

**Problem R-99B**. The <sup>1</sup>H and <sup>13</sup>C NMR spectra (normal and DEPT-135) of R-99A are provided. Your task is to assign the proton and carbon NMR spectra.

(a) All of the protons have been labelled A-L (there is no I). Enter the chemical shift, multiplet structure and approximate coupling constants in the table below. Use the format shown for X:

 $X = e.g.: 3.3 \delta, qt, J = 7, 3 Hz$ 

Α_			
В			

 $H_{A} \xrightarrow{D} O \xrightarrow{H_{A}} O \xrightarrow{K} CH_{3}$   $H_{A} \xrightarrow{D} O \xrightarrow{K} CH_{3}$   $H_{B} \xrightarrow{H_{A}} O \xrightarrow{C} CH_{2}$   $H_{C} \xrightarrow{H_{A}} O \xrightarrow{C} CH_{3}$ 

(b) All of the carbons have been labelled 1-11 in the structure below. Enter the chemical shift in the table. Indicate any ambiguous assignments.

1\_\_\_\_\_

2

3\_\_\_\_

4\_\_\_\_

5\_\_\_\_

6\_\_\_\_

7\_\_\_\_

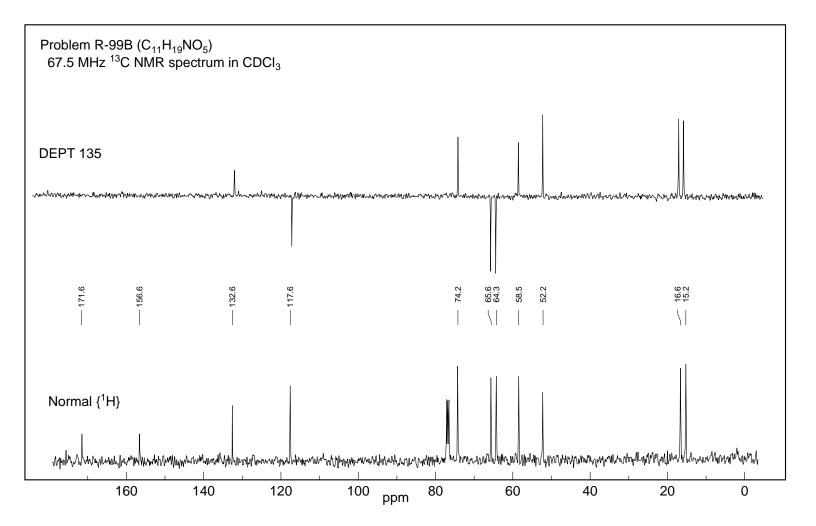
8\_\_\_\_

9\_\_\_\_

10\_\_\_\_

11\_\_\_\_

9 CH<sub>3</sub> 8 0 10 11 1 3 6 0 CH<sub>3</sub> 2 0 4 N 5 6 7



**Problem R-99B**. The <sup>1</sup>H and <sup>13</sup>C NMR spectra (normal and DEPT-135) of R-99A are provided. Your task is to assign the proton and carbon NMR spectra.

(a) All of the protons have been labelled A-L (there is no I). Enter the chemical shift, multiplet structure and approximate coupling constants in the table below. Use the format shown for X:

X e.g.: 
$$3.3 \, \delta$$
, qt,  $J = 7$ ,  $3 \, Hz$ 

A  $5.33$ , dq (dd?),  $J = 16$ ,  $2 \, Hz$ 

B  $5.22$ , dq (dd?),  $J = 10$ ,  $2 \, Hz$ 

C  $5.9$ , ddt,  $J = 16$ ,  $10$ ,  $6 \, Hz$ 

D  $4.58$ , dm (dt?),  $J = 6 \, Hz$ 

E  $5.45$ , broad d,  $J = 8 \, Hz$  (NH, broadened by  $J_{NH}$ )

F  $4.29$ , dd,  $J = 8$ ,  $3 \, Hz$ 

G 3.7, s

15

- H 1.20, d, J = 7 Hz
- J 4.05, qd J = 6, 3 Hz
- 3.33, dq, J = 9.8 Hz; 3.55, dq, J = 9.8 Hz Distereomeric -O-CH<sub>2</sub>-CH<sub>3</sub>
- L 1.10, t, J = 7 Hz

(b) All of the carbons have been labelled 1-11 in the structure below. Enter the chemical shift in the table. Indicate any ambiguous assignments.