[CONTRIBUTION FROM THE COLLEGE OF PHARMACY, UNIVERSITY OF CALIFORNIA]

Sulfophenylarsonic Acids and Certain of their Derivatives. IV. Derivatives of *p*-Sulfonamidophenylarsonic Acid[1]

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We have shown in a previous publication[2] that *p*-chlorosulfonylphenyldichloroarsine reacts with chlorine to form the corresponding tetrachloroarsine which is readily hydrolyzed in ice water to *p*-chlorosulfonylphenylarsonic acid. In this investigation, which represents a continuation of our previous studies, a number of sulfonamide derivatives of *p*-chlorosulfonylphenylarsonic acid have been prepared as indicated in Table I.

Condensations with dimethylamine, *p*-aminobenzoic acid and *p*-aminobenzenesulfonamide were carried out in aqueous media. The required amine (one mole equivalent) dissolved or suspended in warm water, was treated with 0.5 mole equivalent of *p*-chlorosulfonylphenylarsonic acid. Complete reaction was effected by the gradual application of heat.

The diiodoarsines, prepared by treating the corresponding arsonic acids with hydriodic acid, were subsequently hydrolyzed with ammonium hydroxide. The arsineoxides thus obtained, with the exception of *p*-arsenoso-N-phenylbenzenesulfonamide, existed as the hydrates. Doak and co-workers have indicated in a recent publication[3] that certain phenylarsineoxides containing an amide group were obtained in both the arsenoso and arsonoso forms, depending upon the method used for isolation.

TABLE I

DERIVATIVES OF *p*-SULFONAMIDOPHENYLARSONIC ACID

Compound	Recrystallized from	M. p., °C., uncorr.	Yield, %[f]	Formula	As analyses, % Calcd.	Found
R = arsono, -AsO_3H_2, R′ = benzenesulfonamide		softens				
p-R-N-dimethyl-R′	95% EtOH	166–168	57	$C_8H_{12}O_5NSAs$	24.21	24.10
p-R-N-phenyl-R′[a]	Water	.....	33	$C_{12}H_{12}O_5NSAs$	...	...
p-R-N-(*p*-carboxyphenyl)-R′[b]	50% HAc	.....	54	$C_{13}H_{12}O_7NSAs$	18.67	18.62
p-R-N-(*p*-sulfonamidophenyl)-R′	Water	.....	47	$C_{12}H_{13}O_7N_2S_2As$	17.18	17.16
R = diiodoarsino, -AsI_2						
p-R-N-dimethyl-R′	HAc glac.	132.5–134	54	$C_8H_{10}O_2NSAsI_2$	14.60	14.71
p-R-N-phenyl-R′	HAc glac.	125–126	77	$C_{12}H_{10}O_2NSAsI_2$	13.35	13.43
p-R-N-(*p*-carboxyphenyl)-R′	25% EtOH in HAc glac.	234–236	75	$C_{13}H_{10}O_4NSAsI_2$	12.39	12.84
p-R-N-(*p*-sulfonamidophenyl)-R′	HAc glac.	195–197	71	$C_{12}H_{11}O_4N_2S_2AsI_2$	11.71	12.23
R = arsenoso, -As = O						
p-R-N-phenyl-R′[c]	.....	.....	86	$C_{12}H_{10}O_3NSAs$	23.18	23.17
p-R-N-dimethyl-R′[d]	.....	.....	..	$C_8H_{10}O_3NSAs$	27.23	27.20
R = arsonoso, -$As(OH)_2$						
p-R-N-dimethyl-R′[e]	25% MeOH	.....	88	$C_8H_{12}O_4NSAs$	25.56	25.69
p-R-N-(*p*-carboxyphenyl)-R′	25% EtOH	.....	67	$C_{13}H_{12}O_6NSAs$	19.45	19.31
p-R-N-(*p*-sulfonamidophenyl)-R′	25% MeOH	.....	78	$C_{12}H_{13}O_6N_2S_2As$	17.72	17.77

[a] The preparation of this compound by the condensation of aniline with *p*-chlorosulfonylphenylarsonic acid has been described in a previous publication; Oneto and Way, THIS JOURNAL, **61**, 2106 (1939). More recently the compound has been obtained by application of the Scheller reaction (Scheller, Fr. Patent 624,028, *Chem. Zentr.*, **98**, II, 229 (1927)), to N[1]-phenylsulfanilamide. Mixed m. p. of the diiodoarsines, 125–126°. [b] When prepared by application of the Scheller reaction to N[1]-*p*-carboxyphenylsulfanilamide, a 37% yield was obtained as compared to the 54% yield resulting from the condensation reaction. Mixed m. p. of the diiodoarsines, 234–236°. [c] Purified by precipitating the compound several times from alkaline solution with dilute sulfuric acid. [d] Obtained by heating the corresponding arsonoso compound at 135–140° under reduced pressure (10–15 mm.) for four hours. [e] Prepared by Doak, Steinman and Eagle, THIS JOURNAL, **62**, 3012 (1940), by a different procedure. [f] Yields are for purified products.

Summary

The preparation and properties of a number of derivatives of *p*-sulfonamidophenylarsonic acid have been described.

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(1) Aided by a grant from John Wyeth & Brother.

(2) Oneto and Way, THIS JOURNAL, **61**, 2106 (1939).

(3) Doak, Steinman and Eagle, *ibid.*, **62**, 3012 (1940).