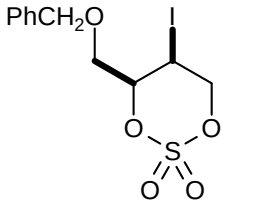
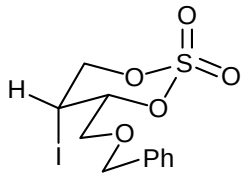
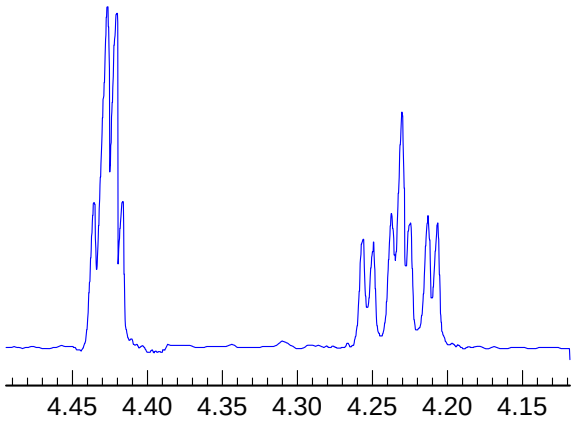
Problem R-07G ($C_{11}H_{13}IO_5S$)
300 MHz ^{1}H NMR spectrum in $CDCl_3$
Source: William Sanders/Kiessling (Reich digitized hard copy) g

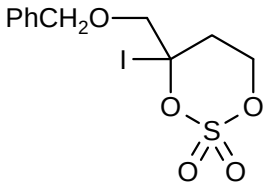






PLT ex-2-2007-08-gq.plt

Problem R-07G ($C_{11}H_{13}IO_5S$). In this problem you are given the part structure of a cyclic sulfate. Your task is to completely assign the ^1H NMR spectrum, and determine position of the substituents and the relative stereochemistry of the iodine and benzyloxymethyl substituents on the ring by interpretation of the spectrum. You may assume that the ring adopts a chair-like conformation.



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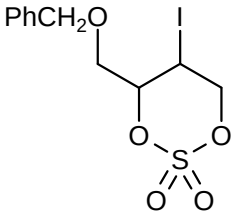

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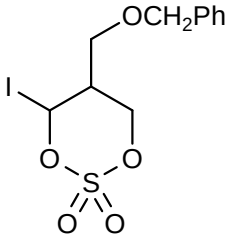

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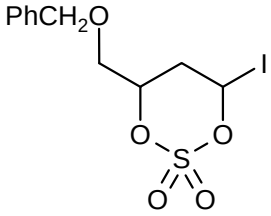


4

(a) Analyze the multiplets **A**-**G**. Report your results in the standard format: δ 9.3, dt, J = 14, 6 Hz, 3H. For each proton indicate which other protons (A-G), are coupled to it. You may use first order analysis.

A (1H) _____ Coupled to: _____

B (1H) _____ Coupled to: _____

C (2H) _____ Coupled to: _____

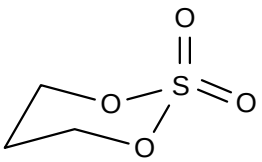
D (1H) _____ Coupled to: _____

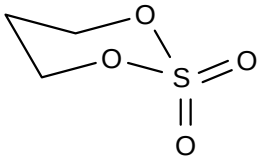
E (1H) _____ Coupled to: _____

F (1H) _____ Coupled to: _____

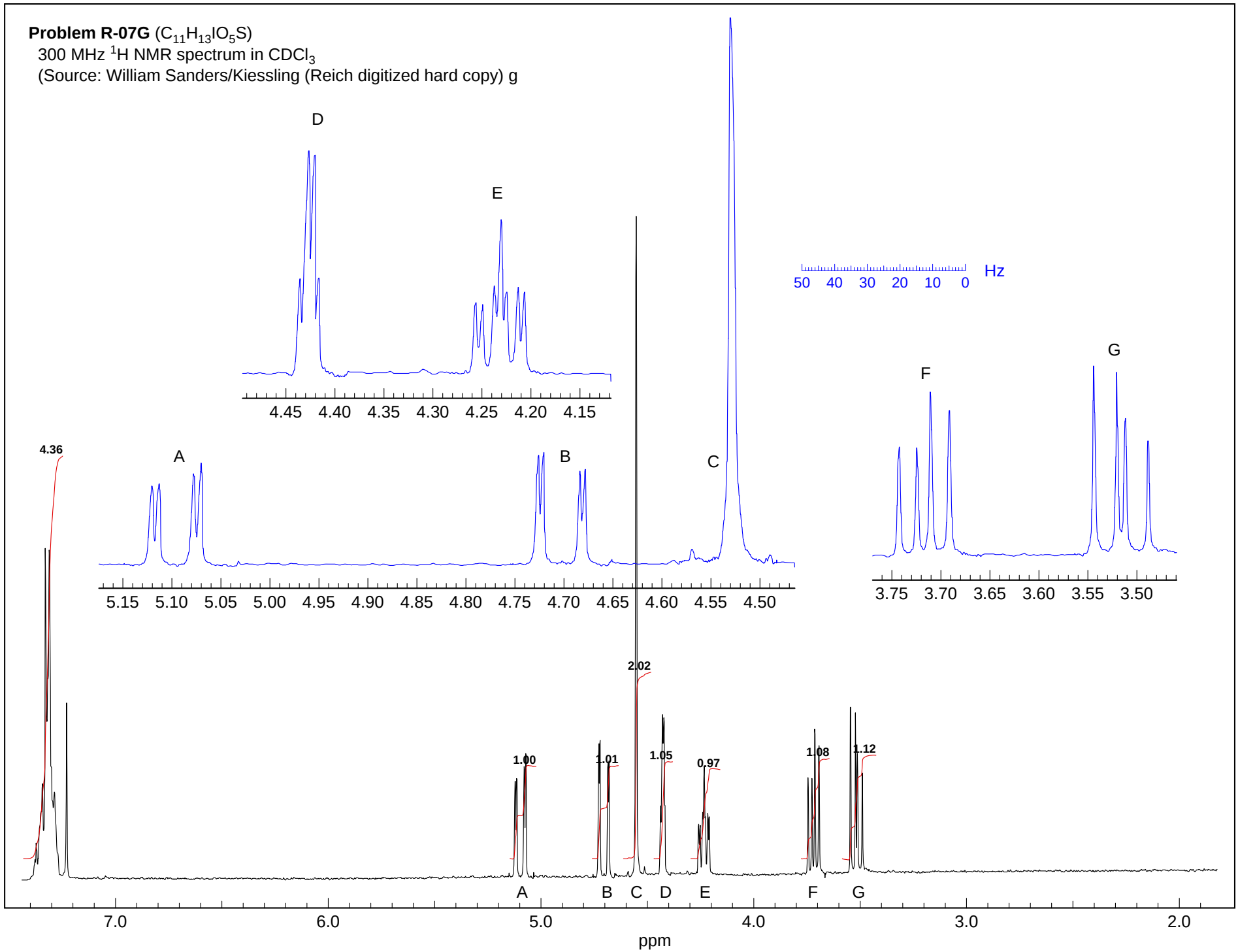
G (1H) _____ Coupled to: _____

(b) Which of the structural isomers is the correct one (**1** - **4**)? ____ If you chose **2**, **3**, or **4**, are the substituents cis or trans? ____ Draw a good representation of compound **R-07G** by adding substituents to one of the structures below, clearly showing the **stereochemistry**. **Label the protons with the etters A-G**.





(c) Describe specifically how you determined the stereochemistry.



PLT ex-2-2007-08-gq.plt

25

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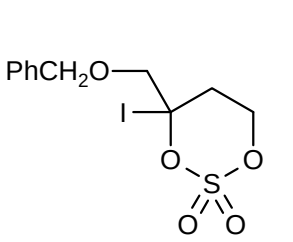

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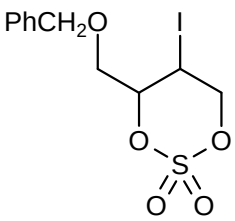

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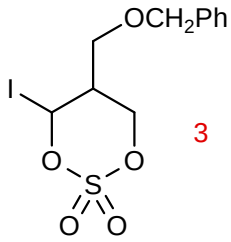

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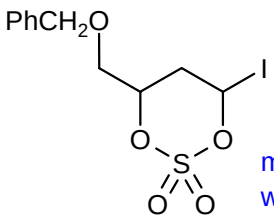


2

most common wrong answer

1 **2** **3** **4**

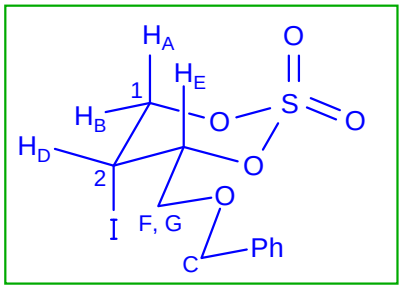
(a) Analyze the multiplets **A**-**G**. Report your results in the standard format: δ 9.3, dt, J = 14, 6 Hz, 3H. For each proton indicate which other protons (A-G), are coupled to it. You may use first order analysis.

11

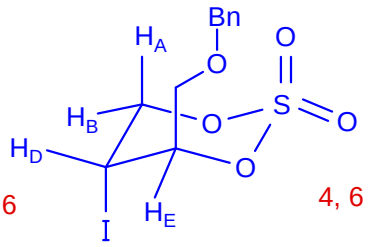
A (1H)	δ 5.1, dd, J=13, 2.5	Coupled to:	B (Jax-ax), D (Jax-eq)
B (1H)	δ 4.7, dd, J=13, 2	Coupled to:	A (Jax-ax), D (Jeq-eq)
C (2H)	δ 4.5, AB quartet, J= 12 Hz	Coupled to:	none
D (1H)	δ 4.4, q, J = 2 (actually a ddd)	Coupled to:	A (Jeq-ax), B (Jeq-eq), E (Jeq-ax)
E (1H)	δ 4.23, ddd, J = 8, 6, 2 Hz	Coupled to:	G, F, D
F (1H)	δ 3.7, ddd, J = 9, 6 Hz	Coupled to:	G, E
G (1H)	δ 3.5 dd, J = 9, 8 Hz	Coupled to:	F, E

(b) Which of the structural isomers is the correct one (**1** - **4**)? 2 If you chose **2**, **3**, or **4**, are the substituents cis or trans? cis Draw a good representation of compound **R-07G** by adding substituents to one of the structures below, clearly showing the **stereochemistry**. **Label the protons with the etters A-G**.

12

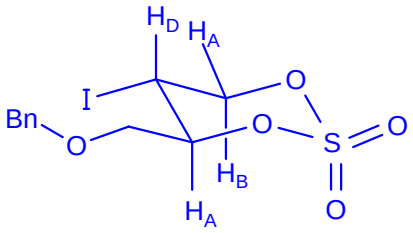


6, 6



4, 6

This would also fit the NMR spectrum, but the molecule would not be in this conformation with two groups axial



Common error: reversing A/B and F/G

(c) Describe specifically how you determined the stereochemistry.

2

H_D (C^2) must be equatorial, since there is an axial proton at C^1 (CH_2) and all couplings to H_D are of the J_{ea} or J_{ee} type. The benzyloxymethyl group could be either axial or equatorial based on the coupling constants, but if it were axial, the ring would flip, which can be ruled out from the couplings