

## Laboratory Report Instructions

### 2,4-Pentanedionato and 1,1,1-Trifluoro-2,4-pentanedionato Complexes of Co(III)

#### Percent Yield

Calculate the approximate percent yields of  $\text{Co}(\text{acac})_3$  and of the mixture of  $\text{Co}(\text{tfa})_3$  isomers, assuming that the hydrated cobalt carbonate has the composition  $\text{CoCO}_3 \cdot 1.2\text{H}_2\text{O}$  and is the limiting reagent.

#### TLC Analysis

Calculate the  $R_f$  values for each component that was seen for TLC analysis of  $\text{Co}(\text{acac})_3$  and  $\text{Co}(\text{tfa})_3$ . Provide a brief, qualitative rationale for the differences in the  $R_f$  values.

#### Infrared Analysis

Identify the C-H stretching and carbonyl stretching bands in the infrared spectra of  $\text{Co}(\text{acac})_3$  and one of the  $\text{Co}(\text{tfa})_3$  isomers. In addition, identify the C-F stretching absorptions in the  $\text{Co}(\text{tfa})_3$  spectrum.

The frequencies of the carbonyl absorptions are shifted to lower wavenumbers than one finds for the carbonyl stretch of an ordinary dialkyl ketone. Provide a brief, reasonable explanation for the direction of the shift.

#### NMR Analysis

##### $\text{Co}(\text{acac})_3$

Assign the signals in both the  $^{13}\text{C}$  and  $^1\text{H}$  NMR spectra. (Note: the integration in the  $^1\text{H}$  spectrum may not correspond exactly to the expected values, probably due to different relaxation times of the different types of protons.)

##### $\text{Co}(\text{tfa})_3$

Decide, on the basis of the of the  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra, which TLC component is *mer*- $\text{Co}(\text{tfa})_3$  and which is *fac*- $\text{Co}(\text{tfa})_3$ . Models or careful drawings of the compounds will be highly useful references for this analysis. Provide a rationale for your decision.

Assign the signals (to the extent possible) for the  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra. In cases where an assignment cannot be made with complete assurance, explain why a decision cannot be made based on the available data. (Note: the integration in the  $^1\text{H}$  spectrum may not correspond exactly to the expected values, probably due to different relaxation times of the different types of protons.)

Assign, to the extent possible, the  $^{13}\text{C}$  NMR signals of the mixture of *mer*- $\text{Co}(\text{tfa})_3$  and *fac*- $\text{Co}(\text{tfa})_3$ . In cases where an assignment cannot be made with complete assurance, explain why a decision cannot be made based on the available data.

In addition, measure and report the  $^{13}\text{C}$ - $^{19}\text{F}$  coupling constant (in Hz) for the trifluoromethyl carbons (the value of the coupling constant will be nearly the same for each of the different trifluoromethyl groups) and for the carbonyl groups that are adjacent to the trifluoromethyl groups (again, the coupling constants will be nearly the same within this group of carbonyl carbons).