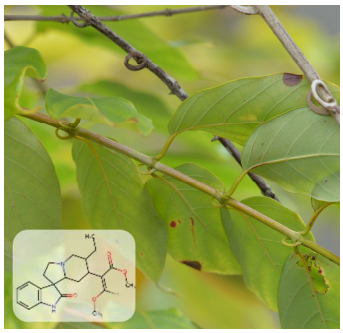
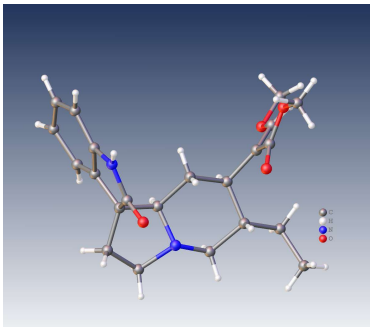
Molecular Structure Analysis of Alkaloids

Product used : Electron Diffractometer

Structure analysis of alkaloids with XtaLAB Synergy-ED, JEOL MS and NMR



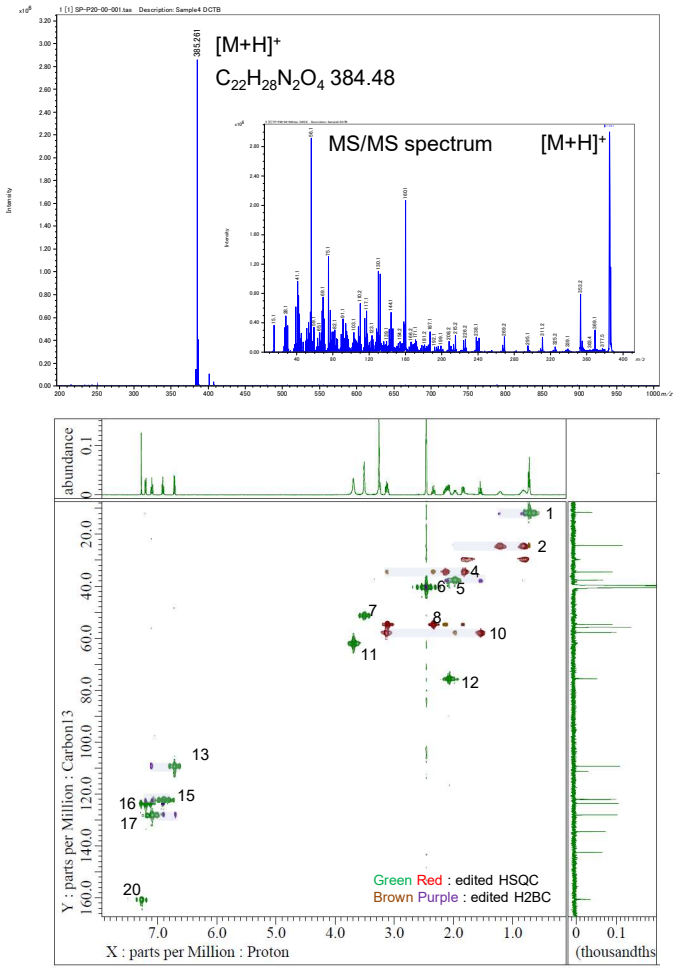
Uncaria rhynchophylla Miquel, Structural formula of Rhynchophylline



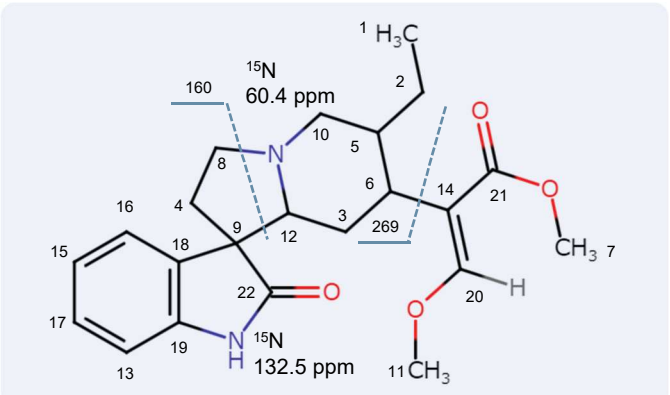
Electron diffraction structure analysis of Rhynchophylline, XtaLAB Synergy-ED

Alkaloids are naturally occurring organic compounds that contain at least one nitrogen atom. They are mostly plant-based natural compounds and often used as medicines. Alkaloid biosynthesis is varied; they are characterized by a great structural variety and a complicated molecular structure. XtaLAB Synergy-ED allows molecular structural analysis of submicron particles of laboratory chemicals, pharmaceutical raw materials and so on. In addition, the comprehensive analysis of the JEOL mass spectrometer (MS) and nuclear magnetic resonance (NMR) spectrometer provides details of the chemical structure information for molecular structure determination.

Chemical structure analysis of Rhynchophylline



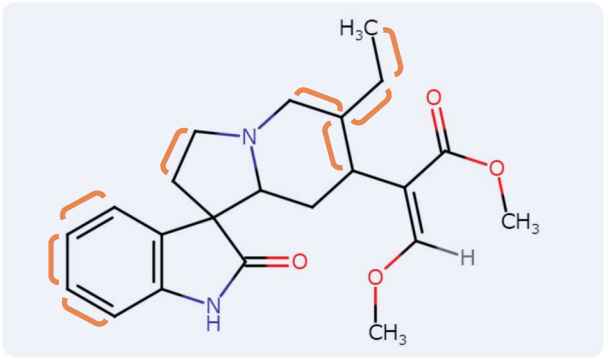
For elucidating the molecular structure of alkaloids, the molecular formula is determined by using MS. In the example, the molecular formula of Rhynchophylline is provided from the Mass spectrum by using JMS-S3000 SpiralTOF™-plus 2.0 (shown above on the left). In addition, NMR analysis provides the details of Rhynchophylline chemical structure (shown below on the left). ^{1}H-^{13}C and ^{1}H-^{15}N connectivity information is available for partial structure analysis of Rhynchophylline. In the example below, the NMR and MS/MS spectra are analyzed complementary to estimate the total chemical structure model of Rhynchophylline.



Chemical structure model of Rhynchophylline obtained through MS and NMR analysis

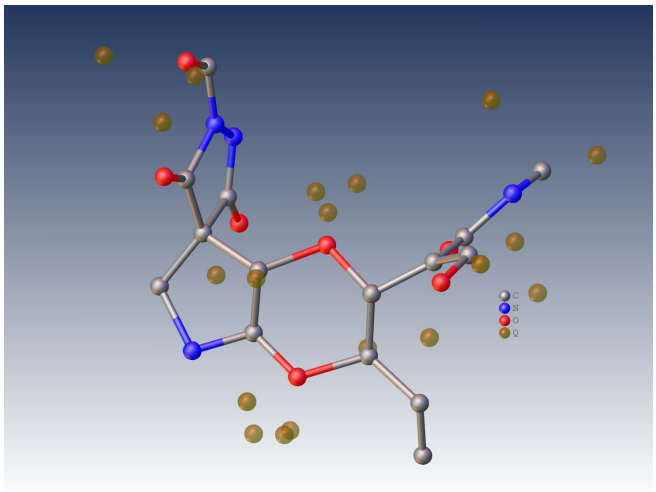
Above : Mass and MS/MS spectra of Rhynchophylline, JMS-S3000 SpiralTOF™-plus 2.0
Below : Edited HSQC and Edited H2BC NMR spectra, JNM-ECZL 500R

Structure refinement of Rhynchophylline

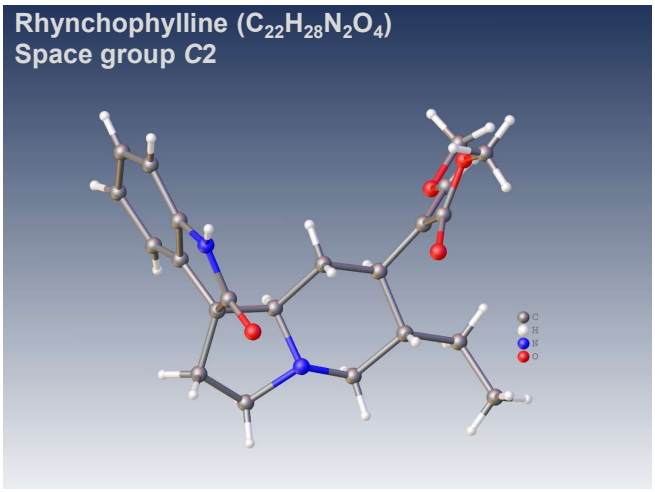


C-H connectivity information of two-bond correlations of Rhynchophylline, Edited H2BC, JNM-ECZL 500R

XtaLAB Synergy-ED allows single crystal analysis of Rhynchophylline with submicron particles. Rhynchophylline contains hydrogen, carbon, nitrogen and oxygen. The molecular structure model is estimated by the electrostatic potential obtained through electron diffraction analysis. An initial structural model is refined with the result of MS and NMR analysis. The chemical structural model of Rhynchophylline obtained through the MS and NMR analysis provides the details of partial structure to elucidate the molecular model correctly. In the example below, the right-hand shows the resulting structure of Rhynchophylline which is refined by the chemical structure model of MS and NMR analysis.



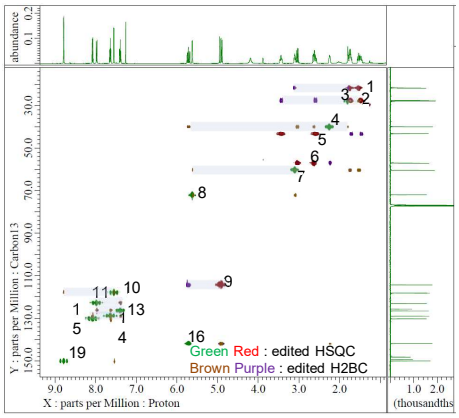
Initial structure model of Rhynchophylline,
Electron diffraction structure analysis, XtaLAB Synergy-ED

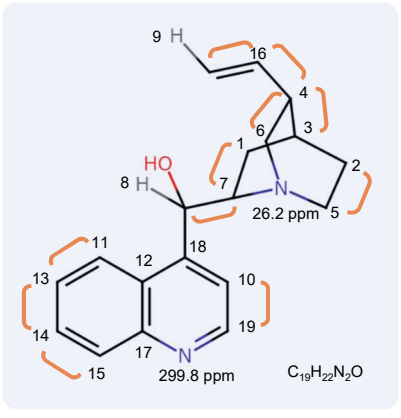


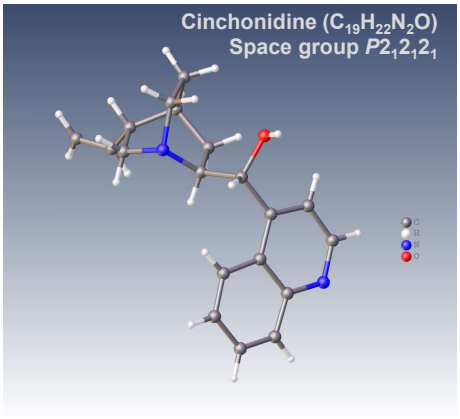
Refined molecular structure of Rhynchophylline

Structure analysis of another alkaloid, Cinchonidine

The structure analysis workflow of Rhynchophylline is available for additional alkaloids. In the following example, the workflow is applied to Cinchonidine. The right-hand is the result of the refined structure of Cinchonidine. This structure refinement is confirmed by the result of MS and NMR analysis. The NMR analysis provides details of the partial structure, ^{1}H-^{13}C and ^{1}H-^{15}N connectivity information, to properly dedicate the molecular model.







Left : Edited HSQC and Edited H2BC NMR spectra of Cinchonidine, JNM-ECZL 500R
Center : C-H connectivity information of two-bond correlations of Cinchonidine, Edited H2BC, JNM-ECZL 500R
Right : Refined molecular structure of Cinchonidine, XtaLAB Synergy-ED

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