Overview

Liquid injection field desorption ionization (LIFDI) is a soft high vacuum ionization technique based on FIDDI. We present a very high resolution Thermo Scientific™ Exactive™ Plus Orbitrap mass spectrometer combined with an LIFDI ion source installed at the back end of the ion path in addition to the ESI source at the front end. This way difficult non-polar compounds such as organometallic catalysts can be characterized which are not accessible by ESI.

Introduction

Organo-Metallic compounds are often subject to reaction with air or moisture and do not tolerate pretreatment or attachment of other ions. This makes their difficult analytical challenges for assay with mass spectrometry.

Field Desorption (FD) has proven an effective way to ionization of these materials resulting in M+ ions. Field Desorption (FD) has proven an effective way to ionization of these materials resulting in M+ ion species via a soft ionization process. LIFDI shares the ionization with FI/FD but transfers the sample by a soft ionization process.

Methods

The sample is placed in a vial. A syringe needle punctures the vial’s septum and the capillary is lowered through the needle into the sample for ca. 3 seconds. The vacuum of the LIFDI ion source pulls a plug of sample into the capillary and forces it to the emitter filament. This process can be done by an autosampler resulting in automated LIFDI.

With the atmospheric source ESI at the front end and the high vacuum source LIFDI at the back end of the ion path, the Exactive Plus LC-MS can be switched back and forth between LIFDI and ESI LC/MS operation without any venting (see Figure 1). Just loading the respective tuning and using the LIFDI or ESI capillary is required.

Figure 1. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source from Linden CMS, Germany.

Figure 2. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source.

Figure 3. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source.

Figure 4. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source.

Figure 5. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source.

Figure 6. Instrument: Exactive Plus Orbitrap LC-MS with LIFDI source.

Results

• Fig. 2 shows clean spectra of an air sensitive rhodium complex with intense molecular ion M+ without fragmentation.
• Fig. 3 shows the spectra of a reactive ruthenium containing metathesis catalyst.
• Fig. 4 shows the measured (above) versus calculated (below) isotope pattern of the catalyst's intact molecular ion.
• Fig 5 shows another Ru species and the loss of CO with a nice match between calculated and measured isotope pattern.
• Fig 6 provides the spectra of a PEG 1500 mixture dominated by singly charged ions.

Conclusion

Reactive air/moisture sensitive compounds can be accessed with the Exactive Plus Orbitrap instrument equipped with LIFDI/ESI combination without excessive experimental effort. Changing from one technique to the other is quick and very convenient.

LIFDI spectra can exhibit intense peaks of intact molecular radical ions M+• from compounds which do not provide meaningful mass spectra by EI or ESI methods.

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