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EFFECT OF PULSED-COLUMN-INVENTORY UNCERTAINTY ON DYNAMIC MATERIALS ACCOUNTING

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ABSTRACT

Reprocessing plants worldwide use the Purex solvent-extraction process and pulsed-column contactors to separate and purify uranium and plutonium from spent nuclear fuels. The importance of contactor in-process inventory to dynamic materials accounting in reprocessing plants is illustrated using the Allied-General Nuclear Services Plutonium Purification Process (PPP) of the now decommissioned Barnwell Nuclear Fuels Plant. This study shows that (1) good estimates of column inventory are essential for detecting short-term losses of in-process materials, but that (2) input-output (transfer) measurement correlations limit the accounting sensitivity for longer accounting periods ($\geq$1 wk for the PPP).

I. INTRODUCTION

Reprocessing plants worldwide use the Purex solvent-extraction process as the standard method for separating and purifying uranium and plutonium from spent nuclear fuels. In practice, this separation and purification from fission products and other impurities is achieved using a series of solvent-extraction contactors (for example, pulsed columns, mixer-settlers, and centrifugal contactors) in which uranium and plutonium are selectively extracted into relatively immiscible countercurrent aqueous and organic streams. Unlike precipitation and ion exchange, which require increased consumption of heat and/or chemicals, the comparatively simple recycle of reagents in Purex solvent extraction makes it a near-ideal process for continuous, multistage, remote operations where high radiation levels are present.

The importance of contactor in-process inventory to dynamic materials accounting systems for reprocessing plants (that is, accounting systems designed to provide near-real-time assessment of the locations and amounts of nuclear materials) has been highlighted in earlier safeguards systems studies.[1-4] The objective of this investigation was to show, for a reference Purex process, that over longer accounting periods, contactor-inventory uncertainty has a relatively small effect as throughput-measurement errors accumulate. The reference process chosen for illustration was the steady-state operation of the continuous portion of the Allied-General Nuclear Services (AGNS) Plutonium Purification Process (PPP), which comprises pulsed-column contactors and other equipment that were constructed and cold-tested as part of the now decommissioned Barnwell Nuclear Fuels Plant. The reference PPP is of special importance to dynamic materials accounting because it was designed to process plutonium nitrate in a relatively pure and concentrated form attractive for diversion.

II. PROCESS DESCRIPTION

The reference PPP is based on the Purex flowsheet of the AGNS Barnwell Nuclear Fuels Plant. This reprocessing plant was designed to receive and process irradiated (spent) power-reactor fuel originally containing UO_2, or UO_2 and PuO_2. Fuel batches having an average burnup not exceeding 40,000 MWd/MTHM (megawatt days per metric ton of heavy metal) were to be processed at rates up to 5 MTHM/day (1,500 MTHM/yr) after a decay period of at least 160 days. As an integral part of the Barnwell facility, the PPP was designed to recover ∿50 kg of plutonium per day (∿2.089 kg Pu/h) from spent nuclear fuel containg ∿1wt % plutonium.

Figure 1 is a block diagram of the PPP. After the first cycle uranium-plutonium codecontamination (removal of fission products and other impurities) and partitioning, the continuous 1BP stream (an aqueous solution of ∿5 g Pu/L as plutonium(III) nitrate and ∿10g U/L as uranyl nitrate) from the 1B electropulse (partitioning) column enters the 1BP surge tank. There the plutonium is reoxidized to the extractable tetravalent state using dinitrogen tetroxide and fed continuously to two serial purification cycles in which aqueous and tributylphosphate

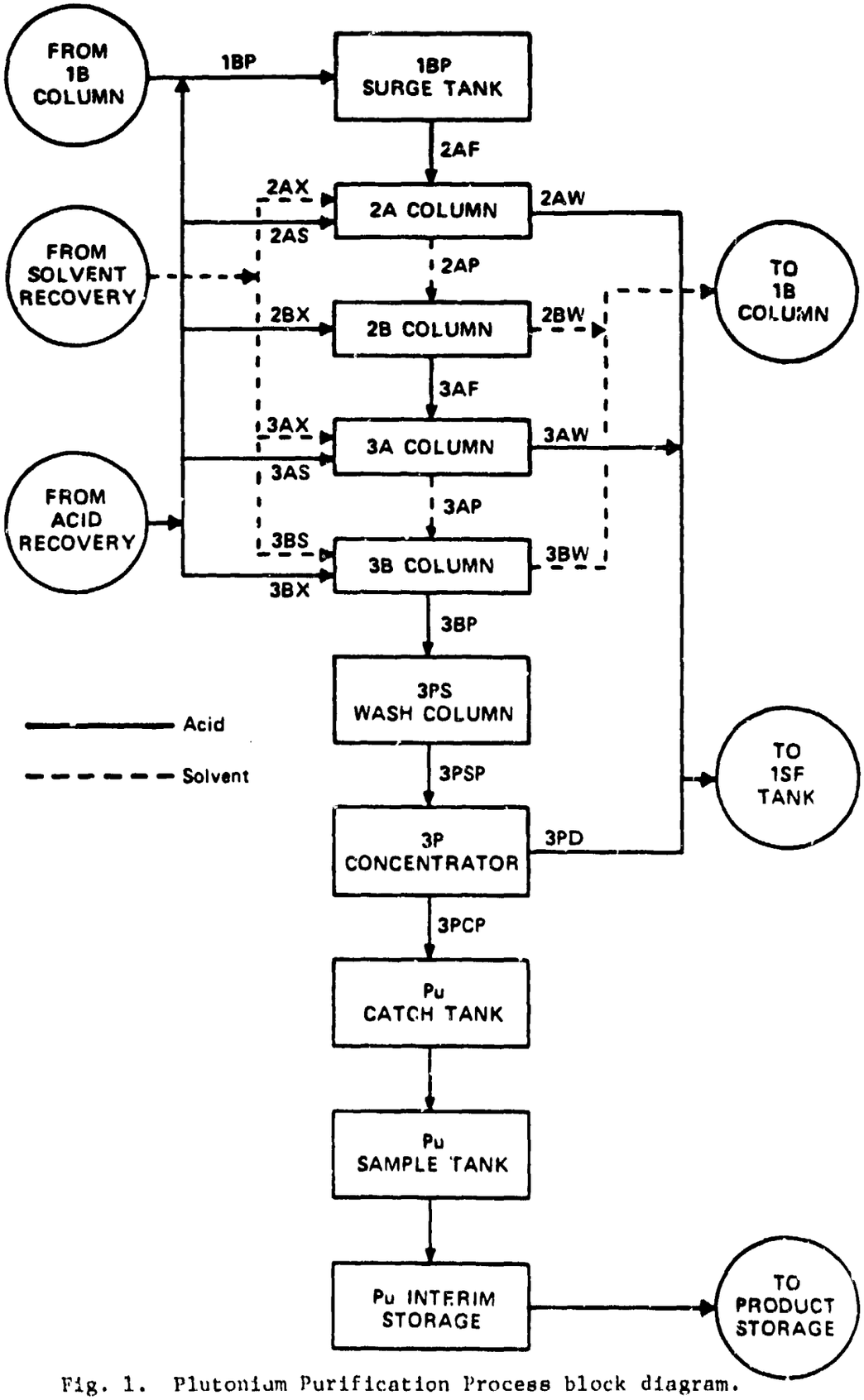

Fig. 1. Plutonium Purification Process block diagram.

(TBP)-kerosene streams are contacted countercurrently in the 2A, 2B and 3A, 3B pulsed columns to effect further decontamination and a higher plutonium concentration (to ∿60g Pu/L). The aqueous and organic waste streams (raffinates) from these four pulsed columns contain virtually all the residual uranium, fission products, and other impurities. The product stream (3BP) from the 3B pulsed column is passed through the 3PS packed diluent-wash column to remove traces of TBP and then concentrated from ∿60 to ∿250 g Pu/L in the 3P concentrator. The concentrator product (3PCP) is collected continuously in the plutonium catch tank, which has an 8-h surge capacity, and, at this point, the PPP is converted to a batch process. After the catch tank is filled, the contents are transferred rapidly to the plutonium sample tank, which has a 24-h surge capacity. The contents of the sample tank, in turn, are transferred to one of three interim product storage tanks, each with a surge capacity of ∿48h, to await transfer to the plutonium product storage and loadout area.

Nominal in-process inventory values and measurement (instrument) precisions for the seven units of equipment in the continuous (reference) portion of the PPP (that portion preceding the catch tank) are given in Table I, along with some mathematical notation used to develop the materials-balance and variance equations in the next section.

TABLE I

REFERENCE PPP IN-PROCESS INVENTORY AND INSTRUMENT PRECISIONS

Identification "j"	Pu Inventory (kg) "I_j"	Instrument Precision (%) "$\sigma_{\varepsilon_{I_j}}$"
1BP Tank	7.413	4.2
2A Column	4.595	2,5,10, or 20
2B Column	2.804	2,5,10, or 20
3A Column	5.422	2,5,10, or 20
3B Column	4.800	2,5,10, or 20
3PS Wash Column	1.174	2,5,10, or 20
3P Concentrator	15.000	1.5

III. DERIVATION OF THE NET-INVENTORY-CHANGE AND NET-TRANSFER VARIANCES

For the reference PPP throughput of ∿50 kg of plutonium per day, it is assumed here that materials balances would be closed every 8h during normal, continuous operation. The dynamic materials balance, MB(N), for a given accounting period containing N materials balances (8h between each) is then given by

$$MB(N) = \sum_{j=1}^{J} [I_j(0) - I_j(N)] + \sum_{\ell=1}^{L} \sum_{n=1}^{N} \sum_{k=1}^{K} T_{k,n,\ell} \qquad (1)$$

$$= \Delta I(N) + T(N),$$

where

J = number of inventory components or pieces of equipment in the reference PPP ($J = 7$ from Table I),

$I_j(0)$ = initial plutonium inventory for component j,

$I_j(N)$ = final (Nth) plutonium inventory for component j,

K = number of hourly transfer measurements at each location ℓ for each materials balance n ($K = 8$),

N = number of materials balances during the accounting period,

L = number of transfer-measurement locations ($L=2$),

$T_{k,n,\ell}$ = kth transfer measurement (hourly) during materials balance n at location ℓ (either the input location or the output location for the reference PPP),

$\Delta I(N)$ = net change in plutonium inventory during the accounting period for the J inventory components, and

$T(N)$ = net transfer of plutonium (inputs minus outputs) across the reference PPP during the accounting period.

If there were no measurement errors, $MB(N)$ would be exactly zero for the steady-state operation of the reference PPP. However, measurement errors exist for the PPP (and other real processes), and they produce an uncertainty in $MB(N)$ having a variance, $\sigma^2_{MB(N)}$, given by

$$\sigma^2_{MB(N)} = \sigma^2_{\Delta I(N)} + \sigma^2_{T(N)} \quad , \qquad (2)$$

assuming no correlation between transfer and inventory measurements.

The derivations that follow for the net-inventory-change and net-transfer variances, $\sigma^2_{\Delta I(N)}$ and $\sigma^2_{T(N)}$, respectively, are basic to the PPP illustration. A multiplicative measurement-error model is used in these derivations, because the measurement errors (standard deviations) associated with the inventory and transfer measurements tend to be proportional to the quantity being measured, and are expressed on a relative basis. The measurement errors are grouped in two categories, instrument precision (ε) and calibration error (η), and are assumed to be uncorrelated, mean-zero random variables having variances σ^2_ε and σ^2_η, respectively. However, whereas the ε error changes each time a

measurement is made, the η error remains the same until the instrument is recalibrated. The η errors (frequently referred to as systematic errors) are the most difficult to estimate because they include uncertainties in standards, calibration parameters, and instrument environment. Moreover, all measurements from the same instrument and having the same η error are correlated.

For the derivation of the net-inventory-change variance, $\sigma^2_{\Delta I(N)}$, recall from Eq. (1) that

$$\Delta I(N) = \sum_{j=1}^{J} \Delta I_j(N), \tag{3}$$

where $$\Delta I_j(N) = I_j(0) - I_j(N). \tag{4}$$

Now, applying the multiplicative measurement-error model[5], and assuming that the initial and final plutonium inventories, $I_j(0)$ and $I_j(N)$, for each inventory component j are measured during the same calibration period (that is, they have the same η error), then

$$I_j(0) = I^*_j(0)\ (1 + \varepsilon_{Ij} + \eta_{Ij}) \tag{5}$$

and $$I_j(N) = I^*_j(N)(1 + \varepsilon_{Ij} + \eta_{Ij}), \tag{6}$$

where $I_j(0)$ and $I^*_j(0)$ are, respectively, the measured and actual jth inventory components at the start of the accounting period, and $I_j(N)$ and $I^*_j(N)$ are, respectively, the measured and actual jth inventory components at the end of the accounting period.

Hence, from Eqs. (4)-(6),

$$\Delta I_j(N) = I^*_j(0)\ (1 + \varepsilon_{I_j} + \eta_{I_j}) - I^*_j(N)\ (1 + \varepsilon_{I_j} + \eta_{I_j}) \tag{7}$$

and from Eqs. (4), (7), and random variable theory,[6]

$$\sigma^2_{\Delta I_j(N)} = \sigma^2_{I_j(0)} + \sigma^2_{I_j(N)} - 2\,\mathrm{Cov}\,[I_j(0)I_j(N)]$$
$$= I^*_j(0)^2(\sigma^2_{\varepsilon_{I_j}} + \sigma^2_{\eta_{I_j}}) + I^*_j(N)^2(\sigma^2_{\varepsilon_{I_j}} + \sigma^2_{\eta_{I_j}}) - 2I^*_j(0)I^*_j(N)\sigma^2_{\eta}. \tag{8}$$

Note, for example, that the variance of the product of a constant C and a random variable η is given by

$$\sigma^2_{C\eta} = C^2\sigma^2_{\eta}\ .$$

Now, substituting the measured j inventory values, $I_j(0)$ and $I_j(N)$, for the actual (but unknown) j inventory values, $I_j^*(0)$ and $I_j^*(N)$, the inventory-change variance, $\sigma^2_{\Delta I_j(N)}$, for each inventory component j, can be estimated by

$$\sigma^2_{\Delta I_j(N)} = [I_j(0)^2 + I_j(N)^2](\sigma^2_{\varepsilon_{I_j}} + \sigma^2_{\eta_{I_j}}) - 2I_j(0)I_j(N)\sigma^2_{\eta} \quad . \qquad (9)$$

Therefore, the net-inventory-change variance, $\sigma^2_{\Delta I(N)}$, is given by

$$\sigma^2_{\Delta I(N)} = \sum_{j=1}^{J} \left\{ [I_j(0)^2 + I_j(N)^2]\sigma^2_{\varepsilon_{I_j}} + [I_j(0) - I_j(N)]^2\sigma^2_{\eta_{I_j}} \right\}, \qquad (10)$$

where $\sigma^2_{\varepsilon_{I_j}}$ and $\sigma^2_{\eta_{I_j}}$ are the ε- and η- error variances, respectively,

of the individual inventory measurements. Moreover, with the assumption that the PPP is at steady state, the initial and final plutonium inventories for each inventory component are equal, $I_j(0) = I_j(N)$, so that $\sigma^2_{\Delta I(N)}$ has the minimum value

$$\sigma^2_{\Delta I(N)} = 2\sum_{j=1}^{J} I_j(0)^2\sigma^2_{\varepsilon_{I_j}} = 2I(0)^2\sigma^2_{\varepsilon_I} \quad . \qquad (11)$$

Looking at Table I, the total in-process inventory, I(0), in the continuous portion of the PPP is ∿41.21 kg of plutonium distributed among seven inventory components, or units of equipment. Thus, the total measured inventory is the sum of the measured inventory in each piece of equipment, seven independent measurements in all. Table II lists values of the net-inventory-change variance, $\sigma^2_{\Delta I(N)}$, corresponding to column-inventory measurement precisions, $\sigma_{\varepsilon_{I_j}}$, of 2,5,10, and 20%

for each of the five columns in the PPP. Precisions for the remaining equipment in the continuous portion of the PPP are listed in Table I.

TABLE II

REFERENCE PPP NET-INVENTORY-CHANGE VARIANCES VS COLUMN-INVENTORY MEASUREMENT PRECISIONS

Column $\sigma_{\varepsilon_{I_j}}$ (%)	$\sigma^2_{\Delta I(N)}$ (kg^2 Pu)
2	0.365
5	0.713
10	1.955
20	6.922

For the reference PPP and most other continuous processes, efficiency and economy dictate that the in-process inventory be held nearly constant during normal operation. Such near-steady-state operation benefits materials accounting by reducing the contribution of inventory measurement errors to the materials-balance uncertainty. Furthermore, the condition $I_j(0) \cong I_j(N)$ implies that the dependence of $\sigma^2_{\Delta I(N)}$ and, hence, of $\sigma^2_{MB(N)}$ on $\sigma^2_{\eta I_j}$ is weak [Eq. (10)]; therefore, a well-known value for $\sigma^2_{\eta I_j}$ is not required. This result is important because standardization of in-process inventory measurements may be difficult, especially for process equipment located in high radiation fields behind heavy shielding.

Now to derive the net-transfer variance, $\sigma^2_{T(N)}$, recall from Eq. (1) that

$$T(N) = \sum_{\ell=1}^{L} \sum_{n=1}^{N} \sum_{k=1}^{K} T_{k,n,\ell} \quad . \tag{12}$$

Also, for simplicity, assume that the input and output transfer measurements, T, and their ε- and η- error variances are equal, but that the two measurement methods are independent, or uncorrelated. Furthermore, assume that the transfer measurement methods are not recalibrated during a PPP accounting period, so that pair-wise correlations exist among the input measurements as well as the output measurements. With these conditions, the variance of the sum of transfer measurements at a single location (input or output of the reference PPP) is derived first, followed by the total (input and output) net-transfer variance.

In general, for the correlated (η) transfer-measurement errors, there are R recalibration intervals through the Nth materials balance, with N_R transfer measurements taking place in each interval. Thus, the total number of transfer measurements, NK, at the end of the Nth materials balance is given by

$$NK = RN_R \quad , \tag{13}$$

where

N = Nth materials balance, and

K = number of transfer measurements per materials balance.

Hence, for no recalibrations during the PPP accounting period, R = 1 and N_R = NK. Now, defining the sum of tranfer measurements at a single location as

$$\Sigma\Sigma T = \sum_{n=1}^{N} \sum_{k=1}^{K} T_{k,n} \quad , \tag{14}$$

and applying the multiplicative measurement-error model to define

$$T_{k,n} = T^*_{k,n} \left(1 + \varepsilon_{T_{k,n}} + \eta_{T_{k,n}}\right) \quad , \tag{15}$$

where $T_{k,n}$ and $T^*_{k,n}$ are, respectively, the measured and actual values of the kth transfer measurement during the nth materials balance, then it follows from Eqs. (13)-(15) that

$$\begin{aligned} \Sigma\Sigma T &= NKT^* + T^* \sum_{n=1}^{N} \sum_{k=1}^{K} \varepsilon_{T_{k,n}} + N_R T^* \sum_{r=1}^{R} \eta_{T_r} \\ &= NKT^* + T^* \sum_{n=1}^{N} \sum_{k=1}^{K} \varepsilon_{T_{k,n}} + NKT^* \eta_T \quad . \end{aligned} \tag{16}$$

Also, recalling that the ε- and η-error variances are uncorrelated (i.e., ε and η are independent random variables), then the variance of $\Sigma\Sigma T$, $\sigma^2_{\Sigma\Sigma T}$, is just the sum of the variances of each term in Eq. (16), where the variance of NKT* (a constant) is equal to zero. Therefore, substituting the measured transfer value, T, for the actual (but unknown) transfer value, T*, the variance of the sum of transfers at a single location can be estimated by

$$\begin{aligned} \sigma^2_{\Sigma\Sigma T} &= NK \text{ Var } (T\varepsilon_T) + \text{Var } (NKT\eta_T) \\ &= NKT^2\sigma^2_{\varepsilon_T} + N^2K^2T^2\sigma^2_{\eta_T} \quad . \end{aligned} \tag{17}$$

The net-transfer variance, $\sigma^2_{T(N)}$, can be derived easily now from Eqs. (1), (2), (14), and (17),

where

$$\sigma^2_{T(N)} = \sum_{\ell=1}^{L} \sigma^2_{(\Sigma\Sigma T)_\ell} \quad . \tag{18}$$

Now, because the two locations (input and output) are uncorrelated and all transfer measurements, T, as well as ε- and η- error variances are equal, then

$$\begin{aligned} \sigma^2_{T(N)} &= 2\sigma^2_{\Sigma\Sigma T} \\ &= 2NKT^2\sigma^2_{\varepsilon_T} + 2N^2K^2T^2\sigma^2_{\eta_T} \quad . \end{aligned} \tag{19}$$

Furthermore, letting

$$\sigma^2_\varepsilon = 2KT^2\sigma^2_{\varepsilon_T} \tag{20}$$

and

$$\sigma^2_\eta = 2K^2T^2\sigma^2_{\eta_T} \quad , \tag{21}$$

then

$$\sigma^2_{T(N)} = N\sigma^2_\varepsilon + N^2\sigma^2_\eta \quad . \tag{22}$$

Here, σ_{ε}^2 and σ_{η}^2 are the ε- and η- error variances of the input and output transfer measurements.

Recall that the throughput of the PPP is ∿2.089 kg of plutonium per hour for a total of ∿16.7 kg during each 8-h materials-balance period. Also, for each input and output measurement, the ε-error relative standard deviation, σ_{ε_T}, is assumed to be 1.414% and the η-error, σ_{η_T}, relative standard deviation is assumed to be 0.583%. Therefore, with PPP input-output measurements made every hour, after 8h (that is, for K = 8)

$$\sigma_{\varepsilon}^2 = (2)\ (8)\ (2.089)^2\ (0.01414)^2 = 0.014\ \text{kg}^2\ \text{Pu} \tag{23}$$

and

$$\sigma_{\eta}^2 = (2)\ (8)^2\ (2.089)^2\ (0.00583)^2 = 0.019\ \text{kg}^2\ \text{Pu} \quad . \tag{24}$$

Then from Eqs. (23) and (24), the net transfer variance for the reference PPP is given by

$$\sigma_{T(N)}^2 = 0.014\ N + 0.019\ N^2 \quad . \tag{25}$$

IV. EFFECT OF PULSED-COLUMN-INVENTORY UNCERTAINTY ON DYNAMIC MATERIALS ACCOUNTING

Looking first at Eqs. (2), (11), and (22), respectively, the variance for the Nth materials balance is given by Eq. (2)

$$\sigma_{MB(N)}^2 = \sigma_{\Delta I(N)}^2 + \sigma_{T(N)}^2 \quad ,$$

the net-inventory-change variance is given by Eq. (11)

$$\sigma_{\Delta I(N)}^2 = 2I(0)^2\sigma_{\varepsilon_I}^2 \quad ,$$

and the net-transfer variance is given by Eq. (22)

$$\sigma_{T(N)}^2 = N\sigma_{\varepsilon}^2 + N^2\sigma_{\eta}^2 \quad .$$

Now, substituting Eqs. (11) and (22) into Eq. (2) yields

$$\sigma_{MB(N)}^2 = 2I(0)^2\sigma_{\varepsilon_I}^2 + N\sigma_{\varepsilon}^2 + N^2\sigma_{\eta}^2 \quad . \tag{26}$$

Table III shows the effect of uncertainties in column-inventory estimates on dynamic (near-real-time) materials accounting in the continuous portion of the PPP. The standard deviations, $\sigma_{MB(N)}$, of the dynamic materials balances are given as a function of the relative uncertainty (2,5,10, or 20%) in estimating the inventory in each column for accounting periods of eight hours (N=1) to seven days (N = 21).

TABLE III

EFFECT OF COLUMN-INVENTORY UNCERTAINTY ON DYNAMIC MATERIALS ACCOUNTING IN THE REFERENCE PPP

Accounting Period	Column-Inventory Uncertainty	Standard Deviation (kg Pu) $\sigma_{\Delta I(N)}$	$\sigma_{T(N)}$	$\sigma_{MB(N)}$
8h (N = 1)	2%	0.60	0.18	0.63
	5%	0.84	0.18	0.86
	10%	1.40	0.18	1.41
	20%	2.63	0.18	2.64
1 day (N = 3)	2%	0.60	0.46	0.76
	5%	0.84	0.46	0.96
	10%	1.40	0.46	1.47
	20%	2.63	0.46	2.67
2 days (N = 6)	2%	0.60*	0.88	1.06
	5%	0.84*	0.88	1.22
	10%	1.40	0.88	1.65
	20%	2.63	0.88	2.77
3 days (N = 9)	2%	0.60*	1.29	1.42
	5%	0.84*	1.29	1.54
	10%	1.40	1.29	1.90
	20%	2.63	1.29	2.93
5 days (N = 15)	2%	0.60*	2.12	2.20
	5%	0.84*	2.12	2.28
	10%	1.40*	2.12	2.54
	20%	2.63	2.12	3.38
7 days (N = 21)	2%	0.60*	2.94	3.01
	5%	0.84*	2.94	3.06
	10%	1.40*	2.94	3.26
	20%	2.63*	2.94	3.95

It can readily be seen from Table III that the column-inventory uncertainty has a significant effect over relatively short accounting periods, but has a much smaller effect over longer periods as throughput-measurement errors (that is, transfer errors) begin to accumulate. In other words, the net-inventory-change variance, $\sigma^2_{\Delta I(N)}$, dominates the Nth materials-balance variance, $\sigma^2_{MB(N)}$, until

$$\sigma^2_{T(N)} \geq \sigma^2_{\Delta I(N)} \quad \text{(see asterisked values in Table III)}, \tag{27}$$

that is, until

$$N\sigma^2_{\varepsilon} + N^2\sigma^2_{\eta} \geq 2I(0)^2\sigma^2_{\varepsilon_I} \quad . \tag{28}$$

The values of N (number of 8-h materials-balance periods) for which Eq. (28) is true can be calculated easily from Table II and Eq.(25) for the column-inventory uncertainties of 2,5,10, and 20% that were selected for illustration. Figure 2 displays the results of solving Eq. (28) for N and clearly shows the effect of PPP column-inventory uncertainty on the time required before

$$\sigma^2_{T(N)} \geq \sigma^2_{\Delta I(N)} \quad .$$

Moreover, Fig. 2 shows the effect, in general, of in-process-inventory uncertainty on dynamic materials accounting.

V. SUMMARY AND RECOMMENDATIONS

In summary, good estimates of column inventory in the reference PPP are essential for detecting short-term losses of in-process materials. In this regard, processing of relatively small batches and operation of the PPP (or any process) near steady state will generally enhance the capability of materials accounting. For longer accounting periods (for example, $\gtrsim$1 wk for the PPP), input - output (transfer) measurement correlations limit the accounting sensitivity. In fact, these correlations can dominate the materials-balance error when the transfer measuring instruments are not recalibrated frequently.

In general, correlations between transfer measurements limit the sensitivity of dynamic materials balances for all high-throughput processes over sufficiently long accounting periods. Therefore, adequate measurement controls must provide for frequent recalibration of the transfer measuring instruments, and ensure well-characterized standards for the transfer measurements. Rapid in-line and at-line assay techniques that provide precise inventory measurements and accurate transfer measurements, with provision for frequent recalibration of the transfer measuring instruments, are generally favored. Finally, because every process is unique, the period between physical inventories should be coupled to the process-specific buildup of transfer measurement correlations; that is, after the materials-balance-error standard deviation, σ_{MB}, for a given process (or materials balance area) becomes unacceptably large, a physical inventory should be taken to "restart" the dynamic accounting system.

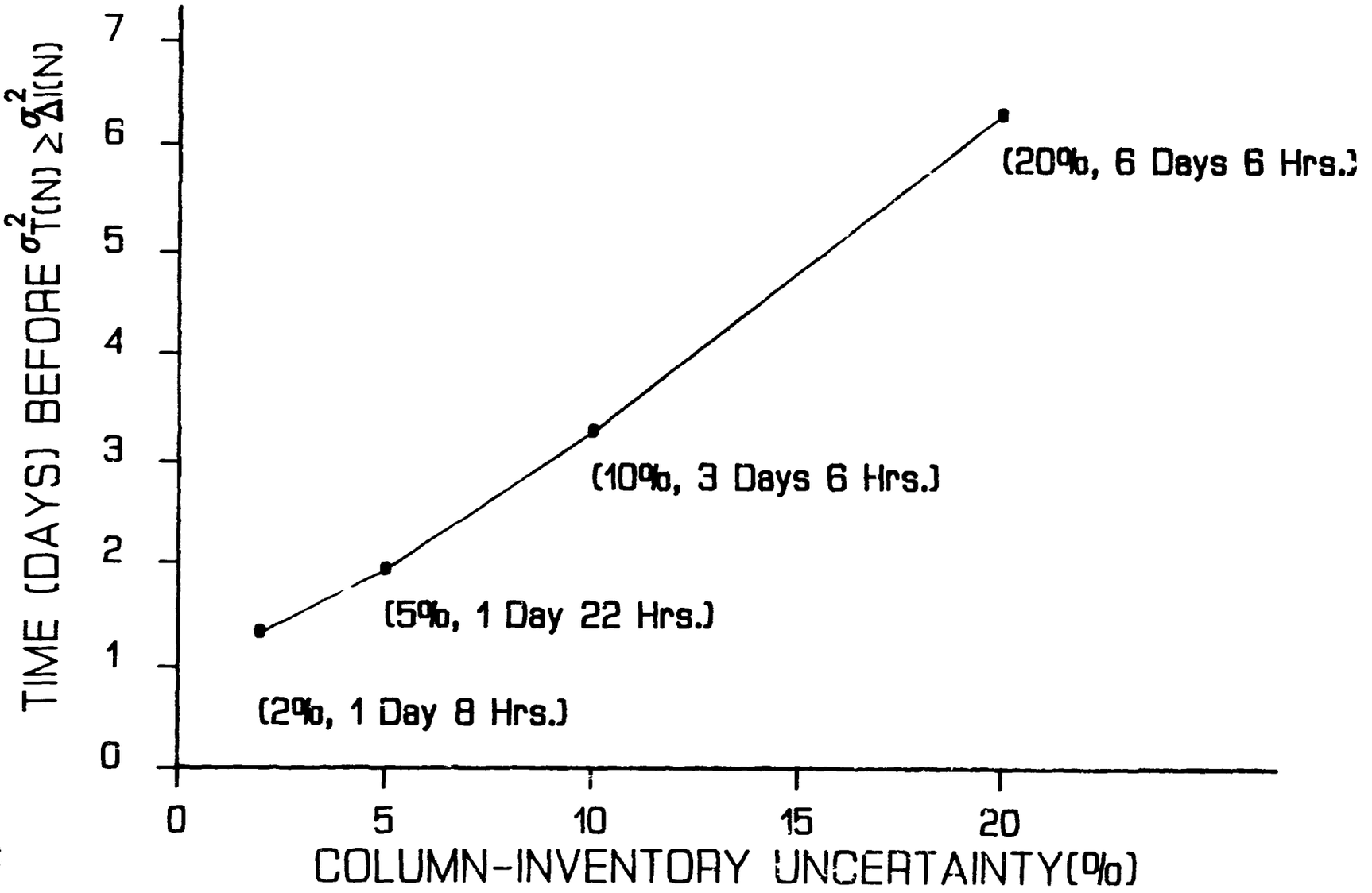

Fig. 2. Effect of cclumn-inventory uncertainty on the time required before $\sigma^2_{T(N)}$ exceeds $\sigma^2_{\Delta I}(N)$.

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