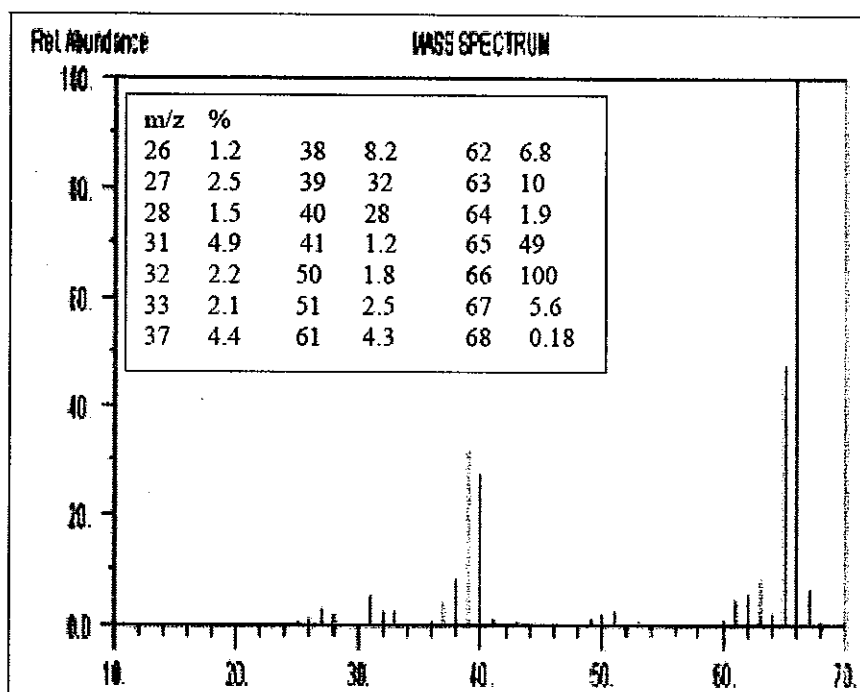


2009.Analytical Chemistry

Part I

- How do you use NMR spectroscopy to distinguish the following two isomers from each other? (a) $\text{CH}_3\text{-CH=C(CN)-CH}_2\text{-CN}$ and (b) $\text{CN-CH=C(CH}_3\text{)-CH}_2\text{-CN}$ (10%)
- IR stretching band for the ordinary CO is found at 2170cm^{-1} , calculate the position of C^{18}O stretching band. (10%)
- Given relative abundance : $^2\text{H}/^1\text{H}=0.016$, $^{13}\text{C}/^{12}\text{C}=1.11$, $^{15}\text{N}/^{14}\text{N}=0.38$, $^{17}\text{O}/^{16}\text{O}=0.04$, $^{18}\text{O}/^{16}\text{O}=0.20$. Find the molecular structure of a compound with the Mass spectrum shown below (with relative intensities of major peaks). You have to give your reasoning. (10%)



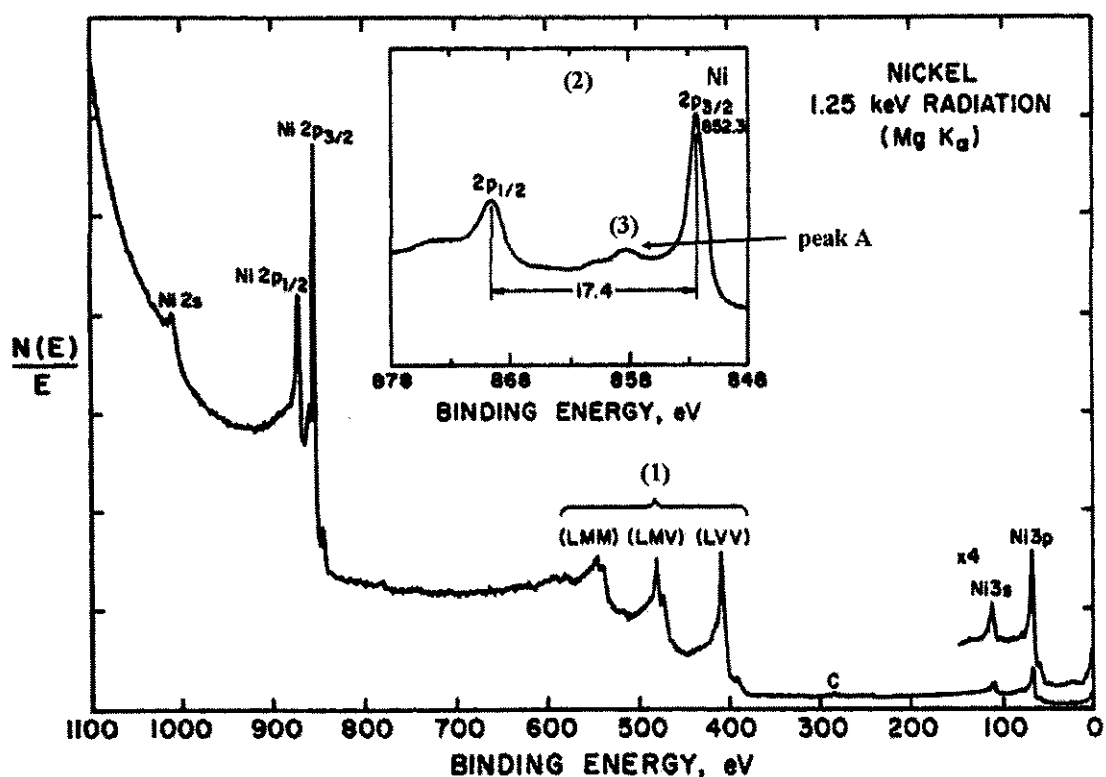
- Please describe the fundamental principle of H-NMR, which should at least include the following: Which part of the electromagnetic spectrum is used for NMR excitation? What compound is used for NMR reference? What's the advantages of using higher magnetic field in NMR? Why the NMR sample tube is usually rotated at high speed? (20%)

Qualify – Advanced Analytical Chemistry

Part II

1. Following is an XPS spectrum of nickel. The X-ray source is Mg $K\alpha$ which the photon energy is 1.25keV. Please answer the questions according to the figure. (40pts)

- (1) There are three peak sets labeled as (LMM), (LMV) and (LVV). What's the name of the process which results in these peaks? What is the mechanism of it? What are the notations (LMM), (LMV) and (LVV) mean?
- (2) The inset of the spectrum is the magnification of Ni2p signals. Please explain the meaning of $2p_{3/2}$ and $2p_{1/2}$. Why there are two peaks of 2p signal?
- (3) There is a small peak labeled as "peak A" which is close to Ni $2p_{3/2}$ peak in the inset. Which of the following specie is responsible for it (peak A)? Please **explain the reason**. (i) NiH (ii) Ni $_2$ O (iii) Ni(OCH $_3$) $_4$ (iv) Ni(NH $_3$) $_6$
- (4) The binding energy of Ni $2p_{3/2}$ is 852.3eV. Assume the workfunction of the XPS spectrometer is 4.5eV, what is the kinetic energy of the electrons which ejected from $2p_{1/2}$?



2. There are three different modes for acquiring Atomic Force Microscopy (AFM) spectra. (1) Contact mode, (2) non-contact mode, and (3) tapping mode. Please explain the differences of them. (You can draw a sketch) (10pts)