

The product was given a quick distillation (1–2 mm.) to yield 6.5 g. of a yellow oil. The oil was extracted with 1.1 *N* sodium hydroxide solution to separate the naphthol as the sodium salt. Acidification of the aqueous phase yielded 4 g. of crude *V*. Recrystallizations from isoöctane gave pure 2-isopropyl-1-naphthol (*V*), m.p. 47–48°.

Anal. Calcd. for $C_{15}H_{14}O$: C, 83.83; H, 7.58. Found: C, 83.5; H, 7.58.

The benzoate of *V* was prepared by reaction with benzoyl chloride in the presence of pyridine (as described by Meyer and Bernhauer¹) and after recrystallization from ethanol and isoöctane was found to melt at 67–68°.

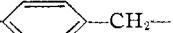
Anal. Calcd. for $C_{20}H_{18}O_2$: C, 82.73; H, 6.25. Found: C, 82.4; H, 6.16.

RESEARCH LABORATORIES
ETHYL CORPORATION
DETROIT 20, MICHIGAN

Polybenzyls from Benzyl Alcohol and Sulfuryl Chloride

BY R. A. GIBBONS,¹ MARIAN N. GIBBONS AND M. L. WOLFROM

RECEIVED JULY 8, 1955

Gladstone and Tribe² noted the production of hydrocarbons of the formula $(C_7H_6)_n$ on pouring benzyl bromide over a copper–zinc couple. Friedel and Crafts³ obtained similar material of the same empirical formula by the action of aluminum chloride upon benzyl chloride, a reaction which was studied further by Jacobson,⁴ who noted that the substance formed with stannic chloride contained 1.4% chlorine. The products were considered to be essentially polybenzyls formed from the polymerizing unit —CH₂—.

What is probably a closely similar material was formed in an attempt to prepare benzyl chlorosulfonate by the action of sulfuryl chloride upon benzyl alcohol. The product was a green solid which slowly turned pink on exposure to air. Its analysis was in accord with the formula $H(C_7H_6)_{10}Cl$. The chlorine may be a chain terminal unit. Hydrogen chloride and sulfur dioxide were evolved in the reaction, the stoichiometry of which is not established.

Experimental

In an attempt to prepare benzyl chlorosulfonate, 21.6 g. (0.2 mole) of benzyl alcohol was added slowly to 27 g. (0.2 mole) of sulfuryl chloride at 0°, according to the general procedure described by Binkley and Degering.⁵ During the addition of benzyl alcohol (about 90 min.), hydrogen chloride was evolved continuously. The resultant pale yellow viscous liquid (which sometimes contained a small amount of solid material) was stable provided that the temperature was maintained below 5°. On warming to room temperature, the mixture underwent a vigorous exothermic reaction. Hydrogen chloride and sulfur dioxide were evolved and a green mud was produced which solidified on cooling and slowly turned pink on exposure to air. The resin as obtained was contaminated by trapped gases and by a sulfur-containing material. Purification was complicated by the tendency of the material to form emulsions. The original resin was dissolved in benzene, washed with water, and precipitated

(1) Fellow of the Foreign Research Scientists Program of the Foreign Operations Administration.

(2) J. H. Gladstone and A. Tribe, *J. Chem. Soc.*, **47**, 448 (1885).

(3) C. Friedel and J. M. Crafts, *Bull. soc. chim.*, [2] **43**, 53 (1885).

(4) R. A. Jacobson, *THIS JOURNAL*, **54**, 1513 (1932); see also M. Kikkawa and S. Tsuruta, *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **53**, 405 (1950); *C. A.*, **47**, 345 (1953).

(5) W. W. Binkley with E. F. Degering, *THIS JOURNAL*, **60**, 2810 (1938).

from the benzene with methanol, a white "milk" being decanted from the pink resinous material. The resin was washed free of organic solvents in boiling water and on cooling a slightly brittle, pinkish-brown resin was obtained; yield 3–5 g., m.p. ca. 60° with preliminary softening. The resin was readily soluble in benzene, dioxane, *N,N*-dimethylformamide, pyridine, chloroform, carbon tetrachloride and benzaldehyde. It was insoluble in water, ethanol, formamide, hexane, 1-butanol and *t*-butyl alcohol. It swelled or was slightly soluble in ether, acetone, ethyl acetate, butanone, diethylamine and benzyl alcohol.

Anal. Calcd. for $H(C_7H_6)_{10}Cl$: C, 89.76; H, 6.46; Cl, 3.79. Found: C, 89.24; H, 6.25; Cl, 3.87.

DEPARTMENT OF CHEMISTRY
THE OHIO STATE UNIVERSITY
COLUMBUS 10, OHIO

The Anticholinesterase Activity of Arylarsonic and Diarylarsonic Acids

BY LEON D. FREEDMAN AND G. O. DOAK

RECEIVED JULY 21, 1955

Certain aromatic phosphonic and phosphinic acids are active as inhibitors of plasma cholinesterase.¹ In the present note we are reporting on the anticholinesterase activity of a series of aromatic arsonic and arsinic acids.²

Experimental

p-Hydroxybenzenearsonic acid was Eastman Kodak White Label; *p*-arsanilic acid was obtained from the B. L.

TABLE I
ANTICHOLINESTERASE ACTIVITY OF ARYLARSONIC AND DIARYLARSONIC ACIDS

Compound	I_{50}^a (moles/l.)
Arylarsonic acids	
$C_6H_5AsO_3H_2$	1.1×10^{-5}
<i>m</i> -ClC ₆ H ₄ AsO ₃ H ₂	2×10^{-5}
<i>p</i> -ClC ₆ H ₄ AsO ₃ H ₂	4×10^{-5}
<i>p</i> -CH ₃ C ₆ H ₄ AsO ₃ H ₂	4×10^{-5}
<i>p</i> -BrC ₆ H ₄ AsO ₃ H ₂	4×10^{-5}
<i>o</i> -ClC ₆ H ₄ AsO ₃ H ₂	7×10^{-5}
<i>p</i> -HOC ₆ H ₄ AsO ₃ H ₂	1.2×10^{-4}
<i>p</i> -NH ₂ C ₆ H ₄ AsO ₃ H ₂	1.2×10^{-4}
<i>o</i> -BrC ₆ H ₄ AsO ₃ H ₂	1.4×10^{-4}
<i>m</i> -O ₂ NC ₆ H ₄ AsO ₃ H ₂	4×10^{-4}
<i>p</i> -O ₂ NC ₆ H ₄ AsO ₃ H ₂	8×10^{-4}
<i>p</i> -NH ₂ O ₂ SC ₆ H ₄ AsO ₃ H ₂	4×10^{-3}
Diarylarsonic acids	
(<i>o</i> -BrC ₆ H ₄) ₂ AsO ₂ H	5×10^{-5} (2×10^{-4}) ^b
(<i>o</i> -ClC ₆ H ₄) ₂ AsO ₂ H	5×10^{-5} (2×10^{-4}) ^b
(<i>o</i> -BrC ₆ H ₄)C ₆ H ₅ AsO ₂ H	5×10^{-4}
(<i>m</i> -ClC ₆ H ₄) ₂ AsO ₂ H	1×10^{-3} (1×10^{-3}) ^b
(<i>p</i> -ClC ₆ H ₄) ₂ AsO ₂ H	2×10^{-3}
(<i>m</i> -O ₂ NC ₆ H ₄) ₂ AsO ₂ H	°
(<i>p</i> -O ₂ NC ₆ H ₄) ₂ AsO ₂ H	°

^a Concentration necessary for 50% inhibition when the enzyme and inhibitor were incubated for 20 minutes prior to the addition of the substrate solution. The value given is the final concentration after the addition of the substrate solution. ^b The value in parentheses was obtained with a second batch of the arsinic acid. ^c No significant inhibition at a concentration of 0.003 *M*.

(1) L. D. Freedman, H. Tauber, G. O. Doak and H. J. Magnuson, *THIS JOURNAL*, **75**, 1379 (1953).

(2) D. Vincent and P. Brygoo, *Bul. soc. chim. biol.*, **28**, 174 (1946), investigated the effect of a number of arsenical drugs on serum cholinesterase and noted that the few aromatic arsonic acids tested were inhibitors.