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**OAK RIDGE  
NATIONAL  
LABORATORY**

**MARTIN MARIETTA**

**PRACTICAL REPORTING TIMES  
FOR ENVIRONMENTAL SAMPLES**

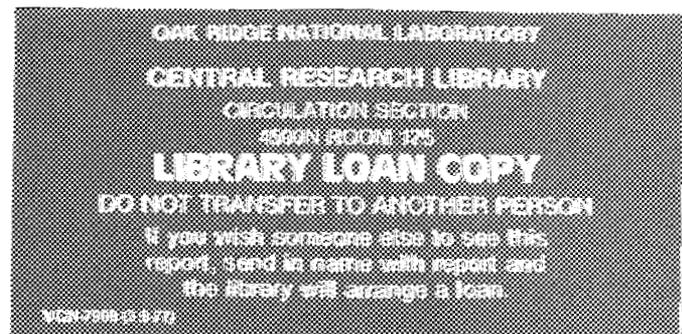
February 1993

Charles K. Bayne  
Denise D. Schmoyer  
Roger A. Jenkins

Supported by

U.S. Army Toxic and Hazardous Materials Agency

DOE IAG No. 1769-A073-A1



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Oak Ridge National Laboratory  
Oak Ridge, Tennessee 37831-6370

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## EXECUTIVE SUMMARY

This report proposes a new definition for how long environmental samples can be held before chemical analysis. Preanalytical holding time is an important factor in obtaining valid information from environmental soil and water samples. The basic concept of holding times is to specify how long a sample can be held with reasonable assurance that the initial concentration has not changed significantly. The definitions of "reasonable assurance" and "changed significantly" are key to holding-time determinations. This paper proposes a "Practical Reporting Time"(PRT) based on statistical definitions of these terms.

A significant change in initial concentration is defined using statistical properties of the measurement system. A critical concentration (CC) is determined on the first day of the holding-time study; it is the concentration below which there is only an  $\alpha\%$  chance, due to measurement error, that a measured concentration would be observed. A significant change has occurred when the concentration falls below this critical concentration. The PRT is defined as the day when there is a risk of  $\gamma\% = 15\%$  that the measured analyte will be below the CC for an  $\alpha\% = 5\%$ .

From 1986-1988, several studies were conducted at Oak Ridge National Laboratory to evaluate holding times for volatile organic compounds and explosives. The data from these studies were used in developing the PRT definition. Maximum holding times (MHT) have previously been calculated for the ORNL holding-time studies by an alternative definition specified by the American Society for Testing and Materials (ASTM). The PRT and ASTM MHT compared favorably with 75% of the cases having holding-time differences of less than 10 days. The PRT and ASTM MHT definitions yield similar holding-time requirements but the PRT definition has the advantage that it is possible to assess the risks for samples held beyond the PRT.

An equation was developed to estimate PRT values from short term holding-time studies. If a chemist is willing to assume a zero-order or first-order kinetics model, a short term experiment could be conducted to estimate the slope of the kinetics model and the standard deviation ( $\sigma$ ) of a single analytical measurement. The PRT value can be estimated from the  $\sigma$ -to-slope ratio and the quadratic polynomial provided in this report. The risks for holding samples beyond their PRT can be determined using the  $\sigma$ -to-slope ratio and the nomograph presented in this report.

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## 1. INTRODUCTION

Analyte concentrations are not always stable in environmental samples. Analytical chemistry laboratories analyze environmental samples as quickly as possible to ensure accurate measurements of the concentration at the time the analyte was sampled. With an increasing number of environmental samples, the time between collecting an environmental sample and its chemical analysis (holding time) may be too long; during this time the analyte may biodegrade (1,2,3) or may decompose. Regulatory agencies have specified holding times for classes of compounds (volatile organics, semivolatile organics, pesticides, explosives) to standardize analytical laboratory procedures. For example, 40 CFR 136 (Code of Federal Regulations, 4), requires that volatile organic compounds (VOCs) samples stored at 4° C must be analyzed within 7 days of collection. This requirement is very stringent for most analytical laboratories. Currently, the holding time has been extended to 14 days for acid-preserved samples (5). The U.S. Army Toxic and Hazardous Materials Agency (USATHAMA, 6) recommends a 28-day holding time for VOCs preserved with sodium bisulfate, and a 56-day (until extraction) holding time for explosives stored at 4° C. These results are based on the Oak Ridge National Laboratory (ORNL) holding-time study (7,8,9,10). The purpose of this study is twofold. First, a statistical definition for a holding time is developed, and second, a method is developed to predict the risks for analyzing an analyte beyond the holding time.

The basic concept of holding times is to specify how long a sample can be held with reasonable assurance that the initial concentration has not changed significantly. The definitions of "reasonable assurance" and "changed significantly" are key to holding-time determinations. This paper proposes a "Practical Reporting Time"(PRT) based on statistical definitions of these terms.

A significant change in initial concentration is defined using statistical properties of the measurement system. A critical concentration (CC) is determined on the first day of the holding-time study; it is the concentration below which there is only an  $\alpha\%$  chance, due to measurement error, that a measured concentration would be observed. A significant change has occurred when the concentration falls below this critical concentration.

Figure 1 illustrates an analyte concentration that is linearly decreasing with time, and measurement variation follows a normal distribution. As the concentration decreases, the chance that an individual sample will be below the critical concentration increases. The probability ( $\gamma$ ) that a sample concentration is below the critical concentration is used as a measure of risk. The PRT is defined as the day when there is a risk of  $\gamma\% = 15\%$  that the measured analyte will be below the CC for an  $\alpha\% = 5\%$ .

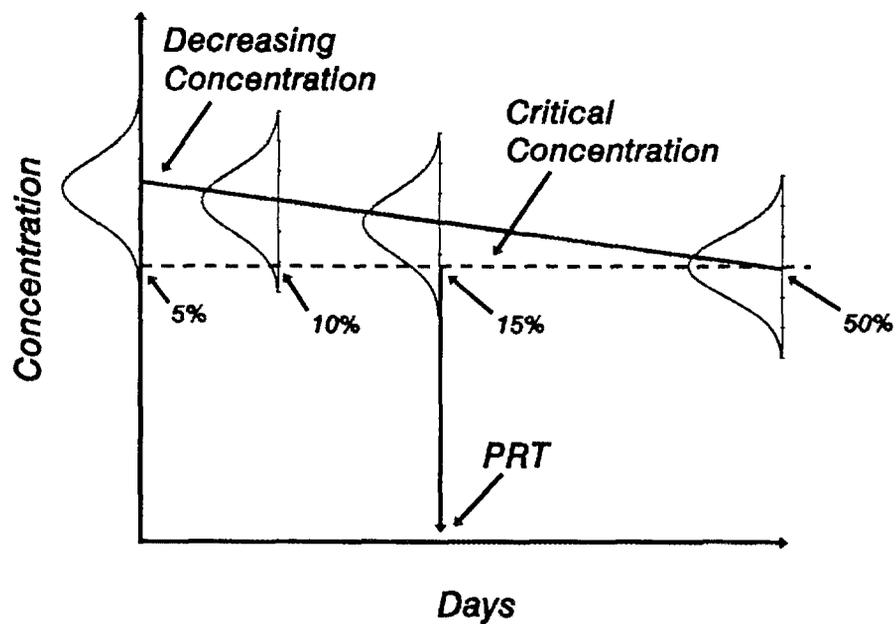


Fig. 1. Practical reporting time (PRT) for an analyte with a linear decreasing concentration.

The PRT depends on the model used to approximate the degradation of an analyte concentration and the precision (standard deviation) of the analytical measurement variation. For a given analyte, large measurement variation will give a longer PRT than those for smaller measurement variation. This result occurs because it is more difficult to detect changes in the initial concentration with larger measurement variation. The chance that a measured analyte is below CC will increase the longer an environmental sample is held past the PRT. The rate of this probability increase reflects the consequences of missing the PRT.

## 2. EXPERIMENTAL

Holding-time studies for volatile organic compounds and explosives were conducted at ORNL (7,8,11) from August 1986 to September 1988. Data from these studies were used to develop the Practical Reporting Time definition.

### 2.1 ORNL Holding-Time Study

Practical reporting times were calculated for 19 volatile organic compounds (VOCs) and 4 explosives for the holding-time studies conducted at ORNL. The ORNL holding-time study made four replicate concentration measurements on days 0, 3, 7, 14, 28, 56, 112 and 365 for VOCs in three water matrices, and four explosives in three water and three soil matrices. For VOCs in three soil matrices, the four replicate measurements were made on days 0, 3, 7, 14, 28, 56 (or 111 days for sterilized USATHAMA soil). Two-spiking levels and four storage temperatures were included in the ORNL holding-time study. The experimental parameters are column and row labels in Tables A.1-A.4. The ORNL holding-time study represents 13,422 concentration measurements over long periods under a variety of matrices and storage temperatures.

Practical reporting times are calculated using the following procedure: (a) an approximating model representing "concentration versus time" is fitted to the data by the method of least squares (12); (b) the one-sided 95% prediction limit for time zero is located for the approximating model (this limit is the critical concentration); (c) a horizontal line is drawn from the critical concentration until it intersects with the one-sided 85% prediction limit; and (d) a vertical line is drawn from the intersection described in (c) to the time-axis. This intersection with the time-axis is the PRT.

Critical concentrations at the 95% prediction limits on day zero represent decision points for analyte concentrations with a normal probability distribution. Concentration measurements below the decision point would be identified as lower than the original concentration. At zero time, about 5% of the concentrations would be below this critical concentration due to measurement distribution. At the PRT, the analyte concentration would have decreased enough to give a 15% chance that a measured concentration would be below the CC.

Maximum holding times (MHT) have previously been calculated for the ORNL holding-time study by two alternative statistical definitions. The first definition was specified by the American Society for Testing and Materials (ASTM, 13). The second definition was specified by Environmental Science and Engineering, Inc. (ESE, 14) for a holding-time study conducted in cooperation with the U.S. Environmental Protection Agency. The ASTM defines the MHT as the time the predicted concentration falls below the lower two-sided 99% confidence interval on the estimated initial concentration. The ESE defines the MHT as the time a one-sided 90% confidence interval on the predicted concentration falls below a 10% change in the initial concentration.

A direct comparison of the ASTM MHT and ESE MHT is difficult because of the two methods for defining critical concentrations and their intersection with the approximating model or the prediction interval. In addition, a good measure is not available for the consequences of measuring analyte concentrations beyond the maximum holding times. The PRT definition has the advantage that a probability of being below the CC can be calculated for a measured analyte concentration at any time period. The increase in this probability can be used to measure the effect of analyzing samples beyond the PRT.

Figure 2 is a histogram of the differences between ASTM MHT and PRT for 360 combinations of the experimental parameters in the ORNL holding-time study. Positive differences show that ASTM values are larger than PRT values, and negative differences show that PRT values are larger than ASTM values. As estimated holding times increase, PRT values are usually larger than ASTM values. All differences are between 18 to -23 days with 75% of the differences between 10 and -10 days. One reason for choosing the value  $\gamma\% = 15\%$  is because at this  $\gamma\%$ , the PRT values closely approximate the ASTM MHT values. A comparison with ESE MHT values show no general trends. The differences between ESE MHT and PRT values range from 256 to -155 days.

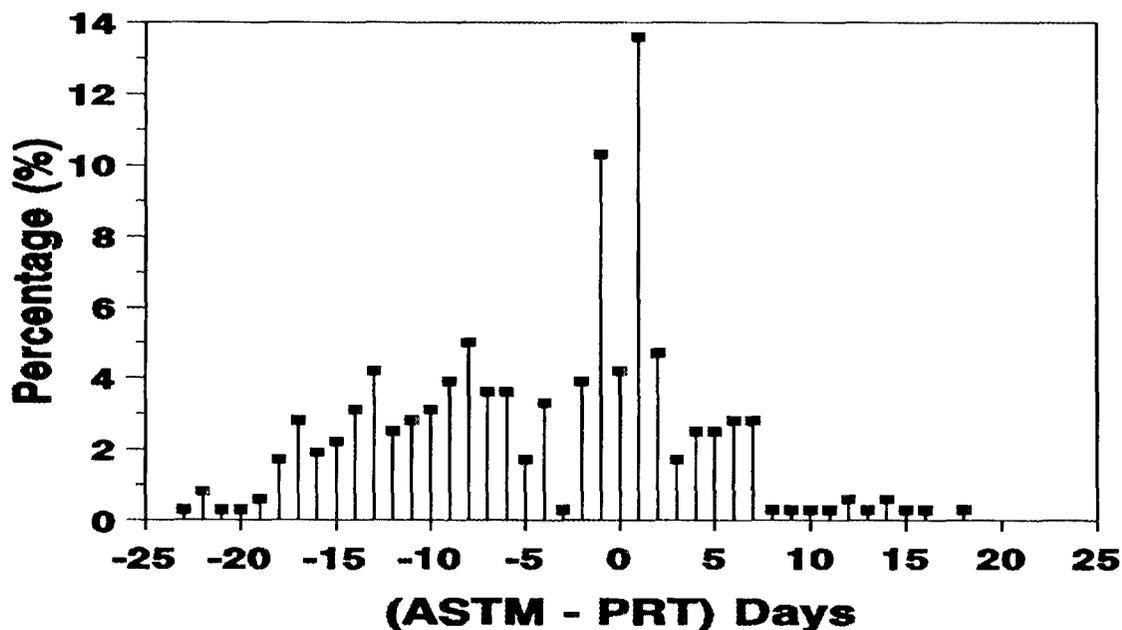


Fig. 2. Frequencies of the differences between ASTM and PRT holding-time estimates.

## 2.2 Approximating Models

Practical reporting times depend on approximating models to represent the degradation of analyte concentrations with time. Approximating models can be either decreasing (most common), increasing, or a combination of a period of stability followed by a rapid decrease. Different approximating models may represent the same analyte under different environmental matrices and storage conditions. Five approximating models were used to represent analyte degradation (zero-order kinetic model, first-order kinetic model, log-term model, inverse-term model and cubic-spline model). Zero-order and first-order kinetic models represent the degradation of concentration or the logarithm (base e) of concentration by a line [ $C = C_0 + B \text{ day}$ , or  $\ln(C) = C_0 + B \text{ day}$ ], respectively. These two models successfully approximated the data for 73% of the ORNL holding-time experimental cases. To approximate more rapidly degrading concentrations, additional terms were added to the zero-order model. The log-term model adds a logarithmic term [e.g.,  $\ln(\text{day})$ , base e] and the inverse-term model adds a reciprocal term [e.g.,  $1/(\text{day})$ ]. The log-term model and inverse-term model can approximate data with rapid concentration degradation for 18% of ORNL holding-time cases. The coefficients for these four models can be estimated by the usual method of least-squares (11). In addition, the least-squares analysis can estimate the precision (standard deviation,  $S_0$ ) for measuring a single analyte by the square-root of the mean square error.

The four linear models couldn't approximate 9% of ORNL holding-time experimental cases that had an initial constant-concentration plateau followed by degradation. An empirical model was applied for these cases which had an initial constant-concentration for days less than  $\text{day} = D_0$ , and a final concentration for days greater than  $\text{day} = D_1$ . The concentrations were modelled by a cubic polynomial between day  $D_0$  and day  $D_1$ . The cubic spline starts with a value of the initial concentration at day  $D_0$  and ends with a value of the final concentration at day  $D_1$ . In addition, the cubic spline is continuous at day  $D_0$  and day  $D_1$ . Coefficients for the cubic spline are estimated by the method of non-linear least-squares (11). Figure 3 illustrates the five approximating models.

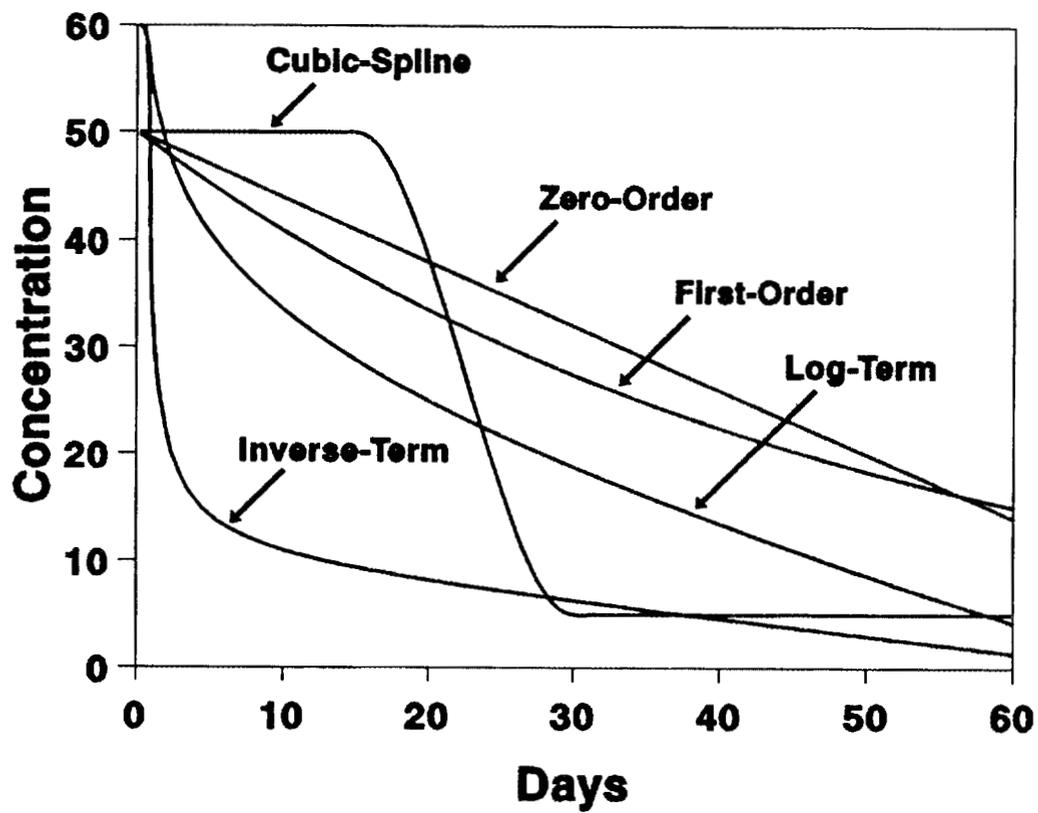


Fig. 3. Five models used to approximate degradation of analyte concentration.

### 3. RESULTS AND DISCUSSION

PRTs from the ORNL holding-time study were used to develop a quadratic equation for estimating PRTs, and a graphical method to assess the risk for samples held past the PRT.

#### 3.1 Sigma-to-Slope Ratios

Holding-time studies at other laboratories may have different spiking levels, environmental matrices, storage conditions and measurement errors. For zero-order and first-order kinetic models, these holding-time results can be related to the new PRT definition through the ratio of the single measurement precision (standard deviation) to the absolute value of the slope of the line that approximates the concentration change (i.e., sigma-to-slope ratios). Figure 4 plots the observed PRT values versus sigma-to-slope ratios ( $S_{\sigma}/|B|$ ) for ORNL holding-time cases approximated by zero-order and first-order models. Sigma-to-slope ratios greater than 385 indicate that precision values are divided by small slope values. These small slope values would not be significantly different from zero at the 5% significance level. Their corresponding PRT values would be set to the maximum experimental time (56, 111, or 365 days). Tables A.1 to A.4 list the sigma-to-slope ratios with the positive (increasing slope) and negative (decreasing slope) signs for the ORNL holding-time experiment. Mathematical formulas to calculate PRT values exactly for zero-order and first-order models are given in Appendix B.

A quadratic polynomial fitted by the method of least-squares to PRT versus the sigma-to-

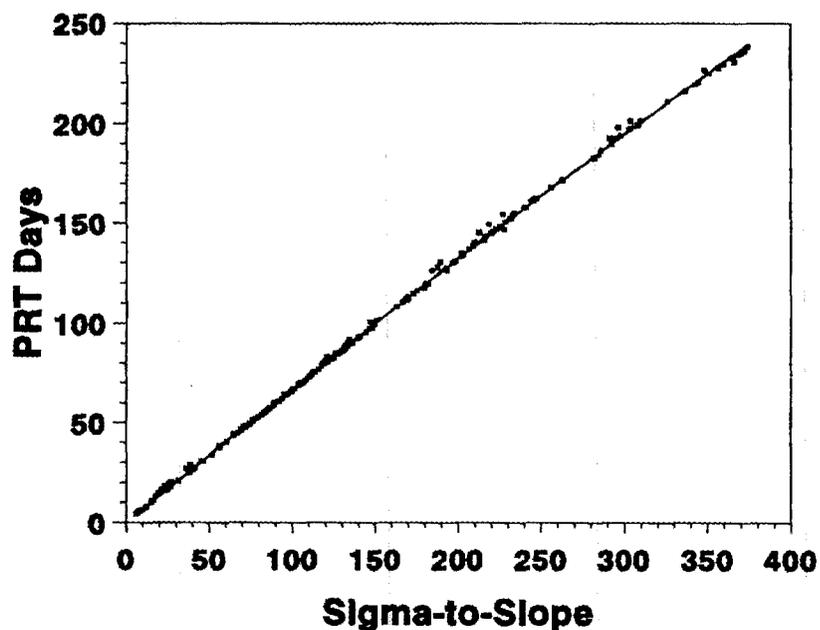


Fig. 4. Quadratic polynomial fitted to PRT values.

slope ratio represents 99.95% of the variation in the PRT values. Predicted PRT values are also shown in Fig. 4 for the quadratic approximation

$$PRT = -0.3051 + 0.6894 \left( \frac{S_0}{|B|} \right) - 0.0134 \times 10^{-2} \left( \frac{S_0}{|B|} \right)^2 .$$

For example, Table A.1 shows that  $(S_0/B) = -151$  for benzene at 50  $\mu\text{g/L}$  concentration in ground water stored at 4°C. First convert this number to sigma-to-slope ratio by taking the absolute value,  $(S_0/|B|) = +151$ . The corresponding PRT value can be approximated either graphically by Fig. 4 or by the quadratic polynomial. Approximate PRT values are rounded down to the next whole number of days:

$$PRT = -0.3051 + 0.6894(151) - 0.0134 \times 10^{-2} (151)^2 .$$

$$PRT = 100.7 \approx 100 \text{ days} .$$

The actual PRT value is 101 days calculated from the mathematical formula in Appendix B. Smaller sigma-to-slope values have shorter holding times. For example, Table A.1 shows  $(S_0/B) = -94$  for benzene at 50  $\mu\text{g/L}$  concentration in surface water stored at 4°C. The quadratic polynomial approximate value is  $PRT = 63$  days and the exact calculation is  $PRT = 62$  days. The margin of error is small between the quadratic polynomial and the exact calculations in Fig. 4 with a maximum difference of 5.6 days at  $PRT = 198$  days.

Estimating PRT values based on the sigma-to-slope ratios is very useful for short term holding-time studies. Suppose a chemist wishes to estimate the PRT value for a compound and their experimental time is limited. If the chemist is willing to assume a zero-order or first-order kinetics model, a short term experiment could be conducted to estimate the slope of the model and the standard deviation of a single analytical measurement. The best procedure is to run replicate concentration measurements at the beginning and end of their experimental time. The chemist may wish to run additional measurements on the third and seventh days to detect rapid degradation, and additional measurements in the middle of their experimental time to detect lack-of-fit. From this experiment, they can estimate the slope and the measurement standard deviation. Using the sigma-to-slope ratio, a PRT value can be estimated for the compound using either Fig. 4 or the quadratic polynomial. This procedure can be used despite analyte, concentration level, sample matrix, or storage condition. The chemist can calculate PRT values for any analyte and conditions for which zero-order and first-order models are used to approximate the analyte concentration degradation. Longer holding-time experiments will give additional confidence that the correct assumptions have been made.

### 3.2 Impact of Missing the PRT

What happens to the risk ( $\gamma$ ) probability if samples are held past the PRT? The risk probability is the probability that an analyte concentration measurement is less than the critical concentration. This risk probability will increase as samples are held past the PRT. The decision maker must decide if the increased risk probability is unacceptable. The rate of increase of the risk probability will depend on the sigma-to-slope ratio. Figure 5 is a nomograph for increasing risk probabilities for days past the PRT value. This nomograph is based on the sigma-to-slope ratios estimated for the ORNL holding-time study. The holding-time nomograph is used in conjunction with the sigma-to-slope ratios in Tables A.1-A.4. For example, the PRT value is 101 days for benzene at 50  $\mu\text{g/L}$  concentration in ground water stored at 4°C with a sigma-to-slope value of  $(S_0/B) = +151$ . Figure 5 shows that at 10 days past the PRT value the risk probability is a little less than 0.17. This increase may be considered acceptable. But, 30 days past the PRT value, the risk probability increases to 0.20 and this increase may be considered unacceptable. This risk probability means the analyte concentration has degraded so that there is a one in five chance an analysis of an environmental sample gives a concentration below the critical concentration. Figure 6 is an enlargement of a section of the nomograph for cases with sigma-to-slope ratios less than 50.

The nomograph illustrates the risk probabilities of concentration changes (below the CC level) rather than the actual concentration change for a single measurement. In addition to measurement precision and slope, the actual concentration change would require the value for the initial concentration which may vary for different holding-time studies. Different holding-time experimental designs would affect the risk probability calculations through the number of degrees of freedom, and the variances and covariances of the intercept and slope. PRT calculations for the ORNL holding-time study show this methodology can be extrapolated to results from a large class of experimental parameters for analytes, spiking levels, matrices, and storage temperatures.

### 3.3 PRT Values for Special Cases

Tables A.1-A.4 list the sigma-to-slope ratios for analytes modelled by the zero-order and first-order kinetic models. Entries designated by "NS" are for cases that have a non-significant slope (i.e., no concentration degradation) at the 5% significance level, and the PRT values are the maximum experimental time. Entries designated by "A" are modelled by the log-term, inverse-term, or cubic spline models and have PRT values less than seven days. In addition, the number of days past PRT is less than seven days for a probability value of  $\gamma\% = 50\%$ . These cases represent rapid degradation of analyte concentration. Entries designated by "B" indicate the number of days past PRT is greater than seven days for a probability value of  $\gamma\% = 50\%$ . All 27 cases designated as "B" are modelled by the cubic-spline model except 50  $\mu\text{g/L}$  of DNT in ground water stored at 4°C which was modelled by a log-term model. PRT values and days past the PRT for these special cases are listed in Table A.5 in Appendix A. Two cases in Table A.5 show unusual jumps in the number of days past PRT. Ethylbenzene spiked at 50  $\mu\text{g/g}$  in USATHAMA soil stored at -20°C shows a jump from 6 days to 90 days, and toluene spiked at 50  $\mu\text{g/g}$  in Mississippi soil stored at -70°C shows a jump from 2 days

to 54 days as the  $\gamma$  probability changes from 30% to 35%. These unusual increases are due to the cubic spline leveling off to a constant concentration. As a result of these cubic spline behaviors the  $(1 - \gamma)\%$  prediction limits less than 70% become parallel to the critical concentration limit. These parallel lines never intersect with the critical concentration limit for  $\gamma\%$  greater than 30%.

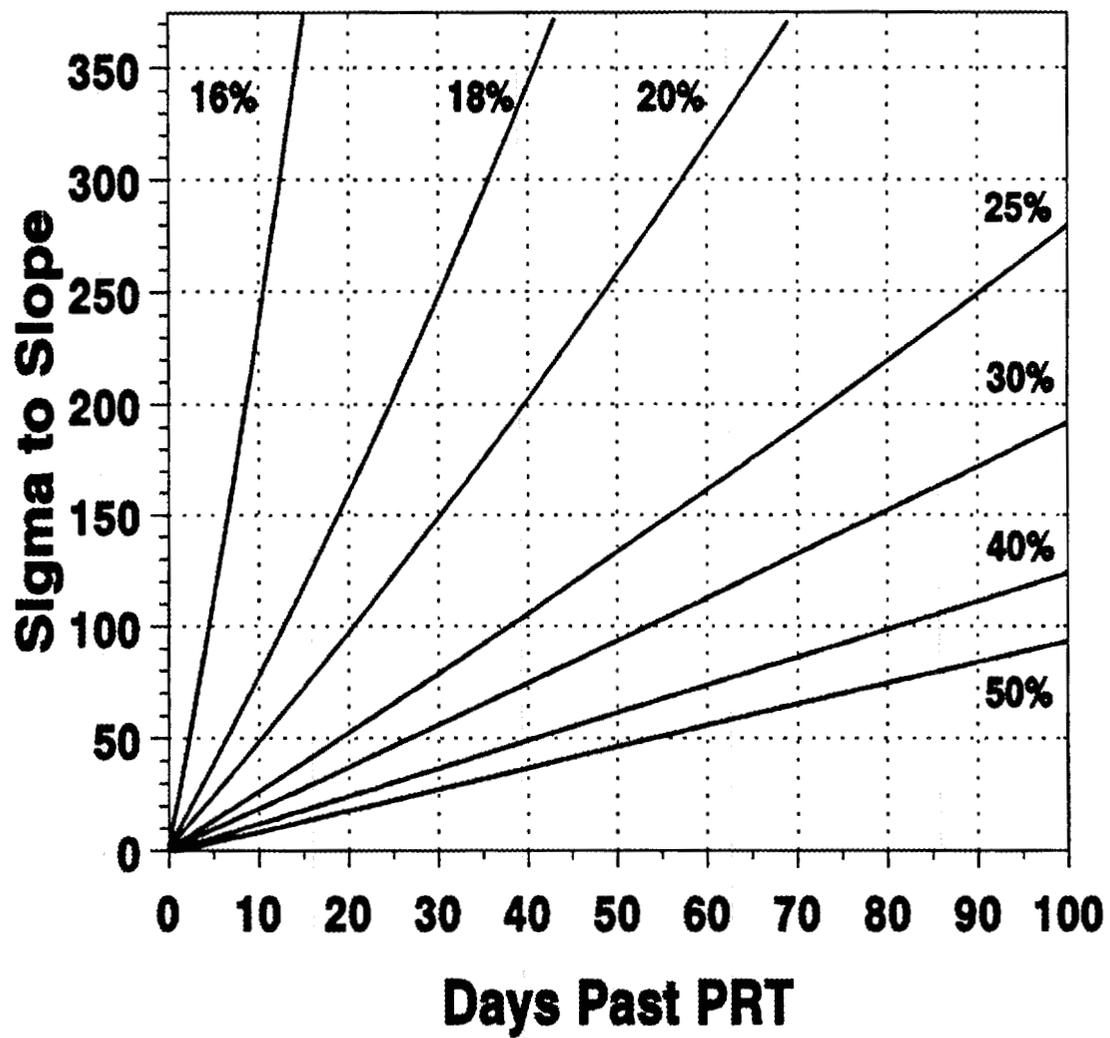


Fig. 5. Holding-time nomograph for days past the PRT. Contours are probabilities of an analyte measurement being less than the critical concentration.

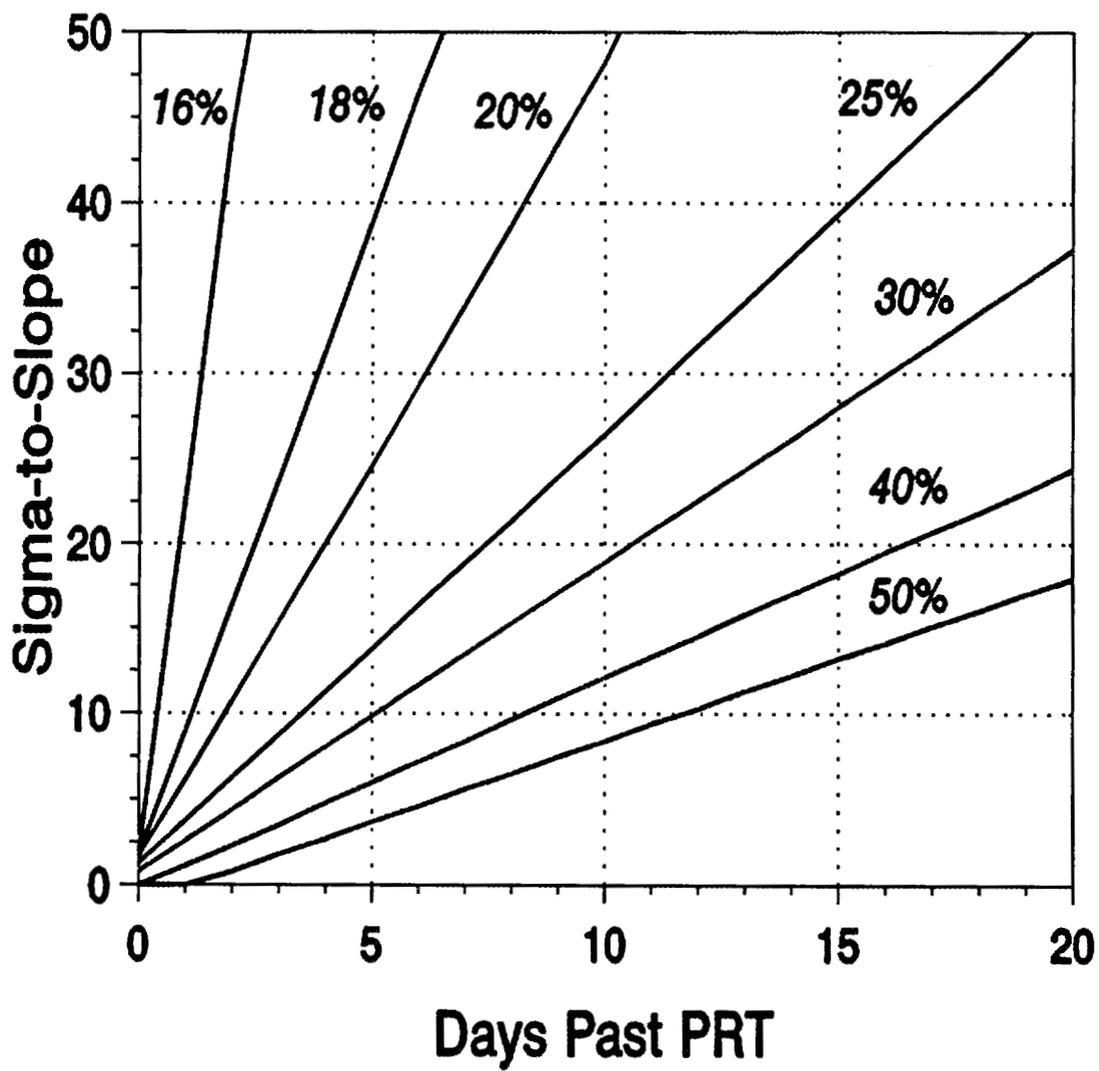


Fig. 6. Holding-time nomograph for sigma-to-slope ratios less than 50. Contours are probabilities of an analyte measurement being less than the critical concentration.

#### 4. CONCLUSIONS

Practical reporting time is a statistically defined holding time to specify how long a sample can be held with reasonable assurance that the initial concentration has not changed significantly. PRT values depend only on the degradation kinetics and the analytical measurement variation. Results from the ORNL holding-time study show that the PRT values are comparable to holding times calculated by the ASTM definition. But, the PRT method can also indicate the consequence of making analytical measurements past the PRT. A quadratic polynomial has been estimated to predict PRT values for a large class of analytes, matrices, and storage temperatures with zero-order and first-order approximating kinetic models. Future holding-time studies with limited resources can calculate PRT values using the sigma-to-slope ratio. The PRT method can indicate the risk of making analytical measurements past the PRT. Finally, a nomograph is presented for decision makers to evaluate the risk of analyzing a sample past the PRT value.

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## **APPENDIX A**

**Sigma-to-Slope Ratios and Past PRT Risks for Special Cases**

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Table A.1 Sigma-to-slope ratios for volatile organic compounds in environmental water samples used in the ORNL holding-time study.

Volatile Organic Compound	Concentration $\mu\text{g/L}$	Water Type					
		Distilled		Surface		Ground	
		Storage Temperature		Storage Temperature		Storage Temperature	
		4 °C	Room	4 °C	Room	4 °C	Room
Benzene	50	NS	NS	-94	-131	-151	-227
	500	233	-256	369	NS	-246	-286
Bromoform	50	NS	-225	-76	-40	126	-121
	500	351	-150	NS	-242	NS	NS
Carbon Tetrachloride	50	NS	NS	-144	B	NS	-189
	500	NS	-207	-308	-72	NS	NS
Chlorobenzene	50	NS	-337	-97	-180	NS	-188
	500	NS	-79	NS	-221	NS	-202
Chloroform	50	-304	-229	-87	-282	NS	219
	500	217	-240	NS	NS	-286	-375
1,1-Dichloroethane	50	NS	NS	-82	-130	NS	NS
	500	304	NS	NS	NS	-309	-366
1,1-Dichloroethene	50	NS	152	-120	-107	349	-213
	500	246	-103	NS	-83	-175	-118
1,2-Dichloropropane	50	NS	NS	-128	NS	133	184
	500	170	220	223	NS	NS	NS
Ethylbenzene	50	NS	-372	B	-79	B	-45
	500	NS	-89	B	-198	-296	-105
Methylene Chloride	50	NS	-326	-108	-148	292	NS
	500	244	-146	-345	-199	-357	-364
Styrene	50	NS	-263	B	A	A	-134
	500	NS	-81	-140	-92	NS	-120
1,1,2,2-Tetrachloroethane	50	B	A	-282	-12	132	-16
	500	A	A	NS	-61	NS	-12
Tetrachloroethene	50	NS	-203	-83	-132	NS	-297
	500	-209	-38	-146	-57	-128	-65
1,1,2-Trichloroethane	50	NS	-15	-131	-263	65	147
	500	303	B	NS	NS	NS	-140
Trichloroethene	50	210	-298	-80	285	NS	NS
	500	207	-86	NS	-193	-343	-163
Toluene	50	NS	-373	-85	-46	-100	-71
	500	NS	-122	-127	-204	-310	-234
o-Xylene	50	NS	-371	-202	-51	-119	-27
	500	NS	-173	NS	NS	NS	-112

NS = Slope not significant  
A = PRT values and days past the PRT ( $15\% < \gamma\% \leq 50\%$ ) are less than seven days.  
B = See Table A.5.

Table A.2 Sigma-to-slope ratios for volatile organic compounds in environmental soil samples used in the ORNL holding-time study.

Volatile Organic Compound (Concentrations < 100 µg/g)	Soil Type							
	USATHAMA		Tennessee			Mississippi		
	Storage Temperature		Storage Temperature			Storage Temperature		
	-70°C	-20°C	-70°C	-20°C	4°C	-70°C	-20°C	4°C
Benzene	A	-112	A	-27	A	A	A	A
Bromoform	NS	95	22	38	-6	NS	NS	A
Bromomethane	A	NS	A	A	A	A	A	A
Carbon Tetrachloride	A	NS	A	-17	-8	A	A	A
Chlorobenzene	A	NS	NS	NS	A	A	A	A
Chloroethane	A	B	A	-22	-8	A	A	A
Chloroform	A	NS	A	B	A	A	A	A
1,1-Dichloroethane	A	NS	A	-19	-7	A	A	A
1,1-Dichloroethene	A	NS	A	-17	A	A	A	A
1,2-Dichloropropane	A	NS	A	B	-6	A	A	A
Ethylbenzene	A	B	A	-36	A	A	A	A
Methylene Chloride	B	NS	NS	NS	-15	NS	A	A
Styrene	NS	NS	NS	NS	A	NS	-23	A
1,1,2,2-Tetrachloroethane	NS	NS	24	26	-6	NS	NS	A
Tetrachloroethene	A	A	A	-21	A	A	A	A
1,1,2-Trichloroethane	-56	NS	NS	NS	-7	B	A	A
Trichloroethene	A	-89	A	B	A	A	A	A
Toluene	A	-104	A	B	A	B	A	A
o-Xylene	A	-64	NS	NS	A	B	A	A

NS = Slope not significant  
A = PRT values and days past the PRT (15% < γ% ≤ 50%) are less than seven days.  
B = See Table A.5.

Table A.3 Sigma-to-slope ratios for explosive compounds in environmental water samples used in the ORNL holding-time study.

Explosive	Concentration μg/L	Water Type					
		Distilled		Surface		Ground	
		Storage Conditions		Storage Conditions		Storage Conditions	
		4°C	Room	4°C	Room	4°C	Room
DNT	50	A	B	-24	A	B	A
	1000	167	197	-74	-110	NS	-124
HMX	100	-100	-91	-25	-40	-105	-86
	1000-2000	B	B	-141	-170	NS	NS
RDX	50	NS	-134	-38	-31	NS	B
	1000	NS	-76	130	99	NS	NS
TNT	50	-108	-10	B	A	-27	A
	1000	360	NS	B	A	-122	B

NS = Slope not significant  
A = PRT values and days past the PRT (15% < γ% ≤ 50%) are less than seven days.  
B = See Table A.5.

Table A.4 Sigma-to-slope ratios for explosive compounds in environmental soil samples used in the ORNL holding-time study.

Explosive	Concentration μg/g	Soil Type								
		USATHAMA			Tennessee			Mississippi		
		Storage Temperature			Storage Temperature			Storage Temperature		
		-20°C	4°C	Room	-20°C	4°C	Room	-20°C	4°C	Room
DNT	10	NS	-367	A	116	-182	A	NS	NS	-110
	100	169	169	231	233	NS	NS	120	181	240
HMX	10	NS	NS	NS	228	NS	-41	125	136	NS
	100	NS	NS	NS	104	97	91	70	89	88
RDX	10	NS	NS	B	144	-193	B	NS	NS	-216
	100	NS	-293	NS	104	132	106	86	108	114
TNT	10	-144	-84	A	NS	-67	A	NS	A	A
	100	-309	-24	A	NS	-83	-24	NS	-247	-78

NS = Slope not significant  
A = PRT values and days past the PRT (15% < γ% ≤ 50%) are less than seven days.  
B = See Table A.5.

Table A.5. Days past PRT corresponding to  $\gamma\%$  probability.

Analyte	Spike	Matrix	Storage	PRT Day	Probability ( $\gamma\%$ ) that a sample is less than CC						
					20%	25%	30%	35%	40%	45%	50%
Carbon Tetrachloride	50 $\mu\text{g/L}$	Surface	Room	17	3	4	6	7	8	10	11
Ethylbenzene	50 $\mu\text{g/L}$	Surface	4°C	13	0	0	1	1	1	1	2
	50 $\mu\text{g/L}$	Ground	4°C	19	2	4	5	6	7	8	9
	500 $\mu\text{g/L}$	Surface	4°C	19	0	1	2	2	3	3	4
Styrene	50 $\mu\text{g/L}$	Surface	4°C	10	0	1	1	1	1	2	2
1,1,2,2-Tetrachloroethane	50 $\mu\text{g/L}$	Distilled	4°C	9	0	0	1	1	1	1	2
1,1,2-Trichloroethane	500 $\mu\text{g/L}$	Distilled	Room	8	0	1	1	2	2	2	3
DNT	50 $\mu\text{g/L}$	Distilled	Room	29	3	5	6	8	9	11	12
	50 $\mu\text{g/L}$	Ground	4°C	2	1	2	3	4	5	7	10
HMX	1000 $\mu\text{g/L}$	Distilled	4°C	20	2	3	4	5	5	6	6
	1000 $\mu\text{g/L}$	Distilled	Room	29	1	2	3	3	4	5	5
RDX	50 $\mu\text{g/L}$	Ground	Room	134	7	13	18	23	28	32	37
TNT	50 $\mu\text{g/L}$	Surface	4°C	18	0	1	1	1	1	1	1
	1000 $\mu\text{g/L}$	Surface	4°C	32	2	4	5	7	8	9	10
	1000 $\mu\text{g/L}$	Ground	Room	0	4	6	8	9	10	11	12
Chloroethane	70 $\mu\text{g/g}$	USATHAMA	-20°C	19	2	3	4	4	5	6	7
Chloroform	60 $\mu\text{g/g}$	Tennessee	-20°C	29	1	2	4	7	12	16	19
1,2-Dichloropropane	60 $\mu\text{g/g}$	Tennessee	-20°C	47	2	5	7	9	9	9	9
Ethylbenzene	50 $\mu\text{g/g}$	USATHAMA	-20°C	21	2	4	6	90	90	90	90
Methylene Chloride	60 $\mu\text{g/g}$	USATHAMA	-70°C	20	1	2	3	3	4	4	5
1,1,2-Trichloroethane	50 $\mu\text{g/g}$	Mississippi	-70°C	0	4	7	9	10	12	13	15
Trichloroethene	50 $\mu\text{g/g}$	Tennessee	-20°C	40	2	3	4	5	5	6	7
Toluene	90 $\mu\text{g/g}$	Tennessee	-20°C	42	0	1	1	2	2	2	3
	50 $\mu\text{g/g}$	Mississippi	-70°C	2	1	1	2	54	54	54	54
o-Xylene	40 $\mu\text{g/g}$	Mississippi	-70°C	14	1	2	2	3	4	4	5
RDX	10 $\mu\text{g/g}$	USATHAMA	Room	18	1	2	3	3	4	4	5
	10 $\mu\text{g/g}$	Tennessee	Room	7	2	4	5	5	6	7	7

## **APPENDIX B**

### **Mathematical Equations for Practical Reporting Time**

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## PRACTICAL REPORTING TIME EQUATIONS

We assume that a linear approximating model with a decreasing slope can represent an analyte concentration ( $C_j$ ) as a model of time (Day,  $D_j$ ).

$$C_j = \alpha + \beta D_j + \epsilon . \quad (\text{B.1})$$

The intercept parameter ( $\alpha$ ) and slope parameter ( $\beta$ ) are estimated by the method of least squares. The estimated values are denoted by "A" and "B" for the estimated intercept and estimated slope, respectively. The measurement error " $\epsilon$ " is a random variable assumed to have a normal probability distribution with zero mean and constant variance ( $\sigma^2$ ). The constant variance assumption means that concentration measurements vary about the same amount for any day. This may not be true if measurement error depends on concentration levels and concentration decreases with time. Measurement variance is estimated by the mean square error for residuals (MSER). The measurement precision is defined by the standard deviation. This precision is estimated by the square root of MSER and is denoted by " $S_o$ ".

The case we are investigating is a linear approximating model with a decreasing slope. Therefore, we restrict the estimated slope to be significantly smaller than zero at a one-sided significance level of 5% {i.e.,  $B < -t(0.05, df)[\text{var}(B)]^{1/2}$ }. We set the PRT to the maximum number of days (e.g., 365 days) in holding-time experiments for estimated slope values that are not significantly different than zero.

The definition for PRT for a linear decreasing approximating model says the 5% percentile point for a predicted measured value at zero time is equal to the 15% percentile value for a predicted measured value at PRT.

$$A - t(0.05, df)\sqrt{\text{var}(A) + S_o^2} = A + BD_{PRT} - t(0.15, df)\sqrt{\text{var}(C_{PRT})} . \quad (\text{B.2})$$

where,

$t(\gamma, df)$  = is the  $(1 - \gamma)$  percentile point of the t-distribution with  $df =$  degrees of freedom.

$\text{var}(A)$  = estimated variance of A. Note  $\text{var}(A) = a_{11}S_o^2$  which is the first row and first column in the 2 X 2 estimated covariance matrix. The value of  $a_{11}$  depends only on the experimental design (i.e., days) used to estimate the linear approximating model.

$\text{var}(C_{\text{PRT}})$  = estimated variance of a predicted concentration at time  $D_{\text{PRT}} = \text{PRT}$ .  
This estimated variance is

$$\text{var}(C_{\text{PRT}}) = \text{var}(A) + D_{\text{PRT}}^2 \text{var}(B) + 2D_{\text{PRT}} \text{cov}(A,B) + S_o^2 . \quad (\text{B.3})$$

$\text{var}(B)$  is the estimated variance of B and  $\text{cov}(A,B)$  is the estimated covariance between A and B. The estimated  $\text{var}(B) = a_{22}S_o^2$  is the second row and second column in the 2 X 2 estimated covariance matrix. The estimated  $\text{cov}(A,B) = a_{12}S_o^2 = a_{21}S_o^2$  is either the first row and second column or second row and first column in the 2 X 2 estimated covariance matrix. The coefficients  $a_{22}$ ,  $a_{12}$ , and  $a_{21}$  depend only on the experimental design (i.e., days) used to estimate the linear approximating model.

Substituting Eq. B.3 into B.2, we can solve for  $D_{\text{PRT}} = \text{PRT}$  as

$$\text{PRT} = \frac{-\Delta \pm \sqrt{\Delta^2 - 4\Lambda\Gamma}}{2\Lambda} , \quad (\text{B.4})$$

where

$$\Lambda = B^2 - t^2(0.15,df)\text{var}(B) ,$$

$$\Delta = 2 [ B t(0.05,df) \sqrt{\text{var}(A) + S_o^2} - t^2(0.15,df) \text{Cov}(A,B) ] ,$$

$$\Gamma = [t^2(0.05,df) - t^2(0.15,df)] [\text{var}(A) + S_o^2] .$$

Equation B.4 shows there are two solutions to a quadratic equation. The PRT is equal to the smallest positive root of Eq. B.4.

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