pressure is photochemically decomposed when irradiated with the light which is transmitted by a thin fluorite window from a hydrogen discharge tube. The effective radiation would appear to lie near the limit of transmission of fluorite. Although no direct measurements of quantum yield have been made, a crude estimate based on a comparison with the rate of the ozone synthesis in the same cell indicates a value of the order of unity.

Among the products we have identified hydrogen and unsaturated hydrocarbons (probably ethylene) in the approximate molecular ratio of 4:1. Higher saturated hydrocarbons may also be produced but have not been detected. The large ratio of hydrogen to unsaturation in the products indicates that the decomposition

$$CH_4 + h\nu \longrightarrow CH_2 + H_2$$
$$2CH_2 \longrightarrow C_2H_4$$

cannot be the only process involved.

DEPARTMENT OF CHEMISTRY
STANFORD UNIVERSITY, CALIF.
PHILIP A. LEIGHTON
ARNOLD B. STEINER

RECEIVED JULY 28, 1936

NEW SYNTHESIS OF GLYCOFURANOSIDES

Sir:

In the last decade considerable progress in sugar chemistry had been made from the study of the glycopyranosides. Further developments can likely be expected from a greater than our present knowledge of the glycofuranosides, or, as E. Fischer originally named them, γ-glycosides. The physical, chemical and biological properties, the conversion into the pyranosides under certain conditions, and the chemical structure in general, of these γ-glycosides should be thoroughly investigated. To be sure, such studies have already been made in the past, in a few isolated instances. However, a review of the literature reveals that only five or six γ-glycosides are known in pure crystalline form obtained mostly in small yields by cumbersome methods. The majority of the γ-glycosides described in the literature were prepared by the original method of E. Fischer [*Ber.*, **28**, 1145 (1895); **47**, 1980 (1914)] yielding only sirupy mixtures that could not be separated. Obviously, a general method which leads to well-defined, crystalline products, would constitute the first step toward a more extensive study of this class of sugar derivatives. We believe that we have found such a method. It represents a modification of a previous general procedure of one of us [Pacsu, *ibid.*, **58,** 509 (1925); Pacsu and Ticharich, *ibid.*, **62,** 3008 (1929)] by which the α-alkylpyranosides were prepared from the sugar mercaptals and mercuric chloride in the *boiling* solutions of the different alcohols. Taking, *e. g.*, galactosediethylmercaptal and ethanol, the reaction can be expressed by the following summary equation

$$C_6H_{12}O_5{\cdot}(SC_2H_5)_2 + 2HgCl_2 + C_2H_5OH = 2HCl + 2C_2H_5S{\cdot}HgCl + C_6H_{11}O_5{\cdot}OC_2H_5$$

This method was shown to yield almost pure (up to 92%) α-alkylpyranosides accompanied only by a slight quantity of the β-isomers. Now we have found that if the same reaction be carried out *at low temperature*, and the hydrochloric acid formed be neutralized at the moment of its development, an excellent yield of crystalline β-ethylgalactofuranoside can be obtained. The substance had m. p. 84.5–86° and $[\alpha]^{25}\text{D}$ $-100.1°$ in water solution. Schlubach and Meisenheimer [*ibid.*, **67,** 429 (1934)] reported m. p. 86° (corr.) and $[\alpha]^{20}\text{D}$ $-97.2°$ for the same compound obtained by a lengthy synthesis from the β-pentaacetate of galactofuranose of Hudson and Johnson. So far it has not been possible to find any isomer in the mother liquor. The same furanoside was obtained when dibenzylmercaptal had been used as starting material. For the instantaneous neutralization of the hydrochloric acid we have found the use of an excess of yellow mercuric oxide to be most excellent. It keeps the reaction mixture neutral during the whole process, and it provides for a desirable excess of the mercuric chloride up to the end of the reaction. For the removal of the mercuric chloride from the solution we have found that pyridine can be used most successfully. It seems very likely that these modifications can also be used with advantage in the procedures developed for the preparation of the open-chain methylated (Levene, 1926) and acetylated (Wolfrom, 1929) aldoses.

Since the formation of glycopyranosides by the original method has proved to be of general validity, we believe that by this modified procedure it will be possible to prepare the hitherto unknown or only difficultly available furanosides of the carbohydrates. It is our purpose to prepare by this method the alkylfuranosides and -thiofuranosides of the aldoses and ketoses from their mercaptals and acetonylated mercaptals. In the forthcoming first paper of this series of investigations conducted in this Laboratory, a detailed description of the procedure will be given.

FRICK CHEMICAL LABORATORY
PRINCETON UNIVERSITY
PRINCETON, NEW JERSEY

EUGENE PACSU
JOHN W. GREEN

RECEIVED AUGUST 14, 1936

NEW BOOKS

Colorimetric Methods of Analysis, Including Some Turbidimetric and Nephelometric Methods. Vol. I. Inorganic. By FOSTER DEE SNELL and CORNELIA T. SNELL. D. Van Nostrand Company, Inc., 250 Fourth Avenue, New York, N. Y., 1936. 766 pp. Illustrated.

In this second edition of Dr. Snell's treatise a much more comprehensive treatment of the field of colorimetry has been made. The book has been divided. This first volume covers general colorimetry and inorganic determinations; a second will deal with organic, biological and miscellaneous methods.

In its general plan and scope the book closely resembles Yoe's "Photometric Analysis." It differs in that nephelometric methods are treated with the colorimetric ones instead of in a separate volume and in that the bibliography occurs in footnotes instead of as a separate section at the end of the book. It contains, of course, methods and references which have appeared in the seven years since the publication of Yoe's book.

In the first nine chapters the general methods of colorimetry are discussed and the multitudinous forms of apparatus which have been devised for the measurement of color and of turbidity are illustrated and explained. Fifty-four chapters are then devoted to different methods for the determination of seventy-four elements or simple inorganic compounds. The last four chapters deal with