steric hindrance. The test is negative for all compounds which contain one of the requisite groupings joined to an aryl radical carrying two ortho substituents. As a matter of fact, the reaction is slow, even with pinacolone. The question as to what is actually formed in the case of hindered methyl ketones is under investigation. In no case is the compound recovered unchanged.

Summary

By the use of dioxane as solvent an iodoform test has been developed which can be used with water-insoluble compounds as well as with those which are soluble in water.

On the basis of results with a large number of compounds a new rule has been formulated concerning the generality of the test.

URBANA, ILLINOIS RECEIVED MAY 21, 1934

NOTES

Pecan Shells as a Source of *d*-Xylose

BY CLIFFORD J. B. THOR AND C. L. SMITH

It has already been pointed out[1] that the alcohol-insoluble residue from samples of pecan shells yielded more than 20% of its dry weight of reducing sugar when subjected to the official method for determining "starch" by direct acid hydrolysis.[2] More recently we have succeeded in isolating crystalline *d*-xylose from such sirups with yields of 11.2 to 12.8% of the untreated air-dry shells.

Our procedure was to sift with cheesecloth and extract with either hot or cold water, hydrolyzing the extracted shell material with about four times its weight of normal sulfuric acid for periods of six to eight hours. The isolation of xylose from the sirups follows essentially the customary methods, with crystallization from ethyl alcohol. The mother liquors gave strong xylose reactions and also yielded some saccharic acid, indicating the presence of glucose, but the amount probably was not great since baker's yeast produced no visible fermentation at a sugar concentration of about 10%. Tests for the other commonly occurring monosaccharides were negative.

A decided advantage of pecan shells as compared with corn cobs or cottonseed hulls is their relative compactness (bulk density about 0.5) and the fact that they can be easily and quickly washed after acid hydrolysis.

BUREAU OF PLANT INDUSTRY
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(1) C. J. B. Thor and C. L. Smith, to appear in *J. Agr. Research.*

(2) "Association of Official Agricultural Chemists, Official and Tentative Methods of Analysis," 2d ed., revised to July 1, 1924 (1925), p. 119 (21).

Some Strychnine Benzoates

BY CHARLES F. POE AND JOHN F. SUCHY

During the course of a series of toxicological and pharmacological experiments, it became necessary to prepare a number of strychnine salts. These were made by the union of strychnine with benzoic acid and the various substituted benzoic acids. Of the salts described in this paper, the benzoate and the salicylate are the only ones mentioned in the literature.

The purest chemicals obtainable were used. Many of these were recrystallized several times, and the melting points were checked in each case. Molecular quantities of strychnine and the various benzoic acids were separately dissolved in alcohol with the aid of heat. After solution the alkaloid and acid were mixed and boiled for about ten minutes. The salt usually crystallized out upon cooling. A number of the benzoic acids, especially those containing two halogen or nitro groups, were rather insoluble in alcohol. In these cases chloroform was used to dissolve the acid, and after mixture with the alcoholic solution of strychnine, the combined solution was allowed to boil until most of the chloroform had volatilized. Each salt was recrystallized until pure. The strychnine content of the salts was determined by the method given in the U. S. Pharmacopoeia,[1] and in cases where the acid radical was not too insoluble, the picrate method of Elmore[2] was also used. The nitrogen was determined by the official Kjeldahl method[3] to include nitrogen of nitrates. The melting points were determined by the usual capillary tube method in conjunction with the Thiele apparatus. Check determinations were made by the "bloc Maquenne" method. With the Thiele apparatus the melting points were very unsatisfactory. Decomposition took place in many cases and no sharp melting point could be obtained. It is well known that the melting point of strychnine varies under different conditions and it is impossible to get a

(1) U. S. Pharmacopoeia, Tenth Revision, 1926.

(2) Elmore, *J. Assoc. Off. Agr. Chem.*, **9**, 224 (1926).

(3) "Official and Tentative Methods of Analysis," Assoc. Official Agric. Chem., 1930, 3d edition, p. 21.

TABLE I

SALTS PREPARED FROM STRYCHNINE AND VARIOUS BENZOIC ACIDS

Benzoate	Formula $C_{21}H_{22}O_2N_2$ +	Color	Strychnine, % Calcd.	Strychnine, % Found	Nitrogen, % Calcd.	Nitrogen, % Found	M. p., °C. (uncorr.), "Bloc Maquenne"
	C_6H_5COOH	White	73.26	73.20	6.14	6.18	231
o-Chloro-	$C_6H_4ClCOOH$	White	68.11	67.88	5.70	5.68	170
m-Chloro-	$C_6H_4ClCOOH$	White	68.11	67.89	5.70	5.60	185
p-Chloro-	$C_6H_4ClCOOH$	White	68.11	67.90	5.70	5.60	251
o-Bromo-	$C_6H_4BrCOOH$	White	62.49	62.72	5.24	5.51	202
m-Bromo-	$C_6H_4BrCOOH$	White	62.49	62.80	5.24	5.21	178
p-Bromo-	$C_6H_4BrCOOH$	White	62.49	62.62	5.24	5.06	252
o-Iodo-	C_6H_4ICOOH	White	57.41	57.70	4.81	4.74	237
m-Iodo-	C_6H_4ICOOH	White	57.41	57.49	4.81	4.86	159
p-Iodo-	C_6H_4ICOOH	Brown	57.41	57.48	4.81	4.82	241
o-Nitro-	$C_6H_4NO_2COOH$	Yellowish	66.65	66.20	8.38	8.40	205
m-Nitro-	$C_6H_4NO_2COOH$	Yellowish	66.65	66.67	8.38	8.16	214
p-Nitro-	$C_6H_4NO_2COOH$	Yellowish	66.65	66.83	8.38	8.23	266
o-Hydroxy-	$C_6H_4OHCOOH$	White	70.77	70.68	5.94	5.91	224
m-Hydroxy-	$C_6H_4OHCOOH$	White	70.77	70.33	5.94	5.99	263
p-Hydroxy-	$C_6H_4OHCOOH$	White	70.77	71.01	5.94	5.93	192
o-Methyl-	$C_6H_4CH_3COOH$	White	71.10	71.05	5.96	5.80	167
m-Methyl-	$C_6H_4CH_3COOH$	White	71.10	70.85	5.96	5.99	168
p-Methyl-	$C_6H_4CH_3COOH$	White	71.10	71.10	5.96	5.96	236
o-Amino-	$C_6H_4NH_2COOH$	Cream	70.92	70.90	8.92	8.87	219
m-Amino-	$C_6H_4NH_2COOH$	Cream	70.92	70.90	8.92	8.88	233
p-Amino-	$C_6H_4NH_2COOH$	Cream	70.92	70.45	8.92	8.87	206
3,5-Dinitro-	$C_6H_3(NO_2)_2COOH$	Yellowish	61.18	61.35	10.26	10.27	267
2,4-Dinitro-	$C_6H_3(NO_2)_2COOH$	Yellowish	61.18	61.45	10.26	10.17	256
2,4,6-Trinitro-	$C_6H_2(NO_2)_3COOH$	Buff	56.55	56.90	11.83	11.71	182 dec.
Salicylate							
5-Iodo-	$C_6H_3OHICOOH$	White	55.87	55.75	4.68	4.68	225
3,5-Dinitro-	$C_6H_2OH(NO_2)_2COOH$	Yellow	59.44	59.47	9.96	9.73	274
Di-iodo-	$C_6H_2OHI_2COOH$	White	46.15	45.95	3.87	3.98	225
Acetyl-	$C_6H_4CH_3CO_2COOH$	White	65.00	65.15	5.45	5.69	142

sharp, definite melting point. This seems to be true also of the benzoate salts of strychnine.

The salts are not very soluble in water, the *o*-chloro salt being the most soluble. The salts made from the acids containing more than one substituted group are almost insoluble in water even at higher temperatures. All of the salts are quite soluble in chloroform, fairly soluble in alcohol and slightly soluble in ether.

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Note on a New Method for the Preparation of Acyclic Unsaturated Hydrocarbons

BY A. L. WARD AND W. H. FULWEILER

In a laboratory investigation requiring the preparation of fairly large quantities of olefinic hydrocarbons, a new method for the removal of a molecule of hydrochloric acid from chloroparaffins was found to be superior to older methods, particularly when used for the preparation of olefins of more than eight carbon atoms.

The method which is similar to one used[1] commercially for the production of terpenic alcohols consists essentially in the treatment of the halogen compound with an alkali phenolate. It has been known that tertiary alkyl iodides,[2] when heated with sodium phenolate in alcoholic solution, give olefins as a by-product of the principal reaction, which is the formation of ethers. By the technique used here only small quantities of ethers were formed from many alkyl halides. The details of the method may be best illustrated by an actual example.

Preparation of Decylene

A quantity of synthetic 2,7-dimethyloctane prepared by the Wurtz reaction was chlorinated. The chlorinated product was fractionated under reduced pressure. The monochloro compound had the constants: b. range 81–83° at 3 mm.; density at 20°, 0.8642; chlorine, by analysis 19.73%, theoretical 20.07%. Potassium cresolate was prepared by treating cresol with 66% of the theo-

(1) Andreau, U. S. Patent 1,478,690 (1923).
(2) Segalier, *J. Chem. Soc.*, **103**, 1154, 1421 (1913).