Acknowledgments.—The authors gratefully acknowledge the kind financial assistance accorded them by the Spruce Falls Power and Paper Company and the Canadian Pulp and Paper Association.

Summary

1. High-pressure, catalytic (copper chromite) hydrogenation of maple ethanol lignin yields water, methanol, ethanol, 4-*n*-propylcyclohexanol, 4-*n*-propylcyclohexanediol-1,2, 3-(4-hydroxycyclohexyl)-propanol-1, an apparently new, unidentified substance, and high-boiling resins in yields of 13.6, 5.0, 8.7, 8.1, 1.9, 3.3, 2.1 and 29.5%, respectively (referred to the weight of the ethanol lignin used). The variation in yield, especially of 3-(4-hydroxycyclohexyl)-propanol-1, as compared with the results obtained by Harris, D'Ianni and Adkins[5] using *aspen methanol* lignin suggests that a difference may exist in the chemical structure of the two types of lignin.

2. Examination of the yields of hydrogenation products from various amorphous fractions of a maple lignin, isolated by ethanolysis, indicates that with increasing solubility of a given lignin fraction and with its increasing susceptibility to depolymerization to simple propylphenol units by ethanolysis there is obtained an increasing yield of water-insoluble, distillable hydrogenation products.

3. This observation is thought to indicate the prevalence of –C–O–C– bonds between the C_6C_3 "lignin building units" in those lignin fractions which are easily soluble and cleavable by ethanolysis or hydrogenolysis into simple units; and, on the other hand, an increasing incidence of –C–C–C– bonds in lignin fractions having these characteristics to a lesser extent.

4. Considering the mechanism of the ethanolysis reaction, the extent of occurrence of –C–O–C– bonds between lignin units as compared to –C–C–C– bonds is believed to determine the degree to which polymerization–depolymerization changes can take place, and, in addition, it is suggested that the –C–O–C– linkage involved is possibly of the acetal (ketal) type.

MONTREAL, CANADA RECEIVED AUGUST 25, 1941

[CONTRIBUTION FROM THE DIVISION OF INDUSTRIAL AND CELLULOSE CHEMISTRY, McGILL UNIVERSITY]

Studies on Lignin and Related Compounds. LXII. High Pressure Hydrogenation of Wood Using Copper Chromite Catalyst (Part 1)

BY HUGH P. GODARD,[1] JOSEPH L. McCARTHY AND HAROLD HIBBERT

The isolation of propylcyclohexanol derivatives by means of the hydrogenation of isolated lignins[2] has provided valuable evidence that propylphenol derivatives are the units from which lignin is derived.[3]

This evidence would be stronger if a higher proportion of the protolignin could be converted into identifiable substances. There was reason to believe this might be accomplished by the high pressure catalytic hydrogenation of wood[4] instead of isolated lignins,[2] since protolignin in wood presumably has a relatively simple structure and should thus give larger amounts of simpler hydrogenated products; and also because wood contains *all* of the protolignin, whereas isolated lignins, obtained by mild alcoholic processes, represent only a fraction of this.[5] An investigation of the hydrogenation of wood was undertaken with the object, after separating the hydrogenated carbohydrates by suitable methods, of isolating and identifying lignin hydrogenation and hydrogenolysis products in higher yields.

In preliminary experiments, various samples of spruce and maple wood, suspended in dioxane, were hydrogenated using a copper chromite catalyst, an initial hydrogenation pressure of about 3500 lb./sq. in., and an average temperature of about 280°. The hydrogen reacted quite rapidly at first, then progressively more slowly until the absorption became complete, usually within ten to twenty hours, depending upon the

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[1] This paper is part of a thesis submitted to the Faculty of Graduate Studies, McGill University, by Hugh P. Godard in partial fulfillment of the requirements for the Degree of Doctor of Philosophy, May, 1941.

[2] (a) Harris, D'Ianni and Adkins, THIS JOURNAL, **60**, 1467 (1938); (b) Harris, Saeman and Sherrard, *Ind. Eng. Chem.*, **32**, 440 (1940); (c) Cooke, McCarthy and Hibbert, THIS JOURNAL, **63**, 3056 (1941).

[3] For comprehensive review of the chemistry of lignin see Hibbert, *Paper Trade J.*, **113**, No. 4, 35 (1941).

[4] Godard, McCarthy and Hibbert, THIS JOURNAL, **62**, 988 (1940).

[5] Brickman, Pyle, McCarthy and Hibbert, *ibid.*, **61**, 868 (1939)

TABLE I

WOODMEAL HYDROGENATION AND PRELIMINARY FRACTIONATION DATA FROM "FRACTIONATION PROCEDURE A"

		Hydrogenating conditions[c]								Total[g]	
Expt.[a]	Wood species and prepn.[b]	Water, g.[d]	Time, hr.	H_2 abs. (moles per 100 g. wood)	Fraction A n_D up to 1.4600^{25} Kl,[f] %	Fraction B n_D 1.4600^{25} to 1.4700^{25} Kl, %	Fraction C n_D 1.4700^{25} to 1.4800^{25} Kl,[f] %	Fraction D n_D 1.4800^{25} to 1.4800^{60} Kl,[f] %	Fraction E n_D 1.4800^{60} Resin Kl,[f] %	Wt., g.	Kl,[f] %
1	ME	2.0	12.5	3.16	47.8	26.3	26.3	8.4	24.2	6.97	133.0
2	ME	3.0	15.5	3.28	22.5	37.3	20.0	5.7	13.7	5.21	99.2
3	MEA	3.0	17	3.32	14.5	32.6	10.4	4.0	9.7	3.74	71.2
4	MEA	3.0	12	3.24	19.6	43.5	21.9	6.5	19.1	5.80	110.6
5	SEA	3.0	15	3.36	55.3	22.3	20.7	...	43.2	6.36	141.5
6	ME	3.0	15	3.20	41.8	30.6	28.0	6.5	47.1	6.98	159.0
7	ME	42.0	7.5	2.28	86.5	11.2	31.2	...	23.2	7.32	152.1
8	ME	10.0	12.5	2.72	39.8	37.6	22.8	...	23.4	6.23	123.6
9[e]	ME	4.1	19.5	3.13							
10[e]	ME	4.1	20.5	3.08							
11[e]	ME	4.1	20.5	3.06							
12[e]	ME	4.1	20	3.13							

[a] Expts. 1–8, 25-g. samples of wood; expts. 9–12, 175-g. samples. [b] "M" = maple, "S" = spruce, "E" = solvent-extracted, "A" = alkali-extracted. [c] Initial hydrogen pressure of 3500 lb./sq. in.; reaction temp. of 280°; dry dioxane solvent. [d] Except for expts. 7 and 8 (water added) the water content was that present in the woodmeal. [e] Hydrogenation products fractionated according to "Procedure B." [f] Original lignin (Klason) in wood. [g] In Expts. 9, 10, 11, 12, there was an insoluble wood residue amounting to 6.3, 6.0, 4.5, 9.6%, respectively.

particular sample of catalyst used. No great difference in the rate of hydrogenation was evident between spruce and maple, or between non-alkali- and alkali-extracted (25°) wood, but with substantial amounts of water present both the rate and amount of hydrogenation were lowered considerably.

After removal of the suspended catalyst, the hydrogenated reaction mixtures were colorless or faintly-yellow liquids, all of the initially suspended wood having been converted into dioxane-soluble products and gases. The solvent and low-boiling products were removed and the remaining liquid was fractionated by distillation using "Fractionation Procedure A" (Experimental Part) into five portions boiling over increasing temperature ranges (Table I). Since the refractive indices of the products could be measured readily and accurately, and since it was found that, in general, this property of the distillates increased with the boiling point, the several fractions are grouped for comparative purposes in Table I on the basis of refractive index measurements.

It is apparent that the nature and yield of the wood hydrogenation products from these preliminary experiments are conditioned by the species, pretreatment, and water content of the hydrogenation medium as well as by the specific catalyst preparation used. Fraction B (Table I) (assumed to be largely 4-*n*-propylcyclohexanol, $n^{25}D$ 1.4650[6]) tends to increase somewhat at the expense of the other fractions when either maple or spruce wood is subjected to a preliminary alkali extraction (Expts. 1 to 4, 5 to 6 in Table I). On the other hand, the amount of lower-boiling products, Fraction A, is doubled and the amount of Fraction B is reduced to about one-third when considerable water (42%) is present in the dioxane medium during hydrogenation (Expts. 1 and 7, Table I), although a moderate amount of water (10%) does not materially alter the results from those obtained in nearly anhydrous media (Expts. 1 and 8, Table I). The residual hydrogenated resin from spruce is much greater (up to 200%) than that from maple wood.

In order to obtain the reaction products in amounts sufficient to permit of a detailed study, four larger batches of solvent-extracted maple woodmeal were hydrogenated (Table I, Expts. 9–12) and the combined products separated according to the scheme illustrated in the Flow Sheet ("Fractionation Procedure (B)").

After separation of the catalyst and a small amount of unhydrogenated woodmeal, the solvent and low-boiling products were removed by distillation. Subsequent refractionation of this low-boiling liquid was then carried out in a Fenske column.[7]

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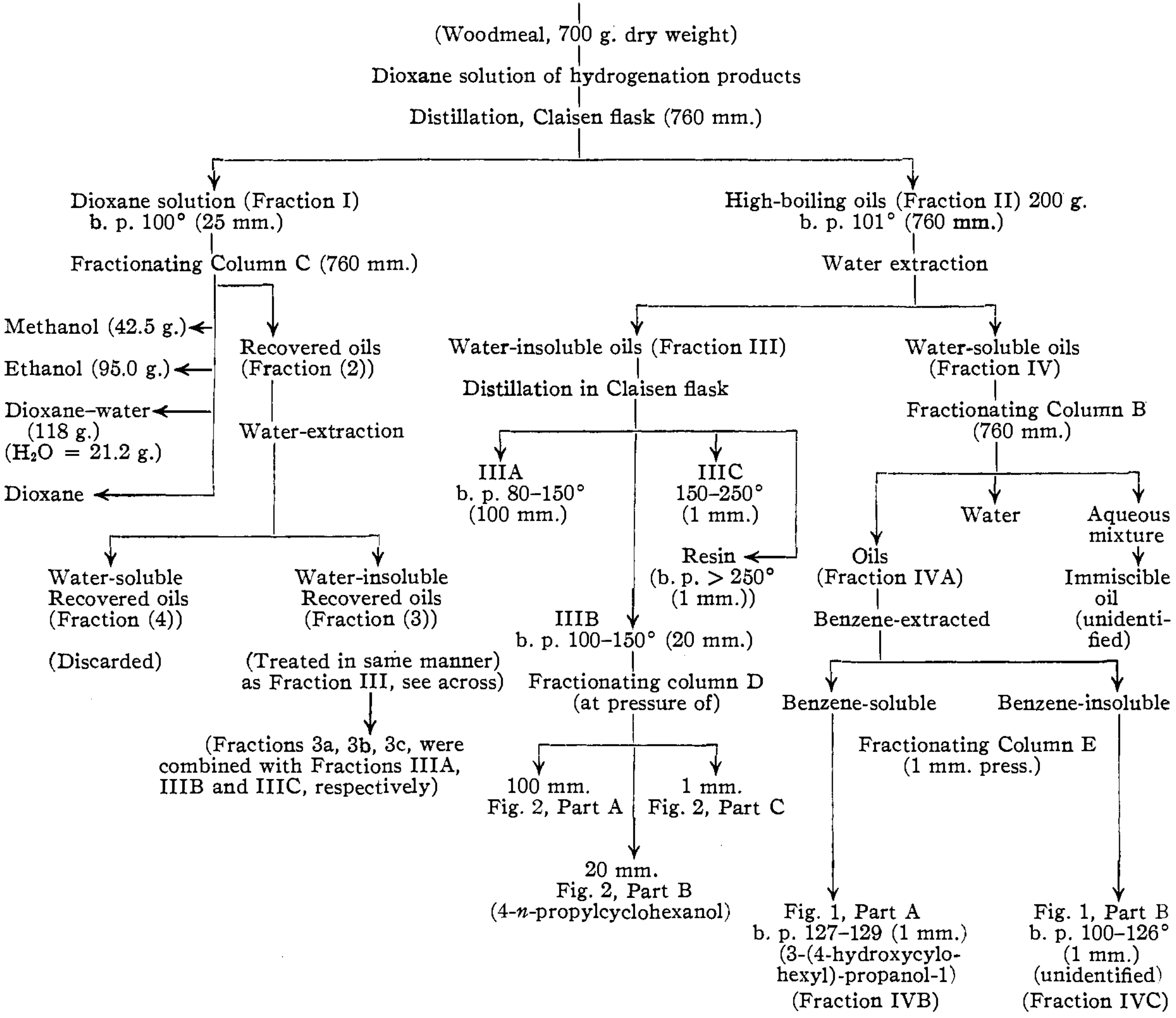
[6] The nature and yields of the wood hydrogenation products, previously reported[4] by the authors, were erroneously interpreted on a basis of the refractive indices of the fractions shown in Table I. The unfortunate inaccuracy of these results, as is evident from the detailed subsequent investigation herein reported, emphasizes the need for caution in characterization based only on physical properties and the essential importance of *chemical* criteria of identity and purity in the investigation of difficultly separable liquid mixtures.

[7] (a) Fenske, Quiggle and Tongberg, *Ind. Eng. Chem.*, **26**, 1213 (1934); (b) Fractionation by J. R. Bower, Jr.

FLOW SHEET

MAPLE WOODMEAL HYDROGENATION "FRACTIONATION PROCEDURE B"



Separation of the dioxane–water azeotrope (b. p. 87.4°, 18% water)[2c] by this fractionation procedure showed the presence of 118 g. representing 21.2 g. of water. Since 4.1 g. of water was already present in the initial wood, at least 18.1 g. of water per 100 g. of wood was formed in the course of the hydrogenation. The presence of methanol and of ethanol is shown by the refractive indices and boiling points tabulated for these in the distillation data, and by chemical methods (see Experimental Part). The amounts of these substances have been found to be 6.3 g. and 14.0 g. per 100 g. of maple wood, respectively.

Since in preliminary work the cellulose hydrogenation products were found considerably more soluble in water than those from lignin,[8] the residual higher-boiling reaction products (Fraction II) were divided into water-*insoluble* (Fraction III) and water-*soluble* (Fraction IV) portions (see Flow Sheet), and the latter concentrated to a small volume by distillation through the Fenske column. A small portion distilling below 100° (760 mm.) and another small portion separating from the aqueous distillate have not, as yet, been identified. The remaining higher-boiling, water-soluble products were extracted with benzene and a large "water-soluble, benzene-soluble" fraction (IVB) isolated, as well as a "water-soluble, benzene-insoluble" fraction (IVC). The former, on fractional distillation (Fig. 1), gave a constant-boiling fraction (b. p. 128–129 (1 mm.); $n^{25}\mathrm{D}$ 1.4865; $n^{54}\mathrm{D}$ 1.4790; Fig. 1A) identified as 3-(4-hydroxycyclohexyl)-propanol-1; the yield was

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(8) Godard, McCarthy and Hibbert, unpublished results.

1.29% of the maple wood or 5.8% of its lignin (Klason) content. Possibly the "water-soluble, benzene-insoluble" fraction (Flow Sheet, Fig. 1B) may have arisen only because the benzene extraction was incomplete. The resin yield was small. The total yield of the water-soluble fraction (IV) amounted to 7.37% of the weight of the original wood, 2.11% being low boiling, 3.16% high-boiling and benzene-soluble, and 2.10% high-boiling and benzene-insoluble. A repetition of the work is expected to give higher yields.

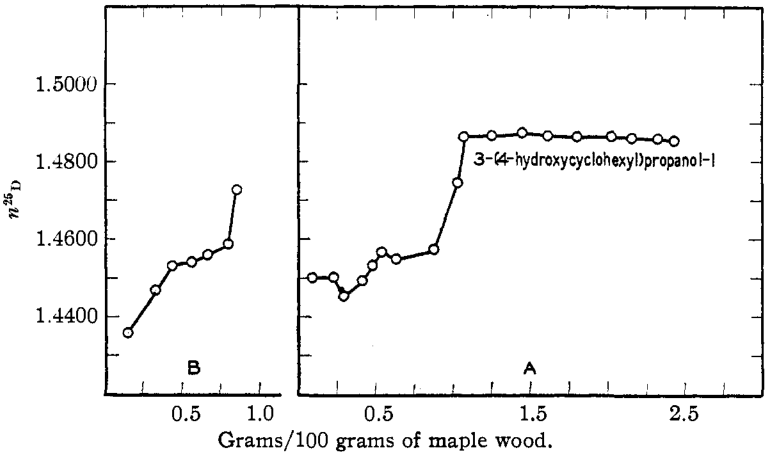

Fig. 1.—Fractional distillation of water-soluble hydrogenation products from maple wood: A, benzene-soluble; B, benzene-insoluble.

The "water-insoluble" products (Flow Sheet, Fraction III) were fractionated in three batches, first from a Claisen flask and then through fractionating column D, to facilitate handling of the material, and because it was desirable to minimize the time of heating and thus to reduce the formation of resin during the distillation. The fractionation curve (Fig. 2B) indicated the presence of considerable amounts of a material of relatively constant refractive index in a yield of 4.4% of the maple wood or 19.5% of the lignin (Klason) therein. The substance was identified as 4-*n*-propylcyclohexanol by careful refractionation as well as by preparation of the crystalline phenyl urethan derivative. The lower-boiling fraction (b. p. 100–150° (100 mm.)) shown in Fig. 2A is now being investigated further. The high-boiling fraction (Fig. 2C; b. p. 100–160° (1 mm.)) has not yet been investigated. The resin (b. p. > 160° (1 mm.)) from this fraction amounted to 3.6% of the wood or a maximum of 16.1% of the lignin (Klason). Seventy-five per cent. of the resin was obtained in the preliminary distillations and the remaining 25% isolated in the fractionation procedure, the major part of the latter being formed from the fraction boiling at the highest temperature. Aside from a small amount collected in cold traps in the vacuum system the remainder of the oils (about 16% of wood weight) is accounted for in the data graphed in Fig. 2.

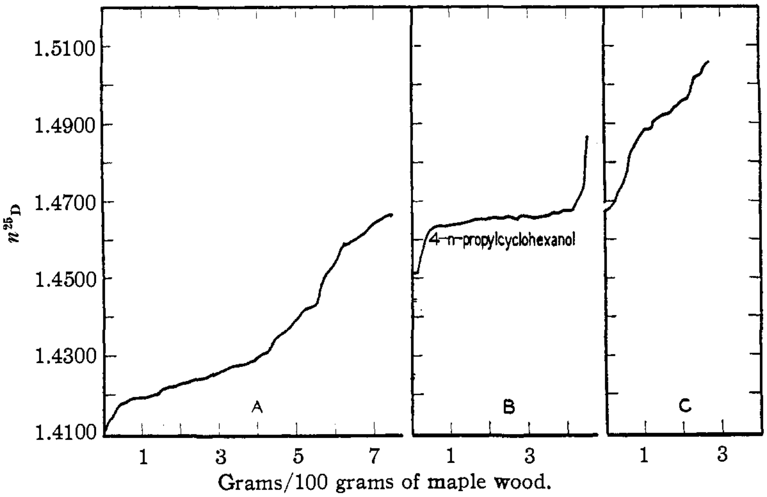

Fig. 2.—Fractional distillation of water-insoluble hydrogenation products from maple wood: A, b. p. 80–150° (100 mm.); B, b. p. 120–122° (20 mm.); C, b. p. 90–150° (1 mm.). Since 65 samples were collected in the range included in part A (and a proportionate number of cuts in B and C) it was impractical to plot the points on these curves.

For purposes of comparison with other published yields of degradation products from lignin, and on the basis of several reasonable assumptions, the yield of cyclohexylpropane derivatives can be calculated on the basis of percentage identified carbon of the methoxyl-free protolignin in the wood. The weight and empirical composition of protolignin in the maple wood is assumed to be that of the lignin isolated by the Klason method,[9] *i. e.*, 22.5 g. per 100 g. of the maple wood used; the carbon content of such lignin is taken as 63.3%[10] and

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[9] Ritter, Seborg and Mitchell, *Ind. Eng. Chem., Anal. Ed.*, **4**, 202 (1932).

[10] Fuchs, "Die Chemie des Lignins," J. Springer, Berlin, 1926.

thus there is (22.5)(0.633) or 14.24 g. of "protolignin" carbon per 100 g. of maple wood. Since the maple wood contained 6.3%[11] methoxyl, 2.44 g. of the lignin carbon was present as methoxyl, leaving, by difference, 11.80 g. of "methoxyl-free protolignin" carbon per 100 g. of wood. The ratio of the carbon content to the molecular weight of 4-*n*-propylcyclohexanol times the weight of this compound (4.41 g.), experimentally isolated, shows 3.36 g. of carbon in this substance per 100 g. of wood. Similarly for 3-(4-hydroxycyclohexyl)-propanol-1 (1.29 g.), there is 0.88 g. of carbon in 100 g. of wood. The sum of these, 4.24 g., represents 36% of the total weight of "protolignin" carbon (11.8 g. per 100 g. of methoxyl-free wood) isolated and identified as propylcyclohexane derivatives.

Experimental

Hydrogenation.—Solvent-extracted woodmeal[12] (25 g., 40 mesh), suspended in absolute dioxane (172 cc.) with copper chromite (10 g.) was placed in the hydrogenation bomb (500 cc. capacity) and hydrogen admitted (after removing air by flushing with hydrogen) at a pressure of 3500 lb./sq. in. The temperature of the bomb was raised to 280° over a period of four hours (initial hydrogen absorption occurred after about one and one-half hours at 190°), this temperature being maintained until the hydrogenation was completed (fifteen to twenty hours). After cooling, the bomb contents were centrifuged to remove the catalyst, leaving a clear, faintly-yellow dioxane solution of the hydrogenation products (stored, when necessary, under nitrogen at 0°). In preliminary experiments the catalyst residue was recovered quantitatively, indicating complete liquefaction of the wood. Other hydrogenation experiments, using alkali-extracted[12] spruce and maple wood, in which the temperature was raised to 280° in about two hours instead of four, proceeded satisfactorily, as was also the case when the bomb charge was increased to 37.5 g. of woodmeal, with the dioxane and catalyst quantities being increased proportionately.

A similar technique was employed in later larger-scale experiments using 175 g. of solvent-extracted maple woodmeal, 100 g. of copper chromite, and 1030 cc. of absolute dioxane in a 2500-cc. bomb. In these runs, however, presumably due to inefficient agitation of the bulky woodmeal-catalyst suspension, approximately 6% of the original wood remained unchanged after hydrogen absorption apparently had ceased; nevertheless, practically colorless dioxane solutions of the hydrogenation products were obtained after the black catalyst mass had been removed by centrifugence.

Fractional Distillation Apparatus.—For solvent removal and crude fractionation a 250 × 4 mm. Vigreux column (A), a 500 × 15 mm. glass-helices-packed column (B), or a 50 × 10 mm. wire-helices-packed, vapor-jacketed Fenske column (C)[7a] was used. Final fractionation of high-boiling liquids was carried out in a 900 × 5 mm. wire-helices-packed column (D) or a 900 × 3.8 mm. Podbielniak gold-spiral-packed column (E), each surrounded by an evacuated jacket and wrapped with resistance wire.

"Fractionation Procedure (A)."—The liquid products obtained from the *small-scale* preliminary hydrogenation experiments were distilled below 90° (25 mm.) to remove low-boiling products and dioxane. The residual oils were then separated by fractional distillation up to 200° (1 mm.) using Fractionating Column A and the fractions listed in Table I obtained.

"Fractionation Procedure (B)" (See Flow Sheet).—The combined dioxane solutions from four *large-scale* maple woodmeal (700 g.) hydrogenations were concentrated (Claisen flask, dry-ice trap) until all the material boiling below 80–100° (25 mm.) (Fraction I), had been removed, leaving 200 g. of a higher-boiling residue (Fraction II). Fraction I was then fractionated at 760 mm. (Column C) giving (a) a low-boiling fraction (below 87°) containing methanol (42.5 g.) and ethanol (95.0 g.)[13] determined by method of Cooke;[14] (b) dioxane–water azeotrope (87–88°) (118 g.); (c) pure dioxane (101.5°) and (d) mechanically entrained oils (Fraction (2)).[15]

Fraction II, dissolved in 30–50° petroleum ether (about 400 cc.) was exhaustively extracted with water (500, 250 and 125 cc. portions, successively), and the water extracts combined and distilled through Fractionating Column B giving (a) a low-boiling (100° (760 mm.)) aqueous mixture composed of an unidentified immiscible oil ($d^{25}_{25} < 1.0000$) and water, and (b) a residue of high-boiling oils (Fraction IVA). The latter was extracted continuously with benzene (seventy-two hours) giving benzene-soluble and -insoluble portions which were then fractionally distilled at 1-mm. pressure through Fractionating Column E (Figs. 1A and 1B, respectively). The latter has yielded, as yet, no identifiable products, but from the former a fraction (b. p. 129–130° (1 mm.); n^{25}D 1.4865) weighing 7.2 g. (1.29% of the maple wood or 5.8% of the Klason lignin content of the wood) was isolated and identified as 3-(4-hydroxycyclohexyl)-propanol-1 by oxidation to β-(4-ketocyclohexyl)-propionic acid and the conversion of this to its semicarbazone, m. p. 201–203°, mixed m. p. with an authentic sample, 201–201.5°.[16]

The water-insoluble oils (Fraction III), after removal of the petroleum ether, were divided into three additional fractions by rapid distillation (Claisen flask), namely, 80–150° (100 mm.)[17] (IIIA), 100–150° (20 mm.) (IIIB), 150–259° (1 mm.) (IIIC), and each (A, B, C) then fractionally distilled through Column D (Fig. 2), the previously described recovered oils (Fraction 3) being added to the appropriate portion of Fraction III as indicated in the Flow Sheet.[18] From Fraction IIIB was obtained 24.1 g. of a material having n^{25}D 1.4630–1.4680 (Fig. 3B) and which was char-

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[11] Analysis by L. M. Cooke.

[12] Peniston, McCarthy and Hibbert, THIS JOURNAL, **62**, 2284 (1940).

[13] Analyses by J. R. Bower, Jr.

[14] Cooke, McCarthy and Hibbert, THIS JOURNAL, **63**, 3052 (1941).

[15] Fraction (2) was extracted with water, as described for Fraction II, yielding water-insoluble Fraction (3) and water-soluble Fraction (4). Fraction (3) was fractionally distilled through Fractionating Column D, the resulting appropriate fractions being added to similar ones from Fraction III. Due to its small volume, Fraction (4) was discarded (see Flow Sheet).

[16] Melting point determination by J. R. Bower, Jr.

[17] Temperatures refer to the heating bath.

[18] An appreciable amount of resinification occurred during each distillation.

acterized as 4-*n*-propylcyclohexanol[19] by conversion of three representative samples to the phenyl urethans (yield 11–28%), m. p. 127°, mixed m. p. with authentic sample, 127°.[16]

Acknowledgment.—The authors wish to thank the National Research Council of Canada for the award of a studentship to one of them (H. P. G.) and also the Canadian Pulp and Paper Association for their kind financial assistance. The authors wish also to express their indebtedness to Dr. L. M. Cooke and Mr. J. R. Bower, Jr., for kind help in the preparation of this manuscript.

Summary

1. Samples of spruce and maple wood were suspended in dioxane and hydrogenated over copper chromite catalyst using an initial pressure of 3500 lb./sq. in. and a temperature of 280° and complete conversion of the wood into dioxane soluble products and gases effected, thus providing, for the first time, a method for the complete liquefaction of wood.

2. From the reaction mixture, 4-*n*-propylcyclohexanol and 3-(4-hydroxycyclohexyl)-propanol-1 have been isolated in yields of 19.5 and 5.8% (based on Klason lignin content of wood), respectively. Using as a basis for calculation the carbon values of these isolated units and of the "methoxyl-free protolignin" content of the wood, the combined yield of propylcyclohexane derivatives represents a recovery of 36%.

MONTREAL, CANADA RECEIVED AUGUST 25, 1941

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[19] Upon refractionation of the 4-*n*-propylcyclohexanol (Column D) 75% of the material was collected between n^{25}_D 1.4630 and 1.4670.

[CONTRIBUTION OF THE DIVISION OF INDUSTRIAL AND CELLULOSE CHEMISTRY, MCGILL UNIVERSITY]

Studies on Lignin and Related Compounds. LXIII. Hydrogenation of Wood (Part 2)

BY JOHN R. BOWER, JR., JOSEPH L. MCCARTHY AND HAROLD HIBBERT

It has been shown in the preceding paper[1] that maple wood can be completely converted, by high-pressure hydrogenation over copper chromite catalyst, into a mixture of dioxane-soluble oils (and gases) containing (a) 4-*n*-propylcyclohexanol and 3-(4-hydroxycyclohexyl)-propanol-1 both of which undoubtedly originate from protolignin (since they have been obtained by direct hydrogenation of isolated lignins[2]), and (b) unidentified distillable oils, originating from either lignin or carbohydrates which are not readily separable by fractional distillation.[3] Although the complete separation and identification of all the wood hydrogenation products will, no doubt, ultimately be accomplished, the present investigation was limited to an attempt to isolate (and, thereby eliminate) woodmeal hydrogenation products of non-lignin origin by comparing the fractionation results from maple holocellulose hydrogenation products with those previously obtained from maple wood.[1]

Maple holocellulose, prepared according to Van Beckum and Ritter,[4] on hydrogenation under the same conditions as those used for maple wood,[1] reacted with approximately 2 moles of hydrogen per 100 g. of material and was completely converted to dioxane-soluble products and gases. However, since the reaction solution was brown in color (as contrasted with a colorless product from maple wood) and the catalyst considerably reduced, the latter could have been poisoned[5] and, accordingly, the crude reaction mixture was retreated with fresh catalyst under the same conditions. Additional hydrogen (0.5 mole/100 g.) was absorbed during eight hours, the total amount being 2.5 moles (compared with approximately 3.1 moles/100 g. for maple wood) and the now colorless reaction mixture was fractionated by the procedure employed previously[1] in the case of maple woodmeal. The results obtained with the two products are shown in Table I.

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[1] Godard, McCarthy and Hibbert, THIS JOURNAL, **63**, 3061 (1941).

[2] (a) Harris, D'Ianni and Adkins, *ibid.*, **60**, 1467 (1938); (b) Cooke, McCarthy and Hibbert, *ibid.*, **63**, 3056 (1941).

[3] This difficulty was not unexpected since Zartman and Adkins [THIS JOURNAL, **55**, 4559 (1933)] have shown that a wide variety of products are formed by hydrogenation of the simplest carbohydrates.

[4] (a) Van Beckum and Ritter, *Paper Trade J.*, **104**, 253 (1937); **105**, 277 (1937). (b) Although the Van Beckum–Ritter holocellulose procedure is essentially a method for the quantitative estimation of the carbohydrate content of wood, it has been applied here as a means of preparing larger quantities of "lignin-free wood."

[5] Difficulty was experienced in removing all traces of chloride (probable catalyst poison) from the holocellulose even when it was re-washed with boiling ethanolamine, hot and cold ethanol, and water, and despite the fact that 5% of the holocellulose itself was dissolved during the treatment. Consequently it was decided to proceed with the examination of the twice-hydrogenated product.