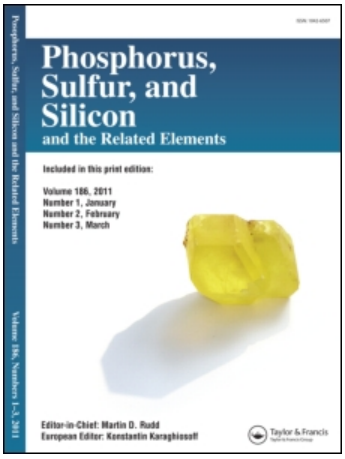

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ETHYLENESULFONANILIDE

Uri Zoller[a]
[a] Division of Chemical Studies, University of Haifa, The School of Education of The Kibbutz, Kiriat Tivon, Israel

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ETHYLENESULFONANILIDE

by

Uri Zoller

Division of Chemical Studies, University of Haifa,
The School of Education of The Kibbutz Movement, Oranim
P.O. Kiriat Tivon, Israel

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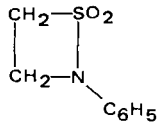
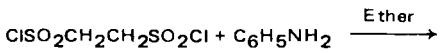
The preparation and the chemistry of ethane-1,2-disulfonyl chloride, has been previously studied and described by Kohler.[1] In his work Kohler claimed the isolation of an "anhydrophenyltaurine" **2** (a four-membered ring β-sultam) from the reaction in the cold of ethane-1,2-disulfonylchloride (**1**) with excess aniline in ether.



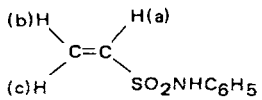
2

This claim was based mainly on the elemental analysis of the product.[1]

In contrast to Kohler's claim, it was found that the reaction of **1** with excess aniline in a cooled ether solution afforded the vinylsulfonamide **3** (which is the non-cyclic isomer of **2**) in 68% yield:



1

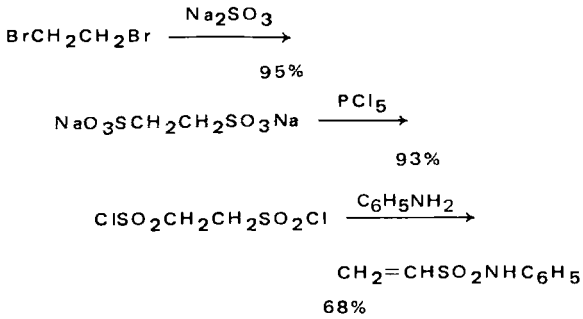


3

The ir spectrum of **3** [(neat); 3250 (NH), 1600 ($CH{=}CH_2$), 1335, 1152 (SO_2N) cm^{-1}] and particularly the nmr spectrum [an ABX type in the vinylic range: δ = 6.78, H(a); 6.36, H(c); 5.9, H(b); and a broad peak at δ = 7.54 (NH) washable with D_2O], unequivocally prove the structure of the product as being identical with that of **3** and not **2**.

Kohler himself was the first to observe that ethane-1,2-disulfonyl chloride reacted with water and alcohols to yield mainly ethylenesulfonic acid.[1] Similarly, ammonium ethylenesulfonate was prepared by treating **1** with ammonia.[2,3] Furthermore, it was shown by others,[4] that esters of ethylenedisulfonic acid cannot be prepared from **1**. Instead, an alcohol in the presence of pyridine or a sodium alkoxide produces ethylenesulfonic acid as does direct hydrolysis or alcoholysis. In all of the above cases, the reaction product of **1** with either a base or a nucleophile contains the vinylic moiety as part of its structure. These results fit very well with the result presented here.

Consequently, the following sequence is suggested as a synthetic alternative route to the ethylenesulfonarylamides otherwise obtained from β-haloethanesulfonyl chlorides:[5]



Experimental Section

Ethane-1,2-disulfonyl chloride (**1**)

Obtained according to Kohler's procedure[1] in 95% yield, mp 91–93°. Ir (KBr): 2995 (CH), 1375, 1160 (SO_2) cm^{-1}. Nmr ($CDCl_3$): δ = 4.26 (s).

Ethylenesulfonanilide (**3**)

To a stirred solution of **1** (2.27 g, 0.01 mol) in anhydrous ethyl ether (50 ml) cooled in an ice bath, was added dropwise aniline (3.73 g) over a period of 30 minutes. The precipitated $C_6H_5NH_2 \cdot HCl$ was separated by filtration, and the ethereal filtrate was washed three times with 1 *N* HCl. Drying of the organic layer ($MgSO_4$), filtration and removal of the solvent under reduced pressure, afforded the crude **3** (1.24 g, 68%). Recrystallization from aqueous ethanol gave pure **3** (mp 68° identical with an authentic sample).[5]

1. E. P. Kohler, *Am. Chem. J.*, **19**, 728 (1897).
2. E. P. Kohler, *Am. Chem. J.*, **20**, 680 (1898).
3. W. F. Whitmore and E. F. Landau, *J. Am. Chem. Soc.*, **68**, 1797 (1946).
4. S. M. McElvain, A. Jelinek, and K. Rorig, *J. Am. Chem., Soc.*, **67**, 1578 (1945).
5. A. A. Goldberg, *J. Chem. Soc.*, 465 (1945).