

Liquid *in situ* Fluorescence Measurements

FL-34

ELEMENTAL ANALYSIS

FLUORESCENCE

GRATINGS & OEM SPECTROMETERS

OPTICAL COMPONENTS

FORENSICS

PARTICLE CHARACTERIZATION

R A M A N

SPECTROSCOPIC ELLIPSOMETRY

SPR IMAGING



Hellma®

No need to sample—simply take your readings directly in place in locations such as reaction vessels

Collecting samples during a continuous process can be tedious and even dangerous if hazardous materials are involved. In applications such as reaction monitoring, sampling can also be difficult to accomplish without interfering with the process being analyzed or altering the data. Sampling can change temperature and pressure conditions, or allow volatile components to evaporate, all leading to errors in the measurement. Furthermore, *in vitro* sample handling and measurement takes time and effort, meaning your data are always behind the actual state of the process.

All of the above make *in situ* measurements very appealing. These can be easily accomplished using a fiber-optic fluorescence probe (model 661.057-UV by Hellma Analytics). (See Figs. 1 and 2.) The fiber-optic cables of this probe are specifically designed to match the beam geometry of our F-3000 series fiber-optic couplers for optimal coupling to HORIBA Scientific fluorometers. Gone are all the opportunities for error during pipetting, mixing, heating, and cleaning, as well as the need for space for a fume hood.

Experimental example

The figure (Fig. 3) on the back of this sheet shows an example of a fiber-optic probe *in situ* measurement using such equipment, in this case for the titration of a nickel-complex by NaOH. The nickel complex is based on an aromatic pyrene structure which has delocalized π -bonds (Fig. 4).

At an excitation wavelength of 340 nm, we observe



Fig. 1. Hellma Analytics fluorescence probe 661.057-UV.



Fig. 2. Specially designed fiber-optic cables for an optimized interface with the HORIBA Scientific F-3000 fiber-optic coupler

emissions at 375 nm and 395 nm. These emissions, seen under acidic conditions (nitric or hydrochloric acid), are derived from the amine protonation on the nickel-monomer complex. When NaOH is added, the ammonium group loses its acidic property. This is indicated by a reduction of the fluorescence, which is caused by photoinduced electron-transfer (PET).

In parallel, a large band centered at about 480 nm appears as the result of a small intermolecular interaction (π - π interaction between moieties of two monomers). (See Fig. 5.) Such “molecules”, also termed “excimers”, only exist in an excited state.

Conclusions

In situ measurements using a Hellma fiber-optic probe and HORIBA Scientific instrumentation enable fluorescence measurements of processes in real-time. Additionally, this combination eliminates the risk of altering the data by errors introduced by sampling or multi-step sample preparation, and helps minimize exposure to hazardous or environmentally-sensitive materials.

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Fig. 3. Experimental set-up for *in situ* measurements: HORIBA Scientific Fluorolog@-3 in the upper-right corner connected to a Hellma fluorescence probe installed in the reaction vessel in the lower-left corner.

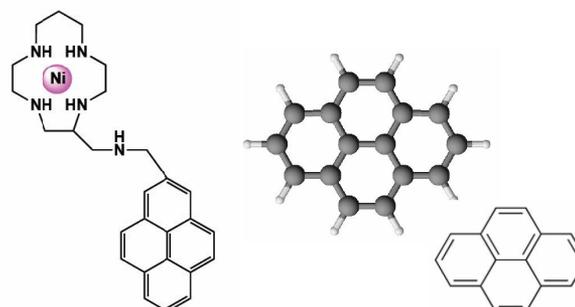


Fig. 4. Nickel complex (left) and pyrene structure, $C_{16}H_{10}$ (center and right) on which it is based. The π -electrons are delocalized throughout the entire pyrene structure.

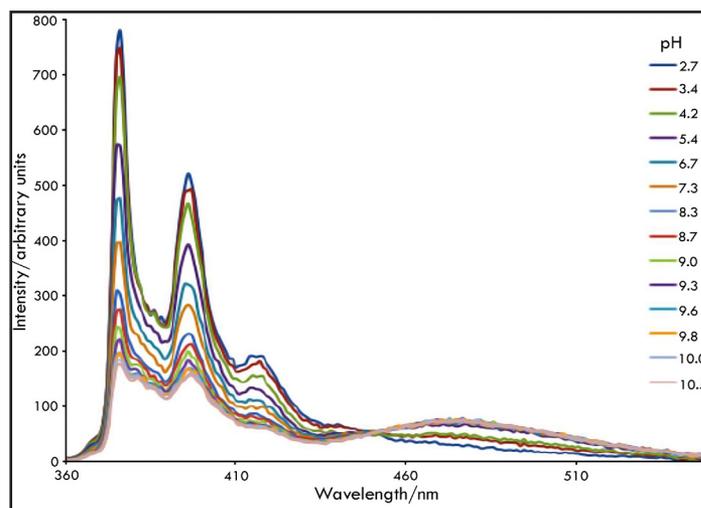


Fig. 5. Evolution of fluorescence spectrum of the nickel complex as pH is raised. $\lambda_{exc} = 340$ nm.



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