

To the ester (50 g.) was added 5 g. of catalyst and the mixture placed under a pressure of 1840 lb. of hydrogen. The temperature was then slowly raised. Hydrogenation of the double bond took place at a pressure of 2550 lb. and a temperature of 146°. If the hydrogenation were interrupted at this point, an approximately quantitative yield (98%) of ethyl 2,3-dimethoxydihydrocinnamate, b. p. 174–176° at 13 mm., could be obtained.

Anal. Calcd. for $C_{15}H_{18}O_4$: C, 65.6; H, 7.57. Found: C, 65.6; H, 7.55.

At a pressure of 2680 lb. and a temperature of 224°, further hydrogen was consumed and the principal product weighing 34 g. (86%), distilled at 160–165° at 13 mm. The product failed to crystallize. For identification, a phenylurethan was prepared. This compound melted at 62.7–63.2° after crystallization from petroleum ether.

Anal. Calcd. for $C_{18}H_{21}O_4N$: C, 68.6; H, 6.70. Found: C, 68.4; H, 6.67.

2,3-Dimethoxydihydrocinnamyl Chloride and Bromide.—The halides corresponding to 2,3-dimethoxydihydrocinnamyl alcohol were prepared by the action of thionyl chloride or hydrogen chloride, and hydrogen bromide or 48% hydrobromic acid. They were separated and subjected to a variety of demethylation procedures without further identification. The dihydric phenols could not be isolated; resins were frequent products. Nor was it found possible to prepare by demethylation the 2,3-dihydroxybenzyl halides. However, during sealed tube demethylations in the presence of concentrated hydrobromic and hydriodic acids, catechol itself was formed and isolated in yields up to 18% from these 3-substituted catechol ethers. This has also been encountered during acid demethylation of 2,3-dimethoxy-*n*-pentadecylbenzene,¹ and by Haworth¹² during the acid demethylations of 3- and 4-substituted catechol ethers. Lability of alkyl substituents in the veratrole molecule under these conditions is thus indicated.

Acknowledgment.—I am indebted to Dr. Arthur T. Ness and to Mr. Charles A. Kinser for the microchemical analyses.

(12) Haworth and Woodcock, *J. Chem. Soc.*, 999 (1947).

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New Sources for Sapogenins

BY RUSSELL E. MARKER, R. B. WAGNER, PAUL R. UL-SHAFAER, EMERSON L. WITTBECKER, DALE P. J. GOLDSMITH AND CLARENCE H. RUOF

The isolations of various steroidal sapogenins from many plant sources have been reported previously from this Laboratory.^{1,2,3} The results of our studies on additional plants are now summarized.

Among the new sources for steroidal sapogenins is the seed of *Trigonella Foenum-graecum* L. (Foenugreek). For their isolation 460 kg. of seeds were processed. There has been isolated

(1) Marker, Turner and Ulshafer, *THIS JOURNAL*, **62**, 2542 (1940).

(2) Marker, Wagner and Ulshafer, *ibid.*, **64**, 1283 (1942).

(3) (a) Marker, Wagner, Ulshafer, Wittbecker, Goldsmith and Ruof, *ibid.*, **65**, 1199 (1943); (b) **69**, 2167 (1947); (c) for supplementary tables, order Document 2384 from American Documentation Institute, 1719 N Street, N. W., Washington 6, D. C., remitting 50¢ for microfilm or \$2.10 for photocopies.

from the mother liquor after the separation of diosgenin (yield, about 1.0 g./kg. dry seed),⁴ two more sapogenins, namely, tigogenin (trace) and gito-genin (yield, 0.1 g./kg. dry seed). The last two sapogenins have occurred jointly in other plants, namely, *Yucca Whipplei* Torr. subsps. *intermedia*, *Agave gracilipes* Trel. and *Agave Schottii* Engelm. However, this is the first and single case of the occurrence of all three in the same plant. The significance of this finding has been discussed.^{3b}

Lilagenin has been isolated from the sapogenin fraction of *Lilium rubrum magnificum*.⁵ In addition, a small amount of yuccagenin was found.

In our preliminary paper,^{3a} we erroneously reported *Samuela Faxoniana* Trel. to be a source for smilagenin. Actually, it is a new source for sarsa-sapogenin.

Other new sources are listed in the accompanying tables.

TABLE I

PLANTS CONTAINING DIOSGENIN AND KRYPTOGENIN		
Plant	Location	Yield g. per kg. (dry) plant Dios. Krypt.
<i>Balanites aegyptica</i> Wall.	Southern Mexico	5.0 1.0
<i>Dioscorea floridiana</i> Bartlett	Southern Georgia	1.7 ...
<i>Dioscorea glauca</i> Muhl.	North Carolina	1.0 ...
<i>Trillium Catesbaei</i> Ell.	North Carolina	... 0.1
<i>Trillium cernuum</i> L.	North Carolina	... 1.0
<i>Trillium decumbens</i> Harbison	North Carolina	... 0.5
<i>Trillium declinatum</i> Gleason	Tennessee	5.0 1.0
<i>Trillium erectum</i> L.	North Carolina	3.0 0.2
<i>Trillium Hugerii</i> Small	North Carolina	3.0 ...
<i>Trillium ludovicianum</i> Harbison	Georgia	5.0 ...
<i>Trillium recurvatum</i> Beck	Mississippi	4.0 Trace
<i>Trillium simile</i> Gleason	North Carolina	4.0 ...
<i>Trillium stamineum</i> Harbison	Georgia	... 0.8
<i>Trillium Vaseyi</i> Harbison	North Carolina	0.4 ...
<i>Trillium viride</i> Beck	North Carolina	... 0.5

PLANTS CONTAINING SITOSTEROL		
Plant	Location	Yield, g. per kg. dry plant
<i>Areca Catechu</i> L.	Commercial	Trace
<i>Arisaema triphyllum</i> Schott	Commercial	0.5
<i>Jatropha palmata</i> Miers	Commercial	Trace
<i>Smilacina racemosa</i> Desf.	State College, Pa.	Trace
<i>Zanthoxylum apiifolia</i> L'Hérit	Commercial	Trace

The identities of the above compounds were established by analyses of the genins and their acetates along with melting point and mixed melting point determinations on both. Generalized isolation procedures have been reported.^{3b}

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(4) Marker, Wagner, Ulshafer, Goldsmith and Ruof, *ibid.*, **65**, 1247 (1943).

(5) Marker, Turner, Shabica, Jones, Krueger and Surmatis, *ibid.*, **62**, 2620 (1940).

(6) Original manuscript received June 26, 1944.

The Use of a Fluorescent Adsorbent for the Chromatography of Colorless Compounds

BY JOHN W. SEASE

When colorless compounds which absorb ultraviolet light are chromatographed on a fluorescent