Report 3. Supplemental Materials Section

A. Solvent study of keto-enol equilibrium of acetylacetone by NMR - online report form

B. Supplemental Material for M (acac)$_x$ - Synthesis, IR and Magnetic Susceptibility

Synthesis and mp:

Pure Product	Mass _____g	Yield _____%	mp _____°C (observed) mp _____°C (expected)
***para*-Toluic acid** (or give standard used)	NA	NA	mp _____°C (observed) mp _____°C (expected)

IR Spectra :

Attach the three labeled IR spectra in the following order. Label the major peaks for applicable functional groups in each spectrum on the hardcopy.

(1) Hacac (in EtOH) ;

(2) M(acac)$_x$ (in EtOH);

(3) M(acac)$_x$ (in KBr pellet.)

C. Supplemental Materials for M(acac)$_x$

Paramagnetic Susceptibility: Attach class handout (Mar 4 or 5) showing the calculation of χ, χ_M^{corr}, μ_{eff} and **n**. Attach the labeled HNMR spectra with $\Delta\nu$ indicated directly in Hz on the hardcopy. (See next page.)

C. Supplemental Materials for M(acac)$_x$ (cont'd)

Paramagnetic Susceptibility:

Find attached the labeled HNMR spectra with $\Delta\nu$ indicated directly in Hz on the chart. (Note: SI units have been used throughout (also for the MW) and the results reported to four significant figures. Please note that there are updated equations (12), (13), (14), (15) and (17) in the CH 362 Lab Manual (p 20-22):

$$\Delta\nu/\nu \approx \Delta\mathbf{B} / (\mu_0 \mathbf{H}) = -1/3\ (\chi_{solution} - \chi_{solvent}) \quad (13)$$

$$\chi_{mass}\ (solute) = -3/m\ \Delta\nu/\nu + \chi_{mass}\ (solvent) \quad (14)$$

$$\chi_M = (MW)\ \chi_{mass}\ (solute) \quad (12)$$

$$\mu_{eff} = 797.8\ (T\ |\chi_M^{corr}|)^{1/2}\ \text{Bohr Magneton} \quad (17)$$

$$\mu_{eff} = (n(n+2))^{1/2}$$

Temperature = _____ K Concentration m = _____kg/m^3

Observed $\Delta\nu$ = _____Hz ν_r for the spectrometer = 400 X 10 ^{6}Hz

Sample calculations using the appropriate number of significant figures, and correct units of measure for each quantity, are given below (note: the solute is M(acac)$_3$ and the solvent is toluene):

χ_{mass} (solute) = χ_{mass} - (-9.0 X 10^{-9} m^3/kg) (solvent) _____

χ_M = _____

$\chi_M^{corr} = (\chi_M - \chi_{diamag}) = \chi_M - \Sigma$ Pascal's Constants _____

where Σ Pascal's Constants = - 2.1682 X 10^{-9} m^3 /mol(Fe complex); -2.1782 X 10^{-9} m^3 /mol (Cr complex) and are used to correct for the diamagnetic contribution from the M(acac)$_3$.

$|\chi_M^{corr}|\ T$ = _____

μ_{eff} = _____

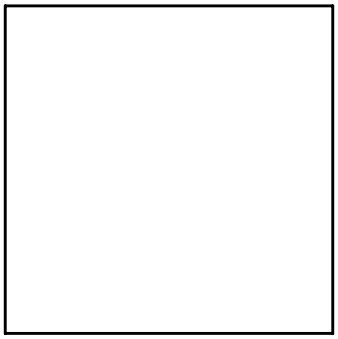
Experimental number for **n** = _____

Theoretical number for **n** in high spin **M(acac)**$_x$ = _____

Theoretical number for **n** in low spin **M(acac)**$_x$ = _____

D. Supplemental Materials for HNMR Assignments for Unknown Aromatic Compound

Here is the proposed structure and compound name(s). The principal functional group is found at position 1 and the subordinate substituent at position 2 or 3 or 4 (depending on whether it is ortho or meta or para):



Compound Name(s):_____

_____

Assignment	Observed Chem. Shift ppm
H-2	
H-3	
H-4	
H-5	
H-6	

Other Significant HNMR Signals (list group and measured ppm)

_____ _____ _____

_____ _____ _____

_____ _____ _____

Comparison of measured CNMR shifts between three possible isomers:

Assignment	Ortho Calculated Chem. Shift ppm	Meta Calculated Chem. Shift ppm	Para Calculated Chem. Shift ppm	Actual Measured Chem. Shift ppm
C-1				
C-2				
C-3				
C-4				
C-5				
C-6				

Other ^{13}C Signals

_____ _____ _____

_____ _____ _____

Pascal's Triangle, drawn to scale for the protons in the _____isomer:

H2

H3

H4

H5

H6

Comparison of Measured vs. Calculated Coupling constants for the _____ isomer:

Identify ortho, meta, and possibly para coupling constants for each proton on the ring. In most cases, 5 coupling constants (J_{mn}) can be obtained: three J_{ortho} and two J_{meta} for the ortho substituted ring, and two J_{ortho} and three J_{meta} for the meta substituted ring. For the para substituted compounds, there in only one J value.

Typical values for J_{ortho} = 6.0-9.0 Hz; J_{meta} = 1.0-3.0 Hz; J_{para} = 0-1.0 Hz. Obtain as many aromatic proton coupling constants as possible from the given peak pick table.

	Measured Coupling Const. for Ortho Substituted, Hz	Measured Coupling Const. for Meta Substituted, Hz	Measured Coupling Const. for Para Substituted, Hz
	J_{3-4} = J_{3-5} =	J_{2-4} = J_{2-6}=	J_{2-3} = J_{3-2} =
	J_{4-3} = J_{4-5} = J_{4-6} =	J_{4-2} = J_{4-5}= J_{4-6}=	
	J_{5-3} = J_{5-4} = J_{5-6} =	J_{5-6}= J_{5-4}=	
	J_{6-5} = J_{6-4} =	J_{6-2}= J_{6-4}= J_{6-5}=	

List any other coupling constants that are observed.

_____

_____

COSY or DQF COSY Give the measured ppm value for the H_N and the H_R cross peaks (N is the label in the standard structure for a hydrogen in the ring and R is the label for the hydrogen correlated to the N hydrogen). Not all isomers will show all correlations.

	Ortho		**Meta**		**Para**	
Assignment **N**	**H_N (ppm)**	**H_R(ppm)**	**H_N (ppm)**	**H_R(ppm)**	**H_N (ppm)**	**H_R(ppm)**
2						H3 =
3		H4 =				H2 =
4		H3 = H5 =		H5 =		
5		H4 = H6 =		H4 = H6 =		H6 =
6		H5 =		H5 =		H5 =

List any other coupling that are observed in the COSY.

_____

_____

HSQC Give the measured ppm value for the H_N and C_N cross peaks for the HSQC (N is the label in the standard structure). Not all isomers will show all correlations.

Assignment **N**	**H_N (ppm) if Ortho**	**C_N (ppm) if Ortho**	**H_N (ppm) if Meta**	**C_N (ppm) if Meta**	**H_N (ppm) if Para**	**C_N (ppm) if Para**
2	X	X				
3			X	X		
4					X	X
5						
6						

Other Significant HSQC Signals

_____ _____ _____

_____ _____ _____

HMBC (Give $\underline{J^3}$ couplings only) Identify the number of the carbon in the ring and in the substituent groups (if any contain carbon and/or hydrogen) that are observed interacting with each hydrogen (not all isomers will show all correlations and there is usually more than one correlation per H_N) .

Assignment H_N	**Correlates with C_N**
2	
3	
4	
5	
6	
Other Significant J^3 HMBC Signals Between H and C	