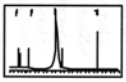
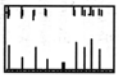
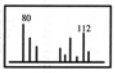
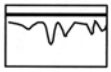
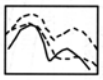
Spectral methods in structure determination

	H-1	*C-13*	*MS*	*IR/RAMAN*	*UV-VIS*	*ORD/CD*	*X-RAY*
•*Radiation type*	RF	RF	Not relevant	IR	UV to visible	UV to visible	X-ray
•*Spectral scale*	0-15 ppm	0-220 ppm	50-4000 amu	400-4000 cm^{-1}	200-800 nm	185-600 nm	Not relevant
•*Average sample*	≅ 1 mg	≅ 5 mg	< 1 mg	< 1 mg	< 1 mg	< 1 mg	Single crystal
•*Molecular formula*	Partial	Partial	Yes	No	No	No	Yes
•*Functional groups*	Yes	Yes	Limited	Yes	Very limited	Very limited	Yes
•*Substructures*	Yes	Limited	Yes	Limited	Limited	No	Yes
•*Carbon connectivity*	Yes	Yes	No	No	No	No	Yes
•*Substituent regiochemistry*	Yes	Yes	No	Limited	No	No	Yes
•*Substituent stereochemistry*	Yes	Yes	No	Limited	No	No	Yes
•*Analysis of isomer mixtures*	Yes	Yes	Yes (by GC/MS LC/MS)	Yes (by GC/IR)	No	No	Yes (if separate)
•*Purity information*	Yes	Yes	Yes	Yes	Limited	Limited	Limited
•*What is measured*	Peak areas Chemical shifts Coupling relaxation	Chemical shifts Coupling relaxation	Singly or multiple charged ions	Vibrational transitions	Electronic transitions	[α]	Relative atom positions *R/S* absolute stereochemistry
•*Typical units*	δ (ppm)	δ (ppm)	*m/z*	cm^{-1}	nm	nm	-
•*Typical representations*							 ORTEP

Hz, 1 H), 3.01 (dd, J = 7.5, 15.3 Hz, 1 H), 1.12–1.00 (m, 21 H); ^{13}C NMR δ 163.9, 161.9, 144.0, 133.5, 79.9, 64.6, 52.2, 31.0, 18.0, 12.5, 12.0; IR (neat) ν 2944, 1748, 1731, 1585, 1324, 1111 cm^{-1}; mass spectrum (FAB) m/z 372.2201 ($C_{18}H_{33}NO_5Si$ + H requires 372.2206).

4-Carbomethoxy-2-(2′*S*-methoxy-4′-hydroxy)-1,3-oxazole, 12d. To a solution of **12c** (12 g, 32 mmol) in THF (70 mL) at 0 °C was slowly added a solution of 1.0 M TBAF (48 mL, 48 mmol) in THF. After 12 h, solvent was removed in vacuo and the crude alcohol was purified via column chromatography eluting with EtOAc (9:1) to give 6.2 g (89%) of **12d** as an orange oil: $[\alpha]_D$ = +17.5° (c 1.0, $CHCl_3$); ^{1}H NMR δ 8.14 (s, 1 H), 3.86 (s, 3 H), 3.80–3.50 (m, 3 H), 3.36 (s, 3 H), 3.12 (dd, J = 6.1, 15.2 Hz, 1 H), 3.05 (ddd, J = 1.0, 6.5, 15.2 Hz, 1 H), 2.28 (brs, 1 H); ^{13}C NMR δ 162.9, 161.5, 144.0, 133.3, 79.0, 63.0, 57.5, 52.1, 29.6; IR (neat) ν 3418, 1732, 1586, 1324, 1110 cm^{-1}; mass spectrum (FAB) m/z 216.0869 ($C_9H_{13}NO_5$ + H requires 216.0872).

4-Carbomethoxy-2-[2′*S*-methoxy-3′(*E*)-penten-5′-al]oxazole, 13. To a solution of the alcohol **12d** (2.5 g, 11 mmol) in CH_2Cl_2 (12 mL) at 0 °C were added DMSO (11 mL, 160 mmol) and Et_3N (11 mL, 80 mmol), and the mixture was stirred for 10 min. Solid SO_3·Pyr (9.1 g, 57 mmol) was then added in one portion, and the reaction was stirred for 30 min at 0 °C and

Unsaturation Number (UN)

Given the molecular formula of an unknown, can guess the combined number of rings and multiple bonds (called the "unsaturation number").

$$UN = \#C - \frac{\#H}{2} - \frac{\#Hal}{2} + \frac{\#N}{2} + 1$$

Unsaturation Number (UN)

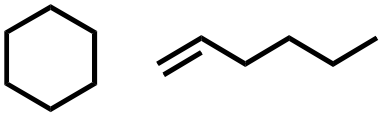
$$UN = \#C - \frac{\#H}{2} - \frac{\#Hal}{2} + \frac{\#N}{2} + 1$$

For $\mathbf{C_6H_{12}}$,

$$UN = 6 - \frac{12}{2} - 0 + 0 + 1$$

$$UN = 1$$ *(ring or double bond)*

Candidate structures for $\mathbf{C_6H_{12}}$:



(1 ring) *(1 double bond)*