Assure 1.5
Raw Material Screening
Release Notes

Current Version: Assure 1.5 PL 2 - IconNMR 4.7.6 Build 25

Software/Hardware Requirements

IconNMR is part of the Bruker TopSpin Software
Please refer to the TopSpin Release letter.

Analysis Assure-RMS release notes are documented following IconNMR.

History of Changes

Only items concerning Assure or of general interest are listed.

IconNMR Changes

Assure 1.5 PL2 (IconNMR 4.7.6 Build 25)

- New: Expert Review Editor Tool available using right click in 'Preceding Experiments'
- New: SST: 13C Sensitivity now with 'Sample Type' configuration for 10%EB, and ASTM EB
- New: Lineshape Test may be switched off for Quantification Samples
- New: RMS and SST configuration separated in IconNMR Configuration
- Fix: Composite Experiments allowed without 'Easy Setup'
- Fix: Not enough storage message at IconNMR shutdown removed

Assure 1.5 PL1 (IconNMR 4.7.4 Build 26)

- Fix: Re-allow setting of Assay type on composite experiments in Experiment Table
- Fix: Lockdown of Data Set Name in easy setup mode fixed
- Fix: IconNMR Accounting calculation problem fixed
- Fix: The Assure-SST report is not created in the log directory because of a permission problem

Assure 1.5 (IconNMR 4.7.4 Build 20)

System Suitability Test improvements

- New: User Defined System Suitability Lineshape/Sensitivity Tests included
- New: SST Reports available in A4 and Letter formats
- Change: SST Temperature Test always runs last
- Change: The System Suitability Tests always run at configurable fixed times
- Change: Updated SST report

Assure 1.4 (IconNMR 4.6.7 Build 25)

- New: Assure now works inside IconNMR's web interface on the iPad
- New: IconNMR Configuration checks Assure Quantification Sample Setup entries to prevent incorrect Experiments from being used.
- New: RMS and SST Parameters are now correctly loaded and saved when using "File->Load" and "File->Save as" in IconNMR Configuration.
- New: The IconNMR Additional Users feature is now available inside Assure

- Fix: Bruker Knowledge Base Item #10855 'IconNMR opens the wrong report for viewing'
- When multiple experiments are selected in the preceding experiments window, the report shown is from one of those experiments but not necessarily the one under the cursor. This issue has now been corrected.

- Fix: Bruker Knowledge Base Item #Item 10770 'Multiple quant samples do not all run'
- The use of multiple quantification samples with only one experiment per sample for testing, as defined under "quantification sample setup" may cause the quantification test to be skipped on the last quant sample. This issue has now been corrected.

- Fix: Bruker Knowledge Base Item 11131 'Assure-SST with SampleTrack repeats experiments'
- Experiments issued from SampleTrack are repeated over and over. Unnecessary experiment entries are created in the experiment list. This issue only This issue has now been corrected.

Assure 1.3 (IconNMR 4.6.5 Build 7)

- No Changes

Assure 1.2 (IconNMR 4.6.3 Build 7)

- Allow Instrument to run even if SST fails (Configuration Fail Safe/Error Handling Option)
- Copy eretic and eretic_concentration files over to RMS Assay from Quant Calibration

Assure 1.2 (IconNMR 4.6.2 Build 27)

- Multiple Assay Samples now handled
- Quantification Sample Test included
- SST now includes F19, 31P Tests
- SST now generates a PDF Report which may be automatically printed

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Previous Versions

Assure 1.1. PL9 (IconNMR 4.6.1 Build 7)

- Latest official Assure 1.1 Version (no changes from PL8)

Assure 1.1. PL8 (IconNMR 4.6.1 Build 7)

- Change: Assure-RMS 1.1 is now delivered together with TopSpin 3.0 PL4
- Change: On Linux, IconNMR attempts to use the KDE QT Theming engine. Use Control Center->Style->Widget Style to select a suitable style for the program's selection elements.
- Change: Solvents D2O and MeOD now use topshim as the default shimming program

Assure 1.1 PL3 (IconNMR 4.6 Build 15)

- Fix: Queued assay experiments now run after a failed TMS/DSS check which interrupted the run with a System Suitability Test.
- Change: Failed DSS/TMS Check reported as fail in processing in 'Preceding Experiments'

Assure 1.1 PL2 (IconNMR 4.6 Build 14)

- Change: System Suitability Test samples run at 298K

Assure 1.1 PL1 (IconNMR 4.6 Build 12)

- Change: Linux permission of System Suitability Test Log files now r—r—r—
- Change: Archiving directory archives according to Configuration->Master Switches->DataSet Management Settings (/data/<user>/nmr is added to the path if so defined)
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- New: Warning when System Suitability Test checks are switched off…"they need to be removed from the queue"
- New: "Delete all experiments in experiment queue" now only possible when the run is stopped.
- New: Assure mode sets default settings for "Temperature Handling"
- New: Deleting all experiments in queue will work for other user's experiments

Assure 1.1 – IconNMR 4.6 Build 1

IconNMR Changes since Assure 1.0

- TMS/DSS/TSP Test via AssureShim AU program added
- Assure Configuration Reorganized
- Fix for no data on SystemSuitability, Lot#, LIMS Container ID, being sent to Analysis software under certain conditions
- Fix for error: "can't read data(systemtest_samples)" on submit during the System Suitability Test(SST) if IconNMR was restarted and the run starts executing a previously queued SST

Assure 1.0 – IconNMR 4.5

Assure 1.0 RC8 – RC11 IconNMR 4.5.2. Build 13

- No changes in IconNMR – Analysis changes follow at the end of this document

Assure 1.0 RC7 - IconNMR 4.5.2. Build 13

- Updates to match changes in standard Bruker TopSpin3.0 PL2/IconNMR 4.5.2 Interface
- Handle errors more gracefully when reloading a setup with samples which are not available for the current sample changer
- React gracefully if the system suitability test can't be queued
- Bruker KB#9702 User ids containing dots are unusable
- Allow for jobs with start at times which are not System Suitability Tests
- Error color scheme improved for 'Preceding Experiments'
- Web-IconNMR iPhone switch corrected
- Preliminary Manual and Release Notes added to Help Menu

RC6 - IconNMR 4.5.1. Build 13

- Disabled stop button may be enabled with the following entry in %HOME%/_iconnmrrc.txt set DISABLE_STOP_AUTOMATION_BUTTON 0
- Disable accelerators <Ctrl-s> etc. when Submit, Cancel… buttons for QC users not visible

Pre RC6- IconNMR 4.5.1. Build 10

- Write .pdf report files directly into expno dir and not in results directory

Analysis Software Notes Follow overleaf

Assure Raw Material Screening Analysis Software

About

Raw Material Screening (RMS) is a new product that automatically detects impurities (known and unknown) in starting materials used in the synthesis of drug and chemical products. It also verifies the presence of the desired starting material.

RMS runs in automation and interactive mode. The automation mode is started from IconNMR. It requires a spectrum to quantify (proton), a quantification method to run die analysis. The result is written into two PDF files: Overview (1 page) and expert report.

The interactive mode is used for method setup. The method contains all calculation parameter and expected compounds. It hold information about the user, host and date of creation, it is secured by a checksum.

Software Requirements

The following software components are required for Windows and Linux operating systems

- PDF viewer, e.g. Adobe acrobat reader
- AMIX3.9.7 for method development

History of Changes

Assure Version 1.5 pl2

- Smoothing of Spectra before Peak Picking
- Expert Review Editor, See addendum.pdf in the Assure-RMS installation directory (subdirectory /docu/assure)

Assure Version 1.5 PL1

- LINUX Version
- PLS Prediction in Quantification Method
- Peak intensity calculation, e.g. required for Heparin Analysis (USP)
- Interactive Method Builder enhanced
- Improvement of Quantification
- Sideband detection

Assure Version 1.4

- Meta Data Handling
- Bucket Table Calculation
- PCA Analysis
- PLS Calibration and Prediction
- New JRES + HSQC Match

- Improvement of Quantification (Fine Tuning of Integrals)
- Interactive Method Builder

Assure Version 1.3

- New quantification strategies
    - Quantify multiplet peaks
    - Quantify line shape
- Fine Tuning option: re-fit peaks/multiplets to get more accurate quantification results
- Spectra preprocessing strategies
    - Underground removal tool for individual regions
    - Reference deconvolution

Assure Version 1.2

- Absolute Quantification based on PULCON principle (ERETIC II)
- Extended to 2H, 13C, 19F and 31P
- Multiple samples can be analysed in one analysis run.
- Customer defined parameters can be defined and used for input, output and user defined calculations
- Customized report templates
- Spectra could be filtered using a moving minimum filter prior to identification / quantification
- Renaming of compound types
- Monitoring of quality parameter
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Assure Version 1.1

- Calculation of peak width updated
- Method could contain a user defined calculation (Python script)
- Report could show decision pass/fail or a number (Concentration of main component or user defined calculation result)
- Handling of empty peak lists updated
- Solvent and NMR reference peaks are not longer removed automatically.
- Support for Adobe Professional
- Handling of illegal settings in method
- Interactive version writes exceptions to logfile

Assure 1.0

Assure 1.0 RC10:

- The following parameters are now accessible in the method:
    - Report / fail peak threshold H/C
    - Noise factor H/C
    - Maximum allowed shift H/C
    - Match threshold H/C