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В сборнике опубликованы материалы XIV международной конференции «Алюминий Сибири — 2008», которые представлены в пяти разделах: «Получение алюминия», «Литье и вторичная металлургия алюминия», «Производство глинозема», «Экология, охрана труда, переработка отходов», «Новости науки и техники». В каждом разделе предложена информация о новейших технических решениях и последних научных достижениях в мировой алюминиевой промышленности.

Сборник предназначен для специалистов алюминиевой промышленности.

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The volume is intended for the experts of aluminium industry.

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The reports maintain the
author's style

LABORATORY SYSTEM FOR ELECTROLYTE COMPOSITION X-RAY ANALYSIS

I.S. Yakimov, P.S. Dubinin, A.N. Zaloga, O.E. Piksina, S.D. Kirik, S.G. Ruzhnikov, A.F. Shimanskiy, M.A. Ozerskiy

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At the present, it is exists the trend to using of reduction cells with raised current, power and density in Russian aluminum smelters. In complex of engineering-technical and technological decisions, it also requires the decisions of the problems to optimization of technological parameters of electrolysis, including optimization and stabilizations of electrolyte chemical composition in the reduction cells. The last becomes more actual, because electrolysis processes run more intensive in baths, previously named, and electrolyte composition changes faster. It follows, that mistakes of the electrolyte composition analytical control bring about greater destabilization of the reduction process, than earlier; and increasing to accuracy and validity of technological control of the electrolyte composition, and also, as probably, its frequency, becomes an actual problem. Together with that, accuracy and validity of the electrolyte composition control, executed by x-ray and chemical analysis, do not increas and remains rather low; our studies in 2002—2006 show, that it's in two or three times lower, than in leading west plants! 11 It brings about hidden loss to efficiency of new generation reduction cells using, and requires a well-timed decision. Concentration of aluminum production within the framework of RUSAL creates the condition for modernization and standardization of the technological control of the electrolyte composition, and it's not required of essential expenses. Opposite of this, our estimation shows the high profitability of these works, which is not difficult to calculate using well studied dependency of the current efficiency from the bath ratio, and the value of BR variation reduction, obtained for account of the reduction of the the BR technological control average error[2).

However, while such standardization is not executed, in aluminum, smelters a need for between laboratories revise of electrolyte x-ray analysis result seasonally appears. Such need can be caused by integer row of reasons: discord in estimation, of accuracy of the BR analysis by technologists and analysts, for example, dew ftorides consumption increasing to electrolyte composition correction; or appeared from expected and real analytical date about BR , re-adjusing of the x-ray measuring equipment in Central Laboratories, deterioration of results of reproductivity and accuracy operative control, and etc. Herewith, it may be reasons of the deterioration to control accuracy, which isn't taken into account, in acting techniques of measurement execution, like an increase of the chemical impurity concentration in electrolyte for account of alumina or circulating electrolyte, what is negatively affecting on measurement techniques, or on crystallizations process of the electrolyte sample; some technological changes, like BR reduction or calcium concentration increasing over measuring limits of the technique of measurement execution; breach of the sampling and automatic sample preparation techniques; measuring equipment drift, and others. Unexplored and important question is an influence of current raised of new reduction cells on to accuracy and optimum frequency of the analysis. In number of the years it has been executed the complex of the studies, which allows to reveal the influence of many factors, previously named, upon to analysis accuracy, and several new methods of the x-ray analysis of the electrolyte with different composition was developed , |.3—6|.

This work communicates about development of the laboratory system for electrolyte composition x-ray analysis in the Siberian Federal University's Centre of Equipment. Collective Using «Scientifi-cally based methods of study and analysis of new materials, nano materials and mineral cheese «. Laboratory system includes three independent ways of analysis.

The First way is the complete and it uses two analytical methods, like the ways, are applied on aluminum smelters: XRF for element analysis and XRD for phase analysis. This way is based on calibration of the XRF spectrometer and diffractornetr (Shimadzu, Japan) by branch standard samples (the WASP) of calcium — magnesium containing electrolyte compozition, designed and qualified by authors earlier [7]. Notice that these WASP are already used for calibration of the x-ray equipment in 5 aluminum plants. Analysis of industrial electrolyte samples is concluded in determination of the full element composition, including impurity elements, by XRF spectrometer, and in following determination of full phase composition by diffractornetr and, finally. BR calculation, using date of both methods. Accuracy of the analysis is provided by using qualified WASP and by application of techniques of the phase composition determination, wich is more exact than techniques is used in smelters. Accuracy of the analysis, made by this way, is higher, than for industrial analysis. Validity of the analysis is checked by correspondence of phase and element balances, and it's excludes the blunders of the analysis regardless of influences factors, previously named.

The second way is also complete and it uses, like the first way, calibration of the XRF spectrometer by WASP. But, diffraction phase analysis in this way is standardless, and it's based on so named

«method of the reference intensities*-, modernized by authors for given problems. This way allows to analyze any varieties of, in particular, calcium-, magnesium-, potassium- and lithium containing electrolytes. Validity of the analysis is checked similarly of previous way, At presence of the exact element analysis (for calcium -magnesium electrolyte it's provided by calibration by WASP), accuracy of BR is close to accuracy of analysis, made by the first way. For example, accuracy of the test BR analysis of calcium-, potassium- and lithium- containing synthetic electrolyte (which have a next composition: BR 2.66, CaF_2 4.0%, MgF_2 1.2% , KF 4.7%, LiF 2.1 % wgh.), has formed 2.64 units of BR [6]. This way requires the measurement of the full x-ray diffraction profile by diffractometr in current of 40—60 minutes, i.e. it's not express.

The third way is purely diffraction and completely standardless, and it is based on the new differential method of the full-profile DDM analysis [8], which is the modernization of the. well-known Rietvelid's method . The essence of the method is modeling and phased interactive refinement of experimental x-ray diffraction spectra of sample by theoretical spectra, calculated on the base of the phases-component atomic crystalline structure. After refinement completion, calculation of the quantitative phase composition is produced . BR, either as in previous way, is calculated from the phase composition. This way also requires the measurement of the sample's full x-ray diffraction profile and hard calculations, but it allows to define BR with high accuracy for any varieties of electrolyte. Figure 1 demonstrates the graph of the correspondence from accounting BR, determined by the third way for WASP, to qualified BR values.

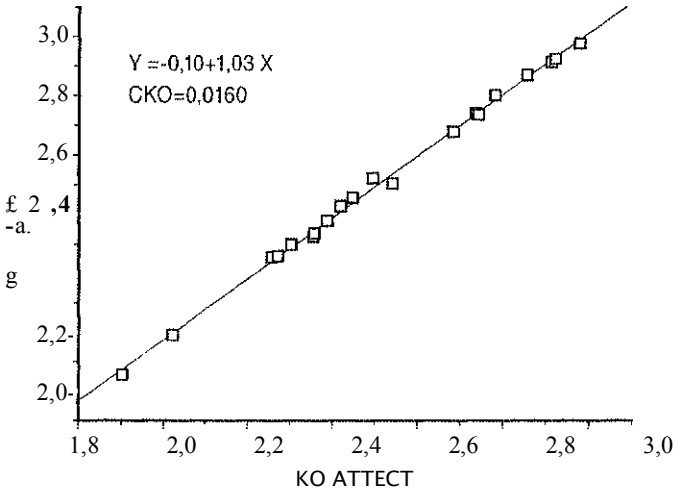

Fig. 1. Correspondence to accounting and qualified BR values for electrolit WASP

Accounting BR values corresponds to qualified BR values with accuracy, characterized by standard defenition of 0.016 BR un., and it's reflecting a total accuracy of the measuring method and qualifications. So far as diffraction measurements data and qualifications data are independent (BR was qualified from between-laboratorys chemical analysis data), and the standard defenition of qualification data are known and forms about 0.008 BR un., it's easy to define the standard definition of diffraction measurements. Diffraction measurements, made by the third way, are characterized by standard defenition about 0.013 BR un., and it practically corresponds to accuracy of the chemical analysis.

Thereby, it was designed system of the 3 independent ways of diffraction analysis for electrolyte composition of any types, characterized by high accuracy and validity of the BR determination. System is intended for arbitration check of electrolyte x-ray analysis results, obtained in Central Laboratories of aluminum smelters; for exploratory integer, and can be effectively used for standardizations of the technological analytical control the electrolyte composition within the framework of corporation or branches.

1,S. Yakimov, P.S. Dubinin, A.N. Zaloc;o, O.E Piks<no, S.D. Kl^ik o' al. Lnbofaiorv S/s.ai; For !:k-.;!r--.-.lyh:- Composition X-Roy Anoiysl:-

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