Palladium-Catalyzed Cross-Coupling Alkylation of Arenediazonium *o*-Benzenedisulfonimides

Margherita Barbero, Silvano Cadamuro, Stefano Dughera*

Dipartimento di Chimica Generale ed Organica Applicata, Università di Torino, Corso Massimo d'Azeglio 48, 10125 Torino, Italy Fax +39(011)6707642; E-mail: stefano.dughera@unito.it

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Abstract: Arenediazonium *o*-benzenedisulfonimides were reacted with tetramethyltin, tetrabutyltin or trialkylboranes. The reactions, carried out in the presence of palladium(II) derivatives as precatalysts, gave the methylation and alkylation products with good overall conversions. The *o*-benzenedisulfonimide was recovered in high yield from all the reactions and could be recycled for the preparation of other salts.

Key words: palladium, Stille reaction, Suzuki reaction, crosscoupling, diazo compounds

In the course of evaluating the synthetic scope of a new family of diazonium salts that are particularly stable in the dry state, namely the arenediazonium *o*-benzenedisulfonimides **1**, we focused our attention on the reactivity of the salts **1** as electrophilic reagents in palladium-catalyzed reactions.¹ In particular, we have studied cross-coupling reactions of **1** with aryltin compounds under Stille conditions,² in order to obtain biaryls.³

Though the literature regarding the palladium-catalyzed cross-coupling reactions between diazonium salts and aromatic or alkenyl organometallic compounds is quite extensive,⁴ to our knowledge, only three examples⁵ have been reported concerning the reactions of diazonium salts with alkyl organometallic compounds, in particular organotin derivatives^{5a,b} or esters of alkylboronic acids.^{5c} These reactions between arenediazonium tetrafluoroborates,^{5a} hexafluorophosphates^{5a} or hydrogen sulfate^{5b} and tetramethyltin, often provide low yields.

The present paper describes an improved procedure for the synthesis of the toluenes **4a–m** or butylarenes **5b**, **5d– f** and **5h**, by the palladium-catalyzed cross-coupling reactions between arenediazonium *o*-benzenedisulfonimides **1a–m** and tetramethyltin (**2**) or tetrabutyltin (**3**) (Scheme 1). Furthermore, we report a new procedure for the synthesis of butylarenes **5a–f**, **5h** and **5n** or ethylarenes **11a–f**, **5h** and **5n**, through palladium-catalyzed reactions between arenediazonium *o*-benzenedisulfonimides **1a–f**, **1h** and **1n** and tributylborane (**10**) or triethylborane (**9**) (Scheme 2).

A range of salts of type **1** were reacted with tetramethyltin (**2**) in the presence of a catalytic amount of palladium(II) acetate (2.5 mol%) using either tetrahydrofuran or aceto-

SYNTHESIS 2008, No. 3, pp 0474–0478 Advanced online publication: 10.01.2008 DOI: 10.1055/s-2008-1032027; Art ID: Z24907SS © Georg Thieme Verlag Stuttgart · New York nitrile as solvent. The results are listed in Table 1; with tetrahydrofuran, the desired products **4** were usually obtained in high yields; however, besides the cross-coupling products, the presence of arenes **6** were nearly always observed and the separation of these by-products was often very difficult (Table 1; entries 1, 14, 19, 22, 26 and 30).



Scheme 1

Products **6** were probably derived from a side reaction of salts **1**, namely hydrodediazoniation.⁶ The best results were obtained using acetonitrile as solvent: under these conditions we observed, except for a few cases (Table 1; entries 4 and 25), the formation of **4** in very good yields both from diazonium salts bearing electron-donating groups and from salts bearing electron-withdrawing groups. Moreover, it seems that the reaction was not influenced by steric effects since the hindered compound **4j** (Table 1; entry 24) was obtained in good yields. This reaction is chemoselective and, in fact, no traces of xylenes were detected in the reactions of salts **1** bearing a halogen atom that could potentially react like diazonium groups

Entry	Product	Solvent	Temp (°C)	Time (min)	Yield ^{a,b} (%)	MS <i>m/z</i> (%) []	M ⁺] Mp ^c /bp (°C/torr)	Lit. mp/ bp (°C/torr)
1	4 a	THF	r.t.	480	d	148 (57)	215/760	82.7-83.3/137
2		MeCN	40	240	77			
3	4b	THF	r.t.	420	82	122 (45)	193/760	e
4		MeCN	40	_f	-			
5	5b	THF	40	120	37	164 (35)	225/760	220-224/760 ⁸
6	4c	THF	40	180	85	137 (100)	175/760	e
7		MeCN	40	30	93 (72) ^g			
8	4d	THF	40	15	90	137 (100)	225/760	e
9		MeCN	40	45	85 (62) ^g			
10	5d	THF	40	180	44	179 (80)	270/760	275/752 ⁹
11	4e	THF	40	30	100	137 (100)	53–55	e
12		MeCN	40	40	88 (81) ^g			
13	5e	THF	40	60	41	179 (85)	214/760	143-145/1510
14	4f	THF	r.t.	210	d	172 (95)	147/760	e
15		MeCN	40	50	80 (38) ^g			
16	5f	THF	40	90	15	214 (68)	223/760	e
17	4 g	THF	40	45	83	218 (100)	202/760	e
18		MeCN	40	960	86 (74) ^g			
19	4h	THF	r.t.	990	d	117 (100)	189/760	
20		MeCN	40	100	89			
21	5h	THF	40	120	20	159 (65)	154/760	e
22	4i	THF	r.t.	100	d	150 (45)	158/760	e
23		MeCN	40	90	90			
24	4j	THF	40	20	87	252 (80)	164/760	58-60/0.811
25		MeCN	40	_f	_			
26	4k	THF	40	30	d	151 (100)	186/760	130/12 ¹²
27		MeCN	40	420	84			
28	41	THF	40	15	68	156 (100)	211/760	214-218/76013
29		MeCN	40	720	62			
30	4m	THF	40	420	d	196 (72)	29–30	2814
31		MeCN	40	120	93			

 Table 1
 Selected Physical Properties of Synthesized Toluenes 4 and Butylarenes 5

^a Yields refer to the pure (GC, GC-MS, TLC, ¹H NMR) products isolated by column chromatography (PE–Et₂O, 9:1) with the exception of entries 11, 17, 20, 23 where the crude residue was virtually pure.

^b GC, GC-MS, TLC analyses of the crude residue showed, besides the main products **4** or **5**, the presence of arenes **6** (Method A; not quantified) and traces of biaryls (Methods A and B).

^c Crystallization solvent: MeOH.

 d It was impossible to separate products 4 from 6 by column chromatography.

^e The products were identical to commercially available samples of analytical purity (Sigma-Aldrich).

^f After 24 h, an azo-coupling test with 2-naphthol showed that the reactions were incomplete.

^g Yields reported in brackets refer to the isolated and purified products **4** reported in the literature,⁵ obtained by the reactions carried out using the corresponding tetrafluoroborates, hexafluorophosphates or hydrogen sulfates instead of salts **1**.

(Table 1; entries 14, 15, 17, 18, 24, 25, 28, 29). We also recovered *o*-benzenedisulfonimide **8** (\sim 80% yield) from all the reactions, which was suitable for reuse in the preparation of salts **1**.

Under our conditions, reacting salts **1b**, **1d**–**f** and **1h** with tetrabutyltin (**3**) in tetrahydrofuran, in the presence of a catalytic amount of palladium(II) acetate (2.5 mol%), we obtained low yields of butylarenes **5b**, **5d**–**f** and **5h**, respectively (Table 1; entries 5, 10, 13, 16 and 21).

It must be stressed that, beside the cross-coupling products, we always detected arenes **6** which, in this case, probably derived from a β -H elimination reaction. In order to both improve the yield of compounds **5** and to avoid the use of toxic tin derivatives, and based on literature reports^{4a-d} regarding palladium-catalyzed cross-coupling reactions between aryl or alkenylboronic acids and diazonium salts, we investigated the reaction of salts **1** with methyl or butylboronic acids.

However, since this approach failed, we turned our attention to readily available alkylboranes **9** and **10** (Scheme 2) in order to obtain alkylarenes **5** and **11** under the Suzuki protocol.¹⁵ In preliminary reactions (see experimental section), we reacted salt **1e** with triethylborane (**9**) in the presence of three different palladium precatalysts. With palladium(II) acetate or dichlorobis(triphenylphosphine)palladium(II), the main product was nitrobenzene **6e**, derived from β -H elimination.¹⁶ In contrast, with [1,1'bis(diphenylphosphino)ferrocene]dichloropalladium(II) (PdCl₂dppf), we obtained only traces of **6e** and the main product was **11e**.



 $Q = Pd(OAc)_2 \text{ or } (Ph_3P)_2PdCl_2 \text{ or } Pd(Cl_2)dppf$





Having found the optimal conditions, variously substituted arenediazonium *o*-benzenedisulfonimides **1** were reacted with **9** or **10**. All the reactions were carried out in tetrahydrofuran, at room temperature for 30 min (Scheme 2). Table 2 shows that products **5a–f**, **5h**, **5n** or **11a–f**, **11h**, **11n** were obtained in very good yields. However, the reaction was not completely chemoselective; in fact, with salt **1f** (Table 2; entries 11 and 12) we detected

Table 2Selected Physical Properties of Synthesized Ethylarenes 11and Butylarenes 5

Entry	Product	Yield ^{a-c} (%)	MS <i>m</i> / <i>z</i> (%) [M ⁺]	Bp (°C/torr)	Lit. bp (°C/torr)
1	11a	83	162 (57)	221/760	_17,,d
2	5a	64	190 (45)	227/760	224–225.5/ 759 ¹⁸
3	11b	75	136 (48)	194/760	e
4	5b	85	164 (35)		
5	11c	79	151 (40)	232/760	e
6	5c	70	179 (45)	258/760	260/760 ⁹
7	11d	82	151 (55)	249/760	115.5/1319
8	5d	81	179 (80)		
9	11e	87	151 (45)	251/760	1251212
10	5e	85	179 (85)		
11	11f	$74^{\rm f}$	186 (55)	207/760	97–98/30 ²⁰
12	5f	77 ^g	214 (68)		
13	11h	88	131 (50)	156/760	e
14	5h	81	159 (65)		
15	11n	87	120 (45)	155/760	e
16	5n	71	148 (57)		

^a Yields refer to the pure (GC, GC-MS, TLC, ¹H NMR) products isolated by column chromatography (PE–Et₂O, 9:1).

^b GC, GC-MS, TLC analyses of the crude residues show, besides the main products **11** or **5**, the presence of traces of arenes **6**.

^c The reaction time was 30 min.

^d The product has previously been reported in the literature but no bp was given.

^e The products were identical to the commercially available samples of analytical purity (Sigma–Aldrich).

^f GC, GC-MS, TLC analyses of the crude residue show the presence of traces of 1,4-diethylbenzene {MS (EI, 70 eV): m/z (%) = 134 (58) [M⁺]}.

^g GC, GC-MS, TLC analyses of the crude residue show the presence of traces of 1,4-dibutylbenzene {MS (EI, 70 eV): m/z (%) = 190 (45) [M⁺]}.

traces of 1,4-diethyl- or 1,4-dibutylbenzene (GC-MS). In these cases compound **8** was also recovered (~70% yield).

In conclusion, the palladium-catalyzed alkylation of arenediazonium *o*-benzenedisulfonimides **1** is general, easy and safe to carry out, and supplies toluenes **4** or alkylarenes **5** or **11** in good yields. Furthermore, the use of the salts **1** instead of the corresponding tetrafluoroborates presents some advantages, namely in the ease of preparation, the high stability of the salts and the possibility of recovering *o*-benzenedisulfonimide at the end of the reactions. Unlike tetrafluoroboric acid, *o*-benzenedisulfonimide is an acid that presents no risk and can be reused to prepare salts **1**. Column chromatography and TLC were performed on Merck silica gel 60 (70–230 mesh ASTM) and GF 254, respectively. Petroleum ether (PE) refers to the fraction boiling in the range 40–70 °C. ¹H NMR spectra were recorded on a Bruker Avance 200 spectrometer. Mass spectra were recorded on an HP 5989B mass-selective detector connected to an HP 5890 GC cross-linked methyl silicone capillary column. Room temperature (r.t.) was 20–25 °C. Chromatographic solvent, yields, physical and spectral data (MS) of the pure (GC, GC-MS, TLC, ¹H NMR) isolated toluenes **4a–m**, butylarenes **5a–f**, **5h**, **5n** and ethylarenes **11a–f**, **11h**, **11n** are reported in Table 1 and Table 2.

Structures of all the products obtained in the course of this research were confirmed by comparison of their physical (mp or bp) and spectral data with those reported in the literature or with those of the commercially available samples (Sigma–Aldrich) of analytical purity.

 $\begin{array}{ll} Me_4Sn, Bu_4Sn, Et_3B \ solution \ (1.0 \ M \ in \ THF), \ Bu_3B \ solution \ (1.0 \ M \ in \ THF), \ MeB(OH)_2, \ BuB(OH)_2, \ Pd(OAc)_2, \ (Ph_3P)_2PdCl_2, \\ Pd(Cl_2)dppf \cdot CH_2Cl_2, \ and \ all \ the \ anilines, \ were \ purchased \ from \ Sigma-Aldrich; \ Dowex \ 50X8 \ ion-exchange \ resin \ was \ purchased \ from \ Fluka. \end{array}$

Arenediazonium *o*-benzenedisulfonimides 1a-n were prepared as described previously.²¹ The crude salts were virtually pure and were used without further crystallization.

CAUTION! In our laboratory there was no case of sudden decomposition during the preparation, purification and handling of salts **1**. Nevertheless, it must be borne in mind that all diazonium salts in the anhydrous state are potentially explosive. Therefore, they must be carefully stored and handled.

$\label{eq:2-Methyl-4-nitrobenzenediazonium} \textit{o-Benzenedisulfonimide} \ (1k)$

Yield: 92%; mp 169-170 °C (dec.) (HCO₂H).

¹H NMR (200 MHz, CF₃CO₂D): δ = 2.35 (s, 3 H), 7.41–7.45 (m, 1 H, H_{arom}), 7.48–8.25 (m, 6 H, H_{arom}).

 ^{13}C NMR (50 MHz, CF₃CO₂D): δ = 18.9, 110.8, 121.5, 126.1, 129.7, 134.9, 135.3, 136.9, 138.1, 148.0.

Anal. Calcd for $C_{13}H_{10}N_4O_6S_2$: C, 40.84; H, 2.64; N, 14.65; S, 16.77. Found: C, 40.93; H, 2.59; N, 14.62; S, 16.68.

3-Chloro-4-methoxybenzenediazonium *o***-Benzenedisulfonimide** (11)

Yield: 99%; mp 113–114 °C (dec.) (HCO₂H).

¹H NMR (200 MHz, CF₃CO₂D): δ = 3.75 (s, 3 H), 6.80–6.82 (m, 1 H, H_{arom}), 7.02–7.25 (m, 2 H, H_{arom}), 7.58–8.05 (m, 4 H, H_{arom}).

¹³C NMR (50 MHz, CF₃CO₂D): δ = 55.9, 115.1, 122.8, 127.8, 129.8, 130.6, 135.0, 136.9, 138.9, 154.7.

Anal. Calcd for $C_{13}H_{10}ClN_3O_5S_2$: C, 43.40; H, 2.80; Cl, 9.85; N, 3.89; S, 17.82. Found: C, 43.36; H, 2.82; Cl, 9.88; N, 3.85; S, 17.86.

Reactions of Arenediazonium $o\mbox{-}Benzenedisulfonimides 1$ with Tetraalkyltins 2 or 3

Synthesis of 4-Nitrotoluene (4e); Typical Procedures

With THF: 4-Nitrobenzenediazonium *o*-benzenedisulfonimide (1e; 3.68 g, 10.0 mmol) was added in one portion with vigorous stirring to a solution of Me₄Sn (2; 1.97 g, 11.0 mmol) in THF (15 mL) at 40 °C. Pd(OAc)₂ (0.25 mmol, 0.05 g) was added in one portion and stirring at 40 °C was maintained for 30 min. Completion of the reaction was confirmed by the absence of azo-coupling with 2-naphthol. GC, GC-MS, and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed 4-nitrotoluene {4e; MS (EI, 70 eV): m/z (%) = 137 (100) [M⁺]} to be the major product. Furthermore, GC-MS analysis showed the presence of *o*-benzenedisulfonylimidotrimethyltin {7;

MS (EI, 70 eV): m/z (%) = 368 (22) [M – Me]⁺}. All attempts to isolate compound **7** failed. The solution was poured into Et₂O–H₂O (200 mL, 1:1), the aqueous layer was separated and extracted with Et₂O (50 mL). The combined organic extracts were washed with H₂O (2 × mL) and dried (Na₂SO₄). After removal of the solvent under reduced pressure, the crude residue was the virtually pure title compound **4e** (GC, GC-MS, TLC, ¹H NMR). The aqueous layer and aqueous washings were collected and evaporated under reduced pressure and the black tarry residue was passed through a column of Dowex 50X8 ion-exchange resin (1.6 g/1 g of product) using H₂O (~50 mL) as eluent. After removal of H₂O under reduced pressure, virtually pure (¹H NMR) *o*-benzenedisulfonimide (**8**) was recovered (1.77 g, 81% yield); mp 192–194 °C (toluene) (Lit.²¹ 192–194 °C).

According to the procedure described above, **1e** (3.68 g, 10.0 mmol) was treated with Bu₄Sn (**3**). GC, GC-MS, and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed 4-nitrobutylbenzene {**5e**; MS (EI, 70 eV): m/z (%) = 179 (37) [M⁺]} and nitrobenzene {**6e**; MS (EI, 70 eV): m/z (%) = 123 (100) [M⁺]} as major products besides *o*-benzenedisulfonylimidotributyltin {**7**; MS (EI, 70 eV): m/z (%) = 452 (11) [M⁺ – Bu]} as minor product. **5e** was obtained (0.74 g, 41% yield) by chromatography (PE–Et₂O, 9:1) of the crude residue. Formation of the volatile compound **6e** was not quantified.

No traces of **5e** were detected when the reaction was carried out with dioxane or MeCN as solvents or with $Pd(Cl_2)dppf$ as precatalyst (0.25 mmol, 0.20 g).

With MeCN: Conducted as above, but using MeCN as solvent. GC,GC-MS and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed **4e** to be formed as the major product, along with traces of 4,4'-dinitrobiphenyl {MS (EI, 70 eV): m/z (%) = 244 (100) [M⁺]} and **6e**. Pure **4e** was obtained (1.20 g, 88%) by chromatography (PE–Et₂O, 9:1) of the crude residue; **8** was also recovered (1.74 g, 79% yield).

Details of the reactions and yields of the pure toluenes **4a–m** and butylarenes **5b,5d–f** and **5h** are listed in Table 1.

Reactions of Arenediazonium o-Benzenedisulfonimides 1 with Trialkylboranes 9 or 10

Synthesis of 4-Nitroethylbenzene (11e); Typical Procedure

4-Nitrobenzenediazonium *o*-benzenedisulfonimide (**1e**; 3.68 g, 10.0 mmol) was added in one portion with vigorous stirring to Et_3B (**9**; 1.0 M in THF, 11 mL) and further THF (4 mL) was added. Pd(Cl₂)dppf (0.25 mmol, 0.20 g) was then added in one portion and stirring was maintained at r.t. for 30 min. The completion of the reaction was confirmed by the absence of azo-coupling with 2-naphthol. GC, GC-MS and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed 4-nitroethylbenzene {**11e**; MS (EI, 70 eV): *m/z* (%) = 151 (94) [M⁺] was formed as the major product, besides traces of **6e**. The workup described above furnished a crude residue which was purified by column chromatography (PE–Et₂O, 9:1) to give the pure title compound **11e** (1.31 g, 87% yield) and recovered **8** (1.78 g, 82% yield).

After carrying out the reaction with Pd(OAc) (0.25 mmol, 0.05 g), GC, GC-MS and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed **6e** was formed as the sole product. When $(Ph_3P)_2PdCl_2$ (0.25 mmol, 0.17 g) was used, only a small amount of **11e** (0.35 g, 23% yield) was recovered; GC, GC-MS and TLC (PE–Et₂O, 9:1) analyses of the reaction mixture showed **6e** to be the major product.

Details of the reactions 1–16 and yields of the pure ethylbenzenes **11a–f**, **11h**, **11n** and butylbenzenes **5a–f**, **5h**, **5n** are listed in Table 2.

Reactions of 1e with Methylboronic or Butylboronic Acid

1e (3.68 g, 10.0 mmol) was added in one portion with vigorous stirring to a solution of $MeB(OH)_2$ (0.66 g, 11.0 mmol) or $BuB(OH)_2$ (1.12 g, 11.0 mmol) in THF (15 mL). Pd(OAc) (0.25 mmol, 0.05 g)

was then added in one portion and the suspension obtained was stirred at r.t. for 24 h; a test for azo-coupling with 2-naphthol was positive. The suspension was further refluxed for 8 h but a test for azo-coupling was still positive. Unreacted **1e** was recovered by filtration.

Using MeCN as solvent or using $(Ph_3P)_2PdCl_2$ (0.25 mmol, 0.17 g) or Pd(Cl_2)dppf (0.25 mmol, 0.20 g), only unreacted **1e** was recovered.

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