Attempts at preparing oxythiamine diphosphate by deamination of cocarboxylase have so far yielded only impure oxythiamine monophosphate.

Oxythiamine reacts with Decalso in the same manner as thiamine. It gives a positive test with the Prebluda–McCollum reagent. Although Slobodin and Ziegel reported that oxythiamine showed a slight antineuritic activity, in our experiments it has been found to produce a toxic effect on mice. Administration of 25–50 µg. of oxythiamine per day resulted after about two weeks in the death of young mice which received a thiamine-low synthetic diet supplemented with 1 µg. of thiamine per day. The effect of oxythiamine on the enzyme system of carp which destroys thiamine was studied. It was found to inhibit the action of the enzyme on thiamine. Additional microbiological studies are in progress.

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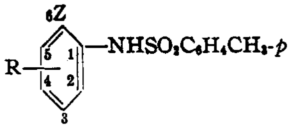
NEW COMPOUNDS

p-Toluenesulfonamides[1]

Two previously unreported *p*-tosylamides derived from substituted *o*-nitranilines and one from 4-amino-1,2-dimethylbenzene have been prepared. It has been reported[2,3] that *o*-nitro-arylamines react anomalously toward arylsulfonyl chlorides, in that the disulfonamides form much more readily than the mono compounds. This was found to be the case, but it was possible to prepare the desired monosulfonamides in a good state of purity, though in somewhat poor yield (Table I).

TABLE I

p-TOLUENESULFONAMIDES



R	Z	Yield, %	M. p., °C., cor.	Formula	Analyses, % Calculated C	Calculated H	Found C	Found H
2-CH$_3$	NO$_2$	15.7	121.5–3.5	$C_{14}H_{14}N_2O_4S$	54.88	4.62	54.69	4.66
2,4-Di-CH$_3$	NO$_2$	20	133.5–5.5	$C_{15}H_{16}N_2O_4S$	56.23	5.04	55.94	5.13
3,4-Di-CH$_3$	H	76	144.5–5.0	$C_{15}H_{17}NO_2S$	65.42	6.22	65.57	6.41

Procedure.—To 0.1 mole of the substituted *o*-nitrâniline suspended in 400 ml. of anhydrous pyridine was added 0.1 mole of *p*-toluenesulfonyl chloride all at once, shaking vigorously to ensure homogeneity. A short air condenser topped by a Drierite tube was inserted in the flask, and the assembly was heated in a boiling water-bath for six hours. At the end of that time, the hot solution was poured into 3000 ml. of ice water with vigorous stirring. An oil admixed with crystals settled out. The supernatant liquor was decanted and the residue was washed several times with water by decantation. Boiling methanol was added to the still moist residue, causing partial solution; the insoluble material was removed by filtration through a steam-jacketed Büchner funnel. Slow cooling of the filtrate caused the deposition of well-formed crystals of the monosulfonamide. Recrystallization from ethanol or methanol, using a small quantity of Darco, gave the pure monotosylamide.

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[1] Abstracted from a thesis submitted by Frank Kipnis to the Polytechnic Institute of Brooklyn, June, 1944, for the degree of Doctor of Philosophy.

[2] Suter, "Organic Chemistry of Sulfur," John Wiley and Sons, Inc., New York, N. Y., 1942, p. 511.

[3] Bell, *J. Chem. Soc.*, 2787 (1929).

(4) The residue, not further investigated, is the disulfonamide.

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1-(4'-Diethylamino-1'-methylbutyl)-2-keto-3-methyl-1,2-dihydroquinoxaline[1]

p-1-Alkyl-3-methyldihydroquinoxalones have been prepared by Kehrmann and Messinger[2] by the interaction of N-alkyl-*o*-phenylenediamines and pyruvic acid in aqueous or alcoholic acid solution. In the present work, the use of boiling cymene in conjunction with an automatic water decanter was found convenient. The final product was isolated in approximately 60% yield and was identified as the dipicrate.

Procedure.—One-tenth mole of 1-(4'-diethylamino-1'-methylbutyl)-amino-2-aminobenzene[3] was dissolved in 200 ml. of anhydrous *p*-cymene in a 1000-ml. interjoint flask. Freshly distilled pyruvic acid (0.105 mole) was added, together with a boiling stone, and the flask vigorously shaken for about five minutes. A calibrated moisture trap (Dean–Stark) topped by an efficient water-cooled reflux condenser was inserted in the flask, and the flask contents heated gently with a free flame. A vigorous reaction ensued and water distilled. Cautious heating was continued until solution was complete (two to three hours). About 80% of the calculated volume of water was eliminated during this period. Longer heating had no effect on the final yield.

The flask contents were cooled, transferred to a separatory funnel and the base separated as the hydrochloride by shaking out three times with 50-ml. portions of 20% hydrochloric acid.

The combined acid layers were run into an excess of ice-cold 15% ammonia water with swirling. The precipitated oil was extracted with four 100-ml. portions of chloroform. The solvent was removed on the steam-bath, and the mixture was steam distilled from saturated sodium chloride solution to remove traces of cymene and unreacted triamine. About 3 liters of distillate was collected before the distillate was free of these materials.

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[1] Abstracted from a thesis submitted by Frank Kipnis to the Polytechnic Institute of Brooklyn, June, 1944, for the Degree of Doctor of Philosophy.

[2] Kehrmann and Messinger, *Ber.*, **25**, 1629 (1891).

[3] Preparation to be reported in a subsequent communication.