nitroacetamide were prepared in the manner described above for the derivatives of ethyl nitroacetate. It was necessary, however, to use the sodium salt of nitromethane in the preparation of the other two compounds in Table I. A typical preparation is that of N-(*o*-chlorophenyl)-α-nitroacetamide (V).

To a solution of 2.2 g. (0.036 mole) of nitromethane in 35 ml. of dry benzene, protected from moisture by a calcium chloride tube, was added 0.7 g. (0.030 mole) of sodium wire. After all the sodium had reacted, the benzene was decanted from the precipitated salt, which was then washed with several portions of dry benzene, and finally suspended in 35 ml. of dry benzene. To this suspension was added 5.0 g. (0.032 mole) of *o*-chlorophenyl isocyanate. The mixture was refluxed (the condenser was protected by a calcium chloride tube) for 2 hours, after which the solid reaction product was filtered off and washed with benzene. The solid was then added, portionwise, to 200 g. of crushed ice; an insoluble residue of 2,2′-dichlorocarbanilide (m.p. 238°)[5] was filtered off, and the aqueous solution was acidified to congo red. A yellow precipitate appeared, and after stirring for an additional half hour the product was filtered off, recrystallized once from water, and obtained as a yellow solid, m.p. 121–122°, in less than 10% yield.

Hydrolysis and Decarboxylation.—About 0.5 g. of the compound obtained by the reaction of ethyl nitroacetate with an isocyanate (except *p*-nitrophenyl isocyanate) was suspended in a large excess of a filtered saturated solution of barium hydroxide, and the mixture was boiled for about 5 minutes. After cooling, the mixture was acidified to congo red, and the solid residue was filtered off and recrystallized from water. In each case, the m.p. of the product was found to be the same as that of the corresponding compound prepared directly from nitromethane; furthermore, no depression of the m.p. was observed in any case for a mixture of a hydrolyzed and decarboxylated product with the corresponding direct condensation product.

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Optical Dispersion of Perdeuterobenzene and Perdeuterocyclohexane

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In the course of some investigations being carried out in this Laboratory[1] on the physical properties of perdeuterobenzene and perdeuterocyclohexane the index of refraction of these compounds has been measured at three different wave lengths of light. It is the purpose of this note to record these index values and the various dispersion constants calculated from them.

The measurements were made with a Bausch and Lomb, Precision Abbe-type refractometer. The temperature of the prisms was maintained at 25.00 ± 0.02°. A sodium vapor light provided the light of wave length 5892.6 Å. (intensity-weighted mean of the doublet D_1,D_2). A mercury vapor lamp together with suitable filters provided the light of wave lengths 5460.7 and 4358.3 Å. The index of refraction of samples of N.B.S. Standard samples of 2,2,4-trimethylpentane and toluene, and of a purified sample of benzene, whose index had been previously checked against an NBS sample, was measured at each of the above wave lengths. Precautions were taken to ensure the purity of the samples, and in all cases the measurements were made on the samples immediately after their removal from sealed glass ampoules. From a comparison of the measured index values at each of the three wave lengths, with the certified values of their indices as reported by the National Bureau of Standards and tabulated by Forziati,[2] suitable instrument corrections were obtained. The instrument corrections were assumed to be a linear function of the refractive index reading, and did not appear to be a function of the wave length of the light used in the measurement. Correction values obtained on this basis were applied to the data reported here. The preparation of these deuterocarbons and their n_D values at 20° and 30° have been reported by Dixon and Schiessler.[3] Linear interpolations at 25° from their reported values are in excellent agreement with the values reported here. Table I lists the data for the deuterocarbons together with the data for the hydrocarbon isomers.

TABLE I

Wave length, Å.	Index of refraction at 25° C_6H_6[3]	C_6D_6[a]	C_6H_{12}	C_6D_{12}[b]
5892.6	1.49792	1.49597	1.42361	1.41909
5460.7	1.50197	1.49998	1.42544	1.42082
4358.3	1.51964	1.51798	1.43304	1.42802

[a] The purity of the C_6D_6 sample was equal to 99.3% replacement of protium with deuterium. Linear extrapolation of the measured value to 100.0% deuteration decreases the measured index by about 0.00001 which is less than the estimated uncertainty in the measurement. [b] The purity of the C_6D_{12} sample was equal to 98.8% deuteration. Linear extrapolation of the measured values to 100.0% purity decreases the measured index by about 0.00005 which is about the same as the estimated uncertainty in the measurement.

Forziati[2] has shown that the optical dispersion of organic compounds can be represented accurately by the modified Hartmann dispersion formula

$$n_\lambda = n_\infty + \frac{C}{(\lambda - \lambda^*)^{1.6}} \qquad (1)$$

where n_λ is the refractive index at light of wave length λ, and C, λ^*, and n_∞ are constants. The constants of this equation have been evaluated (after the construction of suitable tables for the wave lengths involved) by the method described by Forziati,[2] and are tabulated in Table II. Since the characteristic frequency ν_0 in the simplified Sellmeier–Drude[4–6] dispersion equation

$$n^2 - 1 = C/(\nu^2_0 - \nu^2_1) \qquad (2)$$

may be useful in the evaluation of the "London Dispersion Forces,"[7] the constants of this equation have been evaluated by the method of least squares and also are tabulated in Table II.

From equation 2 a value of the refractive index at infinite wave length may be calculated and this is also listed in Table II for comparison with the η_∞ calculated from the Hartmann equation.

Values of the Hartmann equation dispersion constants reported here for benzene are in good agree-

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[1] American Petroleum Institute Project 42. Advisory Committee: H. Sutherland, Chairman, E. M. Barber, J. R. Bates, L. C. Beard, Jr., G. H. Denison, L. M. Henderson, R. F. Marschner, L. A. Mikeska and J. H. Ramser.

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TABLE II

DISPERSION EQUATION CONSTANTS AT 25°

		C_6H_6	C_6D_6	C_6H_{12}	C_6D_{12}
Eq. 1	λ^*, μ	0.12929	0.14981	0.09418	0.09594
	$C \times 10^{+3}$	6.858	5.978	3.779	3.538
	n_∞	1.47417	1.47369	1.41197	1.40813
	$\nu_0 + 10^{-15}$	2.18632	2.10355	2.91743	2.97928
Eq. 2	$C \times 10^{-30}$	5.629058	5.155144	8.471202	8.733591
	η_∞	1.47568	1.47140	1.41254	1.40853

ment with the values given by Forziati[2] even though the values calculated in this research are from measurements made at a combination of wave lengths which are different than those used in the calculations by Forziati. The Hartmann equation reproduces the data with an error of about ±0.00002 refractive index unit. The Sellmeier–Drude equation reproduces the data with an error of about ±0.0004 refractive unit.

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Cellulose Sheets as Chromatographic Supports[1,2]

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During our studies of the various techniques of partition chromatographic analysis[3-7] we have made extensive use of filter papers (*e.g.*, Whatman No. 3)[8] for the separation of relatively large amounts of mixtures of sugars containing mono-, di-, tri- and tetrasaccharides but they have a limited loading capacity.[9,10]

In an extension of this work it has been found that limited use can be made of cellulose sheets,[11] approximately one-eighth of an inch thick, as chromatographic supports for bringing about the separation of a relatively large amount of a mixture of sugars. Due to the poor wet strength of this type of support, which is made from cellulose of short fiber length, it was necessary to carry out the development of the chromatograms in a horizontal direction. For these preliminary experiments a horizontal glass tube, 100 mm. diameter and closed at each end with a rubber stopper covered with aluminum foil, served as the chromatographic chamber and glass T-pieces, inserted across the inside of the tube, formed a horizontal support for the cellulose strip. The developing solvent, contained in a cylindrical dish at one end of the glass tube, was led onto the cellulose strip by means of an adsorbent cotton bridge. This apparatus can also be used for carrying out horizontal partition chromatographic analysis with the usual Whatman No. 1 papers. Methylated sugars show the same R_f value as those determined by either the ascending or descending techniques.

The thick cellulose sheets have been found to have a high loading capacity, *e.g.*, D-xylose in concentrations of 20 mg./cm. moved as a discrete band.

TABLE I

SEPARATION OF SUGARS IN CELLULOSE SHEETS[a]

No.	Sugars	Solvent	Distance moved by solvent front (cm.)	Width of sugar bands (cm.)	R_f values: Cellulose sheet	R_f values: Whatman No. 1 paper
1	D-Glucose (15 mg.)	Phenol–water	26.0	4.1	0.40	0.38
	L-Rhamnose (17 mg.)			4.6	.64	.63
2[b]	2,3-Dimethyl-D-glucose (14.2 mg.)	Methyl ethyl ketone–water azeotrope	34.5	5.5	.28	.28
	2,3,4,6-Tetramethyl-D-glucose (8.6 mg.)			3.2	.94	.82
3[c]	L-Arabinose (2 mg.)	1-Butanol–ethanol–water	24.8	4.1	.27	.14
4	D-Xylose (100 mg.)	1-Butanol–ethanol–water	26.5	4.3	.24	.15
5[c]	D-Glucose (2 mg.)	1-Butanol–ethanol–water	24.8	4.0	.20	.09
6	D-Glucose (14 mg.)	1-Butanol–ethanol–water	..[d]	4.5	1.00[d]	..
	L-Rhamnose (14 mg.)			4.0	1.63[d]	

[a] A cellulose strip 5 cm. in width was used in each experiment. [b] The methylated sugars could not be detected by the usual spray reagents (ammoniacal silver nitrate, *p*-aminodimethylaniline) due to the fact that aqueous solutions wash enough of the sugars into the inside of the sheet to make the level of sugar on the surface too low for detection by these reagents. They were detected, however, by spraying with a 3% solution of ethylleucine hydrochloride in 1-butanol and heating to 130° for about 10 minutes. The sugar bands fluoresced yellow in ultraviolet light. [c] These experiments were carried out in order to determine the R_f values of these sugars on the thick cellulose sheets. Each sugar was put onto the cellulose strip in the form of a spot. [d] The solvent was induced to flow off the end of the paper and the R_f value is quoted in reference to the movement of the glucose.

Moreover, successful separations of D-glucose and L-rhamnose, D-glucose and D-fructose, and 2,3-dimethyl- and 2,3:4,6-tetramethyl-D-glucose have been made using phenol–water, 1-butanol–ethanol–water and methyl ethyl ketone–water azeotrope, respectively. By lap-jointing two or three strips to-

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[1] Paper No. 2849, Scientific Journal Series, Minnesota Agricultural Experiment Station.

[2] A report of work done partially under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act of 1946. The contract is being supervised by the Northern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry.

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