

# An Estimation Problem of Chemical Process with Confluent Parameters: An Interval Approach\*

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## Abstract

We apply interval analysis to a practical problem of estimation of confluent (merged) parameters of a chemical process under the following uncertainty conditions: the process measurements are noisy (corrupted), the probability characteristics of measuring bounded errors are absent, the measurement sample is fatally short, and only approximate *a priori* intervals of the parameters are available. Our algorithm uses a model function describing the chemical process and produces a *containment set* of admissible values of the parameters with an exact description of its boundaries in comparison with the known parallelootope estimation approach.

**Keywords:** interval estimation, parameters, experimental process, measurements, bounded error, approximate *a priori* data

**AMS subject classifications:** 65G40, 93A30

## 1 Introduction

In the paper, we consider a practical problem of processing data obtained in a chemical experiment. We investigate the change in some reagent activity vs. the temperature to estimate a set of admissible values of the process parameters. The process is described by a simple second order polynomial. However, there are serious constraints. Estimation has to be performed under uncertainty conditions: the process activity measurements are noisy (corrupted) with a bounded error whose probability characteristics are completely absent, the measurement sample is fatally short, and only approximate *a priori* intervals of the parameters are available. Further, the three parameters of the process enter into the description function not separately, but merged way into one confluent parameter. As a result, estimation of the set of admissible

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values of the parameters by standard statistical methods (for example, [2, 3, 4]) is difficult or impossible.

We consider an approach based on interval analysis (see [6, 7, 18]). Results are presented in the form of a set of admissible values of parameters with an exact description of its boundaries. As it is customary in interval analysis, uncertainties in measurements are captured by intervals. We seek a set containing parameter values of a certain chemical reaction capturing dependencies on these uncertainty intervals.

Peculiarities of the problem (a simple describing convex function having an inverse and the presence of *a priori* intervals for parameters) allowed us to adjust standard interval algorithms. As a result, simple and fast algorithms for direct construction of a containment set were used successfully for many chemical experiments.

In the well-known parallelotope estimation approach (see, for example, [7] and the powerful SIVIA algorithms presented there), the set boundaries are estimated by a large collection of small parallelotopes. In our approach, the containment set is represented by a collection of its two-dimensional cross-sections with an exact analytical description of their boundaries.

The goal of this work is further development of a new approach for processing the above-mentioned experimental data. The paper has the following structure. In Section 2, the process, its peculiarities, and the input data are described. Section 3 is devoted to interval analysis ideas and the problem formulation. In Section 4, we consider an algorithm for solving the problem and elaborated applied procedures. Section 5 presents results of computation of a containment set of the process parameters. In Section 6, conclusions of the work are given.

## 2 Experimental Process and Input Data

The description of a reagent activity vs. the temperature has the form (see [13, 14]).

$$P(T, a, b, c) = T^2 a b / c, \quad a > 0, \quad b > 0, \quad c > 0, \quad (1)$$

where  $T$  is the temperature (the argument), in  $^{\circ}\text{C}$ ;  $P(\cdot)$  is the reagent activity, a dimensionless value;  $a$ ,  $b$ , and  $c$  are parameters to be estimated with dimensions mole, 1/mole, and  $(^{\circ}\text{C})^2$ , respectively. Measurements of the experiment are presented as the collection of  $N$  measurements of the reagent activity  $P$ ,

$$\{T_n, P_n\}, \quad n = \overline{2, N}, \quad (2)$$

where the time values  $T_n$  are assumed to be known exactly, but the activity values  $P_n$  are measured with bounded error (noise),

$$P_n = P_n^* + e_n, \quad |e_n| \leq e_{\max}, \quad n = \overline{2, N}, \quad \text{and for } T_1 = 0, \quad P_1 = 0, \quad (3)$$

where  $P_n$  is a noisy measurement;  $P_n^*$  is an unknown true value;  $e_n$  is the error in the  $n$ th measurement; and  $e_{\max}$  is the bound of the maximal (by modulus) value of the error. By physical reasoning, the *conditional exact initial measurement*  $P_1$  at  $T_1 = 0$  is zero.

### Conditions for estimation:

- No probabilistic information on errors is known;
- the sample is fatally short:  $N \approx 5 - 7$  measurements only;

- in (1), parameters  $a, b$ , and  $c$  are merged, meaning that it is impossible to estimate of their admissible intervals without some additional information;
- from theoretical estimations and previous experience, only approximate rough *a priori* interval constraints on possible values of the coefficients are available:

$$\begin{aligned} \mathbf{a}^{ap} &= [\underline{\mathbf{a}}^{ap}, \overline{\mathbf{a}}^{ap}], \quad \mathbf{b}^{ap} = [\underline{\mathbf{b}}^{ap}, \overline{\mathbf{b}}^{ap}], \quad \mathbf{c}^{ap} = [\underline{\mathbf{c}}^{ap}, \overline{\mathbf{c}}^{ap}], \\ 0 < \underline{\mathbf{a}}^{ap} < \overline{\mathbf{a}}^{ap}, \quad 0 < \underline{\mathbf{b}}^{ap} < \overline{\mathbf{b}}^{ap}, \quad 0 < \underline{\mathbf{c}}^{ap} < \overline{\mathbf{c}}^{ap}. \end{aligned} \quad (4)$$

The LSQM curve and pointwise estimation of parameters  $a$ ,  $b$ , and  $c$ , and their practically meaningless “cloud-built” intervals can be calculated by *only formal application* of standard statistical procedures [2, 3, 4].

### 3 Interval Approach and Problem Formulation

Ideas and methods of interval analysis theory and application arose from the fundamental, pioneering work of L.V. Kantorovich [8]. Currently, many researchers have written very effective developments of the theory and computational methods, e.g., [5, 6, 7] and in Russia [17, 18, 19, 23]. Special interval algorithms have been developed for estimating parameters of experimental chemical processes [1, 10, 11, 12, 13, 14, 15, 16]. The essence of this “reliable computing” branch of numerical methods theory and application consists of the estimation of parameters under bounded errors (noise or perturbations) in the input information to be processed, and with no further assumptions on the probabilistic characteristics of the errors.

We need the following definitions, using standard interval notation [9].

The *uncertainty set* (interval) of each measurement (USM) is the interval of values of a measured process consistent with the measurement and the error bound,

$$\mathbf{H}_n = [\underline{h}_n, \overline{h}_n] : \underline{h}_n = P_n - e_{\max}, \quad \overline{h}_n = P_n + e_{\max}, \quad n = \overline{1, N}. \quad (5)$$

$\mathbf{H}_1 = \mathbf{H}(0)$  is a thin interval (zero width), since the reagent activity is zero by a physical condition at  $T = 0$ .

The *admissible value* of the parameter vector and corresponding *admissible curve* is

$$(a, b, c) : P(T_n, a, b, c) \in \mathbf{H}_n, \quad \text{for all } n = \overline{1, N}. \quad (6)$$

The *containment set* (in Russian terminology an *information set*) is a totality of all *admissible values* of the parameter vector satisfying the system of interval inequalities (6)

$$\mathbf{I}(a, b, c) = \{(a, b, c) : P(T_n, a, b, c) \in \mathbf{H}_n, \text{ for all } n = \overline{1, N}\}. \quad (7)$$

Because of the very short length of the measurements sample, the absence of probabilistic characteristics of the errors, and measurements uncertainty, it is *impossible* to use (with any good reasoning) the standard statistical methods [2, 3, 4] for constructing the *containment set* from (7).

**Problem formulation:** Using interval methods, we built the containment set  $\mathbf{I}(a, b, c)$  of admissible values of coefficients  $a$ ,  $b$ , and  $c$  *consistent* with the described data.

## 4 Scheme of Solving and Applied Procedures

There are several approaches to solve system (6) of the interval inequalities:

- classic linear programming methods [8] and many others;
- application of parallelotopes method of Fiedler M., *et al* [5], Hansen [6], Jaulin, *et. al* [7], Shary [18];
- “stripes” method of Shary and Sharaya [19], Sharaya [17], Zhilin [23].

The function (1) that describes our chemical process is simple. It is convex, it has an inverse, and the dimension of the parameter vector is only three. Hence, a more convenient and faster *direct grid-analytical* method has been developed for constructing the containment set of parameters; see details in Kumkov and co-authors [1, 10, 12, 13, 14, 16]. This method gives an *exact* representation of boundaries of the containment set (7) cross-sections in two parameters for each node of the grid for the third parameter.

To emphasize, we represent the set  $\mathbf{I}(a, b, c)$  in the form of a collection of its two-dimensional cross-sections  $\{\mathbf{I}_a(b, c)\}$  for nodes of the grid on the parameter  $a$  in its *a priori*  $\mathbf{a}^{\text{ap}}$  or possible minimal outer  $\mathbf{a}^*$  interval of admissible values. The boundaries of each cross-section  $\mathbf{I}_a(b, c)$  are described exactly and analytically.

It is useful to compare our approach with one used in the SIVIA-type programs of Jaulin, *et al* [7]. Their approach is based on an outer approximation of the boundaries of the containment set by a collection of parallelotopes. Their method is *universal* and applicable to *any type* of function describing the process under investigation. However, a multi-dimensional space of parameters with stringent accuracy requirements leads to very large collection of small parallelotopes and expensive computations.

In contrast, our approach is not universal. It is adjusted for *linear dependencies* on estimated parameters, and it applies *hybrid grid-set* techniques. It describes *exact analytical* boundaries of the containment set cross-sections. Moreover, representation of the containment set as a collection of its cross-sections demands substantially fewer computations in practical problems with a low dimensional parameter space.

We solve three auxiliary problems:

1. Introducing the auxiliary merged parameter  $g = ab/c$ , with  $c > 0$ , its containment interval  $\mathbf{g} = [\underline{g}, \overline{g}]$  is calculated [13, 14].
2. If  $d = b/c$ , the interval equation  $ad = \mathbf{g}$  is solved w.r.t. the auxiliary parameter  $d$  as  $\mathbf{d} = \mathbf{g}/\mathbf{a}^{\text{ap}}$ . As a result, in the plane  $a \times d$ , we obtain the containment set  $\mathbf{I}(a, d)$  with the curve (hyperbolic) lower  $\underline{Fr}_d(a)$  and upper  $\overline{Fr}_d(a)$  boundaries as a functions of the parameter  $a$  values from its *a priori* interval  $\mathbf{a}^{\text{ap}}$ .
3. For each value  $a \in \mathbf{a}^{\text{ap}}$ , we have the interval  $\mathbf{d}(a)$ . We construct the containment set  $\mathbf{I}_a(b, c)$  of admissible values for parameters  $b$  and  $c$  for each admissible value of the parameter  $a$ . As a result, having a grid in the interval  $\mathbf{a}^{\text{ap}}$  (in its possible enhanced minimal outer interval  $\mathbf{a}^*$ ), the containment set  $\mathbf{I}(a, b, c)$  is built as a collection  $\{\mathbf{I}_a(b, c)\}$  of its two-dimensional cross-sections.

## 5 Computation Results

For investigation, values of the true model parameters (Fig. 1) were given as  $a_{\text{md}} = 2.0$  mole,  $b_{\text{md}} = 1/140$  mole<sup>-1</sup>,  $c_{\text{md}} = 100$  (°C)<sup>2</sup>, and corresponding  $g_{\text{md}} = 1.4285 \times 10^{-4}$  1/(°C)<sup>2</sup>. The bound on the measurement error is  $e_{\text{max}} = 0.1$  (dimensionless). The

number of measurements is  $N = 7$ ; the sample has values (corresponding to numbers  $n = \overline{1,7}$ ) of temperature  $T_n$  and activity  $P_n$ : (0, 0), (15, 0.0076), (25, 0.096), (35, 0.191), (45, 0.217), (60, 0.474), and (75, 0.858) of dimensions °C and dimensionless, respectively. The approximate *a priori* intervals were given as  $\mathbf{a}^{ap} = [1.8, 2.2]$  mole,  $\mathbf{b}^{ap} = [1/160, 1/120]$  mole<sup>-1</sup>, and  $\mathbf{c}^{ap} = [80, 130]$  (°C)<sup>2</sup>.

Figure 1 shows the input measurements, their uncertainty sets  $\mathbf{H}_n$ , the true model curve, and the LSQM curve. For  $e_{\max}$ , the LSQM curve is admissible in the interval sense.

**Solution of auxiliary Problem 1.** For the initial point-wise uncertainty set  $\mathbf{H}_1$  and each uncertainty set  $\mathbf{H}_n$ ,  $n = \overline{2,7}$ , the *partial* containment interval  $\mathbf{G}_{1,n} = [\underline{g}_{1,n}, \overline{g}_{1,n}]$  is calculated (Fig. 2),

$$n = \overline{2,7}, \underline{g}_{1,n} = \underline{h}_n/T_n^2, \overline{g}_{1,n} = \overline{h}_n/T_n^2. \quad (8)$$

The resulting containment interval  $\mathbf{I}(g)$  of the merged parameter  $g$  is obtained by intersecting the partial intervals  $\mathbf{G}_{1,n}$  (Fig. 2),

$$\mathbf{I}(g) = \cap_{n=\overline{2,7}} \mathbf{G}_{1,n}. \quad (9)$$

We calculate the *a priori* interval  $\mathbf{g}^{ap}$  of the parameter  $g$  and compare it with the interval  $\mathbf{I}(g)$  for analysis of *consistency* of the *a priori* data (4) on parameters  $a$ ,  $b$ , and  $c$  with the given sample of measurements (2) and (3). The data is consistent if the interval  $\mathbf{I}(g)$  has non-empty intersection with the *a priori* interval  $\mathbf{g}^{ap}$  of this parameter (Fig. 2, case *a*). Otherwise (Fig. 2 case *b*), the sample and the *a priori* data are inconsistent.

The solution of the auxiliary Problem 1 and the tube of admissible dependencies (shaded) are shown in Fig. 3. They overlap, so we conclude that the sample is consistent for the given value  $e_{\max}$ .

The well-known interval analysis procedure [20, 21, 22] of regulation of the bound  $e_{\max}$  in (2) and (3) allows one to estimate from below the maximal value  $e_{\max}^*$  of the *actual error level* in the sample to be processed.

The approximate initial value was  $e_{\max}^{\text{init}} = 0.1$  (dimensionless), and the estimated interval was  $\mathbf{I}(g) = [1.34, 1.59] \times 10^{-4}$  1/(°C)<sup>2</sup>. In the limit, the estimate has the value  $e_{\max}^* = 0.0457$  (dimensionless), and the limiting point value of the merged parameter is  $\mathbf{g}^* = 1.449 \times 10^{-4}$  1/(°C)<sup>2</sup>. In this case, the tube of admissible dependencies degenerates to only one curve corresponding to the value  $\mathbf{g}^*$ .

**Solution of auxiliary Problem 2.** The containment set  $\mathbf{I}(a, d)$  of parameters  $a$  and  $d$  for  $d = b/c$  is presented in Fig. 4. Again, we can analyze the consistency of *a priori* data with the measured ones. In Figure 4, the *a priori* interval  $\mathbf{d}^{ap}$  of the auxiliary parameter  $d$ , shown as the thick dash-dotted vertical segment, has been calculated from the *a priori* intervals  $\mathbf{b}^{ap}$  and  $\mathbf{c}^{ap}$ . The thick vertical line in dashes marks the outer interval of  $\mathbf{I}(a, d)$  in  $d$  for the *a priori* interval  $\mathbf{a}^{ap}$ . A non-empty intersection of these two intervals implies the *consistency* of the *a priori* data (4) for parameters  $a$ ,  $b$ , and  $c$  with the given sample of measurements.

In the case of a wider *a priori* interval  $\mathbf{a}^{ap}$ , the solution of Problem 2 from our interval approach enhances (tightens) both the output containment set from  $\mathbf{I}(a, d)$  to  $\mathbf{I}^*(a, d)$  (Fig. 5, shaded) and the initial interval  $\mathbf{a}^{ap} = [1.27, 3.38]$  mole of parameter  $a$  to a tighter  $\mathbf{a}^* = [1.32, 3.21]$  mole.

**Solution of auxiliary Problem 3.** Our calculated containment set  $\mathbf{I}_a(b, c)$  of parameters  $b$  and  $c$  for the fixed value  $a = 1.89$  mole is shown in Fig. 6, built by

intersecting the *a priori* rectangle  $\mathbf{b}^{\text{ap}} \times \mathbf{c}^{\text{ap}}$  (or with possible enhanced intervals) with the cone between the lower  $\underline{c}(b, 1/\underline{d}(a))$  and upper  $\bar{c}(b, 1/\bar{d}(a))$  rays for each  $a \in \mathbf{a}^{\text{ap}}$  and  $b \in \mathbf{b}^{\text{ap}}$ . Here, the set  $\mathbf{I}_a(b, c)$  (the shaded five-vertex polygon) is shown for the value  $a = 1.89$  mole, with the corresponding interval  $\mathbf{d}(1.89)$  from our solution of Problem 2.

Finally, the output containment set  $\mathbf{I}(a, b, c)$  is built as the collection  $\{\mathbf{I}_a(b, c)\}$  of its two-dimensional cross-sections shown in Fig. 7.

## 6 Conclusions

We have considered an estimation problem with confluent (merged) parameters without probabilistic characteristics of the measuring errors. Our interval approach allows us to analyze the consistency of the given sample of measurements itself, to analyze the consistency of the given sample of measurements and the given *a priori* data, and to construct the enclosing set of admissible values of parameters. Our algorithms allow a simple numeric implementation. In special cases, they can give exact estimations of the containment set, and they are faster than previous interval approaches based on parallelotopes.

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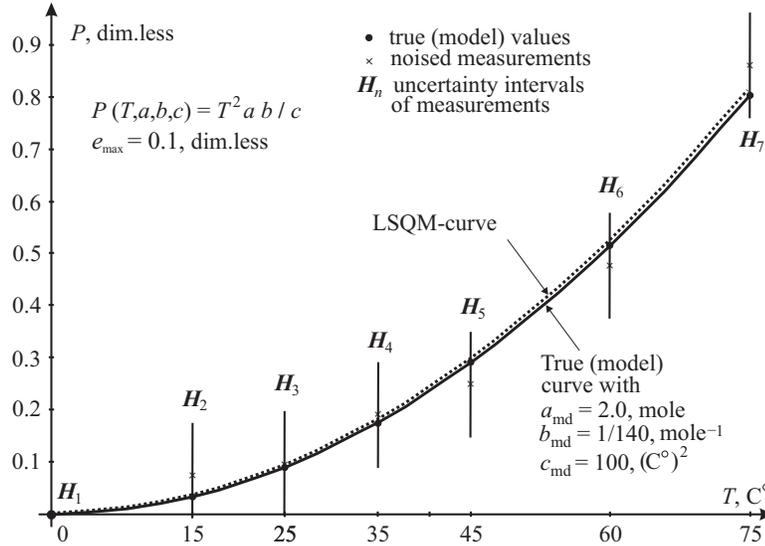


Figure 1: Measurements and their uncertainty sets.

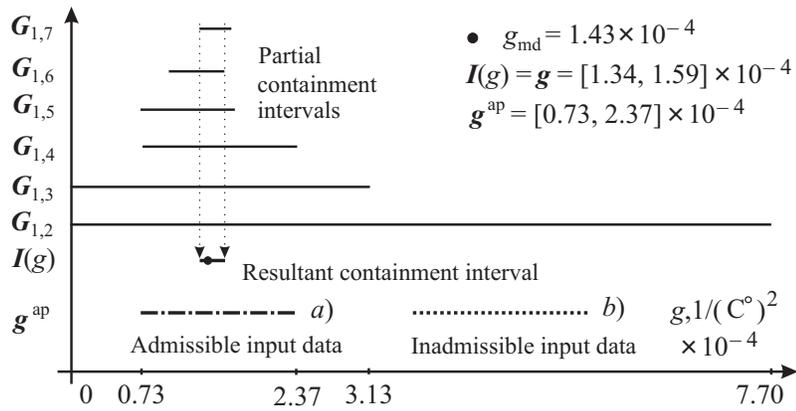


Figure 2: Partial containment intervals  $G_{1,n}$  and the resulting containment interval  $I(g)$ .

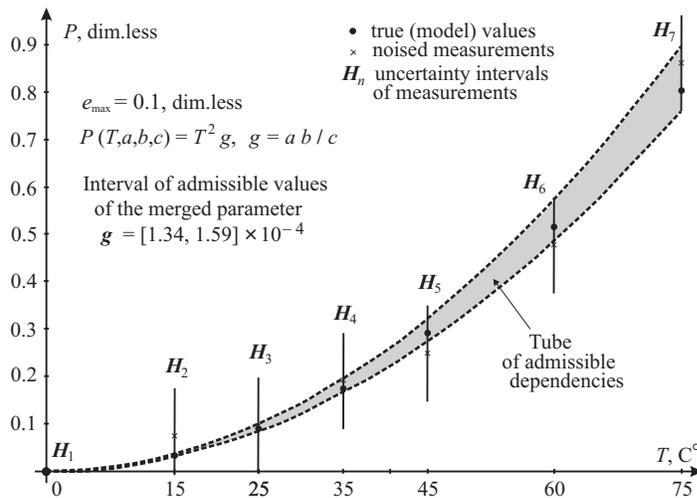


Figure 3: Solution of Problem 1 and tube (shaded) of admissible dependencies.

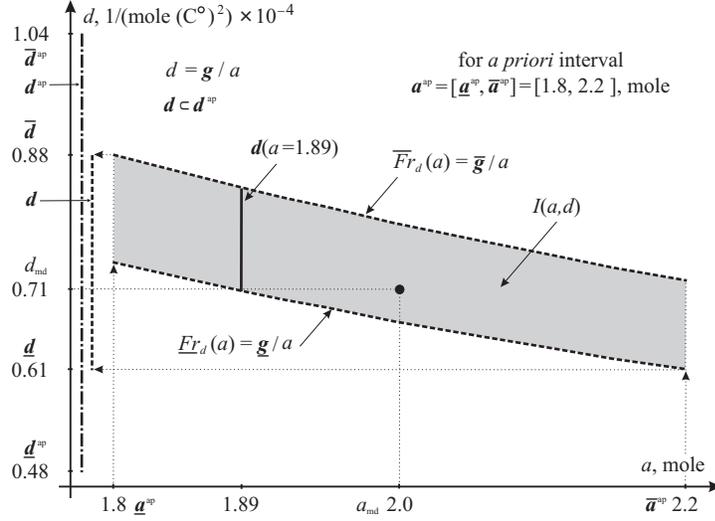


Figure 4: Solution of Problem 2; containment set  $I(a, d)$  (shaded) of parameters  $a$  and  $d = b/c$ .

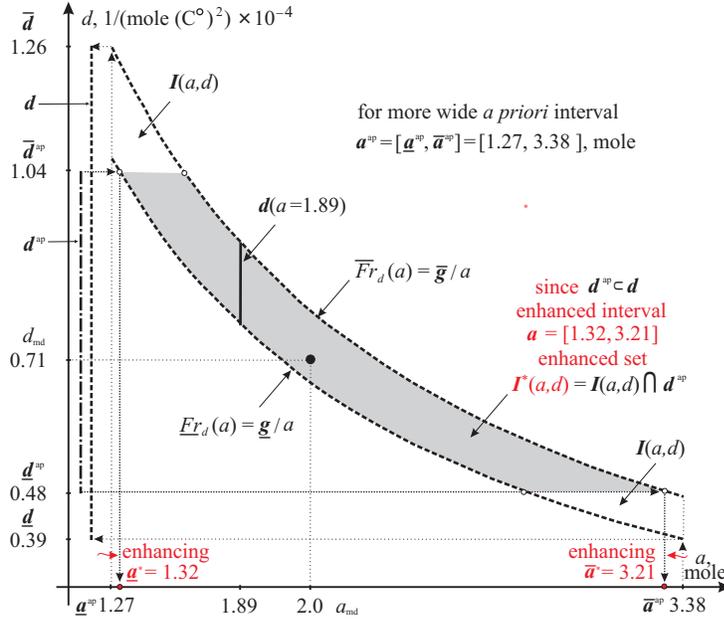


Figure 5: Solution of Problem 2; the case of enhancing (tightening) both the containment set  $I(a, d)$  to a narrower one  $I^*(a, d)$  (shaded) and the initial interval  $a^{ap}$  to a narrower one  $a^*$ .

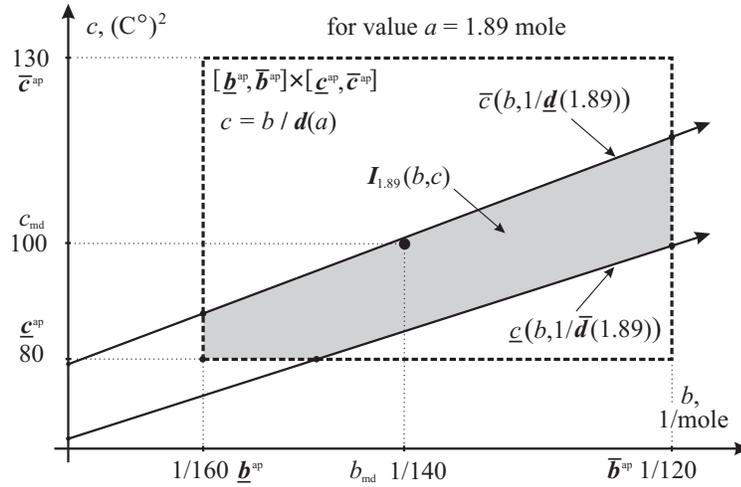


Figure 6: Solution of Problem 3; building the containment set cross-section (shaded) by intersection of the *a priori* rectangle  $\mathbf{b}^{ap} \times \mathbf{c}^{ap}$  with the cone of possible values of the parameters  $b$  and  $c$  for  $a = 1.89$  mole.

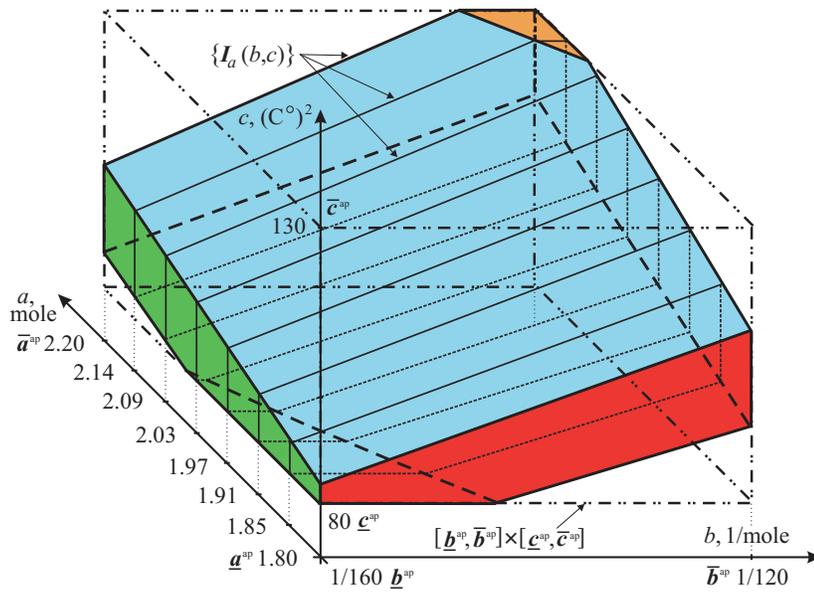


Figure 7: Containment set  $I(a, b, c)$  as a collection of its two-dimensional cross-sections  $\{I_a(b, c)\}$ .

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