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New Methods and Reagents in Organic Synthesis. 26.[1] Reductive Desulfonylation of α-Sulfonylacetates[2]

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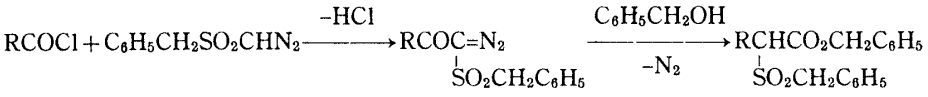
α-Acyl-α-benzylsulfonyldiazomethanes (**2a**—**d**) have been converted to α-benzylsulfonyl-α-substituted-acetic acids (**7a**—**d**) by means of the Wolff rearrangement. Reductive removal of the benzylsulfonyl group of **7b**—**d** can be achieved by the use of sodium–ethanol in tetrahydrofuran. α-Benzylsulfonyl-α-substituted-propionic acids (**9a**—**d**), prepared from benzyl α-benzylsulfonyl-α-substituted-acetates (**3a**—**d**), were also debenzylsulfonylated under similar reductive conditions. The overall process, in combination with the Arndt–Eistert synthesis of α-sulfonylacetates from acyl chlorides and benzylsulfonyldiazomethane (**1**), provides a new, safe method for the homologation of carboxylic acids.

Keywords——reductive desulfonylation; Wolff rearrangement; sodium amalgam reduction; catalytic debenzylation; sodium–ethanol reduction; methylation; α-substituted propionic acid; α-sulfonylacetate

Our recent publications[3,4] have revealed that sulfonyldiazomethanes(α-diazosulfones, RSO_2CHN_2) represent a stable and safe substitute for hazardous diazomethane and can be used for the Arndt–Eistert synthesis of α-sulfonylacetates from acyl chlorides. Thus, benzylsulfonyldiazomethane (**1**) underwent acylation with various acyl chlorides and the Wolff rearrangement of the resulting α-acylated benzylsulfonyldiazomethanes (**2**) with hot benzyl alcohol gave benzyl α-benzylsulfonylacetates (**3**), as shown in Chart 1.



1 **2** **3**

Chart 1

We now describe the reductive desulfonylation of α-benzylsulfonylacetates, which provides a new method for the preparation of α-substituted acetic acids and propionic acids. Aryl, aralkyl, alkyl, and cycloalkyl derivatives were chosen as representative examples for the conversion.

First, benzyl 2-benzylsulfonyl-2-phenylacetate (**3a**) was subjected to various known methods for reductive desulfonylation. Treatment of **3a** with zinc dust in acetic acid[5] resulted in recovery of **3a**. Attempted desulfonylation with Raney nickel in ethanol[6] afforded dibenzylsulfone (**4**) instead of the desired benzyl phenylacetate. Dibenzylsulfone (**4**) was also obtained by the treatment of **3a** with sodium amalgam in phosphate buffer.[7] Surprisingly, alkaline hydrolysis of **3a** with potassium hydroxide afforded **4** again. However, treatment of the phenethyl, isopropyl, and cyclohexyl derivatives of **3** with sodium amalgam in phosphate buffer resulted in ester exchange, giving the corresponding methyl esters (**5b**—**d**), as shown in Chart 2. Without sodium amalgam, transesterification did not occur at all.

Recently, Tsuchihashi and co-workers[8] reported a modification of Bouveault–Blanc's method for reductive desulfonylation using sodium–ethanol in tetrahydrofuran. When **3a** was subjected to this reductive desulfonylation procedure, the proton magnetic resonance

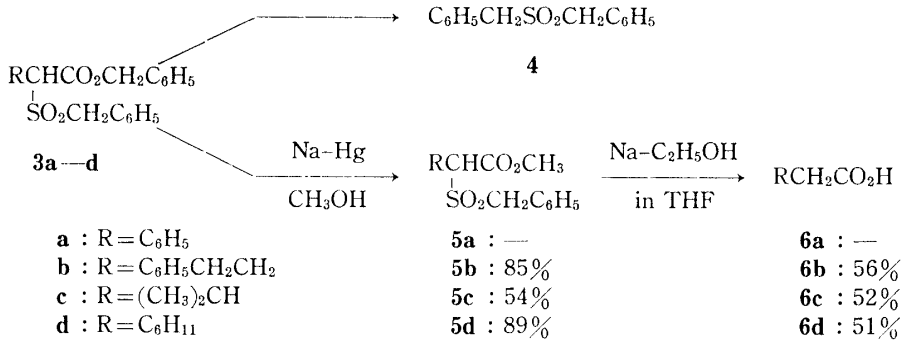


Chart 2

spectrum of the crude product showed the presence of dibenzylsulfone (**4**) as well as the starting material. However, the methyl esters (**5b**—**d**) underwent reductive desulfonylation under the same reaction conditions to give the desulfonylated acids (**6b**—**d**) though in moderate yields.

Next, we turned our attention to the reductive desulfonylation of the corresponding acids, since Tsuchihashi's procedure might cause the reduction of the ester function in addition to the desulfonylation. Thus, the benzyl esters (**3a**—**d**)[3] were catalytically hydrogenated over palladium black in tetrahydrofuran to give α-benzylsulfonylacetic acids (**7a**—**d**). These acids were directly obtained from α-acylsulfonyldiazomethanes (**2a**—**d**) by Wolff rearrangement in toluene containing water. This one-step procedure gave slightly better results than the two-step procedure *via* the benzyl esters (**3a**—**d**). Reductive desulfonylation of the α-benzylsulfonylacetic acids (**7b**—**d**) with sodium–ethanol in tetrahydrofuran smoothly afforded the corresponding desulfonylated acids (**6b**—**d**), shown in Chart 3. However, 2-benzylsulfonyl-2-phenylacetic acid (**7a**) did not yield phenylacetic acid (**6a**) under the same reaction conditions.

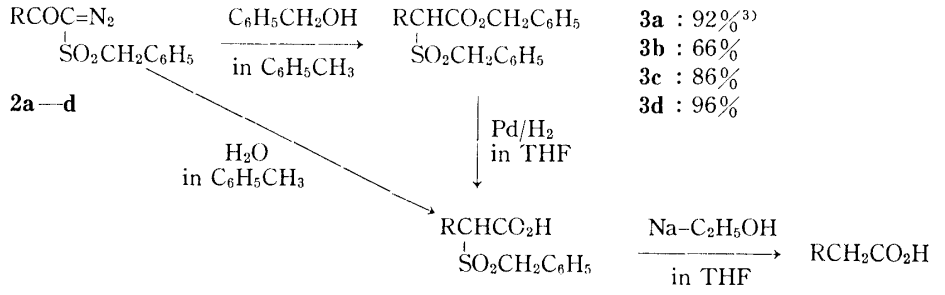


Chart 3

a) Figures in parentheses indicate the yields of **7** directly obtained from **2**.

Since some α-arylpropionic acids show a strong antiinflammatory activity,[9] our efforts were directed to developing a preparative procedure for α-substituted propionic acids.[10] Methylation of the benzyl esters (**3a**—**d**) with methyl iodide has been smoothly carried out using either potassium carbonate in acetone or sodium hydride in dimethylformamide. Reductive debenzylation of the resulting α-methylated benzyl esters (**8a**—**d**) was again catalytically achieved over palladium black. Treatment of the acids (**9a**—**d**) with sodium–ethanol in tetrahydrofuran afforded the desired α-substituted propionic acids (**10a**—**d**), as shown in Chart 4.

In conclusion, α-benzylsulfonylacetates may be desulfonylated under reductive conditions by using sodium–ethanol in tetrahydrofuran. The overall process in combination with the

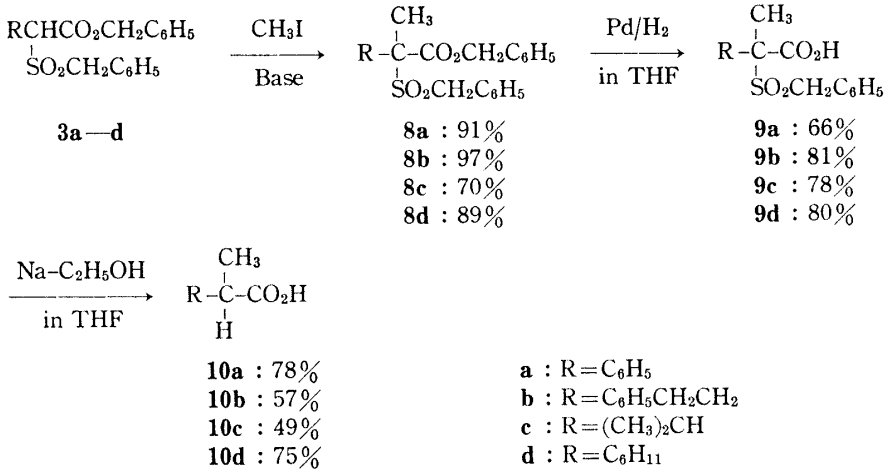


Chart 4

Arndt–Eistert synthesis of α-sulfonylacetates from acyl chlorides and benzylsulfonyldiazomethane (**1**)[3,4] represents a new, safe method for the homologation of carboxylic acids.[11]

Experimental

Melting and boiling points (obtained by Kugelrohr distillation) are uncorrected. All the crystalline products were recrystallized from diethyl ether–hexane, unless otherwise stated. IR spectra were recorded on a JASCO IRA-2 spectrometer (nujol mull for crystals or film for oils). ^{1}H-NMR spectra were recorded on a JEOL PMX-60, MH-100, or FX-100 spectrometer with tetramethylsilane as an internal standard. Silica gel (70—230 mesh ASTM, Merck Art. 7734) was used for column chromatography. Preparative layer chromatography (PLC) was carried out on plates (20 cm × 20 cm, 2 mm thick) precoated with silica gel $60F_{254}$ (Merck). Tetrahydrofuran (THF) and diethyl ether were dried by distillation from benzophenone ketyl and lithium aluminum hydride, respectively.

Attempted Reductive Debenzylsulfonylation of Benzyl 2-Benzylsulfonyl-2-phenylacetate (3a)——(i) With Zinc in Acetic Acid: A mixture of **3a** (380 mg, 1 mmol), zinc dust (326 mg, 5 mmol), acetic acid (1 ml), and ethanol (20 ml) was stirred at room temperature for 24 h. After dilution with water, the mixture was extracted with diethyl ether. The extracts were dried over magnesium sulfate and concentrated *in vacuo* to give a residue, which was recrystallized to yield **3a** (286 mg, 75%).

(ii) With Raney Nickel: A mixture of **3a** (380 mg, 1 mmol) and Raney nickel W-2 (2 ml, suspended in ethanol) in THF (15 ml) was stirred at room temperature for 24 h. After filtration, the filtrate was concentrated *in vacuo*. The residue was passed through a short silica gel column with diethy lether. The ether eluate was concentrated *in vacuo*, and the residue was purified by silica gel (12 g) column chromatography with hexane–ethyl acetate (8: 1) to give dibenzylsulfone (**4**, 192 mg, 78%), which was identified by comparison with an authentic sample.[3]

(iii) With Sodium Amalgam: Pulverized 5% sodium amalgam (3 g) was added to a solution of **3a** (761 mg, 2 mmol) and anhydrous disodium hydrogen phosphate (1.136 g, 8 mmol) in methanol (20 ml). The solution was stirred at room temperature for 24 h. The mixture was poured into water and extracted with chloroform. The extracts were concentrated *in vacuo*, and benzyl alcohol was removed by distillation *in vacuo*. The residue was recrystallized from ethanol to give **4** (352 mg, 71%).

Alkaline Hydrolysis of Benzyl 2-Benzylsulfonyl-2-phenylacetate (3a)——The ester **3a** (190 mg, 0.5 mmol) in methanol (5 ml) was refluxed in 20% aqueous potassium hydroxide (5 ml) for 5 h. The reaction mixture was concentrated *in vacuo*, and the residue was extracted with chloroform. The extracts were concentrated *in vacuo*, and the residue was recrystallized from ethanol to give **4** (108 mg, 87%).

Treatment of Benzyl 2-Benzylsulfonyl-2-substituted-acetates (3b—d) with Sodium Amalgam——Methyl 2-Benzylsulfonyl-4-phenylbutyrate (**5b**): Pulverized 5% sodium amalgam (1.8 g) was added to **3b** (204 mg, 0.5 mmol) and anhydrous disodium hydrogen phosphate (284 mg, 2 mmol) in methanol (40 ml). The solution was stirred at room temperature for 54 h then filtered. The filtrate was concentrated *in vacuo*, and the residue was separated by PLC with hexane–ethyl acetate (5: 3) to give a mixture (168 mg) of **5b** and benzyl alcohol (*Rf* 0.4). Benzyl alcohol was removed by distillation *in vacuo* to give **5b** (141 mg) as a colorless oil. IR ν_{max} cm^{-1}: 1740, 1320, 1210, 1120. NMR δ ppm ($CDCl_3$): 2.1—3.0 (4H, m), 3.80 (3H, s), 3.7—3.96 (1H), 4.23 and 4.50 (2H, AB q, J = 13 Hz), 6.9—7.6 (10H, m).

Methyl 2-Benzylsulfonyl-3-methylbutyrate (**5c**): Pulverized 5% sodium amalgam (1.8 g) was added to **3c** (346 mg, 1 mmol) and anhydrous disodium hydrogen phosphate (568 mg, 4 mmol) in methanol (20 ml).

The mixture was stirred at room temperature for 24 h, then diluted with water. The mixture was extracted with chloroform, and the extracts were washed with saturated aqueous sodium chloride and dried over sodium sulfate. Concentration followed by removal of benzyl alcohol *in vacuo* gave a residue, which was purified by PLC with hexane–ethyl acetate (2: 1) to give **5c** (*Rf* 0.5, 146 mg) as colorless crystals, mp 68.5—70°C. IR ν_{max} cm^{-1}: 1720, 1315, 1305, 1295, 1150, 1110, 1100. NMR δ ppm ($CDCl_3$): 1.25 (3H, d, J=4 Hz), 1.37 (3H, d, J=4 Hz), 2.4—3.0 (1H, m), 3.80 (1H, d, J=8 Hz), 3.98 (3H, s), 7.53 (5H, s). The aqueous layer after extraction with chloroform was acidified with 1 N hydrochloric acid and extracted with chloroform. The extracts were dried over sodium sulfate and concentrated to give 2-benzylsulfonyl-3-methylbutyric acid (**7c**, 51 mg, 20%).

Methyl 2-Benzylsulfonyl-2-cyclohexylacetate (**5d**): The benzyl ester **3d** was treated with sodium amalgam as described for **3b**. The crude residue was dissolved in chloroform, and the solution was passed through a short silica gel column. Concentration followed by removal of benzyl alcohol *in vacuo* afforded **5d** as a colorless oil. IR ν_{max} cm^{-1}: 1740, 1300, 1160, 1140, 1130. NMR δ ppm ($CDCl_3$): 0.8—2.4 (11H, m), 3.64 (1H, d, J=8 Hz), 3.76 (3H, s), 4.26 and 4.50 (2H, AB q, J=13 Hz), 7.36 (5H, s).

2-Benzylsulfonyl-2-substituted-acetic Acids (7a—d)——(i) From Benzyl Esters (**3a—d**): The benzyl ester **3** (1 mmol) in THF (25 ml) was hydrogenated overnight with stirring over palladium black (100 mg) at room temperature and atmospheric pressure. After further addition of palladium black (100 mg) suspended in THF (5 ml), the mixture was stirred in the same manner for 2 d. The catalyst was filtered off, and the filtrate was concentrated *in vacuo*. The residue was dissolved in saturated aqueous sodium bicarbonate (25—30 ml), and washed with chloroform. The alkaline layer was acidified with 1 N hydrochloric acid and extracted with chloroform. The extracts were dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by recrystallization to give **7**.

(ii) From α-Acyl-α-benzylsulfonyldiazomethanes (**2a—d**). The α-acyl-α-benzylsulfonyldiazomethane **2** (1 mmol) was refluxed in toluene (50 ml) containing water (50 mg) for 2—5 h. After cooling, the reaction mixture was extracted with saturated aqueous sodium bicarbonate (30 ml). The alkaline extracts were treated as in (i) to give **7**.

2-Benzylsulfonyl-2-phenylacetic Acid (**7a**): mp 132.5—135°C. IR ν_{max} cm^{-1}: 3300—3000, 1710, 1320, 1310, 1290, 1230, 1145, 1135. NMR δ ppm (CD_3COCD_3): 4.30 and 4.70 (2H, AB q, J=14 Hz), 5.30 (1H, s), 6.8—7.3 (1H, br s), 7.3—8.8 (10H, m). *Anal.* Calcd for $C_{15}H_{14}O_4S$: C, 62.05; H, 4.87. Found: C, 61.96; H, 4.87.

2-Benzylsulfonyl-4-phenylbutyric Acid (**7b**): mp 132.5—134°C. IR ν_{max} cm^{-1}: 3400—3000, 1755, 1740, 1290, 1210, 1120. NMR δ ppm (CD_3COCD_3): 2.1—3.0 (4H, m), 4.17 (1H, t, J=7 Hz), 4.60 and 4.87 (2H, AB q, J=14 Hz), 7.40 (5H, s), 7.57 (5H, s), 8.3—9.3 (1H, br s). *Anal.* Calcd for $C_{17}H_{18}O_4S$: C, 64.13; H, 5.70. Found: C, 64.37; H, 5.84.

2-Benzylsulfonyl-4-phnylbutyric Acid (**7c**): mp 114—116°C. IR ν_{max} cm^{-1}: 3300—3000, 1728, 1300, 1190, 1100. NMR δ ppm (CD_3COCD_3): 1.10 (3H, d, J=3 Hz), 1.23 (3H, d, J=3 Hz), 2.2—2.9 (1H, m), 3.78 (1H, d, J=7 Hz), 4.43 and 4.70 (2H, AB q, J=14 Hz), 7.43 (5H, s), 9.8—10.3 (1H, br s). *Anal.* Calcd for $C_{12}H_{16}O_4S$: C, 56.23; H, 6.29. Found: C, 56.31; H, 6.27.

2-Benzylsulfonyl-2-cyclohexylacetic Acid (**7d**): mp 123.5—125°C. IR ν_{max} cm^{-1}: 3300—3000, 1720, 1300, 1250, 1215, 1170, 1105. NMR δ ppm (CD_3COCD_3): 1.0—2.5 (11H, m), 3.78 (1H, d, J=6 Hz), 4.45 and 4.70 (2H, AB q, J=13 Hz), 7.33 (5H, s), 9.3—10.3 (1H, br s). *Anal.* Calcd for $C_{15}H_{20}O_4S$: C, 60.79; H, 6.80. Found: C, 61.09; H, 7.08.

Reductive Debenzylsulfonylation of Methyl Esters (5b—d) and Carboxylic Acids (7b—d)——(i) From Methyl Esters (**5b—d**): Sodium (230 mg, 10 mmol) was added to the methyl ester **5** (1 mmol) in THF (30 ml), followed by the addition of ethanol (553 mg, 12 mmol). The mixture was stirred at room temperature for 48 h. After addition of water (20 ml), the mixture was washed with chloroform. The alkaline layer was acidified with 1 N hydrochloric acid, and extracted with chloroform. The extracts were dried over sodium sulfate, and concentrated *in vacuo*. The residue was distilled in a Kugelrohr apparatus to give **6**.

(ii) From Carboxylic Acids (**7b—d**). Sodium (230 mg, 10 mmol) was added to the carboxylic acid **7** (1 mmol) in THF (30 ml), followed by the addition of ethanol (507 mg, 11 mmol). The mixture was stirred at room temperature for 24 h. After further addition of ethanol (1 ml), water (20 ml) was added and the mixture was worked up as in (i) to give **6**.

4-Phenylbutyric acid (**6b**),[12a] isovaleric acid (**6c**),[12b] and 2-cyclohexylacetic acid (**6d**)[12c] were identified by comparisons of their IR spectra with those of corresponding authentic samples.

Methylation of Benzylsulfonyl-2-substituted-acetates (3a—d)——(i) With Potassium Carbonate in Acetone: A mixture of **3a** (190 mg, 0.5 mmol), potassium carbonate (138 mg, 1 mmol), and methyl iodide (142 mg, 1 mmol) in acetone (25 ml) was refluxed for 24 h, then concentrated *in vacuo*. The residue was extracted with chloroform. The extracts were concentrated *in vacuo* and the residue was purified by silica gel (25 g) column chromatography with hexane–ethyl acetate (6: 1) to give benzyl 2-benzylsulfonyl-2-phenylpropionate (**8a**, 180 mg), mp 94—95.5°C. IR ν_{max} cm^{-1}: 1735, 1300, 1230, 1135, 1120. NMR δ ppm ($CDCl_3$): 2.08 (3H, s), 3.90 and 4.66 (2H, AB q, J=12 Hz), 5.40 (2H, s), 7.0—7.6 (15H, m). *Anal.* Calcd for $C_{23}H_{22}O_4S$: C, 70.03; H, 5.62. Found: C, 69.77; H, 5.37.

Benzyl 2-benzylsulfonyl-4-phenylbutyrate (**3b**) was similarly methylated to give benzyl 2-benzylsulfonyl-

2-methyl-4-phenylbutyrate (**8b**), mp 94—96°C. IR ν_{max} cm^{-1}: 1735, 1300, 1225, 1140, 1115. NMR δ ppm ($CDCl_3$): 1.72 (3H, s), 2.0—2.8 (4H, m), 4.12 and 4.40 (2H, AB q, J=12 Hz), 5.20 (2H, s), 7.0—7.6 (15H, m). *Anal.* Calcd for $C_{25}H_{26}O_4S$: C, 71.06; H, 6.20. Found: C, 71.12; H, 6.30.

(ii) With Sodium Hydride in Dimethylformamide: Sodium hydride (50%, 116 mg, 2.4 mmol) suspended in dimethylformamide (5 ml) was added to **3c** (693 mg, 2 mmol) in dimethylformamide (5 ml) at 0°C under argon, followed by the addition of methyl iodide (340 mg, 2.4 mmol). The mixture was stirred at room temperature for 30 h. Ice-water was added, and the mixture was extracted with ethyl acetate. The organic extracts were washed with saturated aqueous sodium chloride, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel (60 g) column chromatography with hexane–ethyl acetate (7: 1) to give benzyl 2-benzylsulfonyl-2,3-dimethylbutyrate (**8c**, 512 mg) as a colorless oil. IR ν_{max} cm^{-1}: 1740, 1305, 1240, 1125, 1110. NMR δ ppm ($CDCl_3$): 0.87 (3H, d, J=6 Hz), 1.12 (3H, d, J=6 Hz), 1.53 (3H, s), 2.5—3.1 (1H, m), 4.13 and 4.57 (2H, AB q, J=13 Hz), 5.17 and 5.40 (2H, AB q, J=12 Hz), 7.33 (5H, s), 7.43 (5H, s). *Anal.* Calcd for $C_{20}H_{24}O_4S$: C, 66.64; H, 6.71. Found: C, 66.84; H, 6.62.

Benzyl 2-benzylsulfonyl-2-cyclohexylacetate (**3d**) was similarly converted to benzyl 2-benzylsulfonyl-2-cyclohexylpropionate (**8d**), mp 90.5—92°C. IR ν_{max} cm^{-1}: 1735, 1300, 1220, 1140, 1130, 1100. NMR δ ppm ($CDCl_3$): 0.8—2.7 (11H, m), 1.63 (3H, s), 4.23 and 4.60 (2H, AB q, J=13 Hz), 5.33 (2H, s), 7.40 (5H, s), 7.50 (5H, s). *Anal.* Calcd for $C_{23}H_{28}O_4S$: C, 68.97; H, 7.05. Found: C, 69.13; H, 7.04.

Catalytic Debenzylation of Benzyl Esters (8a—d)——The benzyl ester **8** (1 mmol) was treated as described under procedure (i) for the preparation of **7** using palladium black (200—300 mg) to give **9**.

2-Benzylsulfonyl-2-phenylpropionic Acid (**9a**): mp 165—168°C. IR ν_{max} cm^{-1}: 3300—3000, 1750, 1285, 1215, 1120. NMR δ ppm ($CDCl_3$): 2.10 (3H, s), 4.10 and 4.70 (2H, AB q, J=14 Hz), 7.2—7.8 (10H, m), 8.3—8.5 (1H, s). *Anal.* Calcd for $C_{16}H_{16}O_4S$: C, 63.14; H, 5.30. Found: C, 63.33; H, 5.41.

2-Benzylsulfonyl-2-methyl-4-phenylbutyric Acid (**9b**): mp 116—119°C. IR ν_{max} cm^{-1}: 3300—3000, 1730, 1300, 1140. NMR δ ppm ($CDCl_3$): 1.90 (3H, s), 2.2—3.0 (4H, m), 4.50 and 4.70 (2H, AB q, J=14 Hz), 7.33 (5H, s), 7.50 (5H, s), 9.0—9.3 (1H, br s). *Anal.* Calcd for $C_{18}H_{20}O_4S \cdot H_2O$: C, 61.69; H, 6.33. Found: C, 61.89; H, 6.42.

2-Benzylsulfonyl-2,3-dimethylbutyric Acid (**9c**): mp 165.5—167°C. IR ν_{max} cm^{-1}: 3400—3000, 1745, 1285, 1220, 1125, 1100. NMR δ ppm ($CDCl_3$): 1.07 (3H, d, J=6 Hz), 1.20 (3H, d, J=6 Hz), 1.63 (3H, s), 2.5—3.0 (1H, m), 4.33 and 4.80 (2H, AB q, J=13 Hz), 7.47 (5H, s), 8.7—9.0 (1H, br s). *Anal.* Calcd for $C_{13}H_{18}O_4S$: C, 57.76; H, 6.71. Found: C, 57.57; H, 6.68.

2-Benzylsulfonyl-2-cyclohexylpropionic Acid (**9d**): mp 190.5—192°C. IR ν_{max} cm^{-1}: 3300—3000, 1750, 1280, 1220, 1125, 1090. NMR δ ppm ($CDCl_3$): 0.8—3.0 (11H, m), 1.50 (3H, s), 4.45 and 4.80 (2H, AB q, J=14 Hz), 7.43 (5H, s), 9.8—10.4 (1H, br s). *Anal.* Calcd for $C_{16}H_{22}O_4S$: C, 61.91; H, 7.14. Found: C, 61.70; H, 7.04.

Reductive Debenzylsulfonylation of Carboxylic Acids (9a—d)——Reductive removal of the benzylsulfonyl group from the carboxylic acid **9** was carried out as described under procedure (ii) for the reductive debenzylsulfonylation of the carboxylic acid **7** by the use of sodium–ethanol in THF. The crude acid **10** was purified by distillation.

2-Phenylpropionic Acid (**10a**): Identified by comparison of its IR spectrum with that of an authentic sample.[12d]

2-Methyl-4-phenylbutyric Acid (**10b**): bp 100—110°C (0.8 mmHg). IR ν_{max} cm^{-1}: 3400—3000, 1700. NMR δ ppm ($CDCl_3$): 1.28 (3H, d, J=6 Hz), 1.5—2.9 (5H, m), 7.30 (5H, s), 9.7—10.2 (1H, br s). *Anal.* Calcd for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: 74.03; H, 7.78.

2,3-Dimethylbutyric Acid (**10c**): bp 78—80°C (10 mmHg) (lit.[13] 90°C (16 mmHg)). IR ν_{max} cm^{-1}: 3400—3000, 1705. NMR δ ppm ($CDCl_3$): 0.98 (3H, d, J=2 Hz), 1.02 (3H, d, J=2 Hz), 1.18 (3H, d, J=6 Hz), 1.6—2.6 (2H, m), 10.2—11.0 (1H, br s).

2-Cyclohexylpropionic Acid (**10d**): bp 130—140°C (17 mmHg). IR ν_{max} cm^{-1}: 3400—3000, 1700. NMR δ ppm ($CDCl_3$): 0.92 (3H, d, J=7 Hz), 0.5—2.5 (11H, m), 10.2—10.7 (1H, br s). *Anal.* Calcd for $C_9H_{16}O_2$: C, 69.19; H, 10.32. Found: C, 69.02; H, 10.21.

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