Thermo Scientific ARL 9900 X-Ray Pot Flux Analyzer

The "ARL 9900 Pot Flux Analyzer CaLc" is the latest addition to the ARL 9900 instrument series specifically designed for the needs of the aluminum industry in terms of phase and elemental analysis required for pot flux control. This instrument includes a Full X-ray Diffraction system permitting XRD quantification as well as a specific XRF detection for total calcium.

Innovative solutions for the aluminum industry





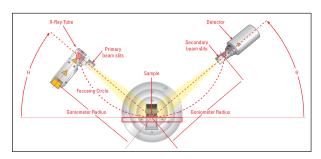


Figure 1: Theta-theta geometry of the ARL 9900 Pot Flux Analyzer CaLc. In addition total calcium detection is fitted close to the sample.



Unique features and advantages

- Includes full XRD technology for unlimited phase determination and fixed XRF Ca detection
- Theta-theta geometry with Co anode X-ray tube rotating around sample (Fig. 1)
- Highest sample throughput thanks to parallel determination of Ca and phases
- 2500W power and Co anode X-ray tube without any peripheral dependence such as water and gas for lower cost per analysis
- Sample changers with 12 positions or up to 98 positions capacity
- Designed for the most demanding applications in process control environment
- Simple automation link to automatic press for full unattended operation.

Bath ratio

Proven instrument derived from the unique ARL 9900 X-ray WorkStation , the ARL 9900 Pot Flux Analyzer CaLc can determine the bath ratio for pot flux control. It is obtained from fluorite (CaF $_2$), chiolite and cryolite phases and the total Ca determination. Other phases like corundum (α -Al $_2$ O $_3$), LiF, MgF $_2$, NaF can also be determined if needed. See typical repeatability in Table 1 overleaf.

Total counting time

- 60 seconds with Ca by XRF analyzed at the same time than the XRD phases
- This counting time can be decreased as fluorite and cryolite phases are not required for bath ratio determination
- Loading and pumping time: 22s unloading time: 15s
- Total turnaround time per sample: 57s excluding fluorite and cryolite = 63 samples per hour
- Total turnaround time per sample: 97s including fluorite and cryolite.



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Channel Sample BA-02	Ca_total XRF %	Excess AIF ₃ XRD %	Corundum α -Al ₂ O ₃ XRD %	Cryolite XRD %	Fluorite CaF ₂ XRD %	Bath Ratio Formula
Run 2	5.77	6.51	0.53	47.24	-0.02	1.264
Run 3	5.74	6.49	0.59	46.10	0.06	1.265
Run 4	5.77	6.64	0.55	46.61	-0.02	1.260
Run 5	5.77	6.70	0.53	46.86	0.02	1.258
Run 6	5.76	6.51	0.54	46.41	0.03	1.264
Run 7	5.77	6.58	0.47	46.39	-0.05	1.262
Run 8	5.77	6.58	0.49	47.12	0.06	1.262
Run 9	5.76	6.49	0.46	46.81	-0.05	1.265
Run 10	5.77	6.64	0.43	46.41	0.06	1.260
Run 11	5.77	6.61	0.57	46.68	0.03	1.261
Average	5.76	6.57	0.52	46.68	0.01	1.262
SD	0.013	0.070	0.051	0.343	0.041	0.0023
Counting Time (s) 40kV-40mA	Simultan.	10	30	10	10	-

Table 1: Typical repeatability test on bath samples with determination of bath ratio using only 1 minute counting time

Lc determination

Additionally the "Pot Flux Analyzer Calc" permits the determination of the crystallite size of calcined petroleum coke (Lc). This factor provides an indication of the electrical conductivity and is related to the quality of the heat treatment. Lc is determined by the resolution at half maximum (FWHM) on the graphite (002) peak.

- Lc = $0.89 \lambda / \beta \cos \Theta$
 - $-\lambda$ = wavelength of the excitation radiation in Å
 - $-\beta$ = full width at half maximum of carbon (002) peak (at d=3.35 Å)
 - Θ = angular position of peak in degree

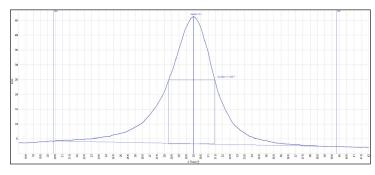


Figure 2: Typical scan of carbon (002) peak acquired in 2 minutes with automatic determination of the resolution at half maximum.

Conclusion

We can provide the aluminum industry with innovative solutions for pot flux control that can fulfill the specific needs of any aluminum electrolysis plant. Typical sample throughput is more than 60 samples per hour for bath ratio determination in pot flux samples.

To see our complete X-ray product portfolio, visit www.thermoscientific.com/xray.

www.thermoscientific.com

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Specifications for the ARL 9900 X-Ray Pot Flux Analyzer CaLc

Complete version includes:

- Co target X-Ray tube
- 2500 W Power supply for 60 KV max. or 60 mA max
- · Closed circuit deionised water cooling system (water-air)
- Vacuum system with rotary pump
- Automatic sample changer for 12 samples
- Sample rotation system, 60 rev./ minute

Detection system includes:

- Sealed detector for element Ca by XRF
- Full X-ray Diffraction System incorporated into the ARL 9900 X-Ray spectrometer for qualitative and quantitative analysis of any phases/compounds.
- Bragg-Brentano goniometer, radius 204 mm
- Long Fine Focus Co anode tube, 1800W power

Opening diameter of cassettes recommended is 33 mm. Samples must be prepared as pressed powders.

Capability for analysis of:

- Cryolite bath samples from aluminum reduction cells
- Cristal lattice (Lc) on baked Anode/Coke according to ASTM D5187
- Alpha Alumina content in Bath
- · Free Alumina in Bath
- Unknown sample scanning and identification of above compounds. Bath ratio calculation through calibration with customer samples

Upgrade to more extended XRF analysis is not possible.



PS41268 F 10/10C

