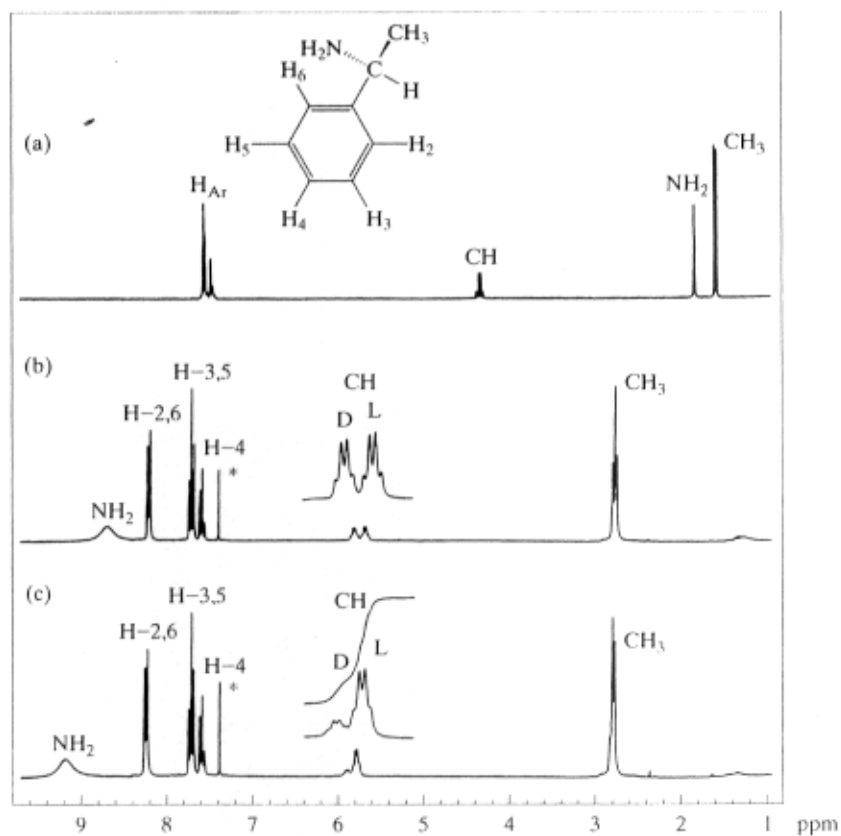
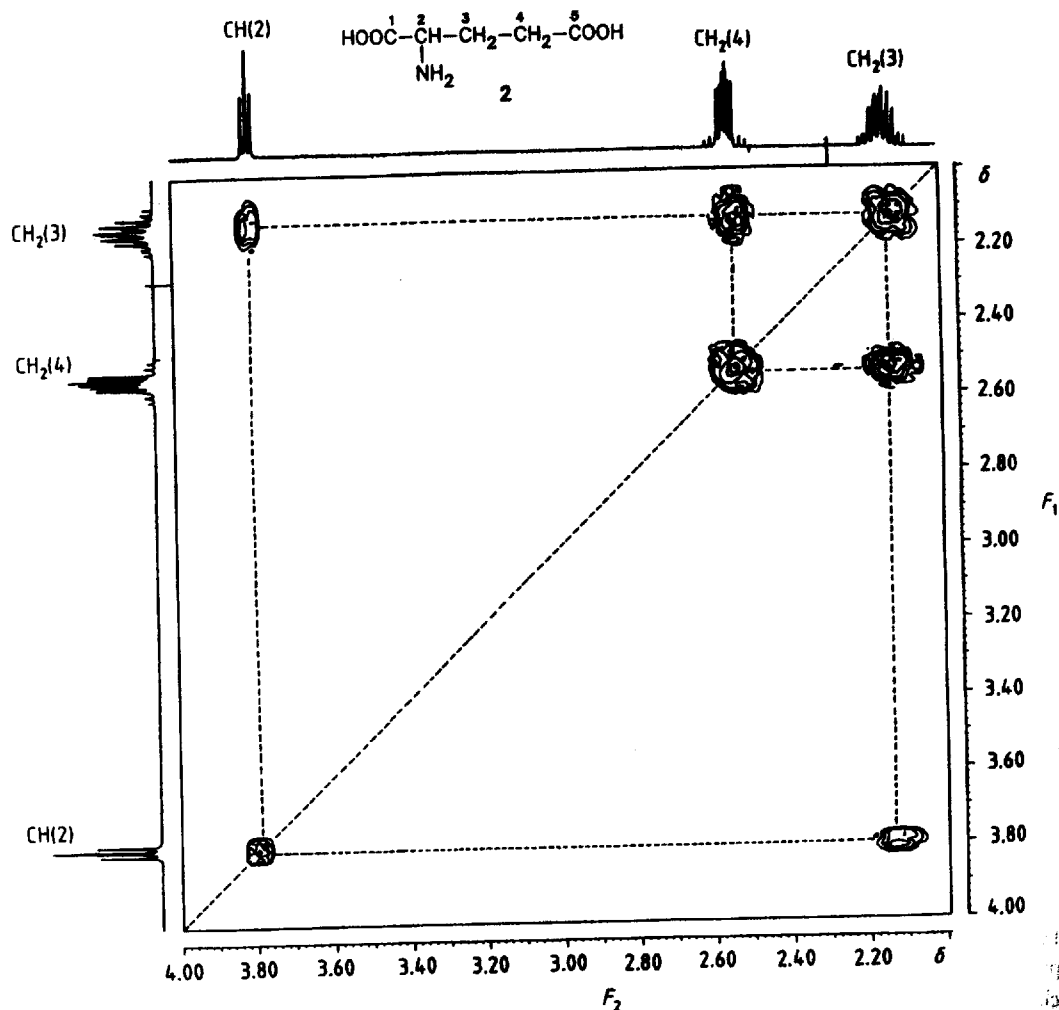


► FIGURE 12.7

250-MHz  $^1\text{H}$  NMR spectra in  $\text{CDCl}_3$  of (A) racemic 1-phenylethylamine, (B) racemic 1-phenylethylamine in the presence of  $\text{D-Eu}(\text{TFC})_3$ , and (C) 4:1 :: L:D-1-phenylethylamine in the presence of  $\text{D-Eu}(\text{TFC})_3$ .\*

\* Adapted from *Basic One- and Two-Dimensional NMR Spectroscopy*, Friebolin, H., VCH, Weinheim, 1993.



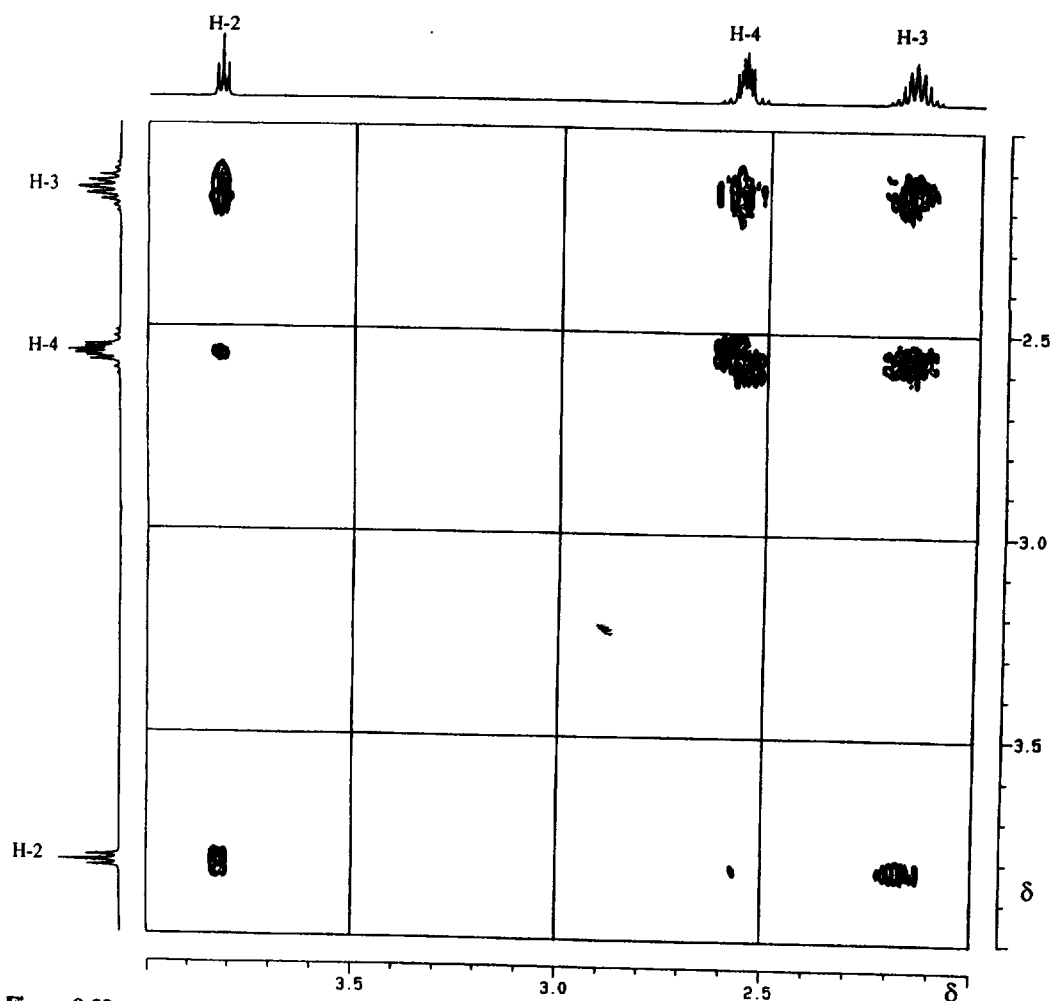
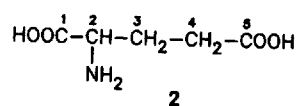


**Figure 9-19.**

500 MHz COSY-90 spectrum of glutamic acid (2) shown as a contour plot. At the left-hand edge and at the top is the one-dimensional  $^1\text{H}$  NMR spectrum with assignments. The diagonal and cross peaks joined by dashed construction lines indicate which protons have a mutual scalar coupling. The diagonal peak of the two protons on C-3 forms a corner of two squares, as these protons are coupled both to the proton on C-2 and to those on C-4.

(Experimental conditions:

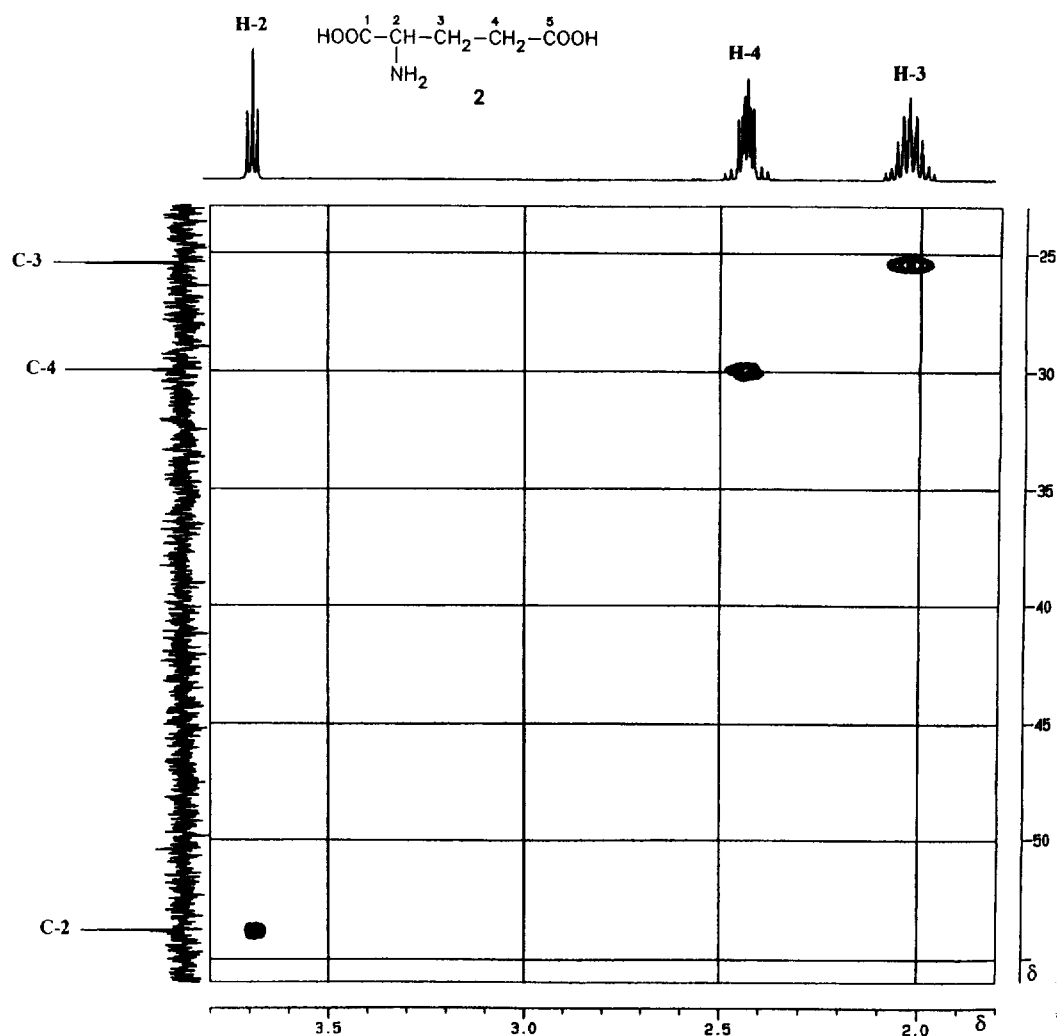
10 mg of the compound in 0.5 ml  $\text{D}_2\text{O}$ ; 5 mm sample tube; 256 measurements with different values of  $t_1$ ; 1 measurement with 16 FIDs; digital resolution 2.639 Hz/data point.)



**Figure 9-22.**

500 MHz long-range H,H-COSY spectrum of glutamic acid (2). The one-dimensional  $^1\text{H}$  spectrum is shown at the top and on the left. Compared with the COSY-90 spectrum (Fig. 9-19) two new signals at approximately (2.6,3.8) and (3.8,2.6) have now appeared. These provide evidence of a correlation between the protons on C-2 and C-4; in the one-dimensional 500 MHz spectrum it is not possible to detect a coupling between these protons. (*Experimental conditions:*

approx. 20 mg in 0.5 ml  $\text{D}_2\text{O}$ ; 5 mm sample tube; 128 measurements with  $t_1$  altered in 600  $\mu\text{s}$  increments; each measurement with 8 FIDs and 1 K data points; total time approx. 40 min.)

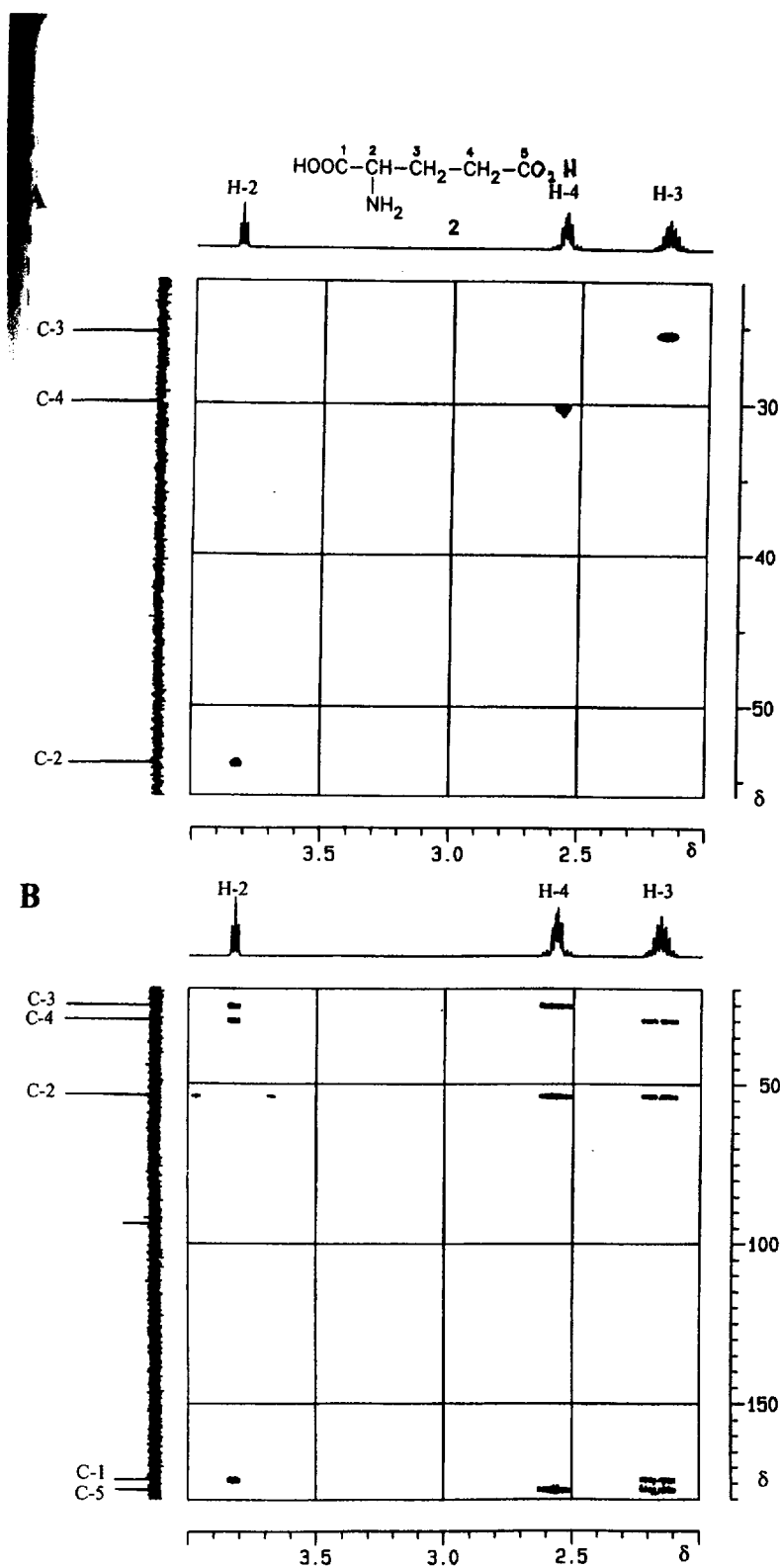


**Figure 9-23.**

Two-dimensional H,C-correlated spectrum of glutamic acid (**2**) in D<sub>2</sub>O, recorded by the HSQC method with <sup>1</sup>H as the observed nuclide. The one-dimensional 500 MHz <sup>1</sup>H NMR spectrum is shown at the top edge and a portion of the 125 MHz <sup>13</sup>C NMR spectrum at the left-hand edge (only the resonances of the two quaternary <sup>13</sup>C nuclei C-1 and C-5 are missing).

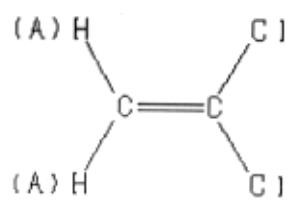
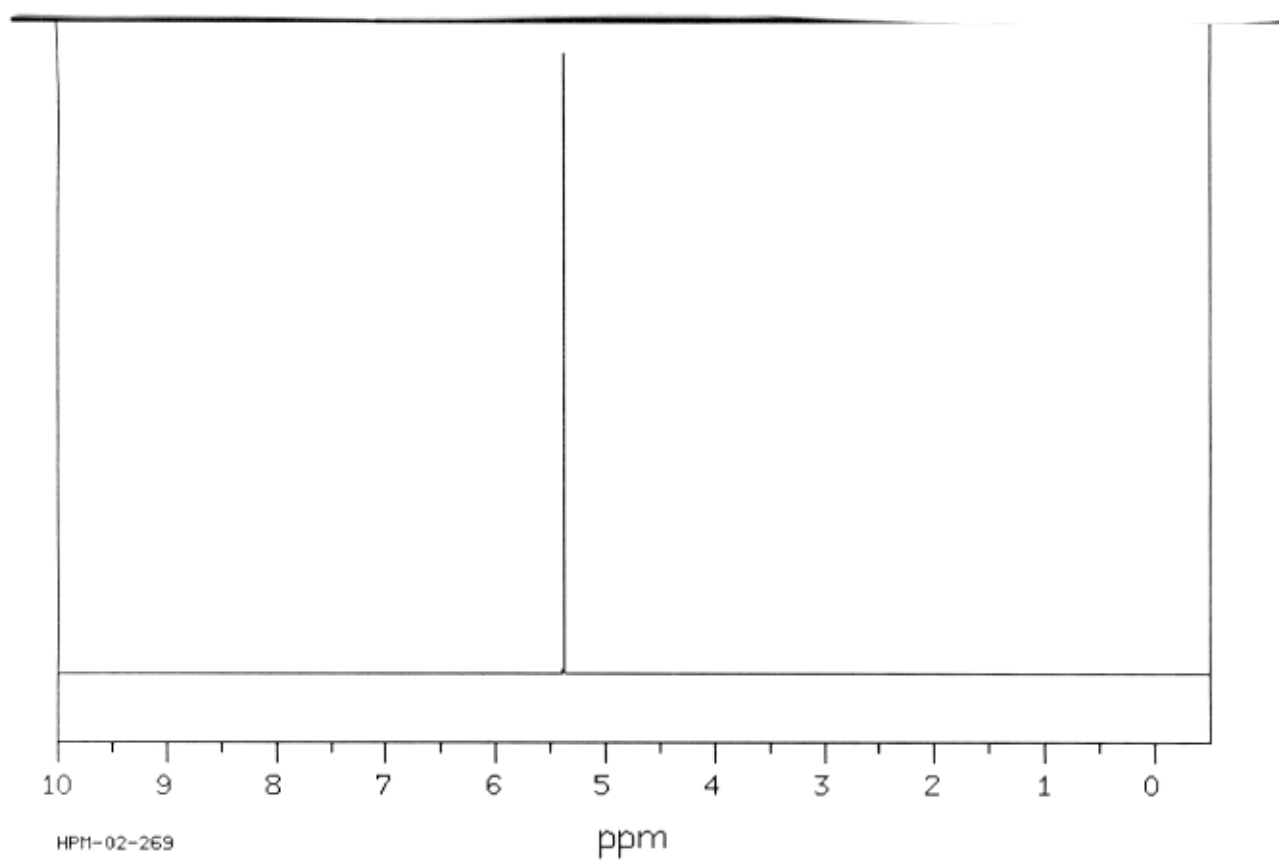
(*Experimental conditions:*

approx. 20 mg in 0.5 ml D<sub>2</sub>O; 5 mm sample tube; 128 measurements with *t*<sub>1</sub> altered in 44 μs increments; each measurement with 4 FIDs and 0.5 K data points; total time approx. 20 min.)



**Figure 9-25.** Two-dimensional H,C-correlated spectra of glutamic acid (2) recorded by different gradient-selected methods. A: Gradient-selected (gs-)HMQC method. The 500 MHz  $^1\text{H}$  NMR spectrum is shown at the top edge and a portion of the 125 MHz  $^{13}\text{C}$  NMR spectrum at the left-hand edge (the C-1 and C-5 resonances are missing). B: Gradient-selected (gs-)HMBC method. The 500 MHz  $^1\text{H}$  NMR spectrum is again shown at the top edge, while at the left-hand edge is the complete 125 MHz  $^{13}\text{C}$  NMR spectrum.

(Experimental conditions: approx. 20 mg in 0.5 ml  $\text{D}_2\text{O}$ ; 5 mm sample tube; for A: 128 measurements with  $t_1$  altered in 47  $\mu\text{s}$  increments; each measurement with 2 FIDs and 1 K data points; total time approx. 10 min; for B: 256 measurements with  $t_1$  altered in 23  $\mu\text{s}$  increments; each measurement with 8 FIDs and 2 K data points; total time approx. 70 min.)



Parameter	ppm	Hz
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D (A)	5.383	
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WHIPPLE, E. B. ET AL. J. CHEM. PHYS. 34, 2136 (1961)