

In attempting to apply this method to the quantitative determination of the azo-dyes, the pigment spots were eluted from the paper with one normal sodium hydroxide and the amount determined with a colorimeter. A complete recovery of the chromatographed spots was not possible, since the azo-dyes of the above compounds are unstable. In chromatograms developed by Lederer's method, recovery was less than 50%, whereas when alkali was used for separation, recovery was about 80%.

The method described in this paper was used in measuring the production of *p*-aminobenzoic acid and anthranilic acid in *Neurospora*.<sup>5</sup>

### Experimental

The azo-dyes were prepared after the method of Schmidt and Kolbl.<sup>6</sup> The arylamines were diazotized, coupled with  $\alpha$ -naphthol in alkaline solution, and the resulting dye extracted from acidified solution with ether. The ether solution was washed in distilled water and condensed to a small volume *in vacuo*; then a drop was deposited on a filter paper strip (Whatman #1).

The chromatograph was developed by the ascending technique: a paper strip was suspended from the lid of a glass jar so that it dipped into one-tenth molar sodium hydroxide which covered the bottom of the jar. This solvent was made by diluting a five normal stock solution, which possibly contained some carbonate. The solvent moves rapidly and in a few hours ascends high enough to separate all the spots.

(5) M. Zalokar, *Genetics*, **35**, 700 (1950) (Abstract).

(6) K. H. Schmidt and C. Kolbl, *Z. Physiol. Chem.*, **281**, 7 (1944).

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## NEW COMPOUNDS

### 2-Tosylaminofluorene Derivatives<sup>1</sup>

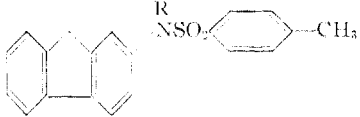
Sodium tosylaminofluorene has been found to react readily with alkyl and acyl halides. These derivatives may be useful in identifying compounds containing active halogen and by hydrolysis monoalkylaminofluorenes may be obtained from some of the derivatives. These alkylamino compounds should prove of value in a study of chemical carcinogenicity. Table I lists these new tosylamino derivatives.

**General Procedure.**—To 0.01 mole of 2-tosylaminofluorene dissolved in 100 ml. of warm neutral ethyl alcohol was added 10 ml. of 1 *N* aqueous sodium hydroxide followed by 0.011 mole of the halide. The mixture was refluxed for one hour. Then 200 ml. of water was added. After cooling, the mixture was filtered. Sometimes an oil was formed; in this case, the mixture was allowed to stand overnight in the cold room and then filtered. Recrystallizing solvent in all cases was methyl alcohol.

The 2-tosylaminofluorene used was prepared by the following improved procedure: To a solution of 5.4 g. (0.003 mole) 2-aminofluorene in 40 ml. of boiling acetic acid was added 9.5 g. (0.0038 mole) of *p*-toluenesulfonyl chloride. To this boiling milky mixture was added 4.1 g. (0.05 mole) of sodium acetate in small portions over 15 minutes. The mixture was refluxed 15 minutes. Finally water was added slowly to the boiling solution until crystals started to come out. The solution was allowed to cool and filtered. The large crystals were recrystallized from aqueous acetic acid. Yield of colorless crystals, melting at 161–162°, was 8.0–8.5 g. (80–85%). This compound can also be crystallized

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TABLE I



RX	Yield, %	M.p., °C.	Nitrogen, %		Sulfur, %	
			Calcd.	Found	Calcd.	Found
Methyl iodide	95 <sup>2</sup>	137–138				
Ethyl iodide	95	185	3.86	3.92	8.81	8.80
<i>n</i> -Propyl bromide	92	125–126	3.71	3.67	8.49	8.52
<i>n</i> -Butyl bromide	65	132–133	3.58	3.65	8.18	8.10
Benzyl chloride	93	200	3.29	3.31	7.53	7.50
Allyl chloride	78	131–132	3.73	3.57	8.53	8.36
Methallyl chloride	74	175–176	3.60	3.81	8.23	8.32
Ethyl chloro-carbonate	50	193	3.44	3.37	7.86	8.05
Acetyl chloride	75	164–165	3.71	3.64	8.49	8.35

from aqueous methyl cellosolve or methyl alcohol; lit. m.p. was 157–158°.<sup>3</sup>

(2) F. E. Ray and J. Little, in press.

(3) N. Campbell, W. Anderson and J. Gilmore, *J. Chem. Soc.*, 446 (1940).

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### Esters of Terephthalic Acid

Four terephthalates were prepared by the following general procedure. The melting points, analyses and yields of the esters are tabulated below.

**Experimental.**—A mixture of 0.5 g. of terephthalyl chloride and 2 ml. of the alcohol was heated gently over a low flame for 10 minutes. A 10-ml. portion of distilled water was then added and the mixture cooled in an ice-bath until the product had solidified. The material was collected by filtration, washed with 2% sodium carbonate solution, dried, and recrystallized from 95% ethanol.

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TABLE I

Ester	Mol. form of ester	Yield, % based on chloride	M.p., °C.	Calcd. C	Analyses, %		Found
					H	C	H
Di- <i>n</i> -heptyl	C <sub>22</sub> H <sub>34</sub> O <sub>4</sub>	31	36	72.89	9.45	72.84	9.54
Di- <i>n</i> -octyl <sup>a</sup>	C <sub>24</sub> H <sub>38</sub> O <sub>4</sub>	34	43	73.81	9.82	74.01	9.92
Di- <i>n</i> -nonyl	C <sub>26</sub> H <sub>42</sub> O <sub>4</sub>	34	46	74.60	10.11	74.85	10.24
Di- <i>n</i> -decyl	C <sub>28</sub> H <sub>46</sub> O <sub>4</sub>	39	57	75.30	10.37	75.28	10.63

<sup>a</sup> J. B. Cohen and H. S. de Pennington, *J. Chem. Soc.*, 113, 63 (1918), report this compound but give no constants.

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### 2,4-Dinitrophenylhydrazones of Some Hexoses and Pentoses<sup>1</sup>

In the course of identification of the carbohydrate residue in the degradation products of nucleic acids, a number of

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