

alone hydrochloride, 77 mg., and noradrenaline, 53 mg., a 28% yield. The activity was 8.5×10^4 c.p.m. per mg. Identity and purity of the noradrenaline were established by microanalysis, pharmacological activity and isotope dilution assay.

Full experimental details for this synthesis are available on microfilm.³

(3) For full experimental details of this synthesis order Document 3847 from American Documentation Institute, c/o Library of Congress, Washington 25, D. C., remitting \$1.25 for microfilm (images 1 inch on standard 35-mm. motion picture film) or \$1.25 for photostats readable without optical aid.

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Temperature Coefficients of Rotation of Some *o*- and *p*-Nitrophenyl Glycosides and their Polyacetates¹

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Pigman² has suggested that the anomalous positive rotations of the *ortho*-substituted phenyl β -D-glycoside tetraacetates are due to "interactions

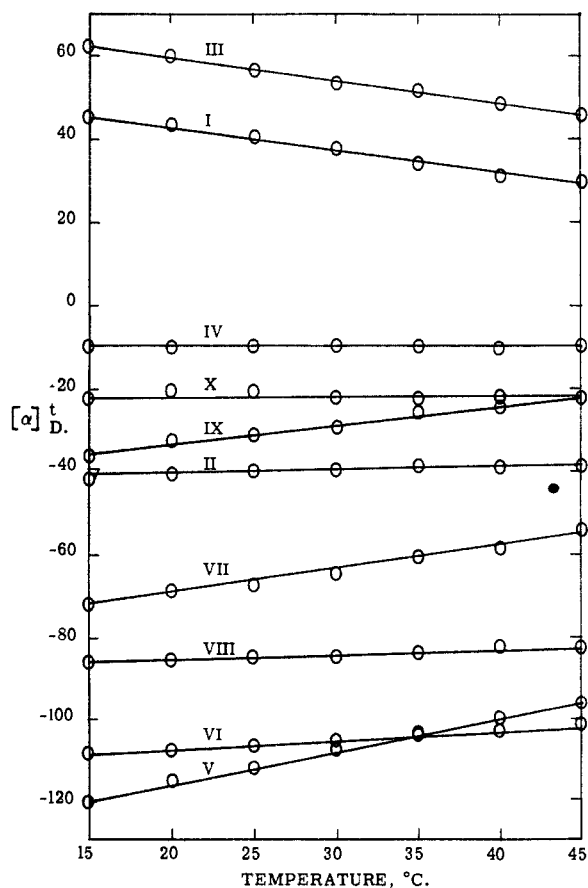


Fig. 1.—Influence of temperature on specific rotation of some *o*- and *p*-nitrophenyl glycosides and their polyacetates.

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(2) W. W. Pigman, *J. Research Natl. Bur. Standards*, **33**, 120 (1944).

between the acetyl and aglycon groups. Such interaction might take the form of weak bonds between these groups or it might operate through steric hindrance to free rotation of the aglycon group about the glycosidic linkage." This was postulated on the basis of the large temperature coefficients of rotation of these glycosides in contrast to those of their *m*- and *p*-isomers. We have determined the rotations of several *o*- and *p*-nitrophenyl glycosides and their polyacetates over the range 15–45°, and find that the *ortho* compounds have large temperature coefficients while the *para* compounds have normal coefficients. This indicates that the acetate groups are not directly concerned with the production of large temperature coefficients and favors their explanation on the basis of steric hindrance.

Experimental

Change of Specific Rotation with Temperature.—The method of preparation of the compounds studied has been reported previously.³ Rotations were determined with a Schmidt and Haensch polarimeter No. 52-b with monochromator. A 2-dm. jacketed tube was used, with water, maintained at $t \pm 0.2^\circ$ by means of a thermostatically controlled water-bath, as the circulating fluid. No correction was made for liquid density change with temperature.

TABLE I

SOLVENTS AND CONCENTRATIONS IN DETERMINATION OF CHANGE OF SPECIFIC ROTATION WITH TEMPERATURE

Compound	Solvent	Concn., %
<i>o</i> -Nitrophenyl β -D-glucoside tetraacetate (I)	Chloroform	1.966
<i>p</i> -Nitrophenyl β -D-glucoside tetraacetate (II)	Chloroform	1.884
<i>o</i> -Nitrophenyl β -D-galactoside tetraacetate (III)	Chloroform	1.865
<i>p</i> -Nitrophenyl β -D-galactoside tetraacetate (IV)	Chloroform	1.983
<i>o</i> -Nitrophenyl β -D-glucoside (V)	Water	0.828
<i>p</i> -Nitrophenyl β -D-glucoside (VI)	Water	0.987
<i>o</i> -Nitrophenyl β -D-galactoside (VII)	Water	1.065
<i>p</i> -Nitrophenyl β -D-galactoside (VIII)	Water	0.980
<i>o</i> -Nitrophenyl α -L-arabinoside (IX)	Water	.290
<i>p</i> -Nitrophenyl α -L-arabinoside (X)	Water	.265

(3) J. A. Snyder and K. P. Link, *THIS JOURNAL*, **74**, 1883 (1952).

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The Sedimentation Constant of Insulin in Acid Solution: A Re-examination¹

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On the basis of their observations on the sedimentation and diffusion constants of bovine insulin in acid solution, Fredericq and Neurath² concluded that the minimum molecular weight of this protein was about 6000. Although this conclusion has received support from the more recent work of Harfenist and Craig³ on counter-current distribu-

(1) This work has been supported by the Lilly Research Laboratories, Eli Lilly and Company, Indianapolis, Indiana, to whom we are also indebted for the supply of crystalline insulin.

(2) E. Fredericq and H. Neurath, *THIS JOURNAL*, **72**, 2684 (1950).

(3) E. J. Harfenist and L. Craig, *ibid.*, **74**, 3087 (1952).