A Facile Synthesis of 1,2,3-Benzoxathiazine 2,2-Dioxides

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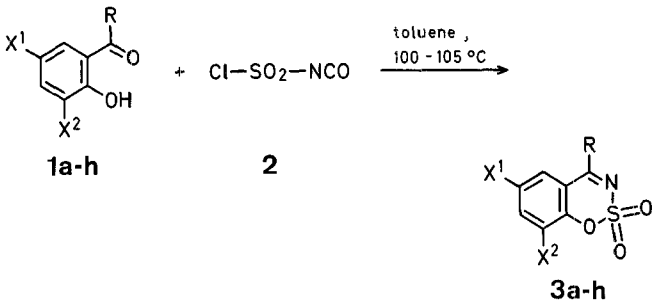
In earlier studies, we reported[1] the reactions of the powerful electrophilic reagent, chlorosulfonyl isocyanate, with substituted 2-aminobenzophenones leading to 4-phenyl-2-(1*H*)-quinazolinones. This work prompted us to investigate further applications of this reagent for the synthesis of e.g. 1,2,3-benzoxathiazine 2,2-dioxides **3** by reacting chlorosulfonyl isocyanate (**2**) with suitably substituted 2-hydroxybenzaldehydes and 2-hydroxyacetophenones or -benzophenones **1**.



1, 3	R	X¹	X²	1, 3	R	X¹	X²
a	H	H	H	e	CH_3	Cl	Cl
b	H	Cl	H	f	C_6H_5	H	H
c	CH_3	H	H	g	C_6H_5	Cl	H
d	CH_3	Cl	H	h	C_6H_5	Cl	Cl

Reaction of chlorosulfonyl isocyanate (**2**) with 2-hydroxybenzaldehydes (**1a, b**), 2-hydroxyacetophenones (**1c-e**), and 2-hydroxybenzophenones (**1f-h**) in toluene at 100–105 °C gave the corresponding benzoxathiazines **3** in yields of 66–83% (Table).

There is only one method reported for the synthesis of the above compounds[2,3,4] which involves the condensation of 2-hydroxyacetophenones and 2-hydroxybenzophenones with large excess of sulfamide. The reaction of 2-hydroxyacetophenone with sulfamide gave 4-methyl-1,2,3-benzoxathiazine 2,2-dioxide[3] in 42% yield, whereas with the present method **3c** is obtained in 68% yield.

The experimental simplicity and the commercial availability of chlorosulfonyl isocyanate make the present method convenient and useful for the preparation of 1,2,3-benzoxathiazine 2,2-dioxides **3**.

1,2,3-Benzoxathiazine 2,2-Dioxides 3; General Procedure:
To a stirred solution of the 2-hydroxy compound **1** (0.046 mol) in toluene (40 ml) at 100–105 °C is added chlorosulfonyl isocyanate (**2**; 4 ml, 0.046

Table. 1,2,3-Benzoxathiazine 2,2-Dioxides (**3a–h**)

Product	Yield [%]	m.p. [°C] (Lit. m.p.)	Molecular formula[a]	I.R. (KBr) ν [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$) δ [ppm]
3a	73	92–94°	$C_7H_5NO_3S$ (183.2)	1590, 1335, 1130	7.3–8.1 (m, 4H); 8.73 (s, 1H)
3b	81	142–143°	$C_7H_4ClNO_3S$ (217.6)	1595, 1335, 1120	7.3–8.0 (m, 3H); 8.95 (s, 1H)
3c	68	119–120° (119–121°)[3]	$C_8H_7NO_3S$ (197.2)	1590, 1340, 1115	2.76 (s, 3H); 7.3–8.1 (m, 4H)
3d	66	134–136°	$C_8H_6ClNO_3S$ (231.6)	1590, 1345, 1120	2.74 (s, 3H); 7.5–8.2 (m, 3H)
3e	68	146–148°	$C_8H_5Cl_2NO_3S$ (266.1)	1600, 1350, 1120	2.71 (s, 3H); 7.60 (d, 1H, J=2 Hz); 7.73 (d, 1H, J=2 Hz)
3f	72	115–116° (115–116°)[4]	$C_{13}H_9NO_3S$ (259.3)	1595, 1340, 1125	—[b]
3g	83	158–159° (156–165°)[4]	$C_{13}H_8ClNO_3S$ (293.7)	1600, 1345, 1125	7.5 (s, 1H); 7.7–8.0 (m, 7H)
3h	75	167–168°	$C_{13}H_7Cl_2NO_3S$ (328.2)	1605, 1340, 1120	—[b]

[a] The microanalyses were in satisfactory agreement with the calculated values: C, ±0.28; H, ±0.17; N, ±0.27.
[b] Aromatic multiplet.

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mol) in toluene (5 ml) over a period of 20 min. Stirring is continued for 3 h at this temperature. The toluene is then removed under vacuum and the residue is added to cold water (50 ml). The solid is filtered, washed with water, and recrystallized from ethanol/methanol to give the desired benzoxathiazine 2,2-dioxide **3** (Table).

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[4] *British Patent* 1073836, Upjohn Co. (1967); *C. A.* **67**, 90818 (1967).