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Unexpected Reaction of 3,6-Bis(vinylsulfonyl)-1,2,4,5-tetrafluorobenzene with Thiosemicarbazide

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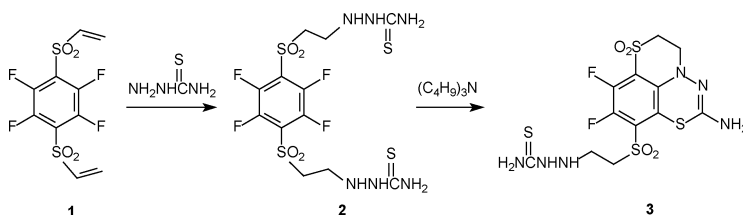
Unexpected Reaction of 3,6-Bis(vinylsulfonyl)-1,2,4,5-tetrafluorobenzene with Thiosemicarbazide

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3,6-Bis(2-thiosemicarbazidoethylsulfonyl)-1,2,4,5-tetrafluorobenzene (**2**) was prepared in up to 77% yield by reacting 3,6-bis(vinylsulfonyl)-1,2,4,5-tetrafluorobenzene (**1**) with a thiosemicarbazide (ratio 1:2) in ethanol or in DMF (20°C). Unexpectedly, with a four-fold amount of thiosemicarbazide at 70–80°C (DMF), the reaction led to the formation of a substituted fused heterocycle, 2-amino-8,9-difluoro-10-(2-thiosemicarbazidoethylsulfonyl)-5,6-dihydrobenzo[h,i]-1,4-thiazino[4,3-d]-1,3,4-thiadiazine-7,7-dioxide (**3**) in 54% yield (Scheme). The use of a strong base, tributylamine, allowed us to obtain the heterocycle **3** from the diadduct **2** in 80% yield along with an 18% yield of 2-amino-8,9-difluoro-5,6-dihydrobenzo[h,i]-1,4-thiazino[4,3-d]-1,3,4-thiadiazine-7,7-dioxide.



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