Substituted Sulfonamides

N^1-Chloroacetyl-*p*-nitrobenzenesulfonamide.—To 10 g. of *p*-nitrobenzenesulfonamide dissolved in 100 cc. of 4.4% sodium hydroxide was added, dropwise with stirring at 5°, 7 g. of chloroacetyl chloride (Eastman Kodak Co.). After fifteen minutes, the solution was neutralized with acetic acid and unchanged *p*-nitrobenzenesulfonamide separated by filtration. The filtrate was acidified to congo red with hydrochloric acid to precipitate the product, which was collected and dried at 60°. It was then recrystallized once from toluene (1 g. per 50 cc.); yield 5 g.

TABLE I

Compound	M. P., °C. (cor.)	Formula	Analyses, %[a] Calcd. C	H	N	Found C	H	N
N^1-Chloroacetyl-*p*-nitrobenzenesulfonamide[b]	172–173	$C_8H_7O_5N_2SCl$			10.1			10.4
N^1-Chloroacetylsulfanilamide[c]	157–158	$C_8H_9O_3N_2SCl$	38.6	3.6	11.3	38.7	3.9	10.9
2-Benzenesulfonamidopyridine	171–172	$C_{11}H_{10}O_2N_2S$			12.0			11.6
2-Benzenesulfonamidopyrimidine	229–230	$C_{10}H_9O_2N_3S$	51.1	3.8	17.9	51.0	4.1	17.9
2-Benzenesulfonamido-4-methylpyrimidine	193–194	$C_{11}H_{11}O_2N_3S$			16.9			16.7
2-Benzenesulfonamidothiazole	171–172	$C_9H_8O_2N_2S_2$	45.0	3.3	11.7	45.0	3.3	11.3
2-Benzenesulfonamido-1,3,4-thiadiazole	188–189	$C_8H_8O_2N_3S_2$			17.4			17.4

[a] Analyses were carried out in these laboratories under the direction of Mrs. Thelma Kirk. [b] Chlorine, calcd. 12.8%; found 12.6%. [c] Chlorine, calcd. 14.3%; found 14.6%.

N^1-Chloroacetylsulfanilamide was prepared from 5 g. of finely divided nitro compound which was added at 35° to 12.25 g. of $SnCl_2·2H_2O$ dissolved in 15 cc. of concentrated hydrochloric acid. Some cooling was necessary at first. After standing for eighteen hours, the solution was cooled and made alkaline with 10% sodium carbonate solution. The precipitate was removed and the filtrate clarified with activated carbon. The product was precipitated, after removal of the carbon, by acidifying the filtrate to congo red. It was dried at 60°, and purified by one recrystallization from benzene–alcohol; yield 1.5 g.

Benzenesulfonamido heterocycles were obtained by the reaction of benzenesulfonyl chloride with the appropriate amino heterocycle in dry pyridine. The general method has been described previously[1]; yields ranged from 75–90%.

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[1] Roblin and Winnek, THIS JOURNAL, **62**, 1999 (1940).

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RECEIVED JULY 31, 1942

COMMUNICATIONS TO THE EDITOR

BARBALOIN

Sir:

The recent note by Gardner and Campbell[1] on some reactions of the aloins emboldens us to place on record some experiments made in 1939. We can confirm Rosenthaler's statement[2] that barbaloin does not give methanol when hydrolyzed with borax and that Cahn and Simonsen's[3] observation is incorrect and we have observed also the formation of furfural under certain conditions. Our most fundamental result is however with reference to the empirical formula of barbaloin which was discussed at some length by Cahn and Simonsen.[3] Dr. E. G. Cox of the University of Birmingham has very kindly determined the molecular weight of barbaloin methyl ether by the X-ray crystal structure method and he finds it to be 521. There can therefore now no longer be any doubt that barbaloin methyl ether has the formula $C_{21}H_{17}O_2(OMe)_7$ from which it would apparently follow that barbaloin itself must be $C_{21}H_{17}O_2(OH)_7$. This formula for the methyl ether is in accord with the analytical data previously recorded (C, 64.5; H, 7.15; OMe, 40.7. Calcd. C, 64.8; H, 7.4; OMe, 41.9). We hope at some future date to be in a position to continue our experiments

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[1] Gardner and Campbell, THIS JOURNAL, **64**, 1378 (1942).

[2] Rosenthaler, *Pharm. Acta Helv.*, **9**, 9 (1934).

[3] Cahn and Simonsen, *J. Chem. Soc.*, 2537 (1932).