s, CH_3CO), 2.69 (2 H, s, C_4H_2), 2.97 (3 H, s, SO_2CH_3), 3.38 (2 H, t, J = 6 Hz, SO_2CH_2), 3.73 (3 H, s, CO_2CH_3), 3.96 (2 H, t, J = 6 Hz, NCH_2), 5.23 (2 H, s, NCO_2CH_2 Ar), 7.52 (2 H, d, J = 9 Hz, Ar H), 8.23 (2 H, d, J = 9 Hz, Ar H); Anal. Calcd for $C_{21}H_{27}N_3O_9S$: C, 50.69; H, 5.47; N, 8.45. Found: C, 50.38; H, 5.42; N, 8.21. Mass spectrum (FD), m/e 497 (M^+).

***p*-Nitrobenzyl (5*R*,6*S*)-6-[(*R*)-1-Hydroxyethyl]-2-([2-(([(*p*-nitrobenzyl)oxy]carbonyl)amino)ethyl]sulfonyl)carbapen-2-em-3-carboxylate (8).** To a stirred solution of 7 (400 mg, 0.68 mmol) in THF (20 mL) was added *m*-CPBA (298 mg, 1.50 mmol) at 0 °C under argon. After stirring for 15 min at the same temperature, Na_2HPO_4 (237 mg, 1.69 mmol) was added to the mixture. The reaction mixture was allowed to warm to room temperature and stirring was continued for 5 h at room temperature. The reaction mixture was diluted with ethyl acetate, washed with 5% $NaHCO_3$ and water, dried (Na_2SO_4), and concentrated. The residue was purified by HPLC [Nucleosil $5C_{18}$ column 20 mm × 30 cm, acetonitrile–water (2:1)] to afford 130 mg (31%) of 8 as a colorless powder: mp 79–92 °C dec; IR (KBr) 1785, 1720, 1700 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 1.32 (3 H, d, J = 6 Hz, CH_3), 3.1–3.4 (2 H, m, C_1H_2), 3.4–3.6 (3 H, m, SO_2CH_2 and C_6H), 3.6–3.8 (2 H, m, NCH_2), 4.28 (1 H, m, C_8H), 4.42 (1 H, m, C_5H), 5.20 and 5.29 (2 H, ABq, J = 14 Hz, CO_2CH_2 Ar), 5.43 and 5.52 (2 H, ABq, J = 14 Hz, CO_2CH_2 Ar), 5.66 (1 H, m, NH), 7.56 (2 H, d, J = 9 Hz, Ar H), 7.66 (2 H, d, J = 9 Hz, Ar H), 8.28 (2 H, d, J = 9 Hz, Ar H), 8.30 (2 H, d, J = 9 Hz, Ar H). Anal. Calcd for $C_{26}H_{26}N_4O_{12}S \cdot {}^1/_2H_2O$: C, 49.76; H, 4.34; N, 8.93. Found: C, 49.45; H, 4.23; N, 8.66.

***p*-Nitrobenzyl (5*R*,6*S*)-6-[(*R*)-1-Hydroxyethyl]-2-[[2-(methylsulfonyl)ethyl]([(*p*-nitrobenzyl)oxy]carbonyl)-amino]carbapen-2-em-3-carboxylate (9).** To a stirred solution of 8 (31 mg, 0.05 mmol) in DMF (0.5 mL) was added DBU (8 mg, 0.05 mmol) at room temperature under argon. After being stirred for 15 min, the reaction mixture was diluted with ethyl acetate, washed with 0.5 N HCl and water, dried ($MgSO_4$), and concentrated. The residue was purified by HPLC [Nucleosil $5C_{18}$ column 20 mm × 30 cm, acetonitrile–water (3:1)] to give 4 mg (14%) of 9 as a colorless powder: mp 70–78 °C dec; IR (KBr) 1780, 1720, 1520, 1350, 1300, 1130 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 1.38 (3 H, d, J = 6 Hz, CH_3), 2.96 (3 H, s, SO_2CH_3), 3.16 (1 H, dd, J = 10, 20 Hz, $C_{1a}H$), 3.26 (1 H, dd, J = 8, 20 Hz, $C_{1b}H$), 3.35 (1 H, dd, J = 2.4, 6.4 Hz, C_6H), 3.40 (2 H, m, CH_2SO_2), 4.02 (2 H, m, NCH_2), 4.2–4.4 (2 H, m, C_5H and C_8H), 5.22 (2 H, s, NCO_2CH_2Ar), 5.18 and 5.36 (2 H, ABq, J = 14 Hz, CO_2CH_2 Ar), 7.42 (2 H, d, J = 9 Hz, Ar H), 7.63 (2 H, d, J = 9 Hz, Ar H), 8.22 (4 H, d, J = 9 Hz, Ar H). Anal. Calcd for $C_{27}H_{28}N_4O_{12}S$: C, 51.26; H, 4.46; N, 8.86. Found: C, 50.82; H, 4.34; N, 9.34. Mass spectrum (FD), m/e 632 (M^+), 630, 586, 546.

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A Convenient Preparation of Cyclopentadiene from Its Dimer

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Cyclopentadiene has been prepared on a laboratory scale by thermolysis of dicyclopentadiene (1), either by reflux (170 °C) of neat 1[1] or by addition of 1 to some high-boiling oil, at 250–260 °C.[2] It is recommended that the vapors are fractionated to remove refluxing, uncracked dimer and entrained liquid. In commercial procedures, the dimer is cracked at 350–360 °C with a short contact time,[2] which avoids the otherwise necessary fractionation to remove the dimer.

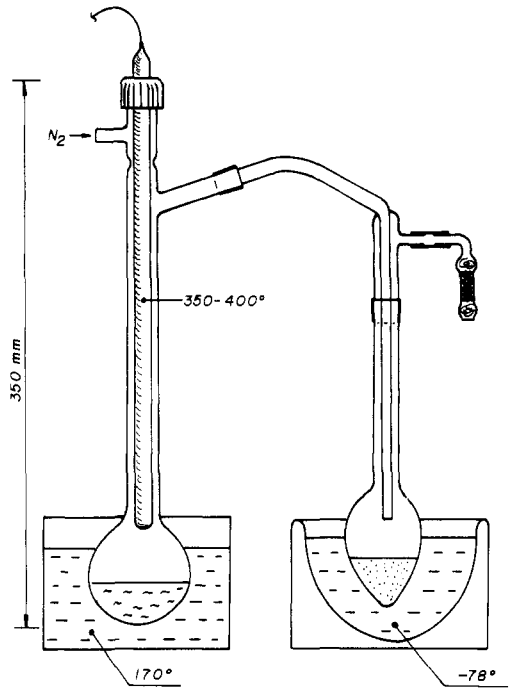

Figure 1. Apparatus for the preparation of cyclopentadiene.

We now report a simplified laboratory procedure which mimics commercial vapor-phase cracking procedures. The hot (350–400 °C) surface is provided by an electrically heated Red-Rod, mounted vertically above the surface of hot (170 °C) dicyclopentadiene. This arrangement permits a rapid and efficient preparation of cyclopentadiene from commercial dicyclopentadiene. In a typical experiment, 5.2 g of dimer gave 4.7 g (91%) of cyclopentadiene within 20 min.

The present apparatus can also be used for the preparation of dimethylketene from its dimer.[3]

Experimental Section

The glassware was dried before use to avoid ice formation in the product. The Red-Rod was purchased from Electrothermal Engineering Ltd.[4]

Dicyclopentadiene (analytical grade; 5.2 g) was charged in the round-bottomed flask (Figure 1). A slow (ca. 30 mL/min) stream of nitrogen was maintained throughout the operation. The Red-Rod was allowed to reach operating temperature (350–400 °C), approximately 25 V to the Red-Rod, and the dicyclopentadiene was heated (oil bath 170 °C). After ca. 20 min, the reaction was completed and cyclopentadiene (4.7 g, 91%) had condensed in the receiver. The product had n_D 1.4435 (lit. n_D 1.433;[1] 1.4429;[2] 1.4440[5]).

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Registry No. 1, 77-73-6; cyclopentadiene, 542-92-7.

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