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IMPROVEMENT OF INFORMATION RELIABILITY AND FAULT DIAGNOSTICS IN PROCESS CHROMATOGRAPHS

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SUMMARY

An effective algorithmic method for improvement of the operational reliability of chromatographic systems is proposed. The method includes algorithms for checking the validity of information, and for fault diagnosis, designed for realization on a microcomputer. The set of the most informative parameters to be monitored is found by analysis of a model in the form of a cause-and-effect graph. For effective operation of control and diagnostic systems, it is necessary to provide a two-level control of the detector signal and of the intermediate and final results of processing, together with the main parameters of the analyzer.

INTRODUCTION

The application of chromatographs to automatic process control systems and computer-aided scientific research systems has highlighted the problem of ensuring high operational reliability of the apparatus. At the same time, more stringent requirements for the quality of analysis have led to still higher complexity of analyzers and consequently to lower reliability. The only way to solve this contradiction is on the basis of both technical and algorithmic methods.

Among the effective algorithmic methods of improvement of the reliability of chromatographic systems are those involving control of the validity of processing information and the automatic diagnosis of faults. In chromatographic systems only sudden faults are usually detected. Fault location is carried out by an operator with the help of manufacturers guides (see, for example, refs. 1 and 2). Nowadays, automatic diagnostic methods are available[3,4], but in chromatographic systems they are normally used only for locating faults in discrete control and data-logging devices (by signature analysis, for example).

In this paper an effective algorithmic method for improvement of the operational reliability of chromatographic systems is proposed. The method includes checks on the information validity which permit the deletion of erroneous data and indicate the presence of a fault (the first stage). If a fault is discovered, a diagnostic algorithm (the second stage) is initiated, followed by automatic identification of the fault.

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METHOD

Information validity check

The information validity of an analytical measurement system is defined here as the probability (D) of absence of any kind of anomalies from the system.

$$D = 1 - P_{\text{err}} \qquad (1)$$

where P_{err} is the probability of appearance of anomalous results. The validity of the results appears to be directly related to the system reliability parameters, especially to its operational reliability: faultless, exact and timely operation.

Two groups of algorithmic methods for improving the reliability may be distinguished. Those belonging to the first group involve estimations of signal parameters (or processing results) that allow correct results to be obtained in the presence of gross errors, spikes, drift, impulse noise, etc., that can be described as distorting factors[5] and make the least squares technique fail. The second group embraces various statistical methods of checking the validity of processed information and implies the application of traditional methods of processing with the use of criteria for diagnosing various anomalies.

Combined application of methods in both groups is most efficient. However, the methods in the second group are preferred as they allow not only the identification and deletion of an anomalous result but also the realization of the fault-location diagnostics of the system.

In order to test the validity of the analytical information in the chromatographic system, a procedure has been elaborated, which includes the following steps[6]:

(a) filling the chromatographic system fault set $A = \{A_i;\ i = 1, \ldots, n\}$ in conformity with the accepted classifier and the fault symptom set $B = \{B_j;\ j = 1, \ldots, m\}$;

(b) specification of faults from the nature of the symptoms; detection of cause-and-effect relationships;

(c) development of a diagnostic model of the chromatographic system in the form of graphs of the cause-and-effect relationships;

(d) analysis of the diagnostic model with the aim of selecting the informative parameters for the validity check.

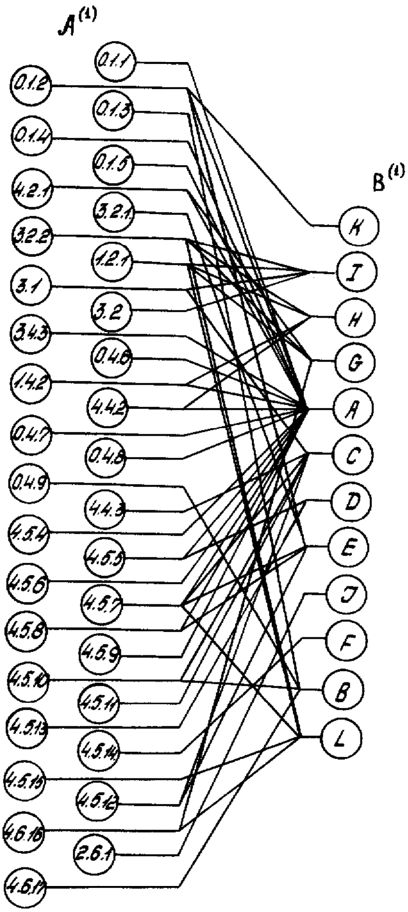
Synthesis the diagnostic model and choice of the informative parameters

When compiling the list of faults, A, in the chromatographic system, typical failures taking place in gas flow chromatographs have been taken into account. At this stage, the opinion of experts was widely sought.

Classification of faults was performed on the basis of the "place of appearance" criterion (detector, column unit, etc.) with further division according to operational characteristics (failures due to temperature or flow deviations, to carrier pollution or line clogging, etc.). This embraced failures of units in the chromatograph proper, in the sample preparation and inlet system, and algorithmic failures (in the course of processing). Computer failures, which are usually monitored by special tests included in the standard software, were not taken into consideration when determining the informative parameters.

A three-value code X, Y, Z is assigned to each failure, where X represents the location, Y is the operational characteristic and Z a serial number for the chromatograph unit under consideration. The failures in a given unit are also arranged in accord with their hazard and significance ranking.

When filling the set of symptoms, *B*, it was ascertained that while faults in the chromatograph itself are revealed mainly by the detector signal, failures of the sample preparation unit and algorithmic failures are revealed sufficiently clearly in the nature of the changes of the results. This led to the elaboration of a diagnostic model of the chromatographic system in the form of a two-level information graph of cause-and-effect relationships. The first and the second level of failure mapping are shown in Figs. 1 and 2, respectively. Here, $A^{(1)}$ is the subset of the chromatograph failures, $A^{(2)}$ the subset of the sample preparation-unit, and algorithmic failures, $B^{(1)}$ the subset of failure symptoms in the parameters of the detector signal and $B^{(2)}$ that in the



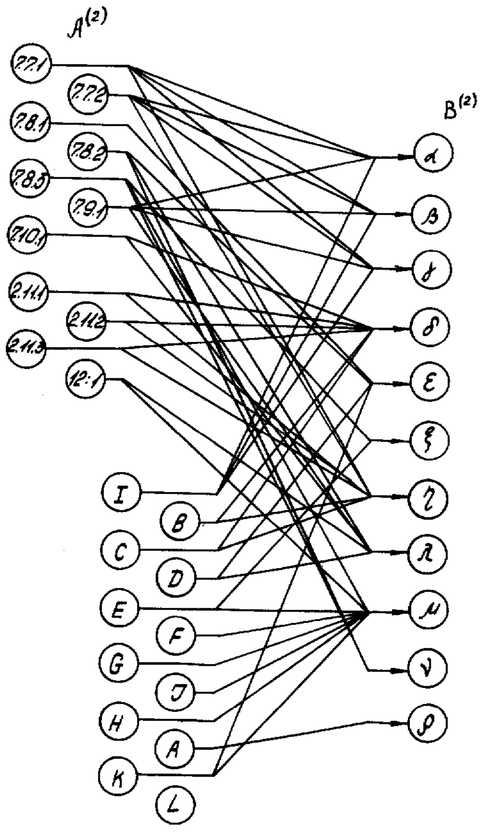


Fig. 1. Information graph of the cause-and-effect relationships between the subset of failures $A^{(1)}$ and the subset of failure symptoms $B^{(1)}$ in the detector signal.

Fig. 2. Information graph of the cause-and-effect relationships between the subset of failures $A^{(2)}$ and the subset of failure symptoms $B^{(1)}$ and $B^{(2)}$ in the detector signal and in the processing results, respectively.

parameters of the processing results. The constituents of those subsets are given in Tables I–IV.

For the purposes of the analysis of the two-level graph of cause-and-effect

TABLE I

SUBSET $A^{(1)}$ OF TYPICAL CHROMATOGRAPH FAILURES

Location of failure	*Code*	*Characteristic*
Detector	4.2.1	Lower temperature
Detector	4.4.2	Pollution
Detector	4.5.8	Filament out of order
Gas system	0.1.2	Carrier gass leakage
Gas system	0.1.4	Irregular hydrogen consumption

TABLE II

SUBSET $A^{(2)}$ OF POSSIBLE FAILURES IN THE PROCESSING AND SAMPLE-PREPARATION SYSTEMS

Location of failure	*Code*	*Characteristic*
Processing algorithm	7.7.1	Detection of false pak
Processing algorithm	7.8.1	Wrong correction for drift
Sample-preparation unit	2.1.13	Invalid sample volume

TABLE III

SUBSET $B^{(1)}$ OF FAILURES IN DETECTOR SIGNAL

Designation	*Description*
A	Higher noise level on the baseline
B	Periodical spikes on the baseline
C	Single surges on the baseline
E	Monotonic drift of the baseline
I	Non-reproducibility of holding time

TABLE IV

SUBSET $B^{(2)}$ OF FAILURES IN PROCESSING OF RESULTS

Designation	*Description*
α	Invalid holding time
η	Anomalous error in concentrations
μ	Monotonic drift of concentrations
λ	Shift in concentrations
ν	Concentration overshooting the limit

relationships, the significance and hazard of all the failures have been conditionally assumed as equiprobable. Then the information content of an element in the set $B = B^{(1)} \cup B^{(2)}$ is determined by the number of links with elements of the set $A = A^{(1)} \cup A^{(2)}$. The qualitative and quantitative analysis of the cause-and-effect relationships diagram revealed that about 90% of all the failures of subset $A^{(1)}$ are mapped on elements A, B, C, E, I (Table III) and over 80% of all the failures of subset $A^{(2)}$ are mapped on elements λ, η, μ, α, ν (Table IV). It is these informative parameters that will be monitored.

Statistical criteria for information validity check algorithm

The detection of anomalies during monitoring of the informative parameters is achieved with the help of statistical criteria[6–9]. The quality and serviceability of a number of criteria have been studied by the Monte Carlo method[7,10].

The comparative analysis of the quality of various criteria has been performed for a number of characteristics[6,9], such as

$$\text{power, } P = Pr\{\tau \in \omega_{cr} | H_1\} \tag{2}$$

where Pr is the probability of proper detection of anomalous values in the information when test samples have those values (the hypothesis H_1 is true), φ (statistics) is some function of sample values (see Table V), and ω_{cr} is the critical region [if the statistics φ get in to this region, of which the border value depends on the previously chosen significance level (α), then hypothesis H_1 is true];

$$\text{relative efficiency, } E_i = P_{i,\alpha}/P_{\text{comp},\alpha} \tag{3}$$

where α = significance level, $P_{i,\alpha}$ and $P_{\text{comp},\alpha}$ the power of the ith and comparison criterion, respectively;

$$\text{threshold of sensitivity, } \mathcal{S} = \text{minimum } (\lambda/S) | P \tag{4}$$

where λ = shift of the mean in the failure model and S = estimation of the standard deviation;

$$\text{labour intensity, } T_i = V_i/V_{\text{comp}} \tag{5}$$

where V_i and V_{comp} are the number of algorithmic operations required to realize the ith and comparison criteria, respectively.

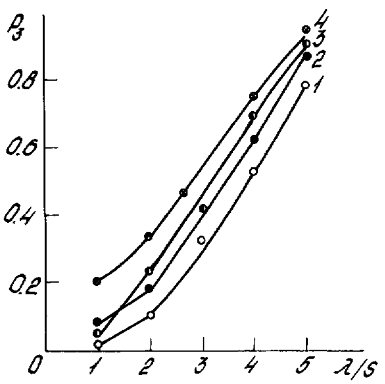
Two types of models have been used for the simulation of failures[7]: the shift of the mean model

$$F_1(X) = N(\mu + \lambda\sigma, \sigma^2) \tag{6}$$

and the variance expansion model

$$F_2(X) = N[\mu, (\lambda')^2\sigma^2] \tag{7}$$

where μ and σ^2 are the mean and sampling variance.



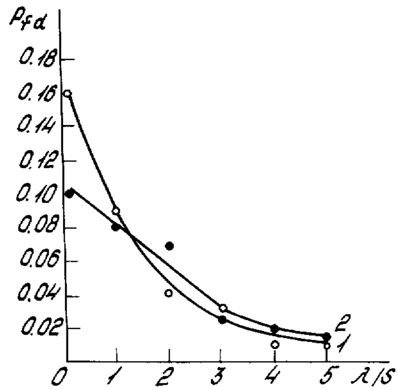


Fig. 3. Power function obtained for (1) the Thomson criterion, (2) the Dickson criterion, (3) the Smirnoff-Grabbs criterion, and (4) the Ferguson criterion 1 ($M = 11$, $\alpha = 0.05$).

Fig. 4. False detections according to (1) the Smirnoff-Grabbs criterion, and (2) the Dickson criterion.

Experimental data for the power, P, and for the probability of false detections, P_{fd}, as obtained for four criteria are illustrated in Figs. 3 and 4. The criteria are given in Table V. The analysis showed that the least labour-intensive though sufficiently powerful criteria for both one- and two-sided problems are those represented by various modifications of the Dickson criterion. The Dickson criterion of labour intensity is 2–3 times less (upper limit, with $M = 15$; lower limit, with $M = 5$) than that of the Smirnoff-Grabbs criterion recommended by the Council for Mutual Economic Assistance, Standard 545-77, while its power is practically no lower and with

TABLE V

PARAMETERS OF CERTAIN CRITERIA USED FOR DETECTION OF SINGLE SPIKES

Failure model (6): $\lambda = 2$; $\alpha = 0.05$; $M = 11$
$X_{(i)}$ = ith value of the sample, ordered by magnitude ($i = 1, \dots, M$); X_i = value of the sample tested; $\bar{X}$ and S = mean and standard deviation, respectively.

Criterion	*Statistics*	*P*	*E*	*𝒮*	*T*
Smirnoff-Grabbs	$\frac{(X_{(M)} - \bar{X})}{S}$	0.24	1	3.1	1
Thomson	maximum $\left\lvert \frac{X_i - \bar{X}}{S} \right\rvert$	0.07	0.29	3.8	1.18
Dickson	$\frac{X_{(M)} - X_{(-1)}}{X_{(M)} - X_{(1)}}$	0.19	0.79	3.3	0.33
	$\frac{X_{(M)} - X_{(M-1)}}{X_{(M)} - X_{(2)}}$	0.22	0.92	3.3	0.33
Ferguson I	$\frac{\sqrt{M}\sum_{i=1}^{M}(X_i - \bar{X})^3}{\left(\sum_{i=1}^{M}(X_i - \bar{X})^2\right)^{3/2}}$	0.41	1.42	2.8	1.29

small shifts, λ, in the model of failures (eqn. 6) it may be even higher (up to 1.5 times). The Smirnoff-Grabbs criterion gives the maximum number of false detections (Fig. 4). The most powerful criterion with a one-sided problem, which is optimal even with small shifts, is the Ferguson criterion. However, the latter has the most elaborate statistics and is very labour-intensive ($T_{av} = 1.29$); it is optimal only for considerable sampling volumes ($M \geqslant 20$).

The characteristics mentioned make it possible to select a criterion most suitable for the specific operating conditions of the system. With a small amount of *a priori* information, it is expedient to use various modifications of the Dickson criterion in succession.

Similarly, the Abbe criterion and special stastistics restriction regions have been investigated and formulated for determination of the monotonic drift and detection of shifts[6].

Synthesis of the diagnostic algorithm

The check of the chromatographic information validity at two levels, the detector signal level and the processing results level, allows one to ascertain only whether the chromatographic system is either serviceable or faulty. Therefore, a diagnostic algorithm is needed for the identification of failures.

The elaboration of this algorithm is based on the same graphs of cause-and-effect relationships, but traced in the reverse direction, *i.e.*, from elements of the set B to elements of the set A. To solve this problem the ancillary information is to be used, such for example as the information on operating parameters of the chro-

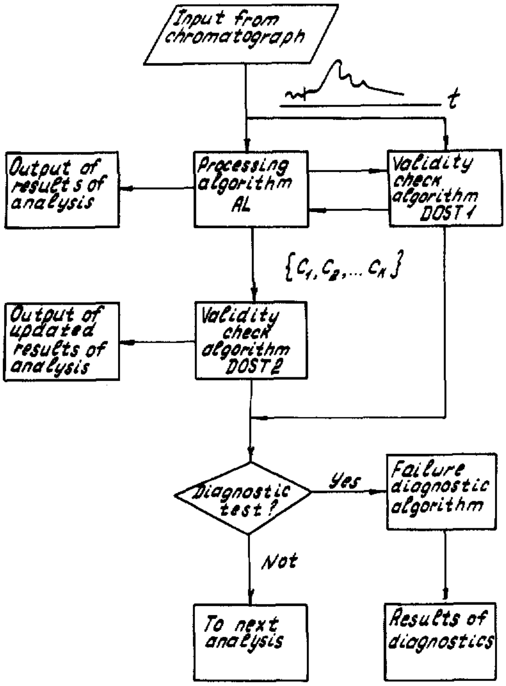


Fig. 5. Block diagram of the supporting algorithms.

matograph systems. Thus, the efficiency of the diagnostic algorithm is determined by the number of indistinguishable failures and the quantity of failures that can be detected and identified depends on what particular ancillary information can be utilized.

For the synthesis of the diagnostic algorithm the diagram shown in Fig. 1 is analysed, resulting finally in detection of: (a) failures identified unequivocally without utilizing the ancillary information; (b) failures identified when utilizing the information furnished by the devices available in the chromatograph; (c) failures that remain indistinguishable and require the provision of additional hardware or intervention of the analyst. The part of the cause-and-effect relationships diagram being from the element of the set *B* on which the maximum number of failures is mapped.

SUPPORTING ALGORITHMS

A typical block diagram of the supporting algorithms is given in Fig. 5. The validity check algorithms DOST1 and DOST2 correspond to the two-level check mentioned earlier (Figs. 1 and 2) and form together with the chromatographic signal processing algorithm, AL, a single complex. The results of obtained by the action of algorithms DOST1 and DOST2 are used as the source information for the diagnostic algorithm which is initiated either during the system check-out or during fault detection.

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