FIGURE 12.7
250-MHz $^1$H NMR spectra in CDCl$_3$
(A) racemic 1-phenylethylamine,
(B) racemic 1-phenylethylamine in
the presence of D-Eu(TFC)$_3$, and
(C) 4:1 : L:D-1-phenylethylamine
in the presence of D-Eu(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 $^1$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 D$_2$O; 5 mm sample tube; 256 measurements with different values of $n$, 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 $^1$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 D$_2$O; 5 mm sample tube; 128 measurements with $t_1$ altered in 600 $\mu$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 $D_2O$, recorded by the HSQC method with $H$ as the observed nuclide. The one-dimensional 500 MHz $^1H$ NMR spectrum is shown at the top edge and a portion of the 125 MHz $^{13}C$ NMR spectrum at the left-hand edge (only the resonances of the two quaternary $^{13}C$ nuclei C-1 and C-5 are missing).

(Experimental conditions:
approx. 20 mg in 0.5 ml $D_2O$; 5 mm sample tube; 128 measurements with $t_1$ altered in 44 $\mu$s 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 1H NMR spectrum is shown at the top edge and a portion of the 125 MHz 13C 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 1H NMR spectrum is again shown at the top edge, while at the left-hand edge is the complete 125 MHz 13C NMR spectrum.

(Experimental conditions: approx. 20 mg in 0.5 ml D2O; 5 mm sample tube; for A: 128 measurements with t1 altered in 47 μs increments; each measurement with 2 FIDs and 1 K data points; total time approx. 10 min; for B: 256 measurements with t1 altered in 23 μs increments; each measurement with 8 FIDs and 2 K data points; total time approx. 70 min.)
Parameter | ppm | Hz
--- | --- | ---
D(A) | 5.383 |  |