

**AN OPTIMAL ION EXCHANGE DESIGN FOR REMOVAL OF CESIUM FROM  
HANFORD WASTE (U)**

by

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## Abstract

Non-elutable crystalline silicotitanate (CST) ion-exchanger materials have been studied for removing cesium from a variety of radioactive wastes at several U.S. DOE sites over the last decade. For the current pretreatment facility design of the River Protection Project (RPP) Waste Treatment Plant (WTP) in Hanford, the removal of cesium from low activity waste (LAW) is achieved by ion-exchange technology based on SuperLig<sup>®</sup> 644 resin. However, due to concerns over potential radiological and chemical degradation of SuperLig<sup>®</sup> 644 resin, IONSIV IE-911 (CST in its engineered form) material is being proposed as a backup ion-exchange material for the removal of cesium from Hanford radioactive waste.

This paper discusses the methodology used to determine the optimal CST ion-exchange column size to process all 16 separate batches of feeds from the ten targeted Hanford waste tanks. The optimal design ensures the best utilization of CST material and therefore results in a minimum amount of spent CST.

## BACKGROUND

A proposed facility is being designed for the immobilization of radioactive waste contained within Hanford underground storage tanks. The waste is pretreated to split it into Low Activity Waste (LAW) and High Level Waste (HLW) streams for separate vitrification. For the current pretreatment facility design of the River Protection Project (RPP) Waste Treatment Plant (WTP), the removal of cesium from low activity waste (LAW) is achieved by ion-exchange technology based on SuperLig<sup>®</sup> 644 resin. The proposed design of the facility consists of two sets of two packed columns placed in series (i.e., a lead column followed by a lag column configuration). During operation, upon reaching a specified cesium concentration criterion at the exit of the lag column, operation is switched to the second set of lead and lag columns. The cesium-loaded lead

column is processed (i.e., washed and eluted) and switched to the lag position. The previous lag column is then placed in the lead position (without eluting) and the system is ready for use in the next cycle.

Due to recent concerns over potentially high rates of radiological and chemical degradation of SuperLig<sup>®</sup> 644 resin and increased pressure drops observed during pilot-scale column studies, an increased interest in developing a potential backup ion-exchanger material has resulted. Crystalline silicotitanate (CST) ion-exchanger materials have been studied for cesium removal at Hanford and Savannah River Site (SRS) over the last six years. Since CST is an inorganic based material (with excellent properties in regard to chemical, radiological, and thermal stability and also little swelling/shrinking) that is considered to be practically non-elutable (while SuperLig<sup>®</sup> 644 is an organic based elutable resin), the overall pretreatment facility flowsheet would be impacted in various ways.

This paper analyzes the performance of proposed backup ion-exchange columns using the CST IONSIV IE-911 material for the removal of cesium from Hanford radioactive waste.

## Objectives

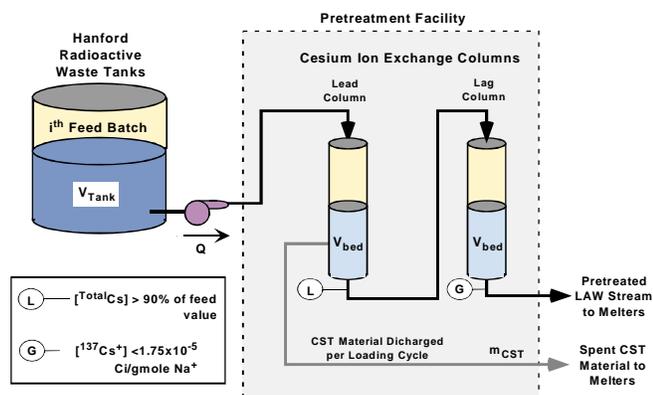
The main objectives of this analysis are:

- To predict the quantity of IONSIV<sup>®</sup> IE-911 CST needed to treat the candidate LAW batch feed solutions (16 feeds in total);
- To predict the number and dimensions of CST ion-exchange columns (i.e., bed geometry) required to reduce the <sup>137</sup>Cs concentrations to  $1.75 \times 10^{-5}$  Ci <sup>137</sup>Cs per gmole of Na after treatment of a selected candidate LAW solution.

## Basic Material Flowsheet

The non-elutable CST material is used only once and then becomes “spent CST” material that must be disposed of by vitrification in the glass melters. A simplified material

flowsheet of the proposed CST based facility is shown in Figure 1. As shown in Figure 1, a spent CST material stream leaves this facility and ultimately contributes to the composite feed entering the glass melters.



**Figure 1.** Simplified material flowsheet of the ion-exchange units used for removal of cesium from a LAW stream

### LAW Batch Feeds

The maximum loading for waste sodium oxide ( $\text{Na}_2\text{O}$ ) currently planned for LAW glass forms is waste-envelope (i.e., waste type) dependent. Therefore, the volumetric flowrate of the LAW stream passing through the ion-exchange facility during the loading phase is waste-envelope dependent. The current processing plan for the Phase 1 LAW inventory is to batch process the entire inventory. The Phase 1 campaign constitutes the processing of ten waste tanks that are broken up into 16 batch feeds (i.e., 11 are Envelope A feeds, 2 Envelope B feeds, and 3 Envelope C feeds). The source tank and volume of solution to be processed on a batch feed are listed in Table 1. The envelope dependent flowrates used in the design calculations are also listed in Table 1 for the 16 batch feeds. The basis for the flowrates centers on the production goals of glass and the allowable limits of waste sodium oxide loading within the glass matrix.

Constraints on the allowable levels of  $^{137}\text{Cs}$  contained within the aqueous effluent stream exiting the ion-exchange facility are also envelope dependent. Based on these  $^{137}\text{Cs}$  concentration limits total cesium exit criteria can be computed based on the isotopic content of cesium within each feed (i.e., it is assumed that the isotopic fraction of  $^{137}\text{Cs}$  to  $^{135}\text{Cs}$  is 25 mole% for Envelopes A and C and 30% for Envelope B). These total cesium exit criteria are also listed in Table 1.

**Table 1.** Key batch processing information on the Phase 1 Low activity waste (LAW) feeds listed in their scheduled order to be processed

Envelope	Source Tank	LAW batch feed (id)	Flowrate <sup>b</sup> (L/min)	Cesium feed conc. [M]	Batch volume to be processed <sup>ac</sup> (m <sup>3</sup> )	Total Cs exit criterion <sup>b</sup> [M]
A	AP-101	LAW-1	52.62	3.598E-05	4,626	2.953E-08
B	AZ-101	LAW-2a	9.4	4.676E-04	2,906	7.032E-08
B	AZ-102	LAW-2b	9.4	4.311E-04	1,755	7.032E-08
C	AN-102	LAW-3	16.2	3.967E-05	4,200	4.894E-08
C	AN-102	LAW-4	16.2	3.779E-05	4,200	4.894E-08
A	AN-104	LAW-5	52.62	6.283E-05	3,820	2.953E-08
A	AN-104	LAW-6	52.62	6.328E-05	3,540	2.953E-08
C	AN-107	LAW-7	16.2	4.455E-05	5,498	4.894E-08
A	AN-105	LAW-8	52.62	4.324E-05	3,700	2.953E-08
A	AN-105	LAW-9	52.62	4.444E-05	3,600	2.953E-08
A	SY-101	LAW-10	52.62	3.692E-05	2,600	2.953E-08
A	SY-101	LAW-11	52.62	3.739E-05	4,600	2.953E-08
A	AN-103	LAW-12	52.62	4.831E-05	4,720	2.953E-08
A	AN-103	LAW-13	52.62	4.831E-05	4,720	2.953E-08
A	AW-101	LAW-14	52.62	4.569E-05	3,940	2.953E-08
A	AW-101	LAW-15	52.62	4.552E-05	5,360	2.953E-08

<sup>a</sup> The volume of each batch feed represents the volume of solution entering the ion-exchange facility at a 5 M sodium basis and includes the volume changes that occur upstream to this facility (i.e., pretreatment activities).

<sup>b</sup> The volumetric flowrates and batch process times are based on the 30 MT/day operation schedule for Envelopes B and C and on the expanded capability of 60 MT/day operation for Envelope A.

<sup>c</sup> The total amount of processing time is ~4.2 years. If the Envelope A feeds are processed at 30 MT/day, then the total amount of processing time is ~5.8 years.

From the viewpoint of determining an optimal ion-exchange facility based on CST packed columns, a minimization of spent CST strategy has been chosen. In this strategy all 16 batch feeds are being addressed individually. Specifically, we address the following key varying attributes: (1) volume of solutions to be processed; (2) feed volumetric flowrates; (3) composition of key constituents; (4) cesium exit criteria; (5) the geometry of the columns; and (6) the number of columns used within a carousel configuration. The methodology used to perform the design (i.e., minimization) is discussed in the remainder of this paper.

## MODEL FORMULATIONS

### Ion Exchange Column Model

For the Cesium-IONSIV<sup>®</sup> IE-911 CST system a porous particle ion exchange column model was considered. In this model we assume that the kinetics associated with local ion exchange at an active resin site are very fast (faster than the various liquid mass transfer mechanisms that transport ions to that site). Assuming radial effects to be negligible within the active region of the packed bed (i.e., a large column-to-particle diameter ratio), a one-dimensional solute (ion) transport equation for the mobile phase (within the bed) becomes

$$\frac{\partial C}{\partial t} = E_b \frac{\partial^2 C}{\partial z^2} - u_o \frac{\partial C}{\partial z} - \frac{3k_f(1-\varepsilon_b)}{R_p \varepsilon_b} (C - C_{p,r=R_p})$$

With boundary and initial conditions

$$z = 0: \quad E_b \frac{\partial C}{\partial z} = u_o (C(t,0) - C_o)$$

$$z = L: \quad \frac{\partial C}{\partial z} = 0$$

$$t = 0: \quad C = C(0, z)$$

Assuming uniformly sized spherical particles with a homogeneous distribution of pores, a one-dimensional species transport equation for the pore phase (within an average sized particle of resin) becomes

$$\left( \varepsilon_p + (1-\varepsilon_p) \frac{\partial Q}{\partial C_p} \right) \frac{\partial C_p}{\partial t} = \varepsilon_p \frac{D_p}{r^2} \frac{\partial}{\partial r} \left( r^2 \frac{\partial C_p}{\partial r} \right)$$

$$r = 0: \quad \frac{\partial C_p}{\partial r} = 0$$

$$r = R_p: \quad \varepsilon_p D_p \frac{\partial C_p}{\partial r} = k_f (C - C_{p,r=R_p})$$

$$t = 0: \quad C_p = C_p(0, r)$$

### VERSE-LC Software

The VERSE-LC (VERsatile REaction SEparation simulator for LIquid CHromatography) code was chosen (Berninger et al., 1991) based on its availability and widespread (and accepted) use in this field. Prior to applying VERSE-LC to the ion exchange modeling the code was thoroughly verified by SRTC researchers. The verification process provided us quality assurance that the installed PC Window95<sup>™</sup> version of VERSE-LC was capable of adequately solving the above mentioned

equations and also helped us to better understand how to accurately use the VERSE-LC code (e.g., mesh refinement requirements and input/output options). For all column results presented in this report numerical errors associated with the results of VERSE-LC should be very small when compared to the uncertainties associated with various model input parameters (bed density, particle radius, pore diffusion, etc.).

### Equilibrium Cesium Isotherms

To perform column transport simulations, an “effective” binary isotherm model in an algebraic form must be available to represent the Q-C<sub>p</sub> relationship. The VERSE-LC Freundlich/Langmuir Hybrid isotherm model was chosen in which the surface loading for cesium on the CST material can be expressed as:

$$Q = \frac{\eta_{df} C_T C_p}{C_p + \beta}$$

where the beta parameter for cesium becomes dependent upon the other ionic competitors for CST adsorption (i.e., K<sup>+</sup>, Na<sup>+</sup>, SrOH<sup>+</sup>, and Rb<sup>+</sup>). The beta parameter contains the selectivity coefficients making it dependent upon temperature and liquid composition of all of the ionic species in solution. The larger the beta parameter the lower loading an isotherm will have. The dilution factor ( $\eta_{df}$ ) is unity when considering a specific powder form and is less than one upon addition of an inert binder (engineered form). The best estimate dilution factor for the engineered form is set to 0.68. The total cesium capacity term, C<sub>T</sub>, is set to 0.58 mmole<sub>Cs</sub>/g<sub>CST</sub>. (Zheng et al., 1996). With the dilution factor and the total cesium capacity set, the beta parameter is determined through nonlinear regression using a database generated for each batch feed by the ZAM model. The ZAM code is a product of over several years development and research in Professor R. G. Anthony's Kinetics, Catalysis and Reaction Engineering Laboratory in the Department of Chemical Engineering Texas A&M University. The ZAM model solves a set of equations for solid-liquid equilibrium. This model includes the competitive ion exchange at CST exchange sites between the following homovalent cations: Na<sup>+</sup>, Cs<sup>+</sup>, H<sup>+</sup>, Rb<sup>+</sup>, K<sup>+</sup>, and SrOH<sup>+</sup>. Non-idealities within the aqueous phase are handled using Bromley's model for calculating activity coefficients of the ions. A description of the current ZAM model is provided by Zheng et al. (1997)

### Axial Dispersion

Axial dispersion in packed columns is the result of mechanical dispersion added onto molecular diffusion. For practical flowrates mechanical dispersion dominates. In the

low Reynolds number range of interest the Chung and Wen (1968) correlation is applicable for sufficiently large columns (i.e., large diameter and length) and is expressed as:

$$E_b = \frac{2R_p u \varepsilon_b}{0.2 + 0.011 \text{Re}^{0.48}}$$

Where the standard deviation of this correlation based on all available data points was reported to be 46%. Equation (8-2) applies for only sufficiently large columns and correction factors must be considered for columns with small diameters and/or short active bed lengths.

## Film Diffusion

For the laboratory-scale column tests and proposed full-scale facility, with the IONSIV<sup>®</sup> IE-911 CST particle size distributions, the Reynolds number range is approximately 0.1 to 1.0. With respect to published literature this is a very low Reynolds number range. Since VERSE-LC has the Wilson and Geankoplis (1966) correlation as an option and this correlation falls somewhat within the spread of available low Reynolds number data we have chosen it for all the column simulations. For each ion species considered, the Wilson and Geankoplis correlation is expressed as:

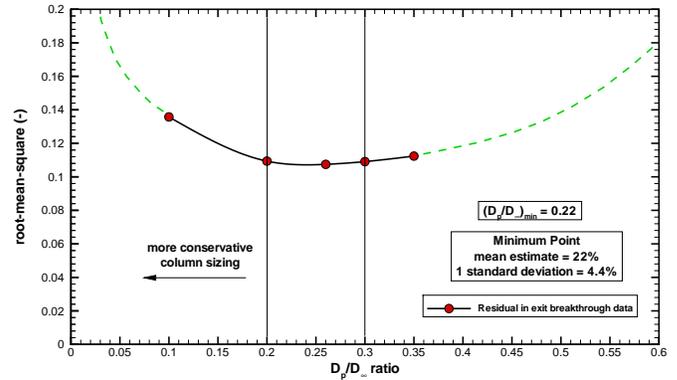
$$J \equiv \left[ \frac{k_{fi}}{u \varepsilon_b} \right] S_{C_i}^{2/3} = \frac{1.09}{\varepsilon_b} \text{Re}^{-2/3}$$

A standard deviation of approximately 25% is reported for this correlation by Wilson and Geankoplis (1966).

## CST Pore Diffusion

CST packed columns are mass transfer limited giving rise to relatively slow “kinetics”. Therefore, in predicting cesium breakthrough behavior for CST packed columns the pore diffusion coefficient should be an important parameter. The “effective” cesium pore diffusion coefficient can be normalized with respect to the free stream “Brownian” diffusion coefficient. The inverse of this ratio is referred to as a tortuosity factor that is primarily dependent upon the internal structure of the porous material. For materials with very large pores a tortuosity factor approaching unity is observed, while for very small pores factors approaching zero can result. The rate of “kinetics” is inversely related to the tortuosity factor. The “best estimate” value of this ratio ( $D_p/D_\infty$ ) was determined by utilizing the breakthrough data for several representative column performance tests. For each test five VERSE-LC simulations were run varying this ratio (i.e., 10%, 20%, 26%, 30% and 35%). For each ratio value an overall residual in calculated breakthrough versus

measured in the form of a root-mean-square was computed. The minimum of this residual curve provided us with a “best estimate” value of this ratio.



**Figure 2.** Overall measure of error in predicting column exit cesium breakthrough as a function of the pore to free stream diffusion coefficient ratio

The composite results of the simulations are summarized in Figure 2. In Figure 2 the solid circles represent the computed composite rms (root-mean-square) values, the solid curve is a spline-fit through the points, and the dashed curves represent estimated behavior outside the tested range. Observable predictive improvement is obtained between ratio values of 20% and 30% as shown in Figure 2. Specific case studies show that with all other parameters being fixed the lower the pore diffusion coefficient the earlier the breakthrough. From a design perspective, lower pore diffusion coefficient values provide more conservative design estimates.

Based on this, we used the lower estimated limit of the rms minimum (i.e., ratio value of 20%). It is believed that a value lower than ~20% would significantly impact column size due to its impact on the leading edge of the concentration wave and a clear systematic (on average) departure from the breakthrough data is seen for values lower than this.

## FULL-SCALE COLUMN PREDICTION AND DESIGN

For column sizing purposes a global optimization strategy is employed where the total amount of spent CST material required to process the entire Phase 1 LAW inventory is minimized. Both 2-column and 3-column carousel configurations are considered, along with a range of possible L/D geometries. All simulations were performed at an operating temperature of 25°C where best estimate feed

conditions for the 16 Phase 1 batch feeds in their scheduled order of processing were applied

The simulations are based on a VERSE-LC model of the lead and lag columns (and guard column for the 3-column configuration) where the parameter settings are consistent with the values used during the assessment of the available laboratory-scale column experiments.

## The Global Optimization Strategy

A global optimization strategy based on simulating the entire processing of the Phase 1 inventory in its schedule sequence was undertaken. The assumption is made that once a batch feed has been completely processed, the next batch feed scheduled will begin without starting with fresh columns (i.e., the batch feeds are stacked inline where the carousel cycling only occurs when the exit lag column criterion is reached). The optimization parameter to be tracked is the amount of spent CST material used to process each batch feed. Since the original basic concern was over the amount of CST ultimately having to be placed in the glass product, this appeared to be the obvious choice for our optimization parameter.

Based on this concept, the total amount of spent CST material required to process the entire Phase 1 LAW inventory can be computed by:

$$m_{CST} = \rho_{bed} V_{bed} (n_{carousel} + n_{cycles})$$

As discussed below, variation in the bed L/D geometry, while holding the bed volume of a column constant, has very little impact on spent CST material amount. Therefore, the spent CST value is a discrete variable that depends primarily on the bed volume of a column. In this way, we are addressing approximately a single-variable optimization problem.

## Spent CST Material Dependency on Geometry

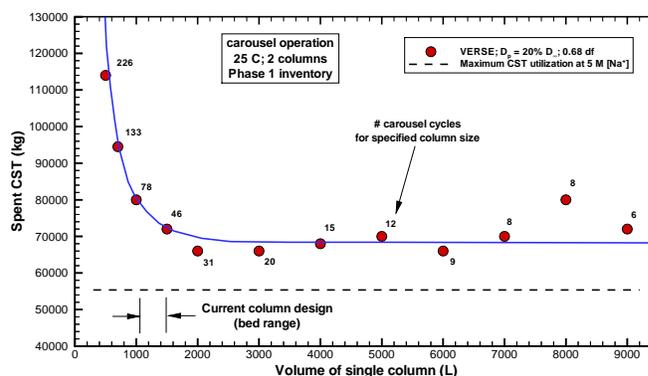
Within a given carousel design, each column is geometrically the same (i.e., the same volume and shape). The two key geometric parameters that define a unique column design are its bed volume and length-to-diameter ratio (L/D).

For CST IE-911 material we find that the overall mass transfer resistance is dominated by pore diffusion, while the resistance due to film diffusion is relatively negligible. Given this information, we would expect that the estimated amount of spent CST required to process the entire Phase 1 LAW inventory would be insensitive to geometric variations

such as L/D. To confirm this expected insensitivity to L/D, VERSE-LC simulations were run for L/D values of 1, 2, 3, 4 and 5. The entire Phase 1 LAW inventory was processed in these simulations. The results obtained showed that the computed spent CST is very insensitive to L/D.

## Analysis Results

Under nominal parameter settings, numerous VERSE-LC simulations were performed where the bed volume of a column was varied from 500 L up to 9,000 L. Both 2-column and some limited 3-column carousel were considered. These nominal case runs were performed to study the loading behavior of a CST packed column carousel and to estimate a near optimal design size. The spent CST results are plotted in Figure 3. The oscillation seen in the data is due to the discrete nature of the computed spent CST required. The solid curve shown in Figure 3 is provided only to highlight the general behavior of the data.

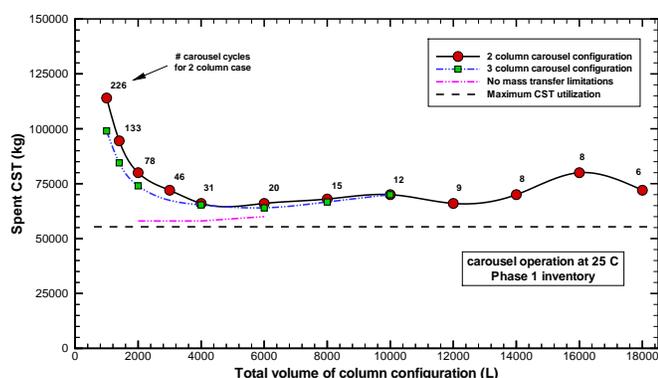


**Figure 3.** Computed total spent CST material required to process the entire Phase 1 LAW inventory based on a two-column carousel configuration at 25°C

As shown in Figure 3, the shape of the spent CST curve is rather flat over a large range of column volumes, becoming steep only at small column sizes. The optimal column size over the flat portion of this curve can be based on capital and operational costs for the facility and its carouseling needs. A near optimal column design is achieved at 2000 L where ~66 MT of spent CST is generated. During Phase 1 the HLW glass melters will generate an estimated 2651 MT of glass product. 66 MT of spent CST adds approximately 2.5 wt% sodium oxide to the HLW glass melter product. Based on the current column design of a bed volume of 1000 L, the estimated spent CST material increases by ~20% to 80 MT. If the bed volume of CST was to be increased in the current design to ~1500 L (i.e., the upper limit of bed volumes in the current design),

the estimated spent CST material increases by ~10% to 72 MT.

Three-column carousel configurations were considered to determine the potential benefit associated with more column stages. In the 3-column carousel runs the total bed volumes are equal to total bed volumes used in the 2-column carousel runs. The spent CST results of these 3-column carousel configurations are plotted in Figure 4, along with the results of the 2-column carousel runs. To put the 2-column and 3-column cases on a common basis for plotting, the computed spent CST is plotted in Figure 4 as a function of total carousel bed volume. Figure 4 shows that only marginal gains can be achieved when a 3-column versus 2-column carousel facility is considered.



**Figure 4.** Computed total spent CST material required to process the entire Phase 1 LAW inventory at 25°C

## CONCLUSIONS

Using VERSE-LC software and an optimization strategy for spent CST material, to process the entire Phase 1 LAW inventory of the Hanford waste, the optimal size of a CST ion exchange column in the 2-column carousel design for cesium removal is determined to be approximately 2m<sup>3</sup>. This corresponds to the smallest column size where only a negligible increase in spent CST results. For column sizes smaller than ~2m<sup>3</sup> a rapid increase in spent CST is observed.

## NOMENCLATURE

C	Mobile phase solute concentration
C <sub>p</sub>	Pore phase solute concentration
C <sub>T</sub>	Total cesium capacity of the CST material
D	Column diameter
D <sub>p</sub>	Intraparticle diffusivity
D <sub>∞</sub>	Brownian diffusivity
E <sub>b</sub>	Axial dispersion coefficient
k <sub>f</sub>	Film mass transfer coefficient
L	Column length

m <sub>CST</sub>	Mass of spent CST
n <sub>carousel</sub>	Number of columns within a carousel
n <sub>cycles</sub>	Total number of carousel cycles required
Q	Solid-phase solute concentration
r	Radial position from center of adsorbent particle
R <sub>p</sub>	Adsorbent particle radius
Re	Reynolds number
Sc	Schmidt number
t	Time
u	Linear interstitial velocity
V <sub>bed</sub>	Bed volume
z	Position in axial direction
β	Freundlich/Langmuir parameter
ε <sub>b</sub>	Bed porosity
ε <sub>p</sub>	Intraparticle porosity
ρ <sub>bed</sub>	Bed density
η <sub>df</sub>	Dilution factor

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