Bruker AM360 with the Aspect 3000 computer; HRFAB, Kratos MS-80RFA, thiothreitol matrix (Chemical Instrumentation Center, Yale University); [α], JASCO DIP-360 Digital polarimeter. ^{1}H NMR chemical shifts are reported as δ values in ppm relative to DMSO-d_6 (2.49 ppm) or methanol-d_4 (3.31 ppm). ^{13}C NMR chemical shifts are reported as δ values in ppm relative to DMSO-d_6 (39.1 ppm) or methanol-d_4 (49.0 ppm). ^{13}C multiplicities were measured using the DEPT sequence. One bond ^{1}H–^{13}C connectivities were determined via the XHCORR experiment and multiple-bond ^{1}H–^{13}C connectivities were determined through the proton-detected HMBC experiment.

Collection and Taxonomy. A sample of *Spongosorites* n. sp. (Phylum Porifera, Class Demospongiae, Order Halichondrida, Family Halichondriidae, Genus *Spongosorites*, HBOI no. 31-III-89-1-010) was collected by the Johnson-Sea-Link manned submersible at a depth of 292 ft at York Bay, St. Vincent, Grenadines. This new species is a massive, amorphous, thickly encrusting sponge, dark yellow alive and dark brown in ethanol preservative. Vermetid gastropods are associated with and incorporated in the sponge. The consistency is firm but crumbly. The genus is characterized by a distinct dermal layer of smaller spicules arranged tangentially to the surface and a confused choanosomal arrangement of spicules with sporadic spicule tracks (30–100 μm in width) running parallel to the surface. In our sponge, there are two size categories of oxeas, some of which are slightly flexed at the mid-point. This new species is most similar to *S. ruetzleri* (van Soest and Stentoft, 1988)[22] from which it is distinguished by the absence of bromotopsentin. A voucher specimen of the sponge has been deposited at the Harbor Branch Oceanographic Museum, catalog number 003:00544.

Isolation of 1. The frozen sponge (100 g) was extracted exhaustively with ethanol by macerating in a Waring blender. The extract was filtered through a bed of Celite and then concentrated to an orange oil by distillation under reduced pressure. The residue was chromatographed under vacuum column chromatographic conditions on an RP-18 stationary phase. The column used had a volume of 360 mL and was 4 cm in height. The column was eluted with a step gradient of acetonitrile–water–trifluoroacetic acid. The extract was applied adsorbed onto a small amount (5 g) of RP-18 packing as a slurry in water containing 0.05% trifluoroacetic acid (TFA) to the top of the column. The column was eluted as follows: fraction 1, 500 mL of water containing 0.05% TFA; fraction 2, 250 mL of water containing 0.05% TFA; fraction 3, 200 mL of water–ACN–TFA (160:40:0.1); fraction 4, 200 mL of water–ACN–TFA (120:80:0.1); fraction 5, 200 mL of water–ACN–TFA (80:120:0.1); fraction 6, 200 mL of water–ACN–TFA (40:160:0.1); fraction 7, 500 mL of ACN. Dragmacidin d eluted in fractions 3, 4, and 5 dependent upon loading of the column. Yield from 100 g of sponge: 534 mg. ^{1}H NMR: see Table I. ^{13}C NMR: see Table I. IR: FT IR (neat, microscope) ν_{max} (cm^{-1} 3165 broad, 1678, 1637, 1531, 1447, 1408, 1244, 1200, 1136, 955, 806. UV: EtOH λ_{max} 213 (47 870), 270 sh, 278 (14 470), 383 (20 740) after addition of 1 drop of HCl to a 2-mL cell; 214 (54 000), 280 (16 400), 452 (19 946). FABMS: 530/532 (M^+), 449/447 (M^+ – (2-aminoimidazole)). HRFABMS: M^+_{obsd} 532.0916, M^+_{calcd} 532.0922.

Biological Methods: Antimicrobial Assays. Minimum inhibitory concentrations (MICs) were determined by standard microdilution broth techniques[23] in a total volume of 50 μL. The growth media used were as follows: *Candida albicans*, Sabouraud dextrose broth; *Cryptococcus neoformans*, Emmon's modification of Sabouraud dextrose broth; Bacteria, Mueller-Hinton broth supplemented with Ca^{2+} and Mg^{2+}. Plates were incubated at 37 °C for either 24 h (bacteria and *C. albicans*) or 48 h (*C. neoformans*). The MIC was determined as the lowest concentration of the drug which completely inhibited growth.

Antitumor and Antiviral Assays. The FeLV assay is an ELISA assay developed by Dr. Sue Cross at HBOI; full details have been published elsewhere.[24] The antitumor assays were run using standard protocols in 96-well plates and MTT to detect cytotoxicity.[25]

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1-(Benzenesulfonyl)- and 1-(*p*-Toluenesulfonyl)-3-methylimidazolium Triflates: Efficient Reagents for the Preparation of Arylsulfonamides and Arylsulfonates

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Arylsulfonyl substituents have served as effective protecting groups for both oxygen and nitrogen functionalities.[1] As arylsulfonates and arylsulfonamides they provide strong chromophores and are stable to a variety of reaction conditions.[1] Subsequently, the arylsulfonyl group can be removed from the amine[2] and the arylsulfonate can be hydrolyzed, displaced, or eliminated. As a consequence of this versatility of sulfonamides, the arylsulfonyl group has found particular application for the masking of amine and guanidine functions[1] and for the protection of α-amino acids in which the carbonyl group is destined to undergo reaction with an organometallic reagent.[3]

The preparation of toluenesulfonyl and benzenesulfonyl derivatives generally relies on the use of the corresponding sulfonyl chloride or anhydride in the presence of pyridine or aqueous base in a Schotten–Bauman type reaction.[1] These procedures fail or are limited when the nucleophiles are insufficiently nucleophilic or sterically encumbered. Side reactions are possible due to the presence of base or the liberated chloride nucleophile, especially under forcing conditions with relatively non-nucleophilic substrates. It would thus be highly desirable to have an arylsulfonating reagent that would operate under mild conditions in the absence of base and competing nucleophile and that could sulfonate relatively non-nucleophilic substrates. Additionally, it would be advantageous if this reagent could be applied in an organic solvent under homogeneous conditions rather than in an aqueous or a mixed phase. With these properties, such a reagent also would be useful for

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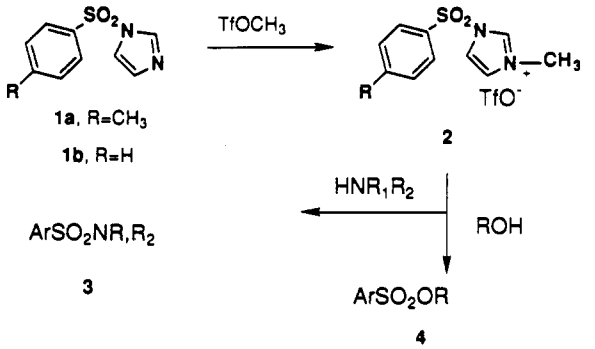
the preparation of alkyl sulfonates which are particularly susceptible to migration, displacement and elimination side reactions.

We have shown in previous work from this laboratory that acylimidazolium salts derived from the reagent 1,1'-carbonylbis-(1,3-dimethylimidazolium) triflate (CBMIT) are highly reactive acyl transfer species.[4] In addition, we have shown that the imidazolium reagent prepared from (benzyloxycarbonyl)imidazole is a greatly enhanced Cbz-transfer reagent to poorly nucleophilic nucleic acid bases.[5] Thus, we projected that (arylsulfonyl)methylimidazolium salts might similarly provide a very reactive arylsulfonyl transfer reagent capable of substituting even poorly nucleophilic substrates under very mild conditions in the absence of base in organic solvents. An additional design feature of this highly reactive arylsulfonating reagent would be that the counterion be nonbasic and non-nucleophilic. We herein describe the easily prepared (toluenesulfonyl)-3-methylimidazolium triflate (**2a**) and (benzenesulfonyl)-3-methylimidazolium triflate (**2b**) and their use in preparing arylsulfonyl substituted amines, phenols, and alcohols under mild conditions.

As used previously to alkylate the imidazole nitrogens of CDI in the preparation of CBMIT,[4] the readily available and excellent alkylating agent methyl triflate was chosen for the alkylation of (toluenesulfonyl)imidazole and (benzenesulfonyl)imidazole, since the trifluoromethylsulfonate (triflate) anion best satisfies the criterion for being nonnucleophilic. These starting (toluenesulfonyl)- and (benzenesulfonyl)imidazolides, **1a** and **1b**, were easily prepared in centigram quantities from the corresponding sulfonyl chloride with imidazole in THF.[6] When the imidazolides **1a** and **1b** were treated in THF solution with 100 mol % of methyl triflate, the charged species **2a** and **2b** were formed immediately. Alcohols or amines, treated with these (arylsulfonyl)imidazolium salts, proceeded to form sulfonate and sulfonamide with ease. Optimized isolated yields in a number of test cases ranged from 90 to 100%, with reaction times ranging from minutes to hours with simple isolation and side product removal.



To illustrate the reagent's versatility, a number of sulfonamides and sulfonates were synthesized with the (arylsulfonyl)imidazolium reagents **2a** and **2b**, with the emphasis on difficult cases (Table I). Since the reaction medium could potentially become acidic from the presence of traces of triflic acid in stored batches of reagent, 1-methylimidazole was added as an acid scavenger. This modification often improved the yields of sulfonates.

In the preparation of sulfonamides, both the hindered secondary amines diisopropylamine and proline were arylsulfonated in good yields under mild conditions (entries 1 and 2). The preparation of alkyl benzenesulfonates proceeded in excellent yield as demonstrated by sulfonating the sterically hindered, secondary alcohol *l*-menthol (entry 7) and tetrasulfonating methyl α-D-glucoside (entry 8). The high reactivity of these arylsulfonyl transfer reagents also has allowed the facile selective sulfonation of a primary alcohol in the presence of secondary alcohols.[7]

We next investigated the arylsulfonation of phenols using phloroglucinol, catechol, and 2,6-dimethylphenol as substrates.[8] Phloroglucinol was triarylsulfonated in quantitative yield in relatively short reaction times (entries 3 and 4) in contrast with the reaction conditions of several days at 60–70 °C in pyridine required for tosyl chloride with yields in the range of 60–70%.[9] The aryl 1,2-dibenzenesulfonate substitution was demonstrated with catechol, which also proceeds in quantitative yield (entry 5). However, slightly lower yields were observed with the benzenesulfonylation of sterically hindered 2,6-dimethylphenol.

The simplicity of reagent preparation and product isolation, along with high yields, mild, nonaqueous, homogeneous conditions and enhanced reactivity, associated with these arylsulfonating reagents should make them applicable for the preparation of a wide variety of sulfonamides and sulfonates.

Experimental Section

Melting points are uncorrected. ^1H and ^{13}C NMR spectra were determined at 400 or 500 MHz in $CDCl_3$ unless otherwise indicated and are expressed as a downfield shift from internal Me_4Si; coupling constants, *J*, are given in hertz. Silica gel 60, 230–400 mesh (EM Reagents), was used for low-pressure chromatography (LPC). Analytical thin-layer chromatography (TLC) was done with aluminum-backed silica plates (Merck). Tetrahydrofuran (THF) was freshly distilled from potassium/benzophenone. *N*-methylimidazole was distilled from calcium hydride. Methyl trifluoromethanesulfonate was prepared as described.[10] 1-Benzenesulfonylimidazole (**1b**) and 1-(4-methylbenzenesulfonyl)imidazole (**1a**, tosylimidazole) were prepared as described from the corresponding sulfonyl chlorides.[6]

1-(Benzenesulfonyl)-3-methylimidazolium triflate (2b) and 1-(toluenesulfonyl)-3-methylimidazolium triflate (2a) were prepared by adding methyl triflate (1.2 mmol, 180 mg) dropwise to the corresponding (arylsulfonyl)imidazole (1.2 mmol, 250 mg and 266 mg, respectively) dissolved in THF (10 mL) at 0 °C. The cloudy solution was stirred for 30 min at 0 °C and then used directly as the sulfonylating reagent.

(–)-Menthyl Benzenesulfonate (4e). A solution of (–)-menthol (1.0 mmol, 156 mg, sublimed) and *N*-methylimidazole (0.80 mL, 1.0 mmol, 82 mg) in THF (5 mL) was slowly transferred via syringe to a stirred solution at 0 °C of 1-(benzenesulfonyl)-3-methylimidazolium triflate (**2b**) prepared as above from 1.2 mmol of (benzenesulfonyl)imidazole (**1b**). The cooling bath was removed, stirring was continued for 24 h, water (30 mL) was added, and the mixture was extracted with ethyl acetate (3 × 30 mL). The combined organic phase was washed with 0.5 M H_3PO_4 (30 mL), saturated $NaHCO_3$ (30 mL), and brine (30 mL), dried over $MgSO_4$, filtered, and evaporated. Sublimation (70–80 °C/0.01 mm) of the residue afforded 280 mg, 95% yield, of **4b**: mp 85–86 °C; $[\alpha]^{20}_D$ –73.1° (*c* 1.0, $CHCl_3$). Anal. Calcd for $C_{16}H_{24}O_3S$: C, 64.8; H, 8.2. Found: C, 64.8; H, 8.4.

***N*-(Benzenesulfonyl)diisopropylamine (3a).** A solution of diisopropylamine (1.0 mmol, 101 mg, freshly distilled from CaH_2) in THF (5 mL) was added to the 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 1.2 mmol), and the

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Table I. Arylsulfonamides and Arylsulfonates

substrate	mol % imidazolium reagent 2[a]	coupling time[b] (h)	yield (%)	product
1. diisopropylamine	120	21	71	*N*-(benzenesulfonyl)diisopropylamine (**3a**)
2. proline	400	10	80	*N*-(benzenesulfonyl)proline (**3b**)
3. 1,3,5-trihydroxybenzene	350	15	100	1,3,5-tris(TsO)benzene (**4a**)
4. 1,3,5-trihydroxybenzene	350	5	100	1,3,5-tris(BsO)benzene (**4b**)
5. catechol	240	1.5	100	1,2-bis(BsO)benzene (**4c**)
6. 2,6-dimethylphenol	125	1.5	76	[(benzenesulfonyl)oxy]-2,6-dimethylbenzene (**4d**)
7. (−)-menthol	125	24	95	(−)-menthyl benzenesulfonate (**4a**)
8. methyl α-D-glucoside	800	21	100	methyl *O*-tetrakis(benzenesulfonyl)-α-D-glucoside (**4f**)

[a] Formation of the imidazolium reagent proceeded for 30–35 min; for entries 3–8, 100 mol % of *N*-methylimidazole was then added. [b] All couplings began at 0 °C and were then allowed to reach rt.

mixture was stirred at 0 °C for 2 h and rt for 21 h. Isolation as for **4a** followed by passage through a short silica column (20% EtOAc/hexanes) afforded 170 mg, 71% yield, of **3a**: mp 88–89 °C; ^{1}H NMR δ 7.87 (d, 1 H, *J* = 6.0, *p*-ArH), 7.48 (m, 4 H, *o*-, *m*-ArH), 3.71 (septet, 2 H, *J* = 6.8, isopropyl-H), 1.25 (d, 12 H, *J* = 6.8, isopropyl-CH_3); ^{13}C NMR δ 142.5, 131.8, 128.7, 127.1, 48.6, 21.9; R_f 0.36 (20% EtOAc/hexanes). Anal. Calcd for $C_{12}H_{19}NO_2S$: C, 59.7; H, 7.9; N, 5.8. Found: C, 59.6; H, 8.0; N, 5.6.

***N*-(Benzenesulfonyl)proline (3b).** To a slurry of *S*-proline (230 mg, 2.0 mmol) in THF (12 mL) was added the 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 8.6 mmol). Following stirring at 0 °C for 1 h and rt for 10 h, the reaction mixture was diluted with half-saturated Na_2CO_3 (40 mL) and extracted with ethyl acetate (2 × 30 mL). The aqueous phase was adjusted to pH 2 with 85% H_3PO_4 and extracted with 2-propanol/$CHCl_3$ (1/4, 3 × 30 mL). The resulting combined organic phase was washed with brine (25 mL), dried over $MgSO_4$, filtered, and evaporated, affording 400 mg, 80% yield, of crystalline *N*-(benzenesulfonyl)proline, mp 83–85 °C (lit.[3b] mp 84–86 °C).

1,3,5-Tris[(*p*-toluenesulfonyl)oxy]benzene (4a). To a solution of 1,3,5-trihydroxybenzene (126 mg, 1.0 mmol) in THF (20 mL) containing *N*-methylimidazole (42 mg, 0.5 mmol) was added the 1-tosyl-3-methylimidazolium triflate reagent (**2a**, 3.55 mmol). Following stirring at 0 °C for 1 and 10 h at rt, the now homogenous solution was subjected to the isolation procedure used for **4a**. Chromatography (20% EtOAc/hexanes) afforded 590 mg, 100% yield, of **4a**: mp 84–86 °C (lit.[11] mp 82–83 °C); R_f 0.53 (50/50 EtOAc/Hex).

1,3,5-Tris[(benzenesulfonyl)oxy]benzene (4b). To a solution of 1,3,5-trihydroxybenzene (151 mg, 1.2 mmol) in THF (25 mL) containing *N*-methylimidazole (82 mg, 1.0 mmol) was added the 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 3.6 mmol). Following stirring at 0 °C for 30 min and rt for 6 h and isolation as for **4a**, LPC (40% EtOAc/hexanes) afforded 655 mg, 100% yield, of **4b**: mp 108–110 °C; TLC (20% EtOAc/hexanes) R_f 0.36. Anal. Calcd for $C_{24}H_{18}O_9S_3$: C, 52.7; H, 3.3. Found: C, 53.4; H, 3.2.

1,2-Bis[(benzenesulfonyl)oxy]benzene (4c). Following the procedure for preparation of **4b**, from the 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 2.4 mmol) and a solution of 1,2-dihydroxybenzene (110 mg, 1.0 mmol) and *N*-methylimidazole (82 mg, 1.0 mmol) in THF (10 mL), was obtained 1,2-bis[(benzenesulfonyl)oxy]benzene (**4c**) in 100% yield, 390 g, after LPC (30% EtOAc/hexanes): mp 153–155 °C; ^{1}H NMR δ 7.74 (m, 6 H), 7.67 (m, 2 H), 7.49 (m, 6 H); ^{13}C NMR δ 141.1, 135.1, 134.5, 129.1, 128.6, 128.4, 124.5. Anal. Calcd for $C_{18}H_{14}O_6S_2$: C, 55.4; H, 3.6. Found: C, 55.6; H, 3.6.

1-[(Benzenesulfonyl)oxy]-2,6-dimethylbenzene (4d). A solution of 2,6-dimethylphenol (122 mg, 1.0 mmol) and *N*-methylimidazole (82 mg, 1.0 mmol) in THF (5 mL) and 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 1.25 mmol) were treated as in the preparation of **4b**. LPC (20% EtOAc/hexanes) and sublimation (80 °C/0.05 Torr) afforded 190 mg, 76% yield, of the benzenesulfonate **4d**: mp 79–80 °C. Anal. Calcd for $C_{14}H_{14}O_3S$: C, 64.1; H, 5.4. Found: C, 64.2; H, 5.3.

Methyl *O*-Tetrakis(benzenesulfonyl)-α-D-glucoside (4f). A slurry of methyl α-D-glucoside (195 mg, 1.0 mmol) and *N*-methylimidazole (330 mg, 4.0 mmol) in THF (10 mL) and the 1-(benzenesulfonyl)-3-methylimidazolium triflate reagent (**2b**, 8 mmol) were treated by the procedure for preparation of **4b**. LPC (EtOAc/hexanes (1/1)) afforded 755 mg, 100% yield, of the tetrabenzenesulfonate **4f**: mp 69–71 °C; $[\alpha]^{20}_D$ +38.1 (*c* 0.5, $CHCl_3$). Anal. Calcd for $C_{31}H_{30}O_{14}S_4$: C, 49.3; H, 4.0. Found: C, 49.4; H, 3.9.

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A Convenient and General Preparation of *N*-Sulfonylimines

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In recent studies we have detailed the effective 4π participation of α,β-unsaturated *N*-sulfonylimines in diastereoselective inverse electron demand Diels–Alder reactions (≥20:1 endo:exo)[2-7] which has proven to be a productive addition to the limited number of useful 1-aza-1,3-butadienes.[8-15] In these studies, the *N*-sulfonylimines have been shown to constitute stable, nonbasic electron-deficient imine derivatives capable of simple isolation and purification. In our initial studies, the *N*-sulfonylimines were prepared through the clean, homolytic rearrangement of in situ generated *O*-sulfinyl derivatives of aldehyde or ketone oximes[16-18] or through the direct

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