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B-XRD1161 - MiniFlex XpC for clinker application

Introduction

The MiniFlex XpC (Xpress connect) is the newest flagship XRD device by Rigaku, designed for the cement industry. Its compact design combines the low infrastructure requirements of a pure benchtop system and the high performance of a floor-standing device. The small goniometer radius of 150 mm combined with the newly developed 800 W X-ray tube and D/teX Ultra250 high-speed 1D detector guarantee perfect results.

In addition, the MiniFlex XpC is specially designed for an automated lab environment, providing a seamless integration. It also offers easy and intuitive hands-on operation empowered by the built-in touch interface, which displays all information concerning running measurements and analytical results.

Powered by Rigaku's SmartLab Studio II software suite, including Rietveld refinement, the entire spectrum of cement materials can be easily measured and analyzed.

Software solutions

Having a high-performing instrument isn't enough. The application software must also be first-class. Having full access and control over the instrument and simultaneously possessing an easy-to-use quantification tool, SmartLab Studio II offers the perfect solution for any requirements. The software is capable of quantifying crystalline and amorphous—down to very low quantities.

In addition, the new EasyX software is designed for fast measurements and easy phase quantifications in only three steps—from measurement to analysis in under three minutes—all easily accessible via the built-in touch interface. The software allows the user to perform Rietveld quantifications based on a template system, plus it delivers detailed statistical information about the calculated quantifications of a series of samples, such as minimum and maximum values and the standard deviation between the measurements.

Application specific

Measurement and phase quantification are performed in a three-step operation in under 3 minutes. Figures 1 and 2 show the Rietveld refinements of clinker and Portland cement (Each standard name: JIS R 5210, CEM I and ASTM Type I). The corresponding quantifications are shown in the tables. The clinker sample shows the typical phases, such as alite (M1 & M3 polymorphs), belite, aluminate, and ferrite. The polymorphs of belite (α, α', β) and aluminate (cubic and orthorhombic) can also be individually quantified if required. The Portland cement quantification yields additional phases, such as quartz or gypsum. Further, critical phases such as lime can also be individually monitored. The quantification of Portland cement is also applicable to blended cement.

Tables 1 and 2 are taken directly from the EasyX software and give the typical repeatability values for both samples, including detailed information about the statistics of a series of measurements for each individual sample. Table 1 shows the quantification result of NIST2688 clinker standard reference material. NIST clinker standard materials are used to compare to the certified values, and this validates the method for process control. Quantification values of each phase are shown to be in good agreement with the certified values. The 1 σ values of each crystalline phase are well below 1 mass%, underlining the precise measurement and evaluation procedure, thus guaranteeing reliable monitoring during the cement production process.

Conclusion

The MiniFlex XpC is the perfect tool for detailed and reliable cement materials analysis in a fast-paced and high-precision environment as demonstrated by the data presented here and details about the instrument's capabilities and advantages. Its low infrastructural demand and the ability for semi- or fully automated setups make the MiniFlex XpC the preferred process control XRD instrument.

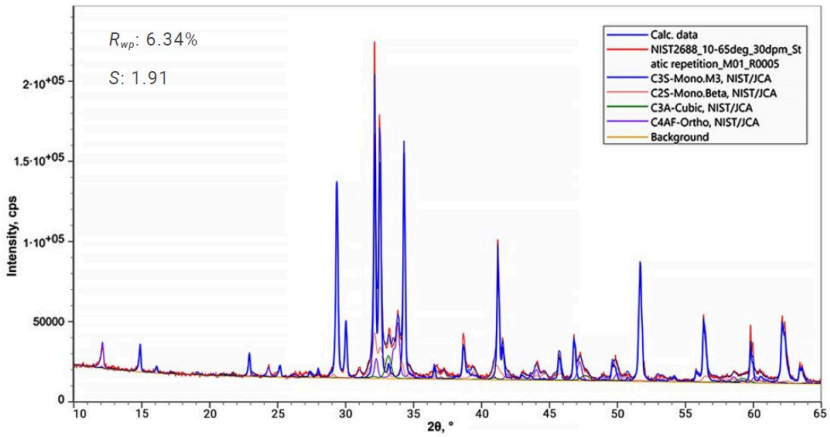

Figure 1: Rietveld refinement result of NIST2688 clinker standard reference material. [R_{wp}: Weighted profile parameter S: Goodness of fit]

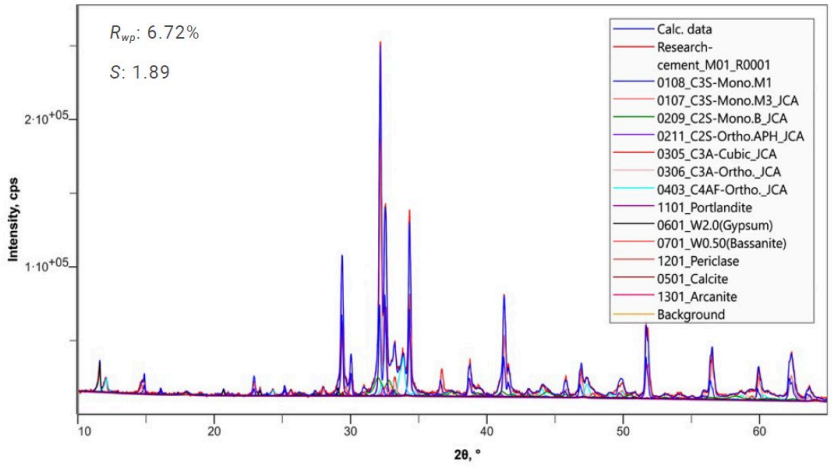

Figure 2: Rietveld refinement result of cement sample.

Table 1: Quantification result and static repetition values for NIST2688 clinker standard reference material (mass%).

Components	**Concentration (n=10)**	**Standard deviation 1 σ , n=10 (3 σ)**	**Certified value**
Alite-M3 (C_3S)	65.4	0.2 (0.6)	64.95 ± 1.04
Belite-β (C_2S)	17.7	0.2 (0.6)	17.45 ± 0.96
Aluminate (C_3A)	4.9	0.3 (0.9)	4.99 ± 0.50
Ferrite (C_4AF)	12.0	0.3 (0.7)	12.20 ± 0.84

Table 2: Quantification result and static repetition values for cement sample (mass%).

Components	Concentration (n=10)	Standard deviation 1 σ , n=10 (3 σ)
Alite-M3 (C_3S)	27.7	0.5 (1.5)
Alite-M1 (C_3S)	39.0	0.5 (1.5)
Total Alite	66.7	0.2 (0.6)
Belite-β (C_2S)	12.7	0.2 (0.6)
Belite-α'H (C_2S)	0.92	0.06 (0.18)
Total Belite	13.6	0.2 (0.6)
Aluminate-Cubic (C_3A)	2.7	0.1 (0.3)
Aluminate-Ortho (C_3A)	2.7	0.1 (0.3)
Total Aluminate	5.4	0.2 (0.6)
Ferrite (C_4AF)	9.1	0.1 (0.3)
Portlandite ($Ca(OH)_2$)	0.3	0.03 (0.06)
Gypsum ($CaSO_4(H_2O)_2$)	1.31	0.06 (0.18)
Bassanite ($CaSO_4(H_2O)_{0.5}$)	2.55	0.07 (0.21)
Periclase (MgO)	0.18	0.04 (0.12)
Calcite ($CaCO_3$)	0.29	0.06 (0.18)
Arcanite (K_2SO_4)	0.52	0.04 (0.12)