X-RAY FLUORESCENCE

Rapid Identification Analysis of Raw Materials with 21CFR Part 11 Compliance

Lab Report 156

Introduction

Pharmaceutical companies are required by regulatory authorities to test incoming materials. The amount of time it takes to perform this testing can be quite significant, and the testing of the material itself can be very rigorous. This can lead to long release times for products to leave the warehouse to begin initial processes. Faster positive material identification (PMI) is needed to decrease production time and play an integral role in allowing products to be released to the market sooner.

Typically, wet chemistry and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) are used to validate raw materials that have been delivered. For sodium chloride to be released to manufacturing a wet chemistry test is required on both sodium and chloride, this test can take up to one hour for each element. For solutions containing trace elements ICP-MS is the analytical tool typically used. ICP-MS is a difficult, time consuming, and expensive instrument to operate. The sample preparation is laborious and can often times introduce errors due to the large dilutions required, the system cannot be operated by a novice user either. A dedicated lab technician is needed to calibrate daily and interpret results.

Raw Material ID with the S2 PUMA Series 2

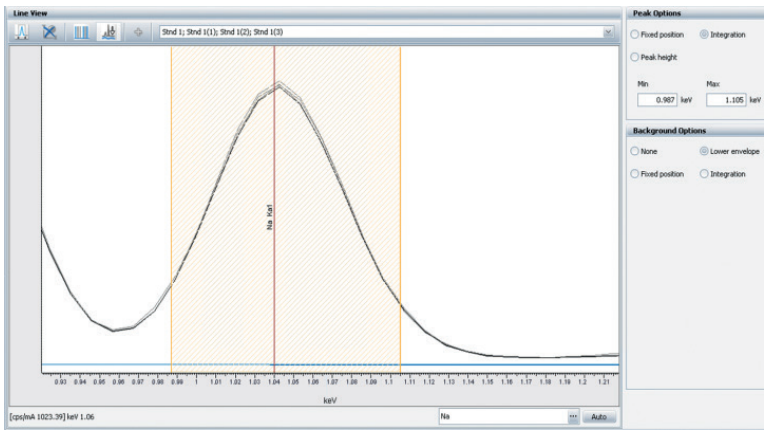
Rapid identification analysis with the Energy Dispersive X-ray Fluorescence (EDXRF) spectrometer S2 PUMA as an alternative has many benefits; easier and faster direct sample preparation, better analysis traceability (computerized audit trails), a non-subjective result, no need for highly trained/educated staff to run the touch button analysis, easy to read sample results, and incredibly fast raw sample to result turn around time.

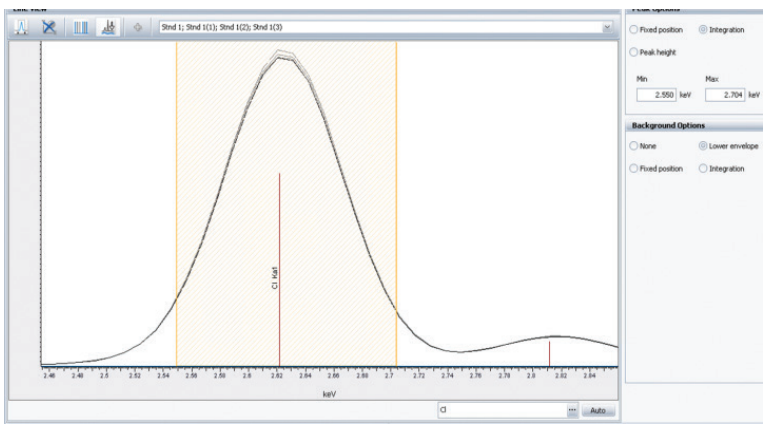
This lab report demonstrates the outstanding analytical performance of the S2 PUMA to positively identify raw materials for pharmaceutical companies. In this specific lab note we will use sodium chloride as one of the many products the EDXRF can quantify.

Instrumentation

The S2 PUMA EDXRF spectrometer is equipped with TouchControl™, SampleCare™, and an easy-to-use touch screen interface. Its unique HighSense™ XP detector easily processes high count rates while ensuring excellent resolution, low dead time and high signal stability, ensuring the best possible performance for this application.

The S2 PUMA spectrometer combines ease-of-use through its unique TouchControl and SampleCare with superior analytical performance. SampleCare ensures reliable operation and high instrument uptime of the S2 PUMA through innovative 4x protection of all spectrometer components against contamination and damage by powders or liquids.





Preparation

EDXRF requires no extensive dilution of the sample; simply add sample to a prepared sample cell and sample is ready to be analyzed. Sample cells can be purchased premade to save even more time, typical powder or liquid sample preparation with a sample cell is approx. 30 seconds.

The sodium chloride raw material was prepared neat in a sample cup. 7 grams of sample was weighed out into a sample cell that was prepared with 3.0 µm Prolene film.

Measurement

The measurement method was developed to optimize the determination of sodium and chloride within the raw material. This method will provide enhanced measurement parameters for tube voltage, current, counting statistical error, and minimize analysis time. Total sample analysis time is only 4 minutes.

Calibration

One validated customer reference sample of sodium chloride was used to calibrate the instrument for sodium and chloride. The standard was prepared in triplicates to account for variation in the sample grain size.

The element lines (Figure 1 and Figure 2) show excellent signal to noise ratios and very clear peak definition with no elemental interference. The signals also depict the excellent resolution of the HighSense detector.

This allows an accurate net intensity value to be used to calculate the concentration of standards and unknowns.

Figure 1
Na KA1 element line

Figure 2
Cl KA1 element line

Results

The instrument is operated directly from the S2 PUMA Touchscreen; no external PC is needed for routine analysis, maintenance checks, or any other functionality. This allows the S2 PUMA to fit virtually on any benchtop surface, the foot print is much smaller than most other analytical techniques, with no waste of strong acids, liquid nitrogen, or argon gas.

A simple Sample Loader interface can then be launched for routine analysis (Figure 3). Merely select open position, pick application method needed, hit start, and then get result. As easy as **1, 2, 3!**

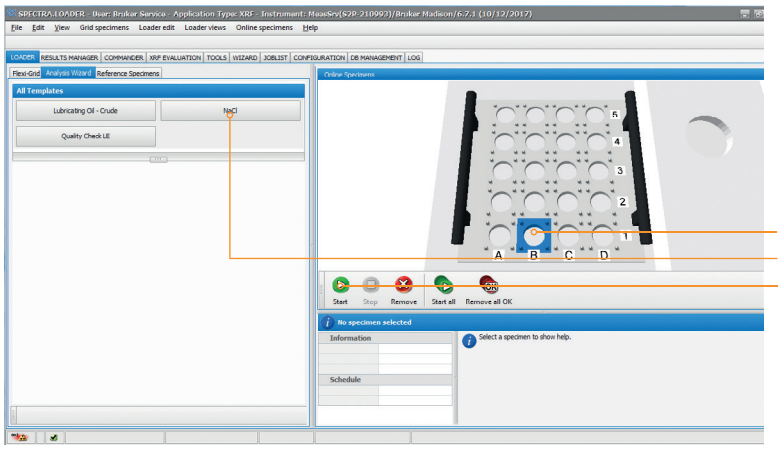


Figure 3
S2 PUMA TouchControl Sample Loader interface

Easy to read result output can be color coded for pass/fail (Figure 4 and Figure 5), written pass/fail indication or a combination of both ensures that an expert or novice user can obtain and report correct results! Clear and concise!

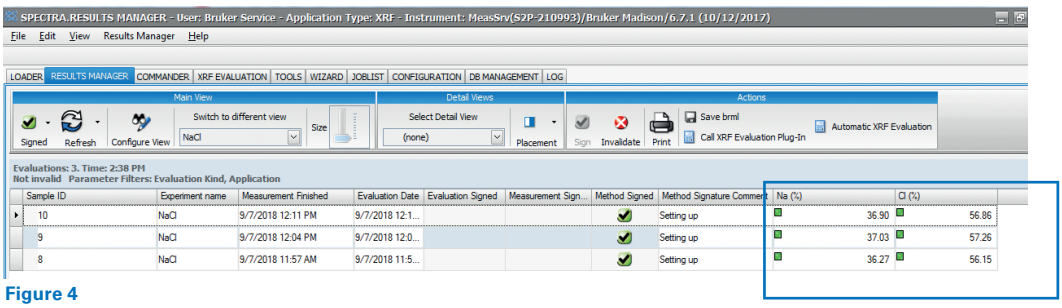


Figure 4
Color coded result output for passed samples

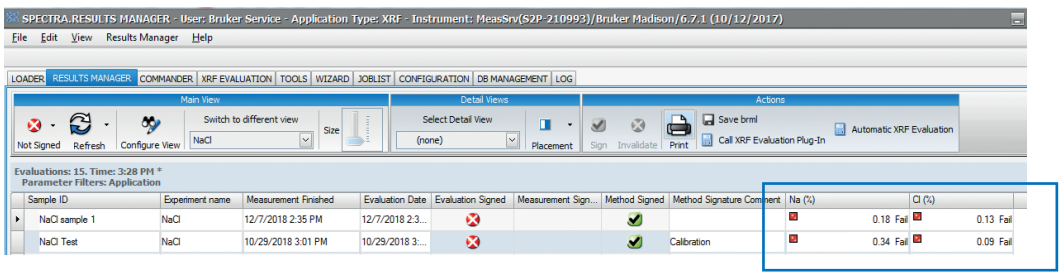


Figure 5
Color coded result output for failed samples

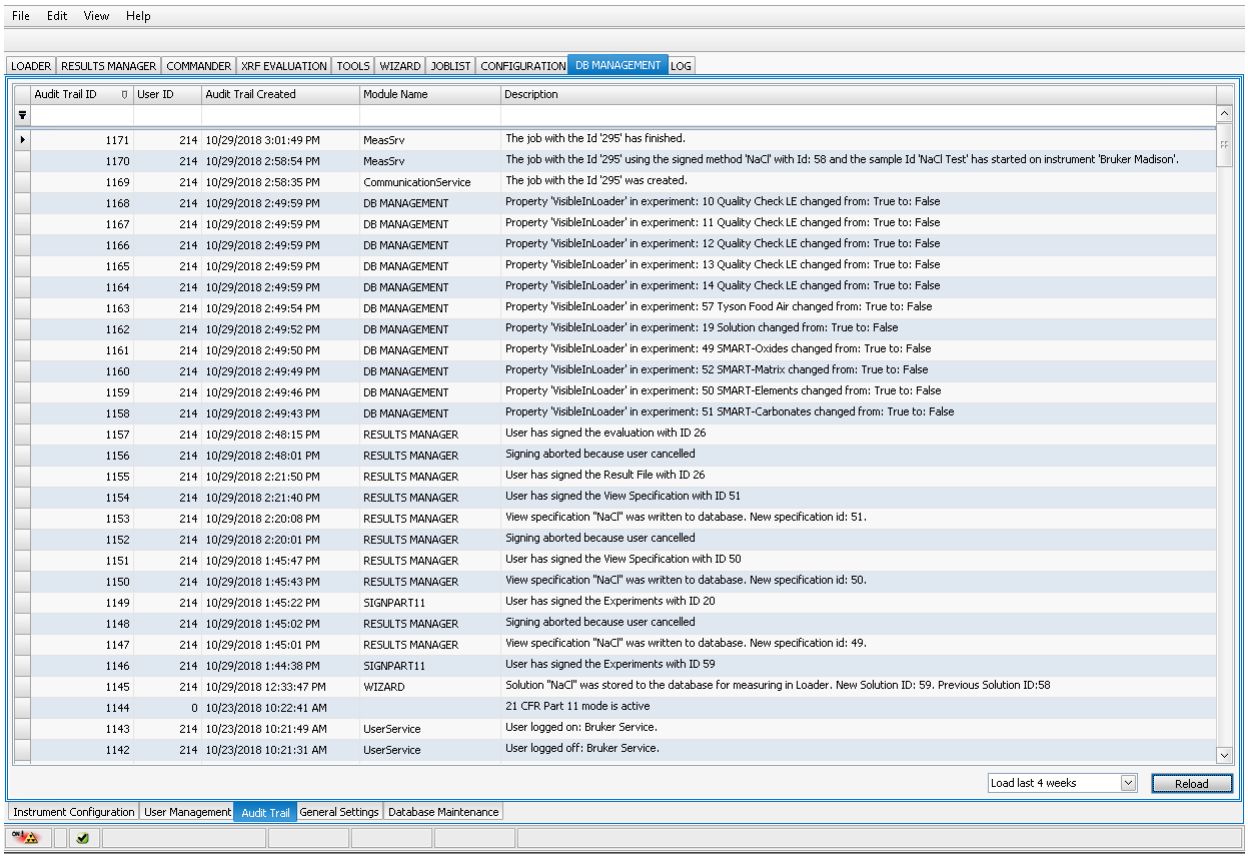
SPECTRA.ELEMENTS Part 11 is specially designed to match the requirements of the 21CFR Part 11 Regulations. This includes a user management feature, separate from Windows user management, which protects files from unauthorized modification or deletion. SPECTRA.ELEMENTS also runs in database mode: all method files, result files, evaluation files, etc. will be stored in a protected database in order to prevent any not tracked modification of these files. After the installation only users with specific rights are allowed to modify applications, views, layouts, view all results, sign evaluations, sign measurements, and sign methods. This prevents errors within the calibration and reporting of results.

The software also offers a comprehensive overview of audit trails (see Figure 6) and user signatures.

Conclusion

The S2 PUMA is a viable, efficient, and simple analytical tool for Positive Material Identification. The user friendly SPECTRA.ELEMENTS software makes calibrating a breeze, while the user-friendly TouchControl Loader makes running an analysis and obtaining results as easy as 1, 2, 3. This instrument is fully compliant with IQ/OQ/PQ as well as 21CFR Part 11, this makes the S2 PUMA a faster, easier, and better option for PMI analysis.

Figure 6
Example of comprehensive Audit Trail



Audit Trail ID	User ID	Audit Trail Created	Module Name	Description
1171	214	10/29/2018 3:01:49 PM	MeasSrv	The job with the Id '295' has finished.
1170	214	10/29/2018 2:58:54 PM	MeasSrv	The job with the Id '295' using the signed method 'NaCl' with Id: 58 and the sample Id 'NaCl Test' has started on instrument 'Bruker Madison'.
1169	214	10/29/2018 2:58:35 PM	CommunicationService	The job with the Id '295' was created.
1168	214	10/29/2018 2:49:59 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 10 Quality Check LE changed from: True to: False
1167	214	10/29/2018 2:49:59 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 11 Quality Check LE changed from: True to: False
1166	214	10/29/2018 2:49:59 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 12 Quality Check LE changed from: True to: False
1165	214	10/29/2018 2:49:59 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 13 Quality Check LE changed from: True to: False
1164	214	10/29/2018 2:49:59 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 14 Quality Check LE changed from: True to: False
1163	214	10/29/2018 2:49:54 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 57 Tyson Food Air changed from: True to: False
1162	214	10/29/2018 2:49:52 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 19 Solution changed from: True to: False
1161	214	10/29/2018 2:49:50 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 49 SMART-Oxides changed from: True to: False
1160	214	10/29/2018 2:49:49 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 52 SMART-Matrix changed from: True to: False
1159	214	10/29/2018 2:49:46 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 50 SMART-Elements changed from: True to: False
1158	214	10/29/2018 2:49:43 PM	DB MANAGEMENT	Property 'VisibleInLoader' in experiment: 51 SMART-Carbonates changed from: True to: False
1157	214	10/29/2018 2:48:15 PM	RESULTS MANAGER	User has signed the evaluation with ID 26
1156	214	10/29/2018 2:48:01 PM	RESULTS MANAGER	Signing aborted because user cancelled
1155	214	10/29/2018 2:21:50 PM	RESULTS MANAGER	User has signed the Result File with ID 26
1154	214	10/29/2018 2:21:40 PM	RESULTS MANAGER	User has signed the View Specification with ID 51
1153	214	10/29/2018 2:20:08 PM	RESULTS MANAGER	View specification "NaCl" was written to database. New specification id: 51.
1152	214	10/29/2018 2:20:01 PM	RESULTS MANAGER	Signing aborted because user cancelled
1151	214	10/29/2018 1:45:47 PM	RESULTS MANAGER	User has signed the View Specification with ID 50
1150	214	10/29/2018 1:45:43 PM	RESULTS MANAGER	View specification "NaCl" was written to database. New specification id: 50.
1149	214	10/29/2018 1:45:22 PM	SIGNPART11	User has signed the Experiments with ID 20
1148	214	10/29/2018 1:45:02 PM	RESULTS MANAGER	Signing aborted because user cancelled
1147	214	10/29/2018 1:45:01 PM	RESULTS MANAGER	View specification "NaCl" was written to database. New specification id: 49.
1146	214	10/29/2018 1:44:38 PM	SIGNPART11	User has signed the Experiments with ID 59
1145	214	10/29/2018 12:33:47 PM	WIZARD	Solution "NaCl" was stored to the database for measuring in Loader. New Solution ID: 59. Previous Solution ID:58
1144	0	10/23/2018 10:22:41 AM		21 CFR Part 11 mode is active
1143	214	10/23/2018 10:21:49 AM	UserService	User logged on: Bruker Service.
1142	214	10/23/2018 10:21:31 AM	UserService	User logged off: Bruker Service.

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