

# Synthesis and Characterization of Some Disulfonyl Azides as Potential Crosslinking Agents for Textile Fibers

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**ABSTRACT:** The syntheses of 1,6-hexanedisulfonyl azide, 1,10-decanedisulfonyl azide, 1,3-benzenedisulfonyl azide, 1,5-naphthalenedisulfonyl azide, 2,6-naphthalenedisulfonyl azide, and 4,4'-biphenyldisulfonyl azide are described in this article. The compounds are characterized according to their melting temperature, infrared spectra, mass spectra, and carbon-13 and proton nuclear magnetic resonance spectra, with the intention of assessing their potential as crosslinking agents for textile fibers. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 79: 1092–1100, 2001

## INTRODUCTION

Disulfonyl azides have been cited widely in the literature, mostly in patent documentation, as crosslinking and blowing agents for hydrocarbon polymers.<sup>1–17</sup> However, the literature has been mainly concerned with end uses for this class of compound, and details of synthesis and characterization are scarce. There are many references to the synthesis of monosulfonyl azides,<sup>18–29</sup> and some texts contain information about the synthesis of a limited number of disulfonyl azides,<sup>4,6,18,30–33</sup> although characterization has been only by melting point and elemental analysis. The aim of this article is to give an account of the syntheses of 1,6-hexanedisulfonyl azide, 1,10-decanedisulfonyl azide, 1,3-benzenedisulfonyl azide, 1,5-naphthalenedisulfonyl azide, 2,6-naphthalenedisulfonyl azide, and 4,4'-biphenyldisulfonyl azide from readily available starting materials and to give a full characterization of these compounds. It is hoped that the contents will serve as a reference work. It was our intention to use these compounds to bring about the crosslinking of textile

fibers following earlier work that reported on their use in crosslinking a variety of polymers.<sup>4–11</sup>

## Basic Chemistry of Synthesis

Depending on the starting material there are two or three general steps involved in the synthesis of a disulfonyl azide.

### Strecker Synthesis

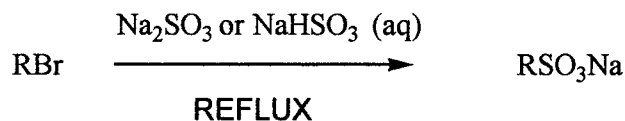
The Strecker synthesis is used for making aliphatic sulfonic acids from bromoalkanes.<sup>34</sup> The reaction was initially formulated in the 19th century and involves the boiling of a saturated aqueous solution of sodium hydrogen sulfite (or sodium sulfite) with a bromoalkane until the organic layer disappears. The reaction (Scheme 1) has been viewed very favorably because yields of the sulfonic acid can be as high as 95%.<sup>35</sup> The reaction is easy to use and is inexpensive.

### Chlorosulfonation of Aromatic Hydrocarbons and Chlorination of Sulfonic Acids

There is little literature on this reaction as it is used mainly in the generation of sulfonyl chlorides as intermediates rather than as products; however, this reaction is very common. It is easily effected at room temperature with efficient stir-

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**Scheme 1** The Strecker Synthesis.

ring and involves two steps<sup>36</sup> (Scheme 2). The first step, which applies only to aromatic nuclei, occurs readily because hydrochloric acid is involved. The second step is an equilibrium and therefore requires an excess of chlorosulfonic acid to push it toward completion. Reaction yields are generally very high, and in some cases a quantitative yield can be obtained; however, careful quenching in iced water is required to minimize the hydrolysis of the sulfonyl chloride. In situations where the product yield is low, a useful alternative procedure is to use the reaction of the sulfonic acid (where available) with a more potent reagent, thionyl chloride.<sup>20,36,37</sup> Aprotic solvents can also be used in order to achieve more efficient mixing. The addition of sodium chloride has been claimed to raise the yields as it quenches the sulfuric acid produced in step 2.<sup>20,36</sup>

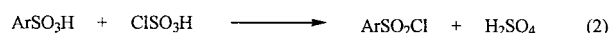
#### Forster–Fiertz Reaction

This is the conversion of a sulfonyl chloride into a sulfonyl azide. Initially, sulfonyl azides were made by nitrosation of the corresponding hydrazides; recently however, the Forster–Fiertz reaction has proved more popular (Scheme 3). The latter procedure generally involves the addition, with efficient stirring, of an ethanolic aqueous solution of the sulfonyl chloride to a saturated aqueous solution of sodium azide. Recent reactions report the use of acetone as a solvent in preference to ethanol, and it is claimed that this increases the purity of the products, the yield, and the ease of the workup.<sup>1,2,18,37</sup>

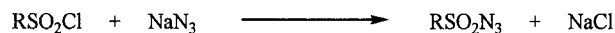
## EXPERIMENTAL

### Materials

1,3-Benzenedisulfonic acid, disodium salt (80%); biphenyl (97%); chlorosulfonic acid (99%); 1,10-



**Scheme 2** Chlorosulfonation.



**Scheme 3** The Forster–Fiertz Reaction.

dibromodecane (97%); 1,6-dibromohexane (96%); 1,5-naphthalenedisulfonic acid, disodium salt (95%); 2,6-naphthalenedisulfonic acid, disodium salt (80%); sodium azide (99%); sodium hydrogen sulfite (ACS reagent); and thionyl chloride (98%) were supplied by Sigma-Aldrich Company, Ltd.

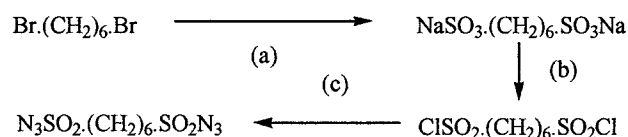
### Instrumentation

Fourier transform infrared spectra were obtained using the KBr disk method on a Perkin-Elmer 1725X FTIR spectrometer. Nuclear magnetic resonance spectra were measured on a hybrid 300-MHz machine with automated sampling, using deuterated dimethyl sulfoxide (DMSO) as a solvent, and tetramethylsilane as a reference for all compounds. Mass spectra were obtained by an electron ionization method that recorded cationic fragments at 190°C on a hybrid VG Autospec mass spectrometer.

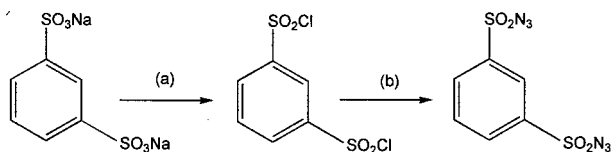
Melting points and decomposition temperatures were measured on a hot-stage microscope using a heating rate of 10°C/min and in most cases were confirmed by differential scanning calorimetry. Differential scanning calorimetry and thermogravimetric analysis measurements were taken from the Du Pont TA2000 system using a nitrogen gas flow of 50 mL/min and a heating rate of 10°C/min.

### Synthesis of 1,6-Hexanedisulfonyl Azide (1,6-HDSA) and 1,10-Decanedisulfonyl Azide (1,10-DDSA)

1,6-Dibromohexane (0.15 mol, 36.7g) was refluxed with a saturated aqueous solution (100 mL) of sodium hydrogen sulfite (0.30 mol, 31.2 g) until the organic layer had disappeared (48 h for 1,6-dibromohexane, 1 week for 1,10-dibromodecane) [Scheme 4(a)]. The mixture was then reduced in volume, the solid product filtered off and dried. The product, 1,6-hexanedisulfonic acid, disodium salt (assumed to be 0.15 mol) was stirred vigor-



**Scheme 4** 1,6-Hexanedisulfonyl Azide.

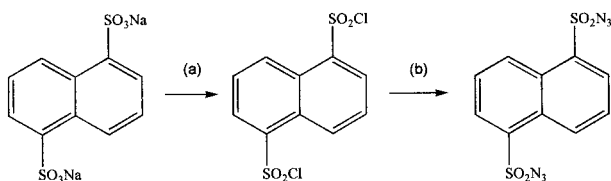


**Scheme 5** 1,3-Benzenedisulfonyl Azide.

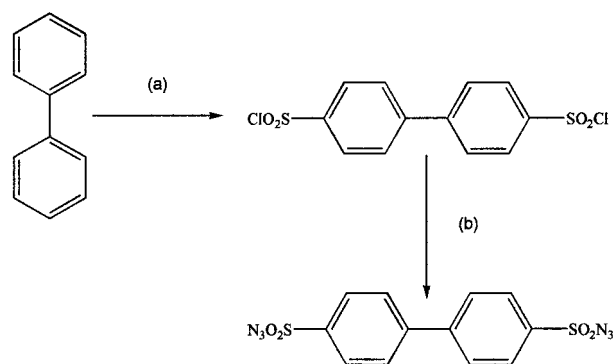
ously with chlorosulfonic acid (1.8 mol, 210 g) for 24 h at room temperature [Scheme 4(b)]. The mixture was quenched slowly in iced water while maintaining the temperature below 20°C. The product was filtered off at the pump and washed with water until the filtrate was neutral.

The recovered 1,6-hexanedisulfonyl chloride (assumed to be 0.15 mol) dissolved in acetone (150 mL) was added to 150 mL of a saturated aqueous solution of sodium azide (0.38 mol, 20.2 g) and mixed vigorously for 24 h at room temperature [Scheme 4(c)]. The acetone was removed by rotary evaporation and the solid product filtered off at the pump, washed with water and then with a little ethanol. Recrystallization was not necessary, as the reaction produced essentially pure, dense white crystals of 1,6-HDSA (yield 29% based on the starting material, 1,6-dibromohexane) with a melting point 89–90°C (lit. 88.5–91.0°C<sup>30</sup>). Elemental analysis was within tolerance [required (%): C, 24.3; H, 4.05; S, 21.6; N, 28.4; found (%): C, 24.5; H, 4.10; S, 21.6; N, 28.6], and mass spectrometry (MS) confirmed a molecular weight of 296. The Fourier transform infrared spectrum (FTIR) showed bands at 2152, 1352, 1213, 1196, 1159, and 804 cm<sup>-1</sup>.

The analogous reaction with 1,10-dibromodecane produced 1,10-DDSA, a 37% yield. Recrystallization was not necessary, as the reaction produced pure, dense off-white crystals with a melting point of 90–91°C (lit. 89.5–91.5°C<sup>30</sup>). Elemental analysis was within tolerance [required (%): C, 34.1; H, 5.68; S, 18.2; N, 23.9; and found (%): C, 34.2; H, 5.75; S, 18.3; N, 23.9], and MS confirmed a molecular weight of 352. Although the molecular ion (M) was not observed, there was a peak at M-42, corresponding to the



**Scheme 6** 1,5-Naphthalenedisulfonyl Azide.

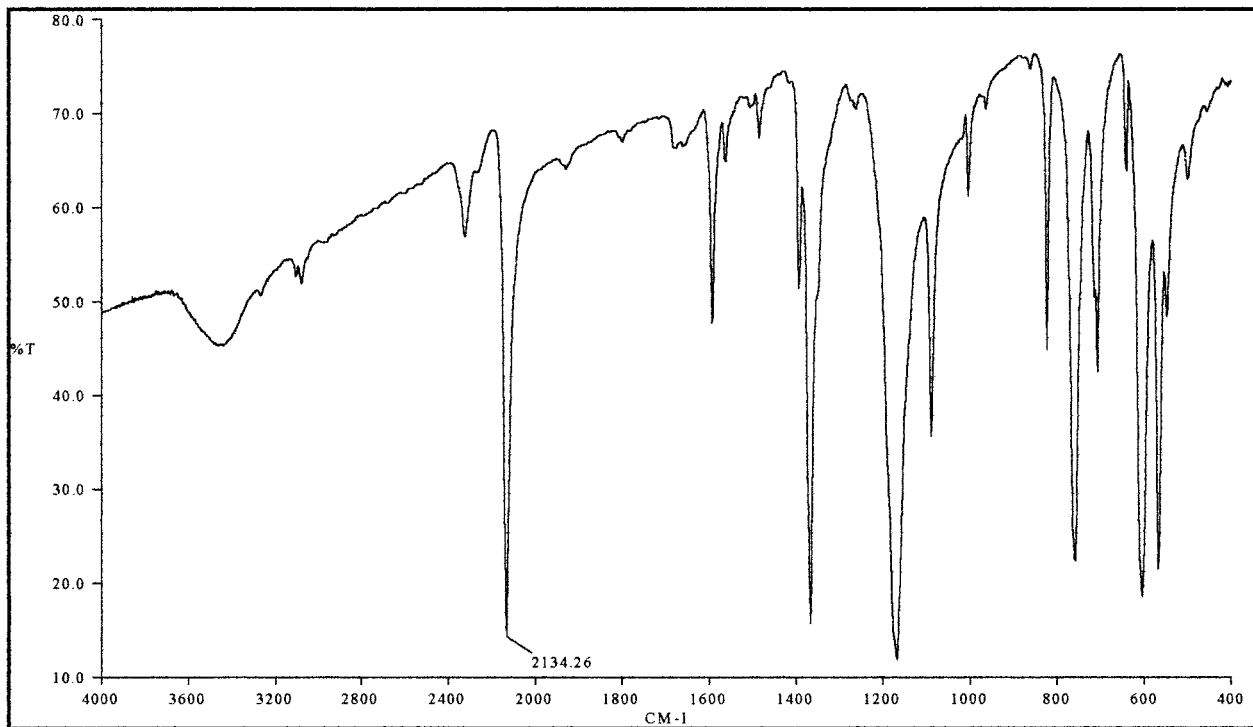


**Scheme 7** 4,4'-Biphenyldisulfonyl Azide.

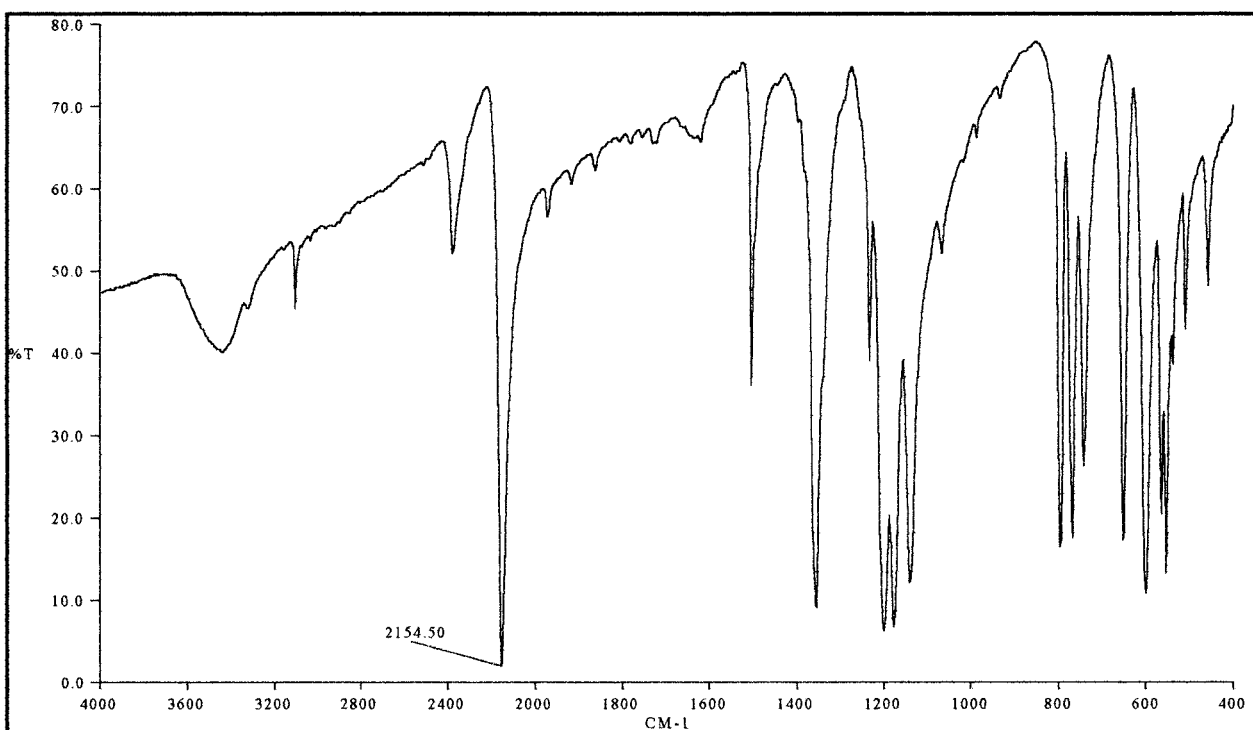
loss of one azide function (N<sub>3</sub>). The FTIR of the compound showed major bands at 2157, 1355, 1208, 1157, and 799 cm<sup>-1</sup>.

### Synthesis of 1,3-Benzenedisulfonyl Azide (1,3-BDSA)

1,3-Benzenedisulfonic acid, disodium salt [1.0 mol, 400 g (~80% pure)] was stirred vigorously with thionyl chloride (12 mol, 1.2 kg, ~1 L) for 3 h at room temperature with an equivalent volume of dimethyl formamide (DMF) [Scheme 5 (a)] (the thionyl chloride and the DMF mixture had been prepared at a temperature below 20°C). The resulting mixture was carefully quenched in iced water while maintaining the temperature below 15°C. The solid product, 1,3-benzenedisulfonyl chloride, was filtered off at the pump, washed with water until the filtrate was at a neutral pH, and then dissolved in acetone (1 L). This solution was then added to 1 L of a saturated aqueous solution of sodium azide (4.0 mol, ~300 g) and stirred vigorously for 24 h at room temperature [Scheme 5 (b)]. The acetone was removed by evaporation, and the solid product was filtered off at the pump and washed with water and then a little ethanol. Recrystallization from cyclohexane produced essentially pure, colorless crystals of 1,3-BDSA (yield 35% based on the starting material) with a melting point 84–85°C (lit. 82–84°C<sup>19,38</sup>). Elemental analysis was within tolerance [required (%): C, 25.0; H, 1.39; S, 22.2; N, 29.2; found (%): C, 25.3; H, 1.45; S, 22.2; N, 29.4], and a molecular weight of 288 was confirmed by MS. FTIR of the compound showed strong absorption at 2143, 1380, 1363, 1176, 1165, and 816 cm<sup>-1</sup>.

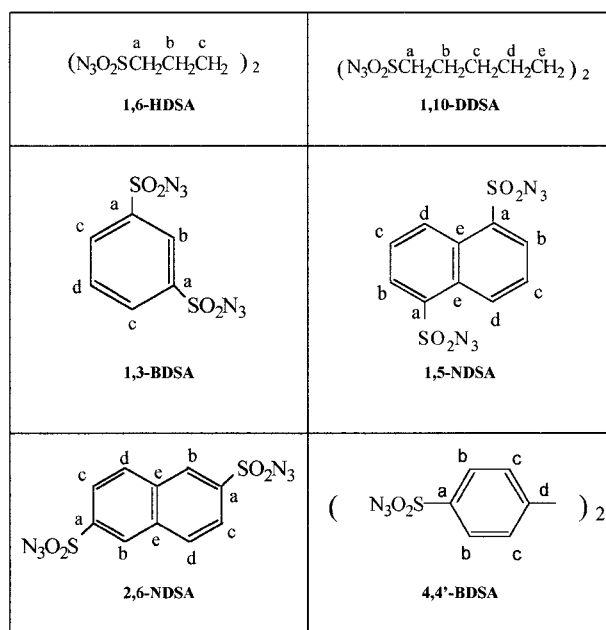


(a)



(b)

Figure 1 FTIR spectra of (a) 4,4'-BDSA and (b) 1,5-NDSA.



**Figure 2** Assignment of carbon and hydrogen nuclei.

### Synthesis of 1,5-Naphthalenedisulfonyl Azide (1,5-NDSA) and 2,6-Naphthalenedisulfonyl Azide (2,6-NDSA)

1,5-Naphthalenedisulfonic acid, disodium salt (1.0 mol, 350 g) was stirred vigorously with chlorosulfonic acid (10 mol, 665 mL) for 24 h at room temperature [Scheme 6(a)]. The mixture was quenched very slowly in iced water while the temperature was maintained below 20°C. The solid product was filtered off at the pump and washed with water until the filtrate was neutral.

The product 1,5-naphthalenedisulfonyl chloride (assumed to be 1.0 mol) dissolved in acetone (3 L) was added to 1.2 L of a saturated aqueous

solution of sodium azide (3.0 mol, ~200 g) and stirred vigorously for 24 h at room temperature [Scheme 6 (b)]. Acetone was removed under reduced pressure and the solid product filtered off at the pump. The product was then washed through with water and a small amount of ethanol. Recrystallization was not necessary (but could be effected by the use of toluene), as the product was essentially pure in the form of fluffy white crystals of 1,5-NDSA (69% yield based on 1,5-naphthalenedisulfonic acid, disodium salt), which melted, with decomposition at 178°C [lit. 177°C<sup>31</sup>]. Elemental analysis was within tolerance [required (%): C, 35.5; H, 1.78; S, 18.9; N, 24.9; found (%): C, 35.8; H, 1.85; S, 18.9; N, 24.9], and MS confirmed a molecular weight of 338. The FTIR spectrum showed main peaks at 2155, 1504, 1355, 1201, 1177, 1140, and 796 cm<sup>-1</sup>.

2,6-NDSA was synthesized in the same manner from 2,6-naphthalenedisulfonic acid, disodium salt. Recrystallization of the final product was not necessary (but again could be effected, as with the 1,5-derivative, by the use of toluene), as the product was essentially pure. The fluffy white crystals of 2,6-naphthalenedisulfonyl azide (94% yield based on 2,6-naphthalenedisulfonic acid, disodium salt) melted, with decomposition at 170°C, in disagreement with the literature value of 152°C<sup>32</sup>. On viewing the compound on a hot stage microscope, crystal cracking and break up was observed at 152°C but no melting. Elemental analysis was within tolerance [required (%): C, 35.5; H, 1.78; S, 18.9; N, 24.9; and found (%): C, 35.8; H, 1.90; S, 18.8; N, 24.7] and MS confirmed the molecular weight at 338. The compound's FTIR spectrum showed strong absorption at 2142, 1378, 1360, 1166, and 760 cm<sup>-1</sup>.

**Table I** Proton NMR Data

Sulfonyl Azide	Chemical Shift (ppm)				
	a	b	c	d	e
1,6-HDSA	3.74 (4p,t) <sup>a,b</sup>	1.76 (4p,q)	1.44 (4p,q)	~	~
1,10-DDSA	3.74 (4p,t)	1.74 (4p,q)	1.39 (4p,q)	1.2–1.3 (4p,q) <sup>c</sup>	1.2–1.3 (4p,q) <sup>c</sup>
1,3-BDSA	~	8.42 (1p,s)	8.50 (2p,d)	8.10 (1p,t)	~
1,5-NDSA	~	8.86 (2p,d)	8.13 (2p,dd)	8.62 (2p,d)	~
2,6-NDSA	~	9.03 (2p,s)	8.22 (2p,d)	8.65 (2p,d)	~
4,4'-BDSA	~	8.15 (4p)	8.15 (4p)	~	~

<sup>a</sup> Number indicates number of protons.

<sup>b</sup> Multiplicity: singlet (s), doublet (d), triplet (t), quintet (q), and doublet of doublets (dd).

<sup>c</sup> Unresolved.

**Table II**  $C^{13}$ -NMR Data

Sulfonyl Azide	Chemical Shift (ppm)				
	a	b	c	d	e
1,6-HDSA	55.06 (2)	26.69 (2)	22.93 (2)	~	~
1,10-DDSA	55.23 (2)	28.89 (2)	28.60 (2)	27.43 (2)	23.32 (2)
1,3-BDSA	139.62 (2)	133.08 (1)	134.04 (2)	126.12 (1)	~
1,5-NDSA	134.52 (2)	131.84 (2)	128.57 (2)	128.81 (2)	131.94 (2)
2,6-NDSA	134.58 (2)	129.61 (2)	124.12 (2) <sup>a</sup>	124.12 (2) <sup>a</sup>	132.99 (2)
4,4'-BDSA	144.75 (2)	129.54 (4)	128.50 (4)	137.96 (2)	~

<sup>a</sup> Unresolved.

### Synthesis of 4,4'-Biphenyldisulfonyl Azide (4,4'-BDSA)

Biphenyl (0.50 mol, 77.1 g) in dry DMF (250 mL) was stirred vigorously with chlorosulfonic acid (6.0 mol, 333 mL) and sodium chloride (30 g) for 24 h at room temperature [Scheme 7 (a)]. The mixture was then quenched in iced water while the temperature was maintained below 30°C. The product, 4,4'-biphenyl disulfonyl chloride, was then filtered off and washed until the acidity of the filtrate was neutral. The filter cake was then used in the synthesis of the diazide derivative.

The product, 4,4'-biphenyldisulfonyl chloride (assumed to be 0.50 mol), was dissolved in acetone and then added to a saturated aqueous solution of sodium azide (2.50 mol, 163 g) and mixed vigorously at room temperature for 24 h [Scheme 7 (b)]. The acetone was removed by rotary evaporation and the solid product filtered off at the pump. The filter cake was washed with water and then with a small amount of ethanol. Recrystallization from DMF produced dense white platelets of 4,4'-BDSA (33% yield with respect to biphenyl, the starting material), which melted, with decomposition at 144–145°C (lit. 144–145°C<sup>4</sup>). Elemental analysis was acceptable [required (%): C, 39.6; H, 2.20; S, 17.6; N, 23.1; found (%): C, 39.8; H, 2.40; S, 17.9; N, 23.2], and MS confirmed a molecular weight of 364. The FTIR spectrum of the compound showed strong absorption at 2134, 1367, 1168, 1089, and 823 cm<sup>-1</sup>.

## RESULTS AND DISCUSSION

### General Observations

Organic azides in general are known to be explosive compounds. However, it's claimed that sulfo-

nyl azides are relatively safe to handle, provided there are more than three carbon atoms per sulfonyl azide group.<sup>18,20,21</sup> No problems were encountered in storing and using these compounds.

Several syntheses were done for each compound, and the results were always reproducible using amounts of starting materials ranging from 0.1 to 5.0 mol, except in the case of 4,4'-BDSA. On several occasions no product could be recovered from the final reaction mixture. Also examined were variations in conditions, such as the use of DMF instead of chloroform and the addition of sodium chloride. It was concluded that it was best to use DMF and NaCl in the chlorosulfonation step, as described in the experimental section. Difficulties in sulfonation of aromatics have been reported previously.<sup>36</sup>

### Fourier Transform Infrared Spectra

Fourier transform infrared (FTIR) spectra for all the compounds were obtained, and two examples are shown in Figure 1. The spectra are all characterized by strong azide bands at 2134–2157 cm<sup>-1</sup> and sulfonyl bands at 1352–1378 cm<sup>-1</sup> and 1140–1167 cm<sup>-1</sup>.

### Nuclear Magnetic Resonance Studies

The spectra were resolved with the assistance of the common literature and expansions of spectra where assignment was difficult. However, the spectra were simple and corresponded with the relevant structures. Figure 2 shows the labeling of the nuclei, and the corresponding nuclear magnetic resonance (NMR) data are shown in Tables I and II.

1,6-HDSA and 1,10-DDSA both had triplets in their proton spectra for Ha of 1:1:1 intensity,

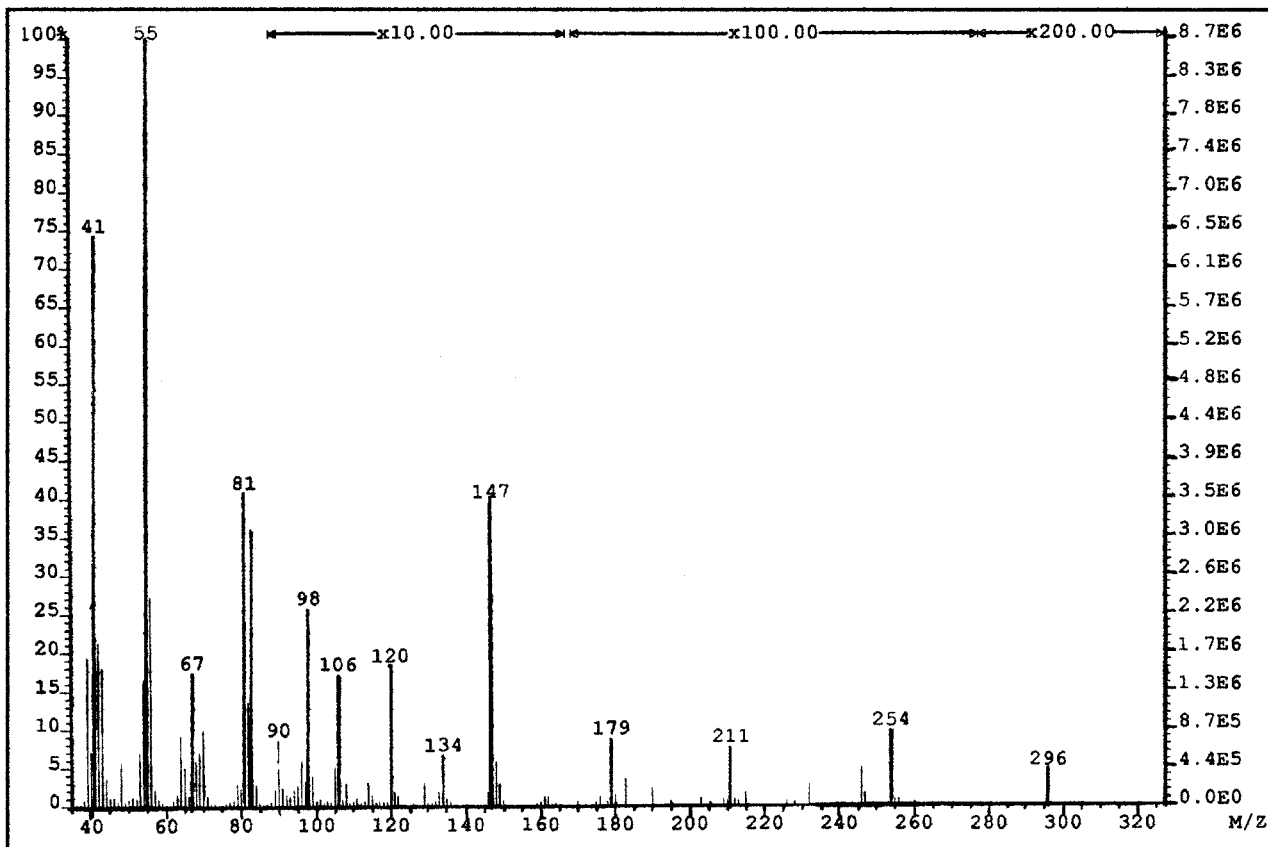


Figure 3 Mass spectrum of 1,6 HDSA.

thought to be a result of the prochirality of the sulfonyl azide group. This behavior is not observed in the aromatic compounds, where the functional groups are attached to the ring. Second-order splitting was observed in the spectra of 1,6-HDSA (Hb and Hc), 1,3-BDSA (Hb and Hc), 2,6-NDSA (Hb and Hc), and 4,4'-BDSA. The latter compound showed a typical para-substituted benzene-type spectrum with leaning; however, because of the similarity in chemical shift of the two types of protons and in the fine structures, assignment was not made.

### Mass Spectra

The molecular ion (M) was observed in all but one of the compounds examined. It was most easily identified within the spectra of the aromatic compounds. For the aliphatic compounds, the spectra were magnified by 200% to detect M, but it was only observed for 1,6-HDSA. However, the signal was weak for all the sulfonyl azides measured. Examples of the spectra are given in Figures 3 and 4.

There was one common peak observed for all the sulfonyl azides at M-42, which corresponds to that of the loss of one azide function, and another at M-198, which was unassigned. All the aromatic compounds' spectra showed a common feature: identical for each were the three largest peaks, at M-42, M-106, and M-212, corresponding to the loss of one azide function, the loss of one sulfonyl azide group, and the loss of two sulfonyl azide groups, respectively. Other fragments common to the aromatic compounds were found at M-132 (weak), M-148 (with a slight base peak variation, possibly corresponding to the loss of one sulfonyl azide function and one azide group), and M-225. No additional peaks were found in common for 1,6-HDSA or 1,10-HDSA.

### Thermal Analysis

#### Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) revealed melting points only for the aliphatic and uniaromatic derivatives, as the other heavier aromatics

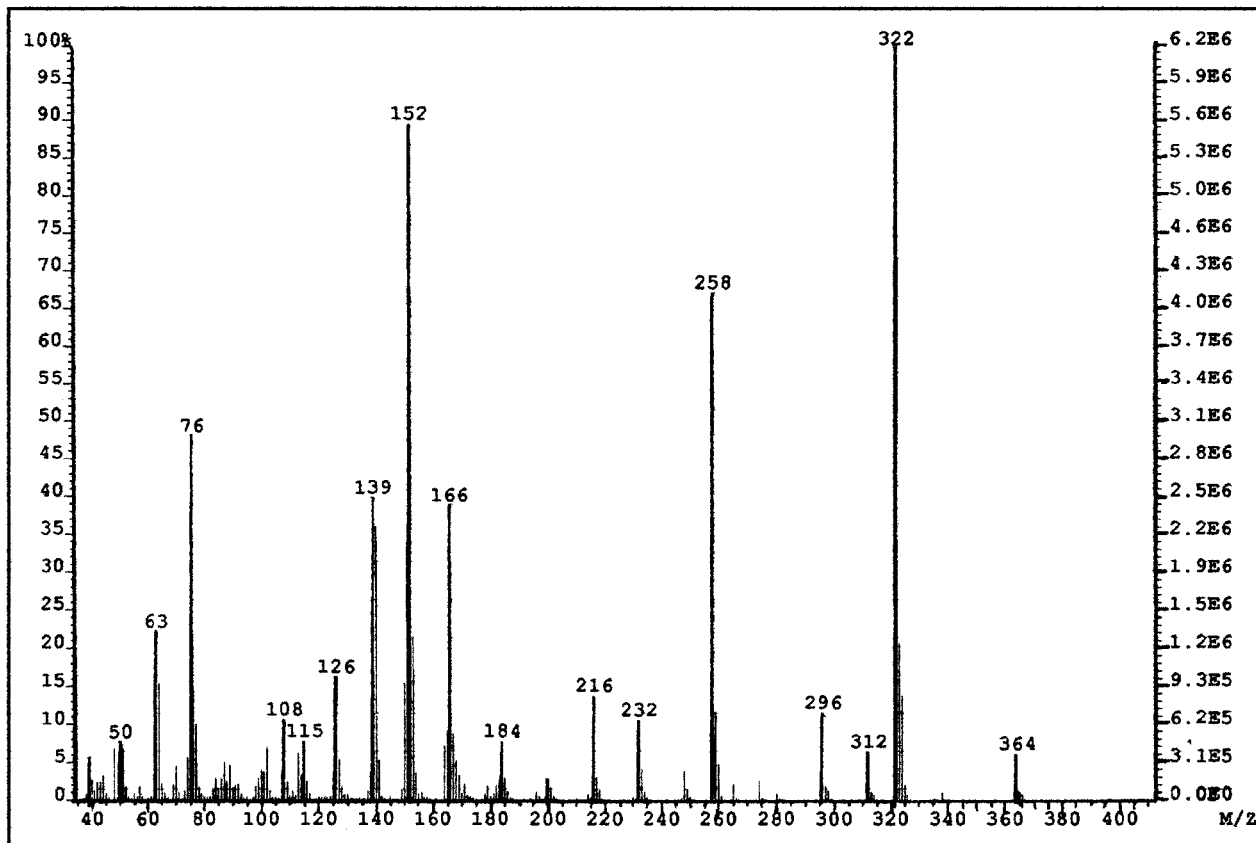


Figure 4 Mass spectrum of 4,4'-BDSA.

melted with decomposition. 1,6-HDSA melted at 90°C, 1,10-DDSA at 91°C, and 1,3-BDSA at 85°C.

#### Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) traces could again only be obtained for the lighter azides, as the heavier aromatics decomposed violently, leaving char in the pan and on the surrounds. 1,6-HDSA and 1,10-DDSA gave similar traces, from which it is apparent that maximum mass loss in each case relates to the loss of both sulfonyl azide groups. 1,3-BDSA (Fig. 5) decomposed with a mass loss less than that of the other two compounds. Many studies have explained that the decomposition of sulfonyl azides as solids can give rise to the loss of many components, such as sulfur dioxide, nitrogen, and other compounds, because of rearrangements, high heat flux, and the radical nature of decomposition of these compounds.<sup>18,21-24,30</sup>

#### CONCLUSIONS

Suitable conditions have been found for the synthesis of six disulfonyl azide compounds from con-

venient starting materials. The compounds have been fully characterized, and their structures have been confirmed. The compounds were made in order to investigate the feasibility of their use as crosslinking agents for synthetic fibers such as acrylic, nylon, polypropylene, and polyester. Preliminary results indicate the compounds can be used to crosslink fibers. The effectiveness of crosslinking is being measured, and the results of

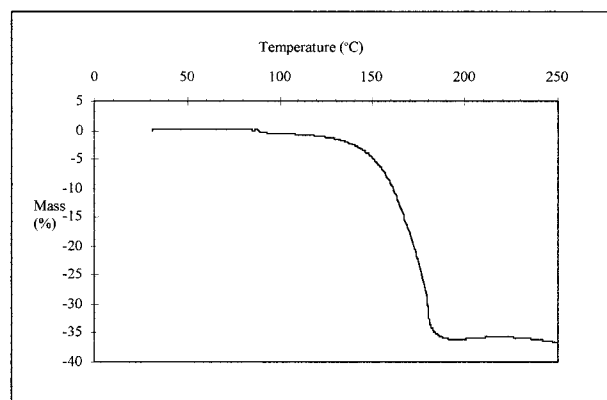


Figure 5 TGA trace of 1,3-BDSA.



this investigation will be published in the near future.

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