A. Labeling of L1-L4 with $^{99m}$Tc(CO)$_3^+$. 
B. Absorption and Fluorescence Studies. 
C. X-ray Structure of L2.
A. Labeling of L1-L4 with $^{99m}$Tc(CO)$_3^+$. Labeling was accomplished in two steps using the readily prepared IsoLink™ kits (Mallinckrodt) to generate the $[^{99m}$Tc(CO)$_3$(H$_2$O)$_3]^+$ precursor (100 µl), which was introduced to a methanol solution (0.5 mL) of the appropriate ligand (0.25 mg). The sealed vial was heated at 90°C for 20 minutes. After cooling, the reaction was checked for purity via HPLC using a Vydac C18 column (4.6mm x 25cm x 5µm) and methanol as eluant. The purity, analyzed using C18 HPLC, was >95% radiochemical purity. The labeling yields were all > 85%, and achievable at levels as low as 1 µg / ml.

![Radiochromatogram](image)

**Figure S1.** Radiochromatogram of the crude reaction product, $[^{99m}$Tc(CO)$_3$L3]$^+$.  

B. Absorption and Fluorescence Studies

*Absorbance Spectra and Extinction Coefficient*

All absorbance measurements were taken using a Varian model CARY -50 Bio UV-Visible spectrophotometer. **Figure 3** shows the absorption spectrum of [Re(CO)$_3$L4]Br in ethylene glycol. Absorption maxima are seen near 321 and 414 nm. The extinction coefficients at 321 and 414 nm are 17729 and 1215 mol$^{-1}$ cm$^{-1}$ respectively.
Figure S2: Absorbance spectra of [Re(CO)₃L₄]Br in ethylene glycol at room temperature.

Fluorescence Spectroscopy

Steady state fluorescence measurements were recorded with a PTI fluorimeter. Emission was monitored from 400 to 800 nm with an excitation wavelength of 321 nm in 1 nm increments with an integration time of 0.5 sec. Samples of [Re(CO)₃L₄]Br were prepared in ethylene glycol (1×10⁻⁵ M).
Figure S3: Emission spectra of [Re(CO)$_3$L$_4$]Br in either air equilibrated or nitrogen equilibrated ethylene glycol.

Emission spectrum of the rhenium probe shows a maximum at 555 nm. As shown in Figure 4, the equilibration of the samples with a nitrogen gas atmosphere drastically increases the quantum yield of the rhenium probe. Using a standard reference material, in this case ruthenium bipyridine, a quantitative value of quantum yield can be calculated by using the following equation where $Q$ and $Q_R$ are the quantum yields, $I$ and $I_R$ are the integrated emission intensities, $OD$ and $OD_R$ are the optical densities, and $n$ and $n_R$ are the refractive indexes of the solvents of the unknown and reference materials (subscript R indicating reference).

$$Q = Q_R \frac{I}{I_R} \frac{OD_R}{OD} \frac{n^2}{n^2_R}$$

Table 1 gives the values measured and resulting quantum yields for [Re(CO)$_3$L$_4$]Br.

Table 1: Quantum Yield Data

<table>
<thead>
<tr>
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<th>Rhenium in Ethylene Glycol (Air Equil.)</th>
<th>Rhenium in Ethylene Glycol (N$_2$ Equil.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Emission Intensity$^2$</td>
<td>$1.99 \times 10^8$</td>
<td>$3.23 \times 10^8$</td>
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</table>
Refractive index | 1.43 | 1.43  
Quantum Yield  | 0.0091 | 0.015 |

†Emission was measured between 400 and 800 nm with an excitation of 321 nm in 1 nm increments with an integration time of 0.5 sec. Excitation bandpass was 1 nm and emission bandpass was 2 nm.

Time resolved fluorescence decays were recorded using a PTI fluorimeter. The 321 nm output of the flash lamp was the excitation wavelength for emission lifetime measurements. The emission of the [Re(CO)₃L₄]Br was monitored at 555 nm. The fluorescence intensity decay was fit to a single exponential function: \( I = \alpha \exp \left(-t/\tau\right) \), where \( \tau \) is the fluorescence lifetime and \( \alpha \) is the preexponential factor. In ethylene glycol under nitrogen atmosphere the fluorescence lifetime of [Re(CO)₃L₄]Br was measured to be 16.7 \( \mu \)sec.

C. X-ray Structure of L2.

![Figure S4. The structure of L2.](image-url)