



Lab Report XRF 173

S2 PUMA Series 2

- Material ID and production monitoring made easy and fast

Quick and reliable identification as well as purity verification of raw materials used to formulate pharmaceutical products is critical to meet stringent quality requirements and to produce efficiently. At-line monitoring of the production process to rapidly identify possible impurities is of similar importance. These are two analytical tasks where energy dispersive X-ray fluorescence (EDXRF) excels. Not only is it possible to rapidly identify substances (often in less than 2 minutes), but also the main contaminants can be determined easily.

The S2 PUMA Series 2 is the optimal solution for such applications:

- XY Autochanger with 22 positions combining high throughput with flexibility,
- Direct analysis of all sample types (liquids, powders, solids, tablets) without sample preparation, SampleCare™ technology, protecting critical system components for low maintenance,
- Intuitive software SPECTRA.ELEMENTS fully compliant with FDA 21 CFR Part 11,
- Sturdy design and robust, high quality components for long lifetime,
- Ergonomic TouchControl™ display for operation without PC peripherals (optional).

S2 PUMA Series 2 – A Powerful Benchtop System with High Sensitivity

The S2 PUMA Series 2 energy dispersive X-ray fluorescence (EDXRF) spectrometer was used for this report. It is ideally suited for process control with its XY Autochanger for unattended analysis of up to 22 samples. The combination of a 50 Watt X-ray tube with closely-coupled beam optics and the new HighSense™ detector technology enables optimal sample excitation, resulting in outstanding analytical performance.

The S2 PUMA runs on Bruker's fast and powerful spectrometer software, SPECTRA.ELEMENTS. Integrated features such as dedicated user levels, tab-based organization, customizable views, loss-eliminated alphas, LIMS compatibility and Auto-Matrix ensure optimal performance and easy operation. A 21 CFR Part 11 version of the software ensures full compliance with FDA regulations, including Audit Trail and Electronic Signature. Optional IQ/OQ procedures are offered with the installation of the system.

QA/QC screening and Material Identification

The optional standardless solution SMART-QUANT FP allows for a quick screening of the elemental composition of all sorts of materials - even if fully unknown. The software returns a simple Yes/No (e.g., NaCl vs. KCl, see Figure 1), enabling immediate actions to save time and money. A measurement like this takes a few minutes.

XY Autochanger - Flexibility meets High throughput

The S2 PUMA allows to measure elements from C to Am. Different chemical compounds can be set up, for example simple oxides, but also other compounds like different salts, or more complicated formulas like talc or dimethicone.

When using the XY Autochanger it is possible to set up an hour's work ahead of time and let the system do the work for you. Liquid samples, powders and solid samples can all be mixed, and the system will switch the measurement mode accordingly.

Sample Preparation

The samples were measured as loose powder in plastic cups with a thin foil (Figure 8). This type of preparation requires minimal handling of the samples, and typically the material can either be reused if needed. It is possible to specify and correct for geometric parameters like the weight or the density of a sample. When measuring light elements (C-Si) in powders or liquids, the Helium mode is used to improve the signal-to-noise ratio. Here, the optimized workflow and small chamber of the S2 PUMA results in lowest helium consumption, minimizing the costs per sample.

A great alternative preparation for powders is pressing the material into pellets. That way they can be measured in vacuum, saving He and achieving optimal sensitivity.

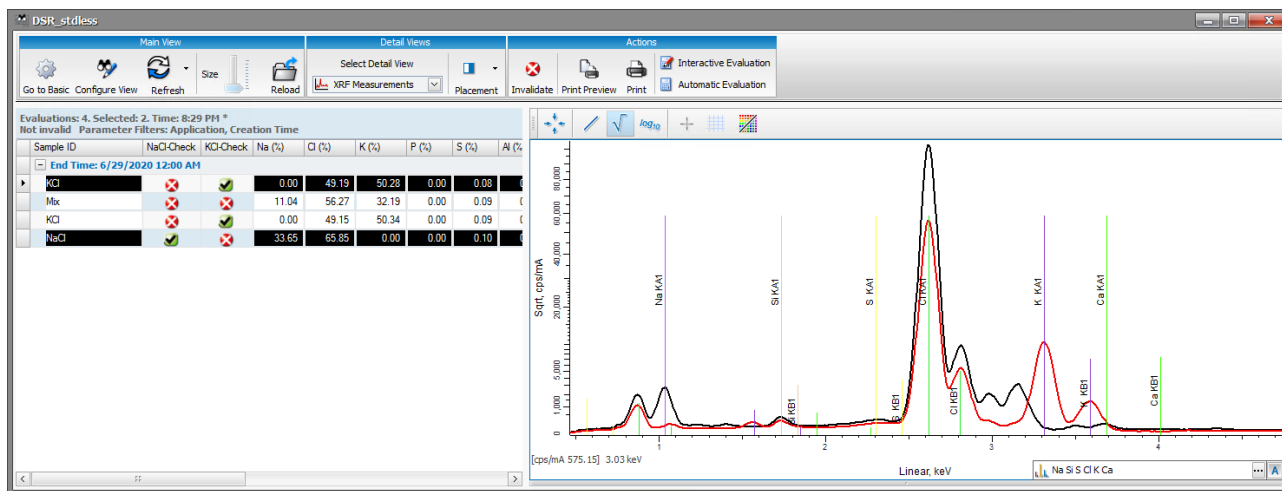


Figure 1: The Results Manager showing different measurements that can be overlaid – here KCl (red) and NaCl (black).

Standardless Application Raw Material ID

SMART-QUANT FP allows to quickly identify salts and common contaminants (Figure 2), including:

- Sr in CaCl_2 and CaCO_3 (antacid)
- Ga in AlPO_4
- As in Na-Borate

Heavier Elements in Creams and Ointments

Two wound healing ointments and one sunscreen using ZnO (and TiO_2) (and TiO_2) as active ingredient have been analyzed to show the capability to measure more viscous samples and determine heavier elements of interest.

The different amounts of ZnO are well resolved (Figure 3). Other ingredients like MgSO_4 , KOH and Dimethicone ($\text{C}_2\text{H}_6\text{OSi}$) in the ZnO ointment, or $\text{C}_2\text{H}_6\text{OSi}$, NaCl, Alumina and of course TiO_2 in the sunscreen can be identified easily.

Even without detailed information about the matrix, SMART-QUANT FP provides a reliable overview of components in the samples (Table 1).

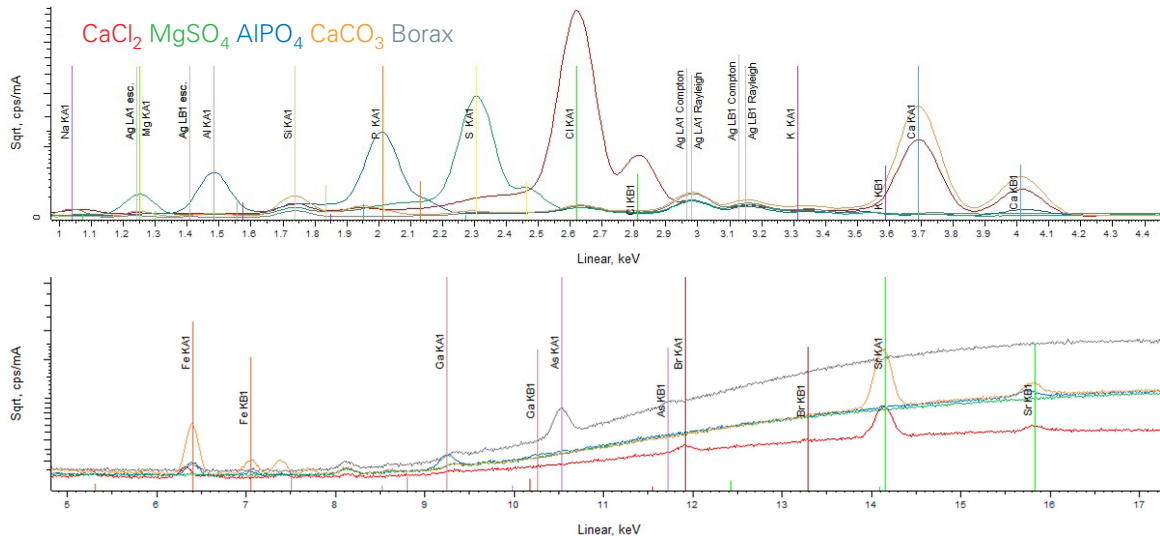


Figure 2: Spectra of various salts used in the pharmaceutical industry (upper plot: major components; lower plot: common contaminants) (upper plot: major components; lower plot: common contaminants).

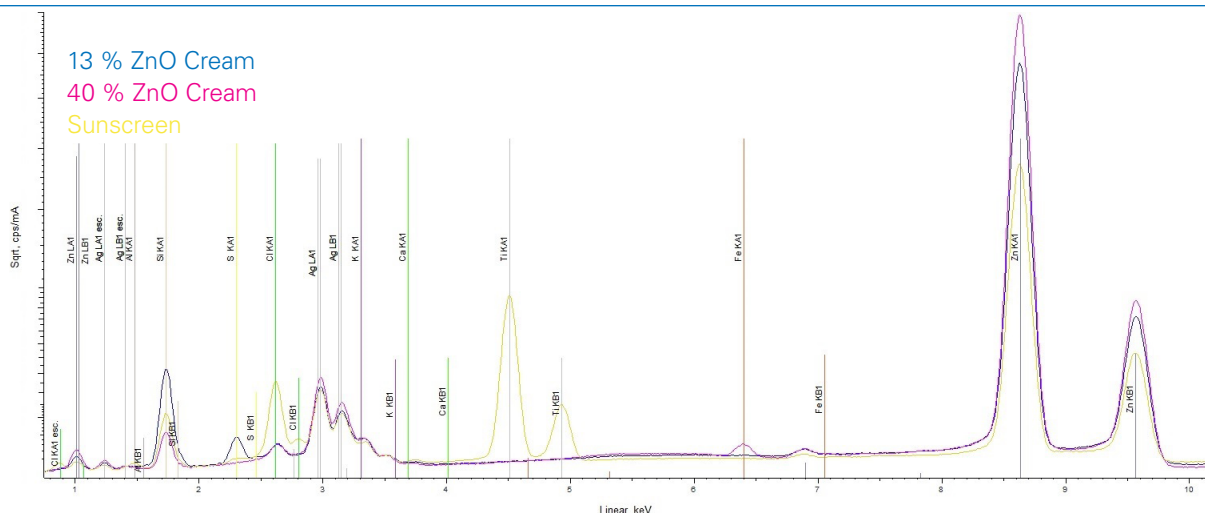


Figure 3: Spectra of ZnO containing ointments (blue and magenta) and sunscreen (yellow).

[%]	Matrix	ZnO	TiO ₂	Al ₂ O ₃	C ₂ H ₆ OSi	MgSO ₄	S	NaCl	Cl
Ointment, low ZnO	76.98	12.06	-	-	10.28	0.57	-	-	0.05
Ointment, high ZnO	60.78	36.16	-	-	2.95	-	-	-	0.07
Sunscreen, 6.5% ZnO, 4.5% TiO₂	83.63	6.36	4.98	0.17	3.84	-	0.02	0.99	-

Table 1: Results of the standardless analysis of ointments and sunscreen containing different levels of ZnO and ZnO+TiO₂ as active ingredients, respectively.

Accurate Quantification with Calibrated Solutions

Bruker's powerful and intuitive spectrometer software, SPECTRA.ELEMENTS allows to quickly build and manage quantitative solutions for all sorts of applications. As an example, a simple calibration for saline solution was setup. The standards were prepared by using a 10 % stock solution. Figure 4 shows the calibration curve. It takes only an hour to set up a calibration like this and routine measurements take < 3 min. The analytical performance can be easily monitored by measuring quality control standards. When a deviation from the expected value is observed, a drift standard delivered with the instrument allows for quick drift correction (no re-calibration necessary!)

The repeatability test highlights the excellent stability of the instrument (Table 2).

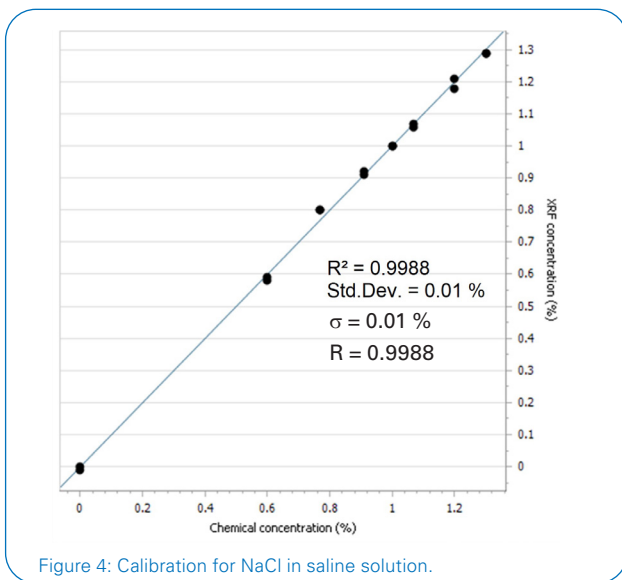


Figure 4: Calibration for NaCl in saline solution.

Preparation	NaCl [%]
# 1	1.00
# 2	0.99
# 3	1.00
# 4	0.99
# 5	0.99
# 6	1.00
# 7	1.00
# 8	0.99
# 9	0.99
# 10	1.00
Average	1.00
Abs. Std. Dev.	<0.01
Rel. Std. Dev.	0.45 %

Table 2: Repetition test.

Conclusion

High sensitivity, excellent repeatability and an intuitive, FDA 21 CFR part 11 compliant software make the S2 PUMA a valuable addition to any pharmaceutical laboratory or production site, be it for initial raw material screening, at-line quality control or in an R&D environment.

The standardless solution SMART-QUANT FP allows for immediate use after installation. For higher accuracy results of specific materials, custom calibrations can be set up quickly. Whether standardless or custom calibration, the S2 PUMA can automatically handle up to 22 samples in various forms and sizes, and switch between four atmosphere modes (vacuum, helium, nitrogen, air).

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Bruker AXS GmbH

info.baxs@bruker.com

www.bruker.com

Worldwide offices

bruker.com/baxs-offices



Online information

bruker.com/s2puma

