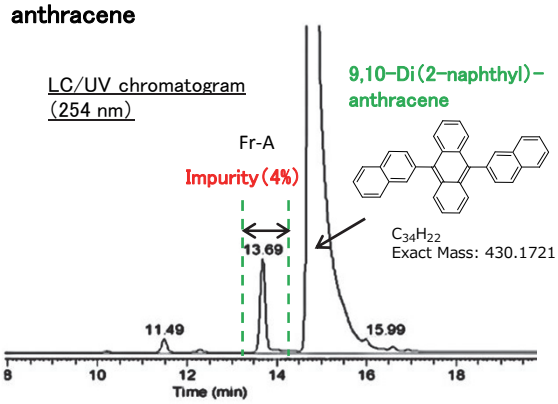
Structural analysis of trace impurity in OLED material by NMR with cryo-cooling probe

It is supposed that the trace impurities (or degradation compounds) in OLED material have a bad influence on the lifetime and emitting properties of OLED. So, it is important to investigate their structures.
In this poster, a trace impurity of OLED material was concentrated by HPLC fractionation and analyzed by NMR with cryo-cooling probe.

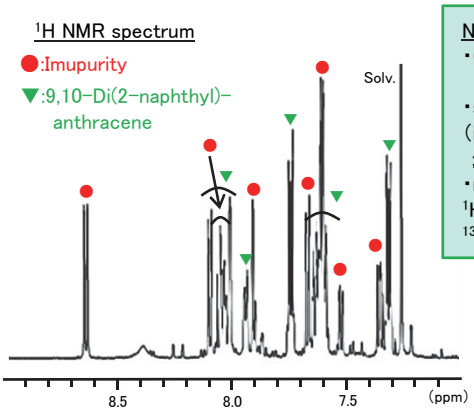
HPLC fractionation

・HPLC fractionation of impurity in 9,10-di(2-naphthyl)-anthracene



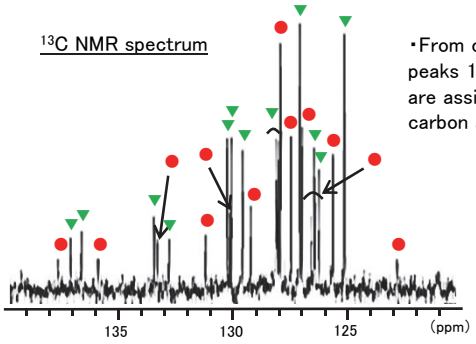
Weight of fractionated impurity ; 0.6 mg

1D NMR



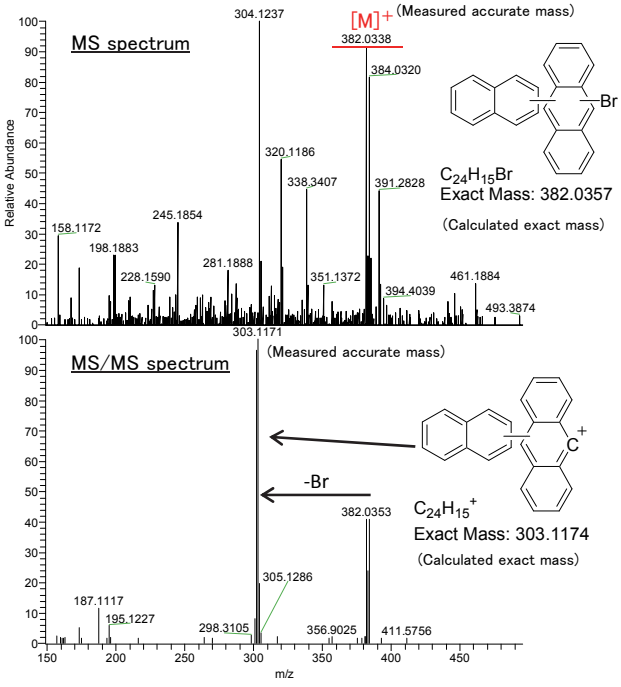
NMR measurements
・600MHz NMR
(＋cryo-cooling probe)
・sample weight;0.6 mg
(impurity absolute weight)
;0.1 mg
・number of scan
^{1}H;128 times (about 20min)
^{13}C;20000 times(about 16hr)

・^{1}H NMR and ^{13}C NMR spectra show that Fr-A is mixture of 9, 10-Di(2-naphthyl)anthracene(▼) and impurity(●).



・From characteristic chemical shift, peaks 123 ppm(^{13}C) and 8.6 ppm (^{1}H) are assigned as the halogen adjacent carbon and proton.

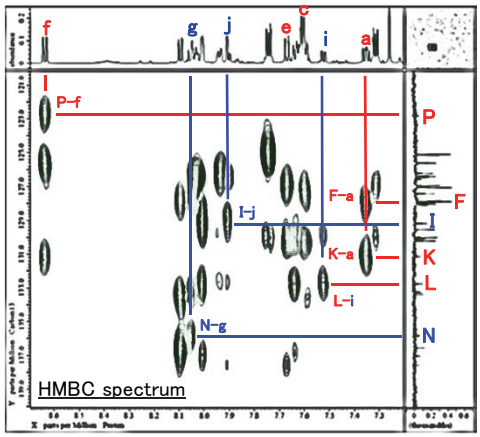
LC/MS

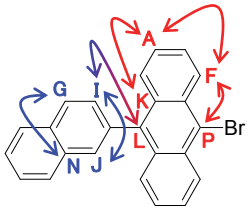


☺ Naphthylanthracene unit and Br are induced.

☹ Substitution sites are undecided.

2D NMR





As a result of correlation peak analysis, it is determined that the impurity is 9-bromo-10-(2-naphthyl)anthracene.

Conclusion

・Trace impurity contained in OLED material was concentrated by HPLC fractionation.
・The elemental composition and partial structure were identified by LC/MS measurement.
・Using NMR with cryo-cooling probe, detailed structure of trace impurity was determined.

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