Development of an Analytical Quality Control System of High-Purity Chemical Substances on the CALS Concept Basis

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ABSTRACT: An information system has been designed for monitoring the quality of chemical reagents and high-purity substances. The information system is based on the international CALS standard (ISO-10303 STEP). It contains the following information for the main elements of analytical monitoring: a list of elements to be monitored (substance classifier), details of the analytical procedure (including the sampling and sample preparation steps), performance parameters of the instruments, metrological support, and normative documentation (GOST standards, technical specifications, and others). J Appl Polym Sci 110: 4016–4021, 2008

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Advances in science and technology are highly dependent on the ability to gain relevant information for research and design work. Lack of information is a particularly serious problem in the priority areas of science and technology, including the technology of chemical reagents and high-purity substances.[1]

The progress of basic research and modern technology is imposing ever more stringent requirements on the purity of substances, which is characterized by an ever increasing number of parameters. Concurrently, the requirements on the analytical techniques for characterizing chemical reagents and high-purity substances are becoming more and more exacting. These issues cannot be addressed without using computer based technologies.[2]

Issues pertaining to the use of computer methods at different stages of the preparation of chemical reagents and high-purity substances received a great deal of attention as early as the 1980s–90s, which led to the development of databases and expert systems for various, but highly specialized projects.[3] In analytic laboratories, computers were then used, for the most part, for primary data processing.[4] At present, however, effective management decision making depends crucially on developing integrated information systems integrating all steps of quality control on the basis of novel information technologies.[5]

At present, Continuous Acquisition and Lifecycle Support (CALS) is the most attractive computer-aided support system. CALS is defined as a strategic management concept that uses the best available information technology, management methods, and international standards (CALS standard ISO-10303 STEP) to increase the effectiveness of organizations. It allows enterprises to be integrated on a worldwide basis, thereby facilitating electronic commerce within and between organizations.[6] Foreign specialists forecast that soon it will be impossible to sell high-technology products without a proper CALS-standardized electronic documentation.[7]

Russian high-technology products that do not meet the CALS standard will lag both economically and qualitatively behind their analogs produced at the West and supported by novel information technologies. Moreover, Russia is a full member of the International Standardization Organization (ISO), and Russian standards and regulations must meet international agreements. Issues pertaining to the quality control of high-purity substances and chemical reagents are particularly critical in the context of the approaching Russia's membership in the International Trade Organization (ITO), one of the conditions being the unification of quality control procedures and the

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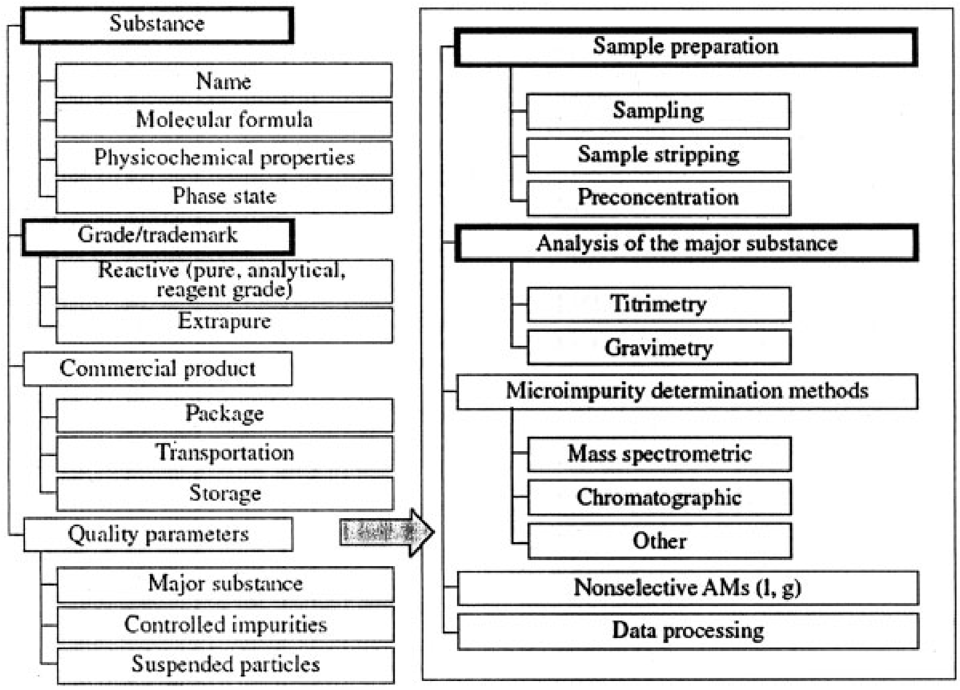

Figure 1 Structure of databases for analytical monitoring of the quality of chemical reagents and high-purity substances.

introduction of statistical data processing standards (GOST R ISO 5725–2002).

The first step in designing the CALS system for monitoring the quality of chemical reagents and high-purity substances was systematic analysis of data[8,9] and expert opinions presented by specialists in analytical procedures and product certification.[10] Those theoretical studies culminated in a structure of databases (Fig. 1).

In the information system being designed, the application area of a high-purity substance or chemical reagent (specified by the customer/user) is represented by the following characteristics:

- controlled impurities,
- maximum acceptable concentrations of controlled impurities, and
- physicochemical characteristics of the major product.

All these parameters are represented by the corresponding files of the database (Fig. 1).

The Commercial Product database contains information on the following product characteristics: package, transportation, and storage (Fig. 1).

One advantage of the proposed database structure is the possibility of rapidly analyzing the effect of microimpurities on the physicochemical properties of the substance and gaining information about their effect on the production process. For example, the important characteristics of a metallic substance are its electrical and thermal conductivity; heat capacity; optical, magnetic, galvanomagnetic, superconducting, and other properties; and defect density.[11] In view of this, only modern computer methods in combination with numerical simulation techniques are capable of predicting the influence of the nature and concentration of impurities on the properties of high-purity substances or chemical reagents. Each entry contains a variety of typical experimental values essential for optimizing the analytical procedure. Thus, the information structure under consideration (Fig. 1) can be used to select the necessary and optimal procedures for qualitative characterization of the product.

In the pilot CALS project developed using systems analysis approaches (Fig. 2), source information is classified into the following groups:

- analyte;
- impurities;
- analytical procedure;
- normative and report documentation.

Each group hierarchically includes several subgroups. For example, the first group, *Analyte* (*Substance*),consists of two subgroups, *Organics* and *Inorganics*, each containing lower-level subgroups: *Acids*, *Salts*, *Oxides*, *Bases*, and other classes of chemical compounds and simple substances (Fig. 2). This classification of high-purity substances conforms to the RF National Standard Classifier, which is a part of the RF Unified System of Classification and Encoding of Technical,

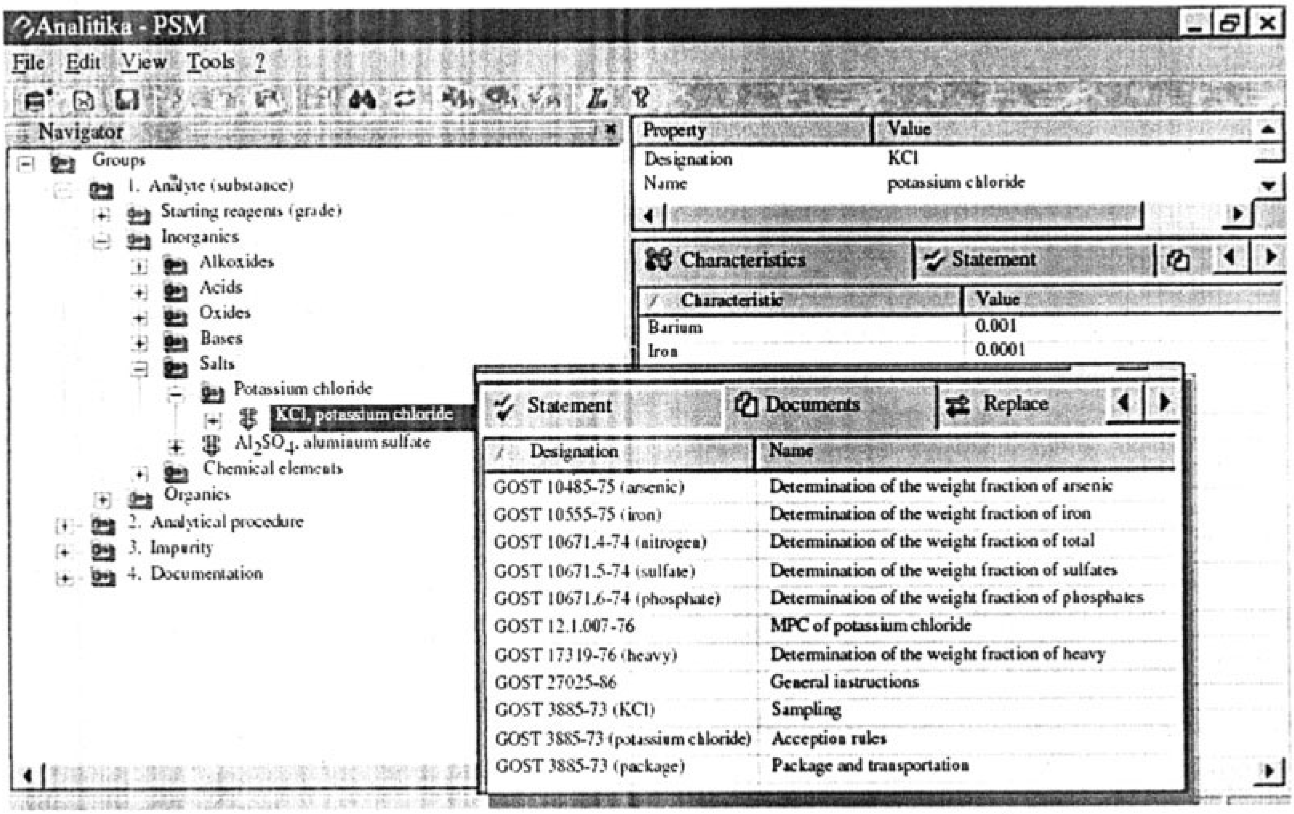

Figure 2 CALS group (inorganic-salts–potassium chloride–GOST-microimpurity content).

Economical, and Social Information. It is also consistent with the International Standard Classifier and the Interstate Standard Classifier.[12]

One of the most important groups in the CALS project is *Normative and Report Documentation* (Fig. 3). Laboratory Information Management System (LIMS),[13] one of the most attractive laboratory information systems today, deals with the following types of input data: external input data – needs and expectations of a customer or market, specifications, contract and normative requirements, national and international standards, branch regulations and rules; internal input data – standards and specifications, qualification requirements, documentation for existing products and services, and other processes' output.

Unfortunately, modern laboratory information systems are unsuitable for the chemical industry, especially for the technology of chemical reagents and high-purity substances. For this reason, we chose another classification of standard documents:

- input: technical specifications, state and branch standards.
- output: laboratory records, test reports, certificates, etc.
- The system has hierarchical structure of databases (substances, normative and technical documentation); the interface is user-friendly. Each functional unit has its special procedures and visual forms, including a set of modern information representation elements and interaction options.
- The information about modern analytical techniques and sample preparation procedures for chemical reagents and high-purity substances is of great importance in creating competitive technologies of high-purity materials. In view of this, the system contains information about various techniques for analyzing both the main substance and impurities. The requirements on the impurity concentration are classed according to the following levels:

- qualitative (determination of impurities that must be controlled, characterization of raw materials);
- quantitative (controllable concentration for each impurity).

For determining the content of the major substance (Fig. 4), the CALS project includes, first of all, absolute (direct) analytical techniques, i.e., procedures that make it possible to evaluate the concentration, with allowance made for the stoichiometric coefficients, directly from the measured parameter. Such approaches (e.g., gravimetric) offer high accuracy but require a long time and are, there-

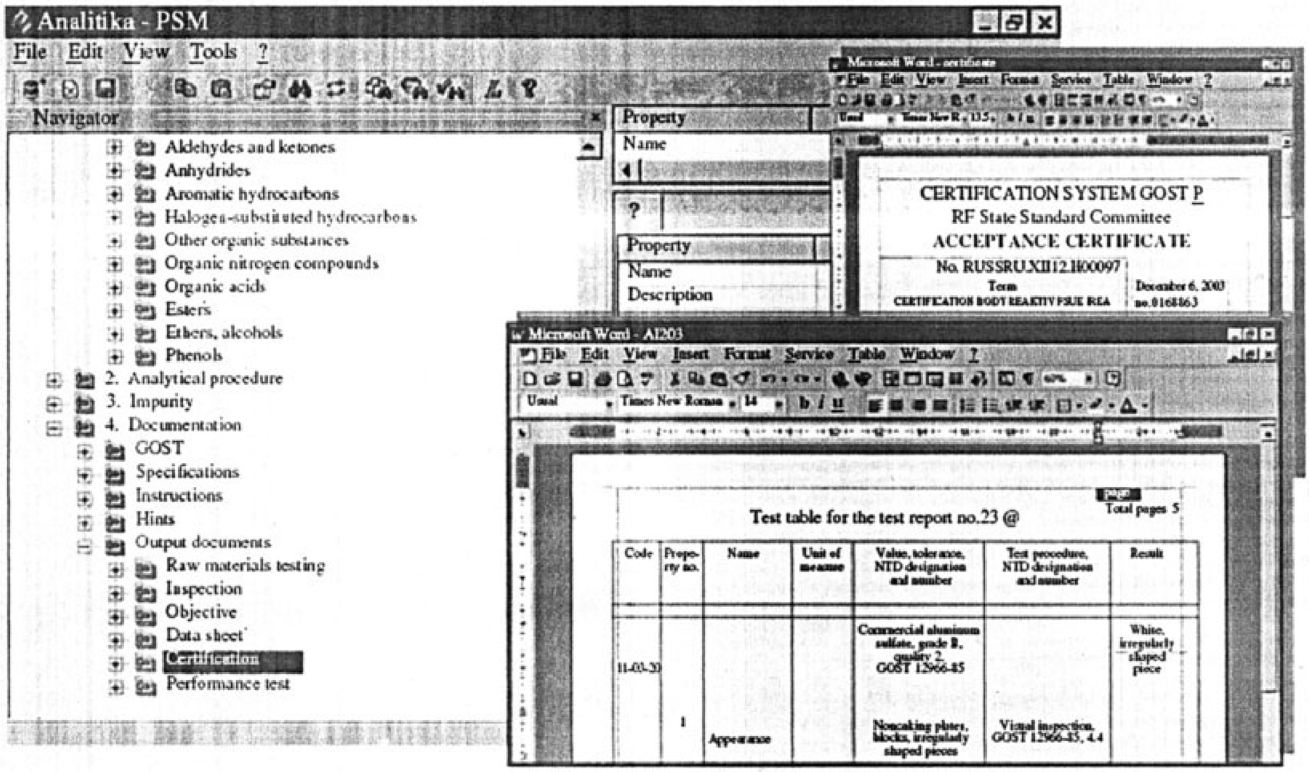

Figure 3 CALS group (test report and certificate).

fore, unsuitable for routine analyses. This group also includes a number of titrimetric methods, often used in chemical analysis by virtue of their simplicity.[8]

In addition, the CALS project includes relative (indirect) analytical techniques in which results are obtained by comparing the analyte and a reference. The uncertainty in such techniques often exceeds 0.5%. For example, the large uncertainties in spectrophotometry and chromatography arise primarily from the very small sample weight, and the large uncertainty in photometry is caused by the strong dilution of the analyte solution. Inorganic impurities in inorganic or organic compounds can readily be determined at contents down to 1–10 ppm. Many impurities can be determined by well-known techniques, such as spectrophotometry or polarography.[8]

The separation of functions between classic chemical and instrumental techniques made it possible to markedly lower detection limits and implement computer-aided data processing, which considerably reduced the analysis time, without impairing the accuracy and reliability of analyses.

In the information structure under consideration (Fig. 1), analytical monitoring of the quality of high-purity substances and chemical reagents includes the following steps (Fig. 4): sampling, sample preparation, analysis by the technique chosen, and data processing. At the same time, the choice of the analytical technique depends primarily on the expected concentration range, especially on its lower boundary, and on metrological characteristics of the technique, such as accuracy and reproducibility. An important role in selecting the analytical technique is also played by practical factors, e.g., the number of analyzed impurities, analysis time, availability of apparatus, and personnel qualification. In connection with this, expert systems with extended databases have recently become a necessity.[5] In addition to formalized entity-attribute relationships, extended databases include empirical rules according to which subjects are classified and preparatory operations and methods of analysis are chosen.

As the requirements imposed by technology on analytical techniques were becoming more and more exacting, instrumentalization of the final stages of analyses was approaching completion, increasing the role of preconcentration step in sample preparation.[8] In view of this, in the case of complex analytes, the Sample Preparation (thermal or chemical stripping of poorly soluble substances) and Preconcentration (preparation of a representative solution for a particular technique) subgroups are included in the Analytical Procedure group.

The effectiveness of preparatory steps determines in many respects the success of the entire analytical technique. The use of CALS technology, in turn, makes it possible to achieve the following results:

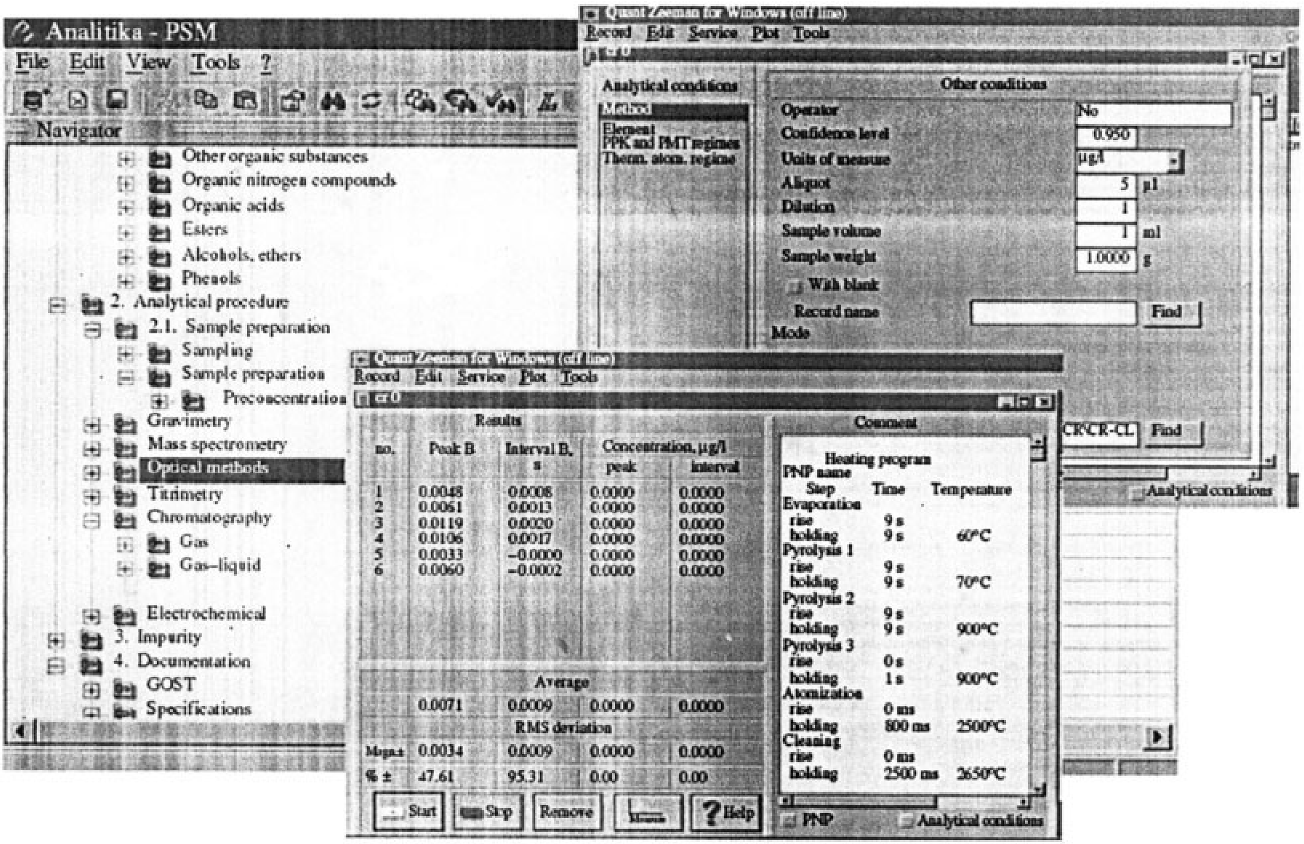

Figure 4 CALS group (electrothermal atomization atomic absorption spectrophotometry).

- to develop a database for the measuring facilities used, including the sample preparation step (the set of facilities, their performance characteristics, measuring ranges, standardized measuring procedures, accuracy class, etc.);
- to carry out comparative analysis of measuring facilities;
- to analyze the compatibility of the already existing and new equipment; and
- to enhance technical documentation using modern software (Word, AutoCad, etc.).

The instrumental analytical techniques included in the CALS project involve information about the appropriate instruments (Fig. 4):

- schematic diagram,
- service documentation,
- calibration check schedule and certificate, and
- calibration plot.

The CALS system provides solutions to some of the associated problems:

- automatic order of materials and replacement;
- planning and maintenance record keeping;
- diagnostics of equipment and trouble shooting;
- provision of reference data on the design and operating principle of equipment;
- provision of reference data necessary for running the equipment, performing maintenance work, and repairing instruments;
- provision of information about the personnel; and
- training of personnel to run, maintain, and repair the equipments.
- The PDM STEP Suite 2.5 system allows forming and using a unified electronic product description on the basis of the ISO-10303 STEP standard. Determining the structure of stored data and software interfaces using the international CALS-standard makes it possible to concurrently retrieve necessary information on different knowledge domains and to integrate with any other information system. No other PDM system offers such opportunities.[7]
- A standard CALS project is a set of functional models involving a description of the sample life-cycle stages, monitored at the Analytical Research Center. In the pilot CALS project, these models represent typical computer structures[14]: marketing, design, manufacturing, operation, repair, and others.

- At the marketing stage, it is necessary to take certain measures aimed at analyzing the market for high-purity substances; to compare the products with Russian and foreign analogs; to assess the demand and potential customers' requirements on quality; to plan measures to ensure necessary analytical monitoring, without exaggerating the advantages of the product; to plan measures intended to upgrade existing technologies and promote the product to the market; to assess users' and potential manufacturers' requirements on control over limiting impurities; and to study the effect of such impurities on the properties of the high-purity substance and the operation of related devices.
- At the design stage, one considers existing sampling, stripping, and characterization techniques and carries out research work aimed at devising new, more promising techniques.
- The manufacturing stage involves the laboratory measures described earlier (Analytical Procedure group). In addition, this stage includes internal and external analysis quality control in conformity with the relevant metrological requirements, including those on data processing.
- The operation stage involves the use and maintenance of equipment and the documentation involved (Fig. 4). This stage also includes disposal elements, in particular, normative and technical documentation pertaining to the disposal of samples and controls carrying some hazard (toxic, explosion-hazardous, and flammable). The repair stage is structured at three levels[15]:

- malfunction of analytical equipment,
- origin of malfunction, and
- correction of the malfunction.

The CALS system allows one to easily find a substance of the desired grade using a standard classification (Fig. 2). Next, a normative documentation is searched for by its definition for each controlled impurity (table in Fig. 2). This enables rapid gaining of information about the analytical technique and equipment of interest (Fig. 4), together with typical output documents (Fig. 3).

The Analyte group of our system is very similar in many respects to the analogous group in the High-Purity Substances and Materials (Exhibition-Collection of Extrapure Substances) information system.[1] At the same time, the Analytical Procedure and Documentation groups in the CALS system contain substantially more information.

The use of the CALS standard in designing monitoring information systems allows one to enhance the effectiveness of data processing and, hence, the quality and speed of analyses. Therefore, CALS technology offers the possibility of considerably reducing the cost of analyses and enhances the quality and serviceability of the information systems. On the whole, this information technology allows one to create not only an effective quality control system meeting international standards but also an effective system for coordinating the analytical centers involved throughout the research and production cycle.

Potential users of such information include workers and supervisors at science and production institutions, inspecting state bodies, investors, etc.

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