

## Singlet-Oxygen Measurements using the Fluorolog® System

FL-15

ELEMENTAL ANALYSIS

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### Introduction

The chemistry, spectroscopy, and reactivity of singlet oxygen,  $^1\text{O}_2$ , is of considerable interest in environmental applications, novel organic syntheses and mechanisms of photo-oxygenation.<sup>1</sup> Recently, research has been directed towards understanding the biological effects of  $^1\text{O}_2$ , such as the photodynamic destruction of cells in the presence of a dye, oxygen, and light.

An understanding of the mechanisms that produce  $^1\text{O}_2$  is needed for the synthesis of new anti-cancer agents used in photodynamic therapy. Spectroscopic properties of the dye and  $^1\text{O}_2$  may be characterized using fluorescence spectroscopy. The near-IR emission of  $^1\text{O}_2$  is around 1280 nm. The Fluorolog® system easily detects this signal with appropriate emission gratings and suitable detector. The standard R928P photomultiplier tube responds to photons up to 800 nm. InGaAs detectors extend this range to nearly 1700 nm.

The DSS-IGA020L InGaAs detector is an excellent choice for  $^1\text{O}_2$  measurements. It operates at liquid- $\text{N}_2$  temperature to minimize thermal noise. Usable from 800–1700 nm, the InGaAs detector provides a smooth overlap with the R928P photomultiplier tube.

### Experimental method and results

To demonstrate  $^1\text{O}_2$  detection generated using a transition-metal complex, tris-2'-2'-bipyridine ruthenium(II) chloride,  $[\text{Ru}(\text{bpy})_3]\text{Cl}_2$ , in  $\text{D}_2\text{O}$ , was used as a  $^1\text{O}_2$  generator.<sup>2</sup> The optical density of the solution was 0.3 A over the  $\text{D}_2\text{O}$

baseline at 450 nm. To enhance the  $^1\text{O}_2$  yield, the solution was saturated with  $\text{O}_2$  by bubbling pure  $\text{O}_2$  through it for 5 min before measurement. The  $\lambda_{\text{max}}$  of the  $[\text{Ru}(\text{bpy})_3]^{2+}$  complex is near 450 nm.

A Fluorolog® spectrofluorometer, equipped with a 450-W xenon lamp, single-grating monochromators, and near-IR accessories (DSS-IGA020L InGaAs detector and emission grating at 600 grooves  $\text{mm}^{-1}$ , blaze = 1000 nm) was used to measure the  $^1\text{O}_2$  emission spectrum. A high-pass filter ( $\lambda = 780 \text{ nm}$ ) acted as an order-sorter, necessary when such large wavelength-ranges are spanned. Fig. 1 shows the emission spectrum with an integration time of 0.1 s and a bandpass of 12 nm. Note the excellent signal-to-noise ratio.

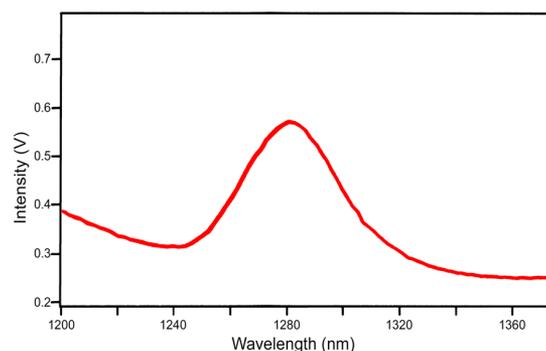


Fig. 1. Emission spectrum of  $^1\text{O}_2$  generated by  $[\text{Ru}(\text{bpy})_3]\text{Cl}_2$  in  $\text{D}_2\text{O}$ .

<sup>1</sup> Wasserman, H.H.; Murray, R.W., in *Singlet Oxygen*; Academic Press: New York, 1979.

<sup>2</sup> Mulazzani, et. al. *J. Phys. Chem.* **1994**, 98, 1145.

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