as described by Emil Fischer and by Friedrich, has been acetylated and crystalline β-gentiobiose octa-acetate has been obtained in a yield corresponding to about 1 g. of gentiobiose per 100 g. of *d*-glucose. Evidence is presented that the disaccharide osazone which Fischer obtained in low yield from such a sirup, and named isomaltose osazone, is gentiobiose osazone.

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NOTE

***p*-Nitrobenzoyl Esters of β-Phenylethyl, γ-Phenylpropyl, and δ-Phenylbutyl Alcohols.**—In the preparation of normal ω-phenyl-alkyl chlorides containing from one to seven carbon atoms in the chain, the corresponding alcohols were obtained as intermediates.[1] In order to characterize these alcohols definitely it was desirable to convert them into solid derivatives for the purpose of analysis.

It had been found that the phenylurethan derivatives were not suitable for this purpose, inasmuch as these substances all had relatively low melting points which decreased with increasing length of the carbon chain. The decrease in melting point is not regular but gives indication of alternation. ε-Phenylamyl alcohol did not yield a solid phenylurethan even when cooled to low temperatures. Table I gives the melting points of the phenylurethan derivatives.

TABLE I

MELTING POINTS OF PHENYLURETHAN DERIVATIVES

Bz = benzyl; Ph = phenyl; Et = ethyl; Pr = propyl; Bu = butyl; Am = amyl; Hx = hexyl; Hp = heptyl.

Phenylurethan of	Phenol[a]	Bz alc.[b]	β-PhEt alc.[c,d]	γ-PhPr alc.[c]
M. p., °C.	125.5	78	79–80	47–48
Phenylurethan of	δ-PhBu alc.[c]	ε-PhAm alc.[e]	ζ-PhHx alc.	η-PhHp alc.
M. p., °C.	51–52	uncrystallizable	?	?

[a] A. W. Hoffmann, *Ber.*, **4**, 249 (1871). Eckenroth, *Ber.*, **18**, 517 (1885). Leuckhart, *Ber.*, **18**, 875 (1885). Cazeneuve and Morel, *Bull. soc. chim.*, [3] **19**, 696 (1898). A. Morel, *ibid.*, **21**, 827 (1899).

[b] Soden and Rojahn, *Ber.*, **34**, 2809 (1901).

[c] H. Walbaum, *Ber.*, **33**, 2300 (1900).

[d] Soden and Rojahn, *Ber.*, **33**, 3065 (1900).

[e] von Braun, *Ber.*, **44**, 2872 (1912).

A few of the *p*-nitrobenzoyl esters of this series of alcohols were prepared with the hope that perhaps they would yield solids of higher melting point than the phenylurethans. It was found, however, that in case of these derivatives also the melting points decreased rapidly, but without alternation, and that even the ester of δ-phenylbutyl alcohol had a melting point

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[1] Conant and Kirner, THIS JOURNAL, **46**, 240 (1924).

so low as to render this derivative quite inconvenient for purposes of identification.

The preparation of the esters was carried out as follows.

To 2.5 g. of the alcohol in a small flask were added 10 g. of pyridine and then a 10% excess of *p*-nitrobenzoyl chloride in portions. The flask was connected to a reflux condenser protected by a calcium chloride tube and heated on the steam-bath for two to three hours. On cooling overnight the contents of the flask crystallized. Ether was first added, then 25 cc. of dil. hydrochloric acid and the mixture was transferred to a small separatory funnel and vigorously shaken. The aqueous layer was run off and the ether layer extracted twice with dil. hydrochloric acid. The combined aqueous layers were then extracted twice with small portions of ether. The combined ether layers were washed twice with dil. sodium carbonate solution and the ether was removed by evaporation.

The residue from the β-phenyl ethyl alcohol crystallized and was purified for analysis by twice recrystallizing from 95% alcohol. The residues from the γ-phenylpropyl and δ-phenylbutyl alcohols did not crystallize after the ether was removed, so they were taken up in the least quantity of 95% alcohol necessary to dissolve them and the solution was cooled to —20° in a freezing mixture. Crystals were obtained which were filtered off through a funnel surrounded with a freezing mixture and washed with cold alcohol.

Table II gives the melting points and data obtained from the analyses.

TABLE II

ETHERS

p-Nitrobenzyl ester of	M. P., °C.	Analysis, %			
		Calcd.		Found	
		C	H	C	H
Phenol[a]	129	...	..	...	..
Bz alc.[b]	83.5–84	...	..	...	..
β-PhEt alc.	62–63	66.40	4.83	66.12	4.81
γ-PhPr alc.	45–46	67.35	5.30	66.80	5.78
δ-PhBu alc.	18–20	Not analyzed			

[a] Raiford, Taft and Lankelma, THIS JOURNAL, **46,** 2056 (1924).

[b] Bamberger and Renauld, *Ber.*, **30,** 2288 (1897).

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