

```

batch time assay...
1      1      0      103.8
2      1      3      104.6
3      1      6      103.6
4      1      9      103.7
5      1     12      103.8
6      1     18      104.8
7      1     24      104.9
8      2      0      103.7
9      2      3      103.7
10     2      6      103.4
11     2      9      103.8
12     2     12      103.9
13     2     18      104.2
14     2     24      104.5
15     3      0      103.2
16     3      3      104.1
17     3      6      103.4
18     3      9      103.9
19     3     12      103.4
20     3     18      104.3
21     3     24      104.7

```

```

*****
*                               Now, Go to analyze the data                               *
*****

```

Drug product with upper acceptance criteria of ____ % of label claim
1: 105

Drug product with lower acceptance criteria of ____ % of label claim
1: 95

<<Output: ANCOVA model: batch vs. time vs. assay (%)>>

Analysis of Variance Table

Response: assay

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
batch	2	0.42286	0.21143	1.4869	0.2575582
time	1	2.37990	2.37990	16.7373	0.0009634 ***
batch:time	2	0.02722	0.01361	0.0957	0.9092641
Residuals	15	2.13287	0.14219		

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

<< ANCOVA Output: Testing for poolability of batches >>

The tests for equality of slopes and equality of intercepts do not result at a level of significance of 0.25 (there is no significant difference in slope and intercepts among the batches).

<<Suggestion>>

The data from all batches can be combined.

<<Output: linear regression model: time vs. assay (%)>>

Call:

lm(formula = assay ~ time, data = ANCOVAdata)

Coefficients:

(Intercept)	time
103.52934	0.04298

Analysis of Variance Table

Response: assay

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
time	1	2.37990	2.37990	17.506	0.0005033 ***
Residuals	19	2.58295	0.13594		

 Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

<< Output >>

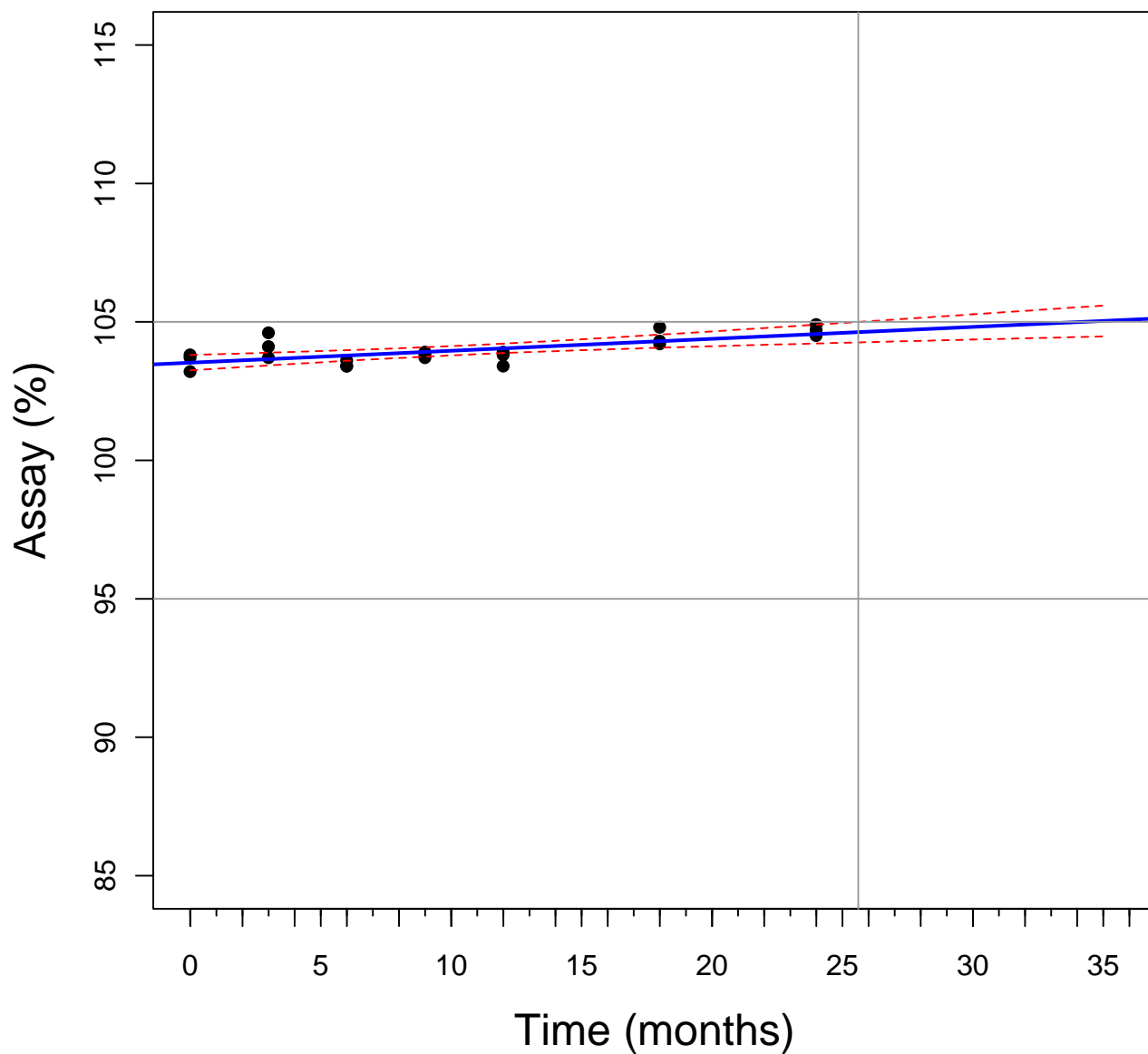
<<Summary: linear regression model>>

Y= 103.5293 +(0.04298071) X

	time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	103.8	103.5293	0.270658683
2	3	104.6	103.6583	0.941716567
3	6	103.6	103.7872	-0.187225549
4	9	103.7	103.9162	-0.216167665
5	12	103.8	104.0451	-0.245109780
6	18	104.8	104.3030	0.497005988
7	24	104.9	104.5609	0.339121756
8	0	103.7	103.5293	0.170658683
9	3	103.7	103.6583	0.041716567
10	6	103.4	103.7872	-0.387225549
11	9	103.8	103.9162	-0.116167665
12	12	103.9	104.0451	-0.145109780
13	18	104.2	104.3030	-0.102994012
14	24	104.5	104.5609	-0.060878244
15	0	103.2	103.5293	-0.329341317
16	3	104.1	103.6583	0.441716567
17	6	103.4	103.7872	-0.387225549
18	9	103.9	103.9162	-0.016167665
19	12	103.4	104.0451	-0.645109780
20	18	104.3	104.3030	-0.002994012
21	24	104.7	104.5609	0.139121756

Drug product with Upper acceptance criteria of 105 % of label claim
Shelf life = 25.60840 months

Shelf Life= 25.61 months



SAS
Stability Analysis

TIME	_1	_2	_3
0	103.8	103.7	103.2
3	104.6	103.7	104.1
6	103.6	103.4	103.4
9	103.7	103.8	103.9
12	103.8	103.9	103.4
18	104.8	104.2	104.3
24	104.9	104.5	104.7

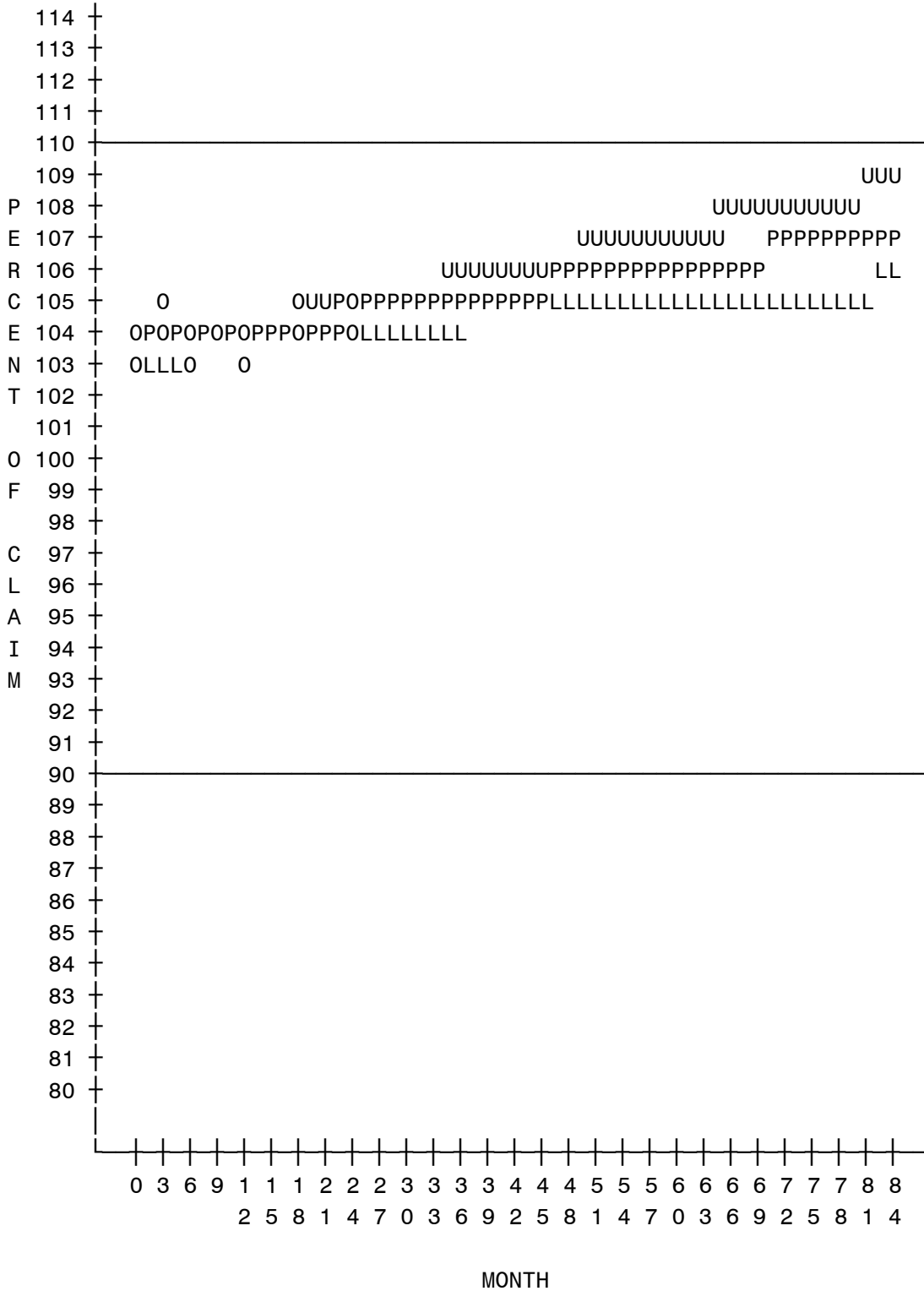
Stability Analysis

SOURCE	SS	DF	MS	F	P
A	0.45	4	0.11	0.79133	0.54877
B	0.42	2	0.21	1.48693	0.25756
C	0.03	2	0.01	0.09573	0.90926
D	2.13	15	0.14		
E	227014.05	6	37835.67		

```
*****  
* Statistical Analysis: *  
* Key to sources of variation *  
* A = sep. intercep, sep slope | com intercep, com slope *  
* B = sep. intercep, com slope | com intercep, com slope *  
* C = sep. intercep, sep slope | sep intercep, com slope *  
* D = Residual *  
* E = Full Model *  
*****
```

Stability Analysis

Plot of LEVEL*TIME. Symbol used is 'O'.
 Plot of PREDICT*TIME. Symbol used is 'P'.
 Plot of L_BOUND*TIME. Symbol used is 'L'.
 Plot of U_BOUND*TIME. Symbol used is 'U'.



NOTE: 78 obs had missing values. 182 obs hidden.

Stability Analysis

Batch All

Fitted Line : $Y = 103.52934132 + 0.0429807053 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	103.251	103.529	103.807	0.13281	
0	103.251	103.529	103.807	0.13281	
0	103.251	103.529	103.807	0.13281	
1	103.311	103.572	103.834	0.12479	
2	103.370	103.615	103.860	0.11712	
3	103.428	103.658	103.888	0.10989	
3	103.428	103.658	103.888	0.10989	
3	103.428	103.658	103.888	0.10989	
4	103.485	103.701	103.917	0.10316	
5	103.541	103.744	103.947	0.09707	
6	103.595	103.787	103.979	0.09172	
6	103.595	103.787	103.979	0.09172	
6	103.595	103.787	103.979	0.09172	
7	103.648	103.830	104.013	0.08725	
8	103.698	103.873	104.049	0.08381	
9	103.746	103.916	104.087	0.08154	
9	103.746	103.916	104.087	0.08154	
9	103.746	103.916	104.087	0.08154	
10	103.791	103.959	104.128	0.08051	
11	103.833	104.002	104.171	0.08079	
12	103.873	104.045	104.217	0.08236	
12	103.873	104.045	104.217	0.08236	
12	103.873	104.045	104.217	0.08236	
13	103.910	104.088	104.266	0.08515	
14	103.945	104.131	104.317	0.08905	
15	103.977	104.174	104.371	0.09391	
16	104.009	104.217	104.425	0.09960	
17	104.038	104.260	104.482	0.10598	
18	104.067	104.303	104.539	0.11293	
18	104.067	104.303	104.539	0.11293	
18	104.067	104.303	104.539	0.11293	
19	104.094	104.346	104.598	0.12036	
20	104.121	104.389	104.657	0.12819	
21	104.147	104.432	104.717	0.13634	
22	104.172	104.475	104.778	0.14476	
23	104.197	104.518	104.839	0.15340	
24	104.221	104.561	104.900	0.16224	
24	104.221	104.561	104.900	0.16224	
24	104.221	104.561	104.900	0.16224	
25	104.245	104.604	104.962	0.17123	
26	104.269	104.647	105.024	0.18036	*

27	104.293	104.690	105.087	0.18961	*
28	104.316	104.733	105.149	0.19896	*
29	104.340	104.776	105.212	0.20840	*
30	104.363	104.819	105.275	0.21791	*
31	104.386	104.862	105.338	0.22749	*
32	104.408	104.905	105.401	0.23713	*

Stability Analysis

Batch All

Fitted Line : $Y = 103.52934132 + 0.0429807053 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
33	104.431	104.948	105.464	0.24681	*
34	104.454	104.991	105.528	0.25655	*
35	104.476	105.034	105.591	0.26632	*
36	104.499	105.077	105.655	0.27613	*
37	104.521	105.120	105.718	0.28597	*
38	104.543	105.163	105.782	0.29585	*
39	104.566	105.206	105.846	0.30574	*
40	104.588	105.249	105.909	0.31567	*
41	104.610	105.292	105.973	0.32561	*
42	104.632	105.335	106.037	0.33557	*
43	104.654	105.378	106.101	0.34555	*
44	104.676	105.420	106.165	0.35555	*
45	104.698	105.463	106.229	0.36557	*
46	104.720	105.506	106.293	0.37559	*
47	104.742	105.549	106.357	0.38563	*
48	104.764	105.592	106.421	0.39569	*
49	104.786	105.635	106.485	0.40575	*
50	104.808	105.678	106.549	0.41582	*
51	104.830	105.721	106.613	0.42591	*
52	104.852	105.764	106.677	0.43600	*
53	104.874	105.807	106.741	0.44610	*
54	104.895	105.850	106.805	0.45621	*
55	104.917	105.893	106.869	0.46632	*
56	104.939	105.936	106.933	0.47644	*
57	104.961	105.979	106.998	0.48657	*
58	104.983	106.022	107.062	0.49670	*
59	105.004	106.065	107.126	0.50684	*
60	105.026	106.108	107.190	0.51699	*
61	105.048	106.151	107.254	0.52714	*
62	105.070	106.194	107.319	0.53729	*
63	105.091	106.237	107.383	0.54745	*
64	105.113	106.280	107.447	0.55761	*
65	105.135	106.323	107.511	0.56778	*
66	105.156	106.366	107.576	0.57795	*
67	105.178	106.409	107.640	0.58813	*
68	105.200	106.452	107.704	0.59830	*
69	105.221	106.495	107.769	0.60848	*
70	105.243	106.538	107.833	0.61867	*

Stability Analysis

95% Two-Sided Confidence Limit

Common Intercept and Common Slope

BATCH	ESTIMATED DATING PERIOD (MONTHS/WEEKS)
All	25

```

batch time assay...
1      1      0      100
2      1      3      101
3      1      6      101
4      1      9      100
5      1     12      99
6      1     18      98
7      2      0      99
8      2      3      100
9      2      6      100
10     2      9      99
11     2     12      99
12     2     18      97
13     3      0      101
14     3      3      100
15     3      6      99
16     3      9      99
17     3     12      98
18     3     18      97

```

```

*****
*                               Now, Go to analyze the data                               *
*****

```

Drug product with upper acceptance criteria of ____ % of label claim
1: 105

Drug product with lower acceptance criteria of ____ % of label claim
1: 95

<<Output: ANCOVA model: batch vs. time vs. assay (%)>>

Analysis of Variance Table

Response: assay

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
batch	2	2.7778	1.3889	3.2407	0.07493 .
time	1	16.8397	16.8397	39.2926	4.139e-05 ***
batch:time	2	0.8508	0.4254	0.9926	0.39910
Residuals	12	5.1429	0.4286		

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

<< ANCOVA Output: Testing for poolability of batches >>

The test rejects the hypothesis of equality of intercepts but fails to reject that the slopes are equal (there is a significant difference in intercepts but no significant difference in slopes among the batches).

<<Suggestion>>

The data can be combined for the purpose of estimating the common slope.

<<Output: linear regression model: time vs. assay (%)>>

Call:

```
lm(formula = assay ~ batch + time, data = ANCOVadata)
```

Coefficients:

(Intercept)	batch2	batch3	time
101.1413	-0.8333	-0.8333	-0.1635

Analysis of Variance Table

Response: assay

```

      Df  Sum Sq Mean Sq F value    Pr(>F)
batch   2  2.7778  1.3889  3.2442  0.06956 .
time    1 16.8397 16.8397 39.3342 2.051e-05 ***
Residuals 14  5.9937  0.4281

```

```

---
Signif. codes:  0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

```

```
<< Output and Linear regression model>>
```

$$Y = 101.1413 + (-0.1634921) X$$

time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	100	101.14127 1.1412698
2	3	101	100.65079 -0.3492063
3	6	101	100.16032 -0.8396825
4	9	100	99.66984 -0.3301587
5	12	99	99.17937 0.1793651
6	18	98	98.19841 0.1984127

```
<< Output and Linear regression model>>
```

$$Y = 100.3079 + (-0.1634921) X$$

time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	99	100.30794 1.3079365
2	3	100	99.81746 -0.1825397
3	6	100	99.32698 -0.6730159
4	9	99	98.83651 -0.1634921
5	12	99	98.34603 -0.6539683
6	18	97	97.36508 0.3650794

```
<< Output and Linear regression model>>
```

$$Y = 100.3079 + (-0.1634921) X$$

time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	101	100.30794 -0.6920635
2	3	100	99.81746 -0.1825397
3	6	99	99.32698 0.3269841
4	9	99	98.83651 -0.1634921
5	12	98	98.34603 0.3460317
6	18	97	97.36508 0.3650794

```

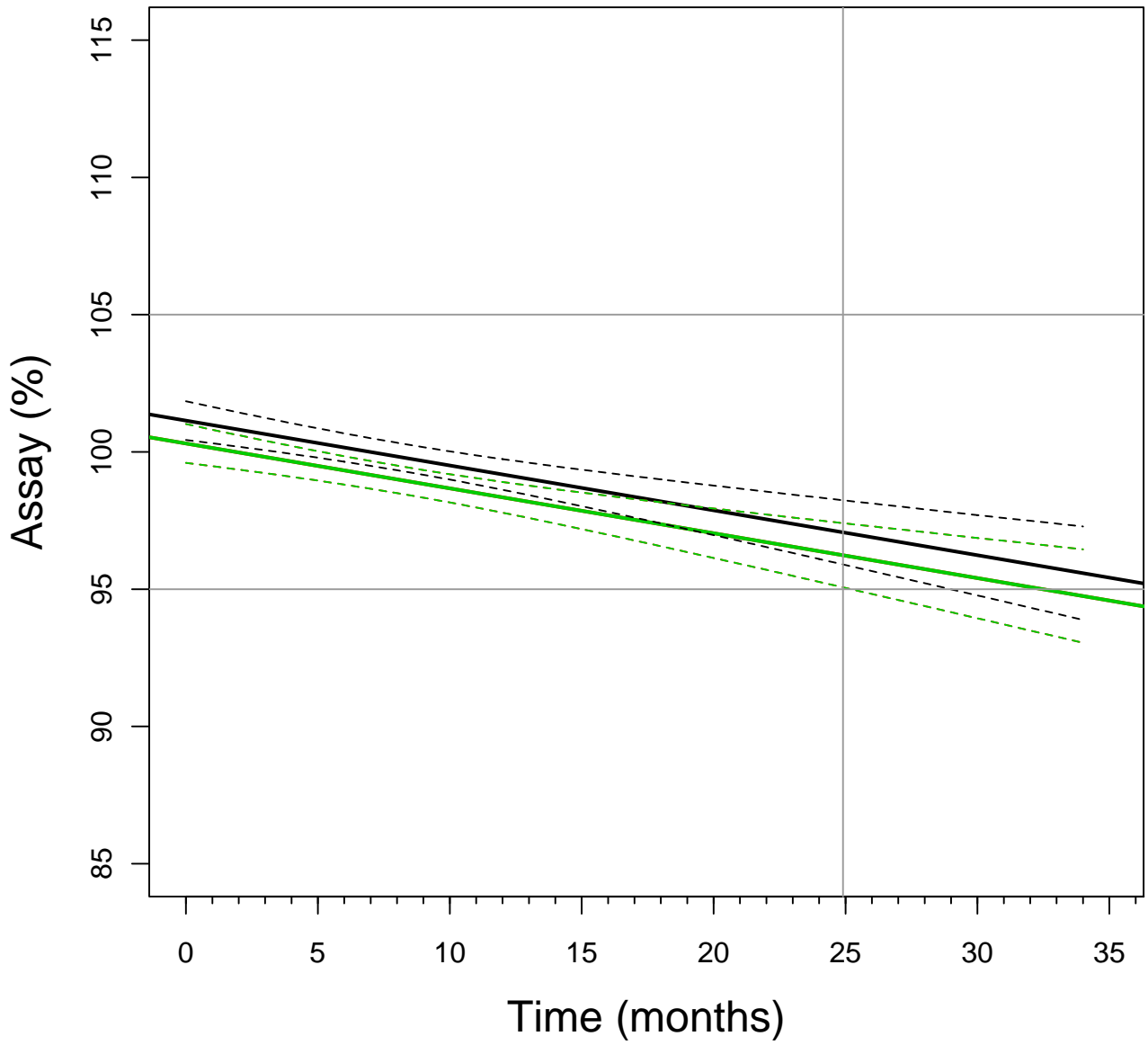
*****
<< Output >>
-----

```

Drug product with lower acceptance criteria of 95 % of label claim
Shelf life = 24.90487 months

```
*****
```

Shelf Life= 24.90 months



SAS
Stability Analysis

TIME	_1	_2	_3
0	100	99	101
3	101	100	100
6	101	100	99
9	100	99	99
12	99	99	98
18	98	97	97

Stability Analysis

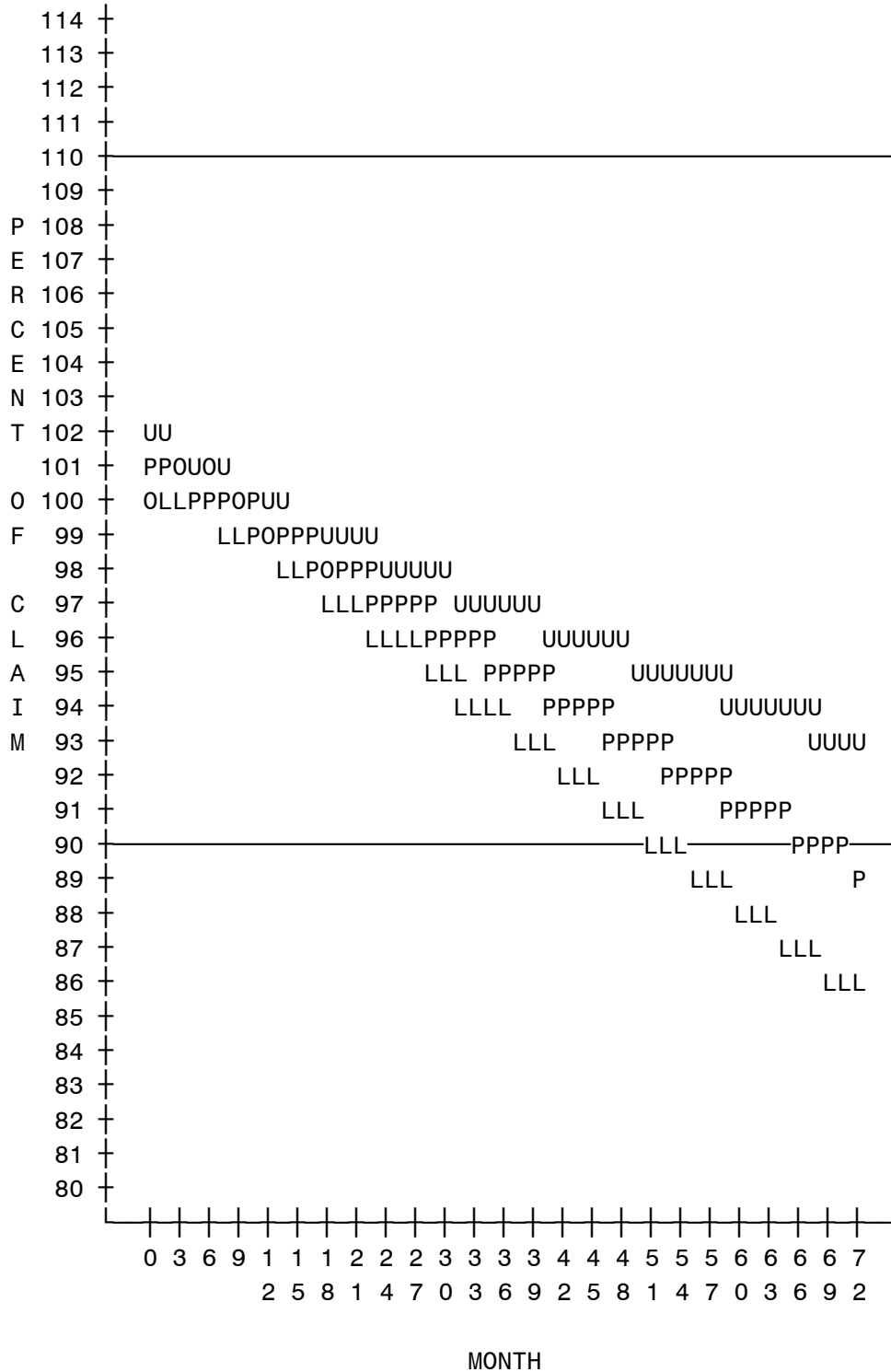
SOURCE	SS	DF	MS	F	P
A	3.63	4	0.91	2.11667	0.14146
B	2.78	2	1.39	3.24074	0.07493
C	0.85	2	0.43	0.99259	0.39910
D	5.14	12	0.43		
E	177429.86	6	29571.64		

```
*****  
* Statistical Analysis: *  
*   Key to sources of variation *  
* A = sep. intercep, sep slope | com intercep, com slope *  
* B = sep. intercep, com slope | com intercep, com slope *  
* C = sep. intercep, sep slope | sep intercep, com slope *  
* D = Residual *  
* E = Full Model *  
*****
```

Stability Analysis

----- BATCH=1 -----

Plot of LEVEL*TIME. Symbol used is 'O'.
Plot of PREDICT*TIME. Symbol used is 'P'.
Plot of L_BOUND*TIME. Symbol used is 'L'.
Plot of U_BOUND*TIME. Symbol used is 'U'.

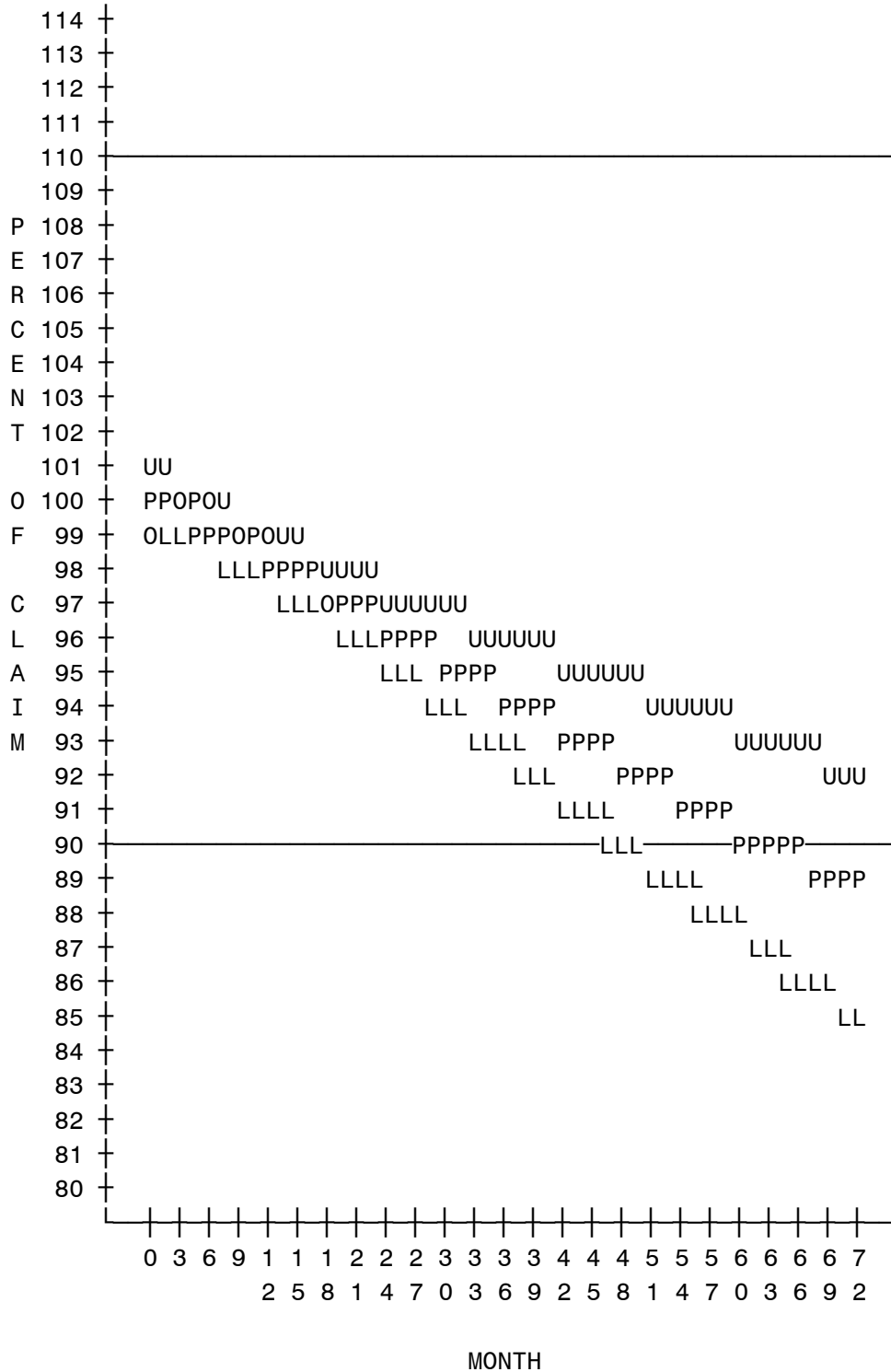


NOTE: 67 obs had missing values. 76 obs hidden.

Stability Analysis

----- BATCH=2 -----

Plot of LEVEL*TIME. Symbol used is 'O'.
Plot of PREDICT*TIME. Symbol used is 'P'.
Plot of L_BOUND*TIME. Symbol used is 'L'.
Plot of U_BOUND*TIME. Symbol used is 'U'.

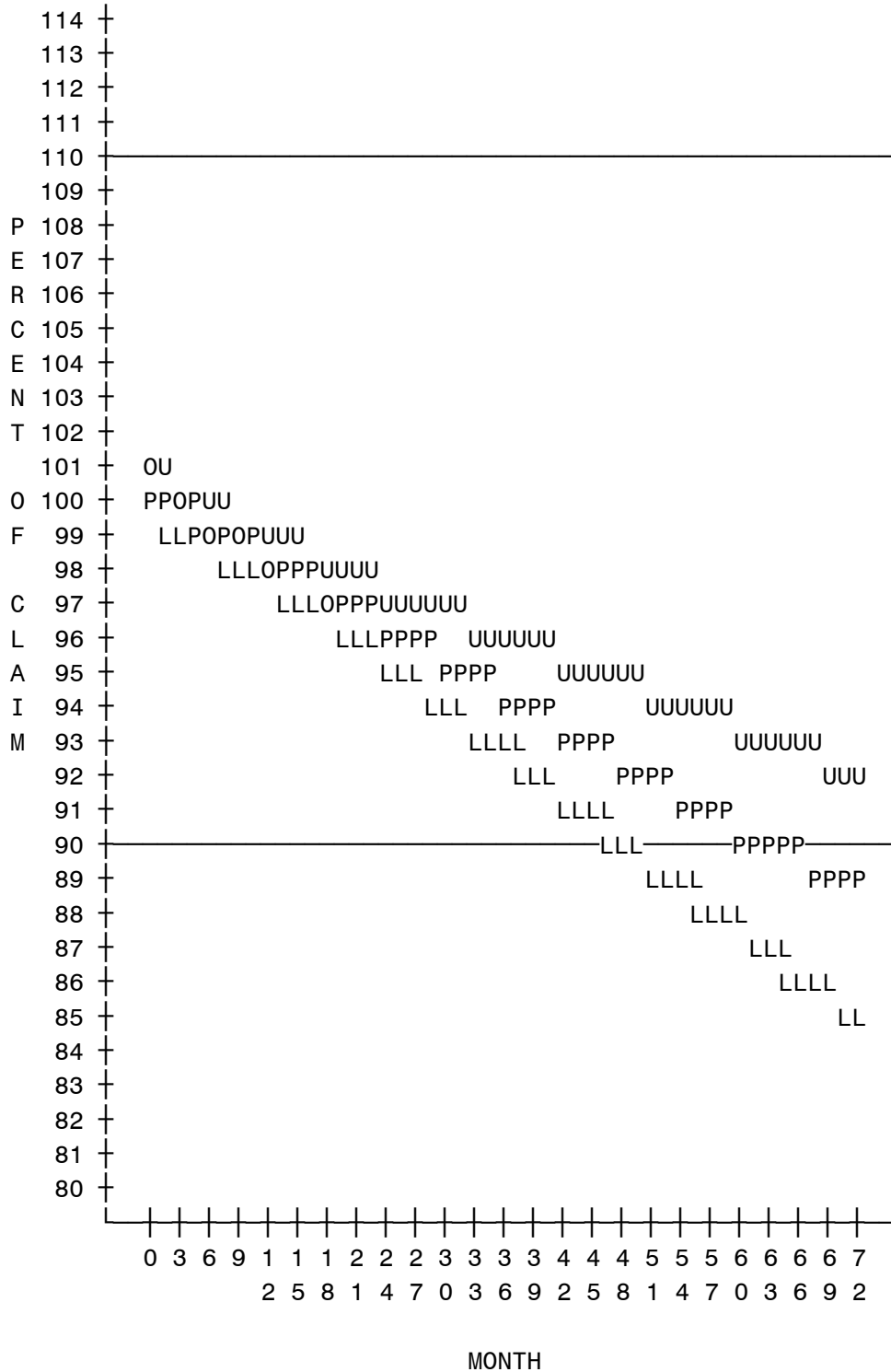


NOTE: 67 obs had missing values. 138 obs hidden.

Stability Analysis

----- BATCH=3 -----

Plot of LEVEL*TIME. Symbol used is 'O'.
Plot of PREDICT*TIME. Symbol used is 'P'.
Plot of L_BOUND*TIME. Symbol used is 'L'.
Plot of U_BOUND*TIME. Symbol used is 'U'.



NOTE: 67 obs had missing values. 204 obs hidden.

Stability Analysis

Batch 1

Fitted Line : $Y = 101.14126984 + -0.163492063 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	100.414	101.141	101.868	0.33889	
1	100.284	100.978	101.672	0.32350	
2	100.150	100.814	101.478	0.30954	
3	100.013	100.651	101.288	0.29722	
4	99.872	100.487	101.102	0.28675	
5	99.727	100.324	100.921	0.27833	
6	99.577	100.160	100.744	0.27216	
7	99.421	99.997	100.572	0.26839	
8	99.260	99.833	100.406	0.26712	
9	99.094	99.670	100.245	0.26839	
10	98.923	99.506	100.090	0.27216	
11	98.746	99.343	99.940	0.27833	
12	98.564	99.179	99.794	0.28675	
13	98.378	99.016	99.653	0.29722	
14	98.188	98.852	99.516	0.30954	
15	97.995	98.689	99.383	0.32350	
16	97.799	98.525	99.252	0.33889	
17	97.599	98.362	99.124	0.35552	
18	97.398	98.198	98.999	0.37324	
19	97.194	98.035	98.875	0.39189	
20	96.989	97.871	98.754	0.41135	
21	96.782	97.708	98.633	0.43151	
22	96.574	97.544	98.514	0.45227	
23	96.365	97.381	98.397	0.47355	
24	96.155	97.217	98.280	0.49530	
25	95.944	97.054	98.164	0.51744	
26	95.732	96.890	98.049	0.53993	
27	95.520	96.727	97.934	0.56274	
28	95.307	96.563	97.820	0.58581	
29	95.094	96.400	97.706	0.60913	
30	94.880	96.237	97.593	0.63266	*
31	94.665	96.073	97.481	0.65638	*
32	94.450	95.910	97.369	0.68028	*
33	94.235	95.746	97.257	0.70432	*
34	94.020	95.583	97.145	0.72851	*
35	93.804	95.419	97.034	0.75283	*
36	93.589	95.256	96.923	0.77725	*
37	93.372	95.092	96.812	0.80178	*
38	93.156	94.929	96.701	0.82641	*
39	92.940	94.765	96.591	0.85112	*
40	92.723	94.602	96.480	0.87591	*

41	92.506	94.438	96.370	0.90077	*
42	92.289	94.275	96.260	0.92570	*
43	92.072	94.111	96.150	0.95069	*
44	91.855	93.948	96.040	0.97573	*
45	91.638	93.784	95.931	1.00083	*
46	91.420	93.621	95.821	1.02598	*

Stability Analysis

Batch 1

Fitted Line : $Y = 101.14126984 + -0.163492063 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	91.2026	93.4571	95.7117	1.05117	*
48	90.9850	93.2937	95.6023	1.07640	*
49	90.7673	93.1302	95.4930	1.10167	*
50	90.5495	92.9667	95.3838	1.12698	*
51	90.3317	92.8032	95.2747	1.15232	*
52	90.1138	92.6397	95.1656	1.17770	*
53	89.8958	92.4762	95.0566	1.20310	*
54	89.6778	92.3127	94.9476	1.22853	*
55	89.4597	92.1492	94.8387	1.25399	*
56	89.2415	91.9857	94.7299	1.27947	*
57	89.0233	91.8222	94.6211	1.30497	*
58	88.8051	91.6587	94.5124	1.33050	*
59	88.5868	91.4952	94.4037	1.35605	*
60	88.3685	91.3317	94.2950	1.38162	*
61	88.1501	91.1683	94.1864	1.40720	*
62	87.9317	91.0048	94.0778	1.43280	*
63	87.7133	90.8413	93.9693	1.45842	*
64	87.4948	90.6778	93.8608	1.48406	*
65	87.2763	90.5143	93.7523	1.50971	*
66	87.0577	90.3508	93.6438	1.53537	*
67	86.8392	90.1873	93.5354	1.56105	*
68	86.6206	90.0238	93.4270	1.58674	*
69	86.4020	89.8603	93.3187	1.61244	*
70	86.1833	89.6968	93.2103	1.63816	*
71	85.9647	89.5333	93.1020	1.66388	*
72	85.7460	89.3698	92.9937	1.68962	*

Stability Analysis

Batch 2

Fitted Line : $Y = 100.30793651 + -0.163492063 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	99.5811	100.308	101.035	0.33889	
1	99.4506	100.144	100.838	0.32350	
2	99.3170	99.981	100.645	0.30954	
3	99.1800	99.817	100.455	0.29722	
4	99.0389	99.654	100.269	0.28675	
5	98.8935	99.490	100.087	0.27833	
6	98.7433	99.327	99.911	0.27216	
7	98.5879	99.163	99.739	0.26839	
8	98.4271	99.000	99.573	0.26712	
9	98.2609	98.837	99.412	0.26839	
10	98.0893	98.673	99.257	0.27216	
11	97.9126	98.510	99.106	0.27833	
12	97.7310	98.346	98.961	0.28675	
13	97.5451	98.183	98.820	0.29722	
14	97.3551	98.019	98.683	0.30954	
15	97.1617	97.856	98.549	0.32350	
16	96.9652	97.692	98.419	0.33889	
17	96.7661	97.529	98.291	0.35552	
18	96.5646	97.365	98.166	0.37324	
19	96.3611	97.202	98.042	0.39189	
20	96.1558	97.038	97.920	0.41135	
21	95.9491	96.875	97.800	0.43151	
22	95.7411	96.711	97.681	0.45227	
23	95.5319	96.548	97.563	0.47355	
24	95.3218	96.384	97.446	0.49530	
25	95.1108	96.221	97.330	0.51744	
26	94.8991	96.057	97.215	0.53993	*
27	94.6867	95.894	97.101	0.56274	*
28	94.4737	95.730	96.987	0.58581	*
29	94.2602	95.567	96.873	0.60913	*
30	94.0463	95.403	96.760	0.63266	*
31	93.8319	95.240	96.647	0.65638	*
32	93.6171	95.076	96.535	0.68028	*
33	93.4021	94.913	96.423	0.70432	*
34	93.1867	94.749	96.312	0.72851	*
35	92.9711	94.586	96.200	0.75283	*
36	92.7552	94.422	96.089	0.77725	*
37	92.5391	94.259	95.978	0.80178	*
38	92.3228	94.095	95.868	0.82641	*
39	92.1063	93.932	95.757	0.85112	*
40	91.8896	93.768	95.647	0.87591	*

41	91.6728	93.605	95.537	0.90077	*
42	91.4558	93.441	95.427	0.92570	*
43	91.2388	93.278	95.317	0.95069	*
44	91.0215	93.114	95.207	0.97573	*
45	90.8042	92.951	95.097	1.00083	*
46	90.5868	92.787	94.988	1.02598	*

Stability Analysis

Batch 2

Fitted Line : $Y = 100.30793651 + -0.163492063 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	90.3693	92.6238	94.8783	1.05117	*
48	90.1517	92.4603	94.7690	1.07640	*
49	89.9340	92.2968	94.6597	1.10167	*
50	89.7162	92.1333	94.5505	1.12698	*
51	89.4984	91.9698	94.4413	1.15232	*
52	89.2804	91.8063	94.3323	1.17770	*
53	89.0625	91.6429	94.2232	1.20310	*
54	88.8444	91.4794	94.1143	1.22853	*
55	88.6263	91.3159	94.0054	1.25399	*
56	88.4082	91.1524	93.8966	1.27947	*
57	88.1900	90.9889	93.7878	1.30497	*
58	87.9718	90.8254	93.6790	1.33050	*
59	87.7535	90.6619	93.5703	1.35605	*
60	87.5351	90.4984	93.4617	1.38162	*
61	87.3168	90.3349	93.3531	1.40720	*
62	87.0984	90.1714	93.2445	1.43280	*
63	86.8799	90.0079	93.1359	1.45842	*
64	86.6615	89.8444	93.0274	1.48406	*
65	86.4430	89.6810	92.9190	1.50971	*
66	86.2244	89.5175	92.8105	1.53537	*
67	86.0059	89.3540	92.7021	1.56105	*
68	85.7873	89.1905	92.5937	1.58674	*
69	85.5686	89.0270	92.4853	1.61244	*
70	85.3500	88.8635	92.3770	1.63816	*
71	85.1313	88.7000	92.2687	1.66388	*
72	84.9126	88.5365	92.1604	1.68962	*

Stability Analysis

Batch 3

Fitted Line : Y = 100.30793651 + -0.163492063 X

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	99.5811	100.308	101.035	0.33889	
1	99.4506	100.144	100.838	0.32350	
2	99.3170	99.981	100.645	0.30954	
3	99.1800	99.817	100.455	0.29722	
4	99.0389	99.654	100.269	0.28675	
5	98.8935	99.490	100.087	0.27833	
6	98.7433	99.327	99.911	0.27216	
7	98.5879	99.163	99.739	0.26839	
8	98.4271	99.000	99.573	0.26712	
9	98.2609	98.837	99.412	0.26839	
10	98.0893	98.673	99.257	0.27216	
11	97.9126	98.510	99.106	0.27833	
12	97.7310	98.346	98.961	0.28675	
13	97.5451	98.183	98.820	0.29722	
14	97.3551	98.019	98.683	0.30954	
15	97.1617	97.856	98.549	0.32350	
16	96.9652	97.692	98.419	0.33889	
17	96.7661	97.529	98.291	0.35552	
18	96.5646	97.365	98.166	0.37324	
19	96.3611	97.202	98.042	0.39189	
20	96.1558	97.038	97.920	0.41135	
21	95.9491	96.875	97.800	0.43151	
22	95.7411	96.711	97.681	0.45227	
23	95.5319	96.548	97.563	0.47355	
24	95.3218	96.384	97.446	0.49530	
25	95.1108	96.221	97.330	0.51744	
26	94.8991	96.057	97.215	0.53993	*
27	94.6867	95.894	97.101	0.56274	*
28	94.4737	95.730	96.987	0.58581	*
29	94.2602	95.567	96.873	0.60913	*
30	94.0463	95.403	96.760	0.63266	*
31	93.8319	95.240	96.647	0.65638	*
32	93.6171	95.076	96.535	0.68028	*
33	93.4021	94.913	96.423	0.70432	*
34	93.1867	94.749	96.312	0.72851	*
35	92.9711	94.586	96.200	0.75283	*
36	92.7552	94.422	96.089	0.77725	*
37	92.5391	94.259	95.978	0.80178	*
38	92.3228	94.095	95.868	0.82641	*
39	92.1063	93.932	95.757	0.85112	*
40	91.8896	93.768	95.647	0.87591	*

41	91.6728	93.605	95.537	0.90077	*
42	91.4558	93.441	95.427	0.92570	*
43	91.2388	93.278	95.317	0.95069	*
44	91.0215	93.114	95.207	0.97573	*
45	90.8042	92.951	95.097	1.00083	*
46	90.5868	92.787	94.988	1.02598	*

Stability Analysis

Batch 3

Fitted Line : $Y = 100.30793651 + -0.163492063 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	90.3693	92.6238	94.8783	1.05117	*
48	90.1517	92.4603	94.7690	1.07640	*
49	89.9340	92.2968	94.6597	1.10167	*
50	89.7162	92.1333	94.5505	1.12698	*
51	89.4984	91.9698	94.4413	1.15232	*
52	89.2804	91.8063	94.3323	1.17770	*
53	89.0625	91.6429	94.2232	1.20310	*
54	88.8444	91.4794	94.1143	1.22853	*
55	88.6263	91.3159	94.0054	1.25399	*
56	88.4082	91.1524	93.8966	1.27947	*
57	88.1900	90.9889	93.7878	1.30497	*
58	87.9718	90.8254	93.6790	1.33050	*
59	87.7535	90.6619	93.5703	1.35605	*
60	87.5351	90.4984	93.4617	1.38162	*
61	87.3168	90.3349	93.3531	1.40720	*
62	87.0984	90.1714	93.2445	1.43280	*
63	86.8799	90.0079	93.1359	1.45842	*
64	86.6615	89.8444	93.0274	1.48406	*
65	86.4430	89.6810	92.9190	1.50971	*
66	86.2244	89.5175	92.8105	1.53537	*
67	86.0059	89.3540	92.7021	1.56105	*
68	85.7873	89.1905	92.5937	1.58674	*
69	85.5686	89.0270	92.4853	1.61244	*
70	85.3500	88.8635	92.3770	1.63816	*
71	85.1313	88.7000	92.2687	1.66388	*
72	84.9126	88.5365	92.1604	1.68962	*

Stability Analysis

95% Two-Sided Confidence Limit

Separate Intercepts and Common Slope

BATCH	ESTIMATED DATING PERIOD (MONTHS/WEEKS)
1	29
2	25
3	25

```

batch time assay...
1      1      0      100
2      1      1      101
3      1      2      102
4      1      3      101
5      1      6      103
6      1      9      105
7      1     12      103
8      2      0      102
9      2      1       99
10     2      2       98
11     2      3       98
12     2      6       97
13     2      9       98
14     2     12       97
15     3      0      100
16     3      1       97
17     3      2       97
18     3      3       96
19     3      6       98
20     3      9       97
21     3     12       96

```

```

*****
*                               Now, Go to analyze the data                               *
*****

```

Drug product with upper acceptance criteria of ____ % of label claim
1: 110

Drug product with lower acceptance criteria of ____ % of label claim
1: 90

<<Output: ANCOVA model: batch vs. time vs. assay (%)>>

Analysis of Variance Table

```

Response: assay
      Df Sum Sq Mean Sq F value    Pr(>F)
batch    2  90.286   45.143  28.2016 8.276e-06 ***
time     1   0.493    0.493   0.3078 0.587230
batch:time 2  21.497   10.748   6.7147 0.008269 **
Residuals 15  24.011    1.601

```

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

```

-----
<< ANCOVA Output: Testing for poolability of batches >>
-----

```

The test rejects the hypothesis of equality of slopes (there is a significant difference in slopes among batches).

<<Suggestion>>

It is not considered appropriate to combine the data from all batches.

<<Output: linear regression model: time vs. assay (%)>>

```

Call:
lm(formula = assay ~ batch * time, data = ANCOVAdata)

```

```

Coefficients:
(Intercept)      batch2      batch3      time  batch2:time
  100.7105      -1.0526      -2.6974      0.3038      -0.5646
batch3:time
  -0.4581

```

Analysis of Variance Table

Response: assay

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
batch	2	90.286	45.143	28.2016	8.276e-06 ***
time	1	0.493	0.493	0.3078	0.587230
batch:time	2	21.497	10.748	6.7147	0.008269 **
Residuals	15	24.011	1.601		

 Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

<< Output and Linear regression model>>

Y= 100.7105 +(0.3038278) X

	time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	100	100.7105	0.71052632
2	1	101	101.0144	0.01435407
3	2	102	101.3182	-0.68181818
4	3	101	101.6220	0.62200957
5	6	103	102.5335	-0.46650718
6	9	105	103.4450	-1.55502392
7	12	103	104.3565	1.35645933

<< Output and Linear regression model>>

Y= 99.6579 +(-0.2607656) X

	time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	102	99.65789	-2.3421053
2	1	99	99.39713	0.3971292
3	2	98	99.13636	1.1363636
4	3	98	98.87560	0.8755981
5	6	97	98.09330	1.0933014
6	9	98	97.31100	-0.6889952
7	12	97	96.52871	-0.4712919

<< Output and Linear regression model>>

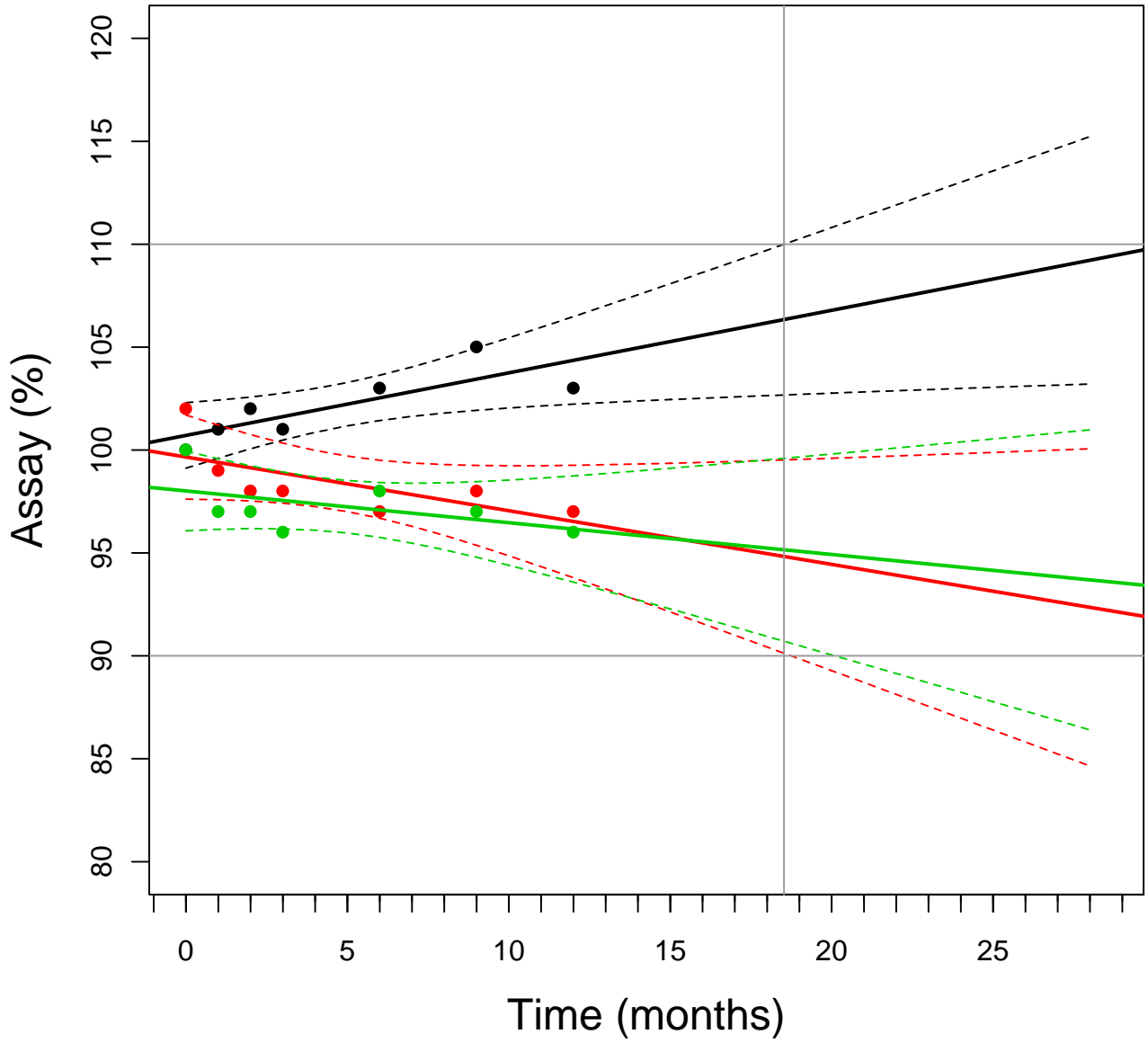
Y= 98.01316 +(-0.1543062) X

	time	Observed assay(%)	Calculated assay(%)	Residuals
1	0	100	98.01316	-1.9868421
2	1	97	97.85885	0.8588517
3	2	97	97.70455	0.7045455
4	3	96	97.55024	1.5502392
5	6	98	97.08732	-0.9126794
6	9	97	96.62440	-0.3755981
7	12	96	96.16148	0.1614833

 << Output >>

Drug product with Upper acceptance criteria of 110 % of label claim
Shelf life = 18.52295 months

Shelf Life= 18.52 months



SAS
Stability Analysis

TIME	_1	_2	_3
0	100	102	100
1	101	99	97
2	102	98	97
3	101	98	96
6	103	97	98
9	105	98	97
12	103	97	96

Stability Analysis

SOURCE	SS	DF	MS	F	P
A	111.78	4	27.95	17.4582	.0000163
B	90.29	2	45.14	28.2016	.0000083
C	21.50	2	10.75	6.7147	.0082686
D	24.01	15	1.60		
E	207122.99	6	34520.50		

```

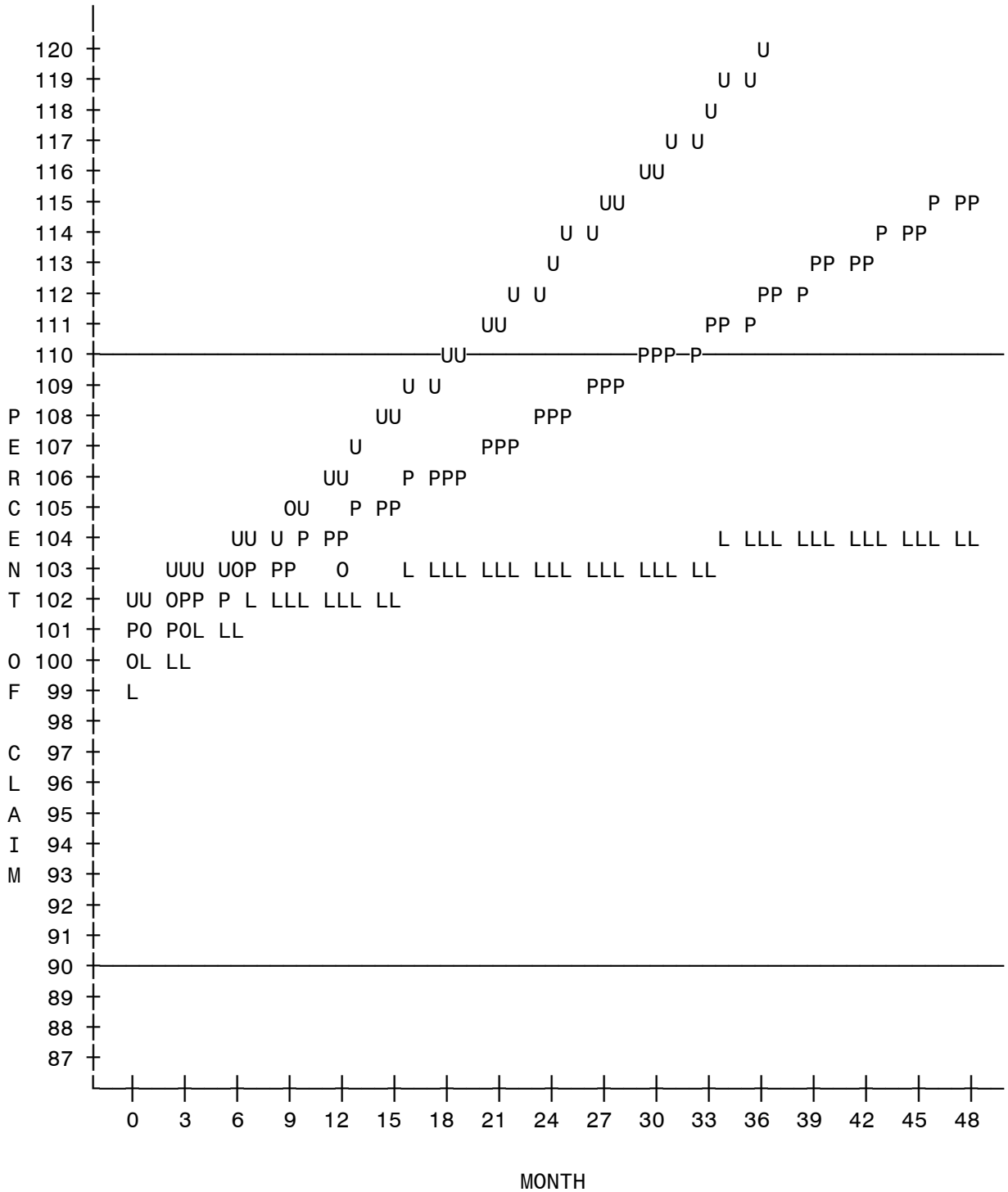
*****
* Statistical Analysis: *
*   Key to sources of variation *
* A = sep. intercep, sep slope | com intercep, com slope *
* B = sep. intercep, com slope | com intercep, com slope *
* C = sep. intercep, sep slope | sep intercep, com slope *
* D = Residual *
* E = Full Model *
*****

```

Stability Analysis

BATCH=1

Plot of LEVEL*TIME. Symbol used is 'O'.
 Plot of PREDICT*TIME. Symbol used is 'P'.
 Plot of L_BOUND*TIME. Symbol used is 'L'.
 Plot of U_BOUND*TIME. Symbol used is 'U'.

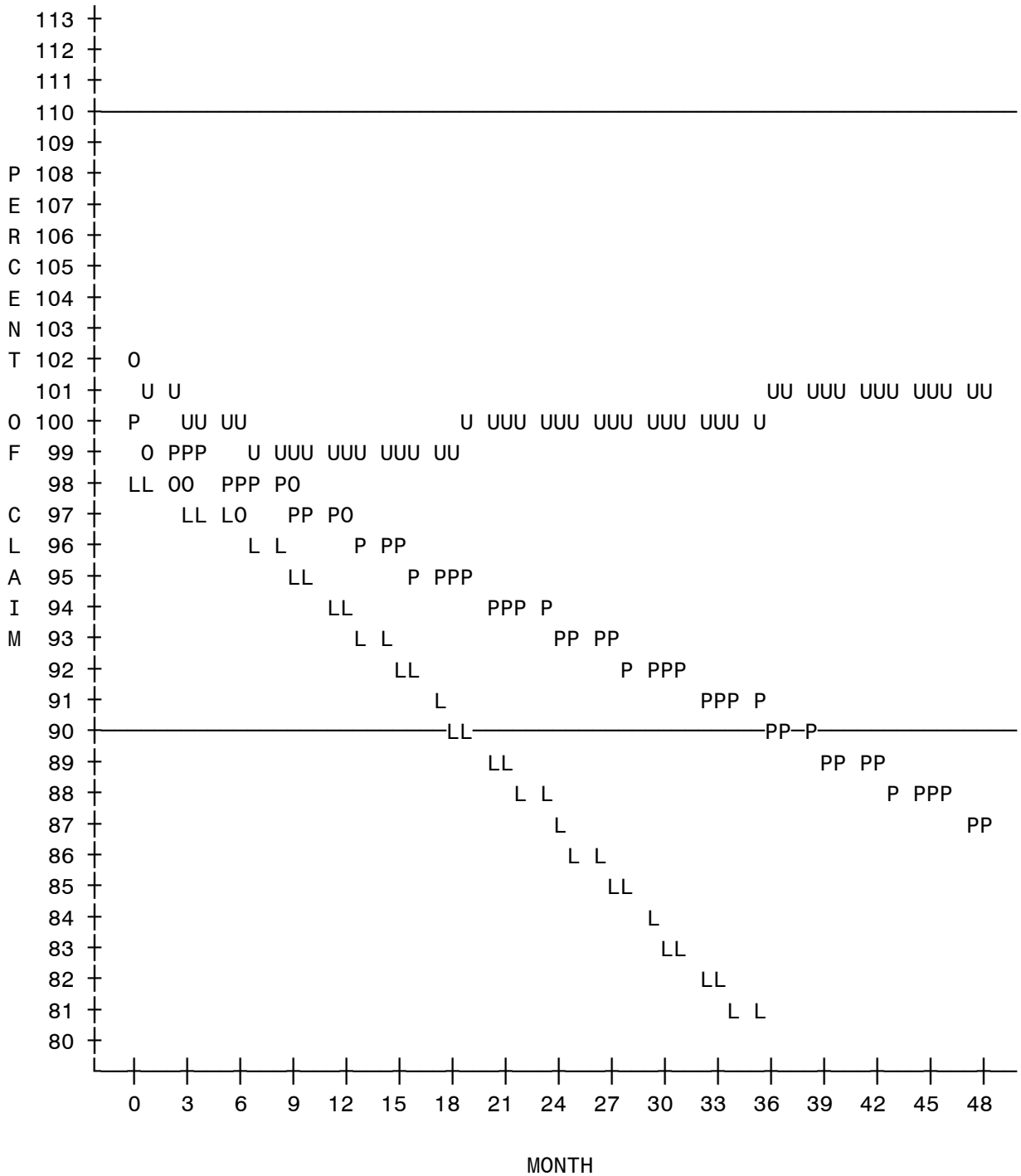


NOTE: 42 obs had missing values. 3 obs hidden. 12 obs were out of range.

Stability Analysis

----- BATCH=2 -----

Plot of LEVEL*TIME. Symbol used is 'O'.
Plot of PREDICT*TIME. Symbol used is 'P'.
Plot of L_BOUND*TIME. Symbol used is 'L'.
Plot of U_BOUND*TIME. Symbol used is 'U'.

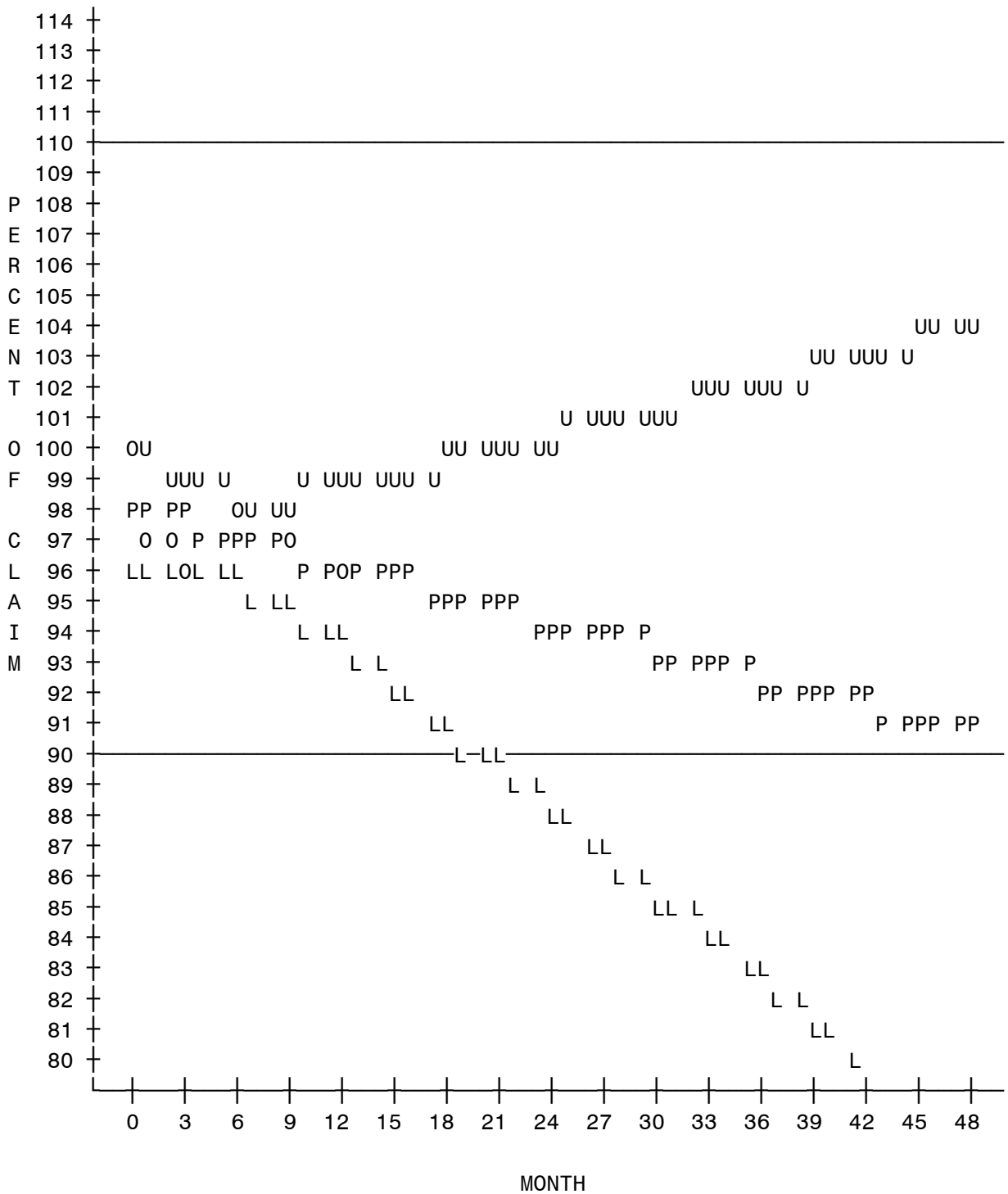


NOTE: 42 obs had missing values. 5 obs hidden. 25 obs were out of range.

Stability Analysis

----- BATCH=3 -----

Plot of LEVEL*TIME. Symbol used is 'O'.
Plot of PREDICT*TIME. Symbol used is 'P'.
Plot of L_BOUND*TIME. Symbol used is 'L'.
Plot of U_BOUND*TIME. Symbol used is 'U'.



NOTE: 42 obs had missing values. 5 obs hidden. 32 obs were out of range.

Stability Analysis

Batch 1

Fitted Line : $Y = 100.71052632 + 0.3038277512 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	99.118	100.711	102.303	0.61945	
1	99.603	101.014	102.426	0.54899	
2	100.063	101.318	102.574	0.48847	
3	100.486	101.622	102.758	0.44198	
4	100.861	101.926	102.991	0.41428	
5	101.178	102.230	103.282	0.40920	
6	101.434	102.533	103.633	0.42754	
7	101.638	102.837	104.037	0.46656	
8	101.800	103.141	104.482	0.52162	
9	101.933	103.445	104.957	0.58826	
10	102.045	103.749	105.453	0.66297	
11	102.142	104.053	105.963	0.74334	
12	102.229	104.356	106.484	0.82772	
13	102.308	104.660	107.012	0.91499	
14	102.382	104.964	107.546	1.00441	
15	102.452	105.268	108.084	1.09544	
16	102.519	105.572	108.625	1.18773	
17	102.583	105.876	109.168	1.28099	
18	102.645	106.179	109.714	1.37502	
19	102.705	106.483	110.261	1.46969	*
20	102.764	106.787	110.810	1.56487	*
21	102.823	107.091	111.359	1.66048	*
22	102.880	107.395	111.910	1.75645	*
23	102.936	107.699	112.461	1.85271	*
24	102.992	108.002	113.013	1.94924	*
25	103.047	108.306	113.566	2.04598	*
26	103.102	108.610	114.119	2.14291	*
27	103.156	108.914	114.672	2.24001	*
28	103.210	109.218	115.226	2.33726	*
29	103.263	109.522	115.780	2.43463	*
30	103.316	109.825	116.334	2.53211	*
31	103.369	110.129	116.889	2.62970	*
32	103.422	110.433	117.444	2.72738	*
33	103.475	110.737	117.999	2.82513	*
34	103.527	111.041	118.554	2.92296	*
35	103.579	111.344	119.110	3.02085	*
36	103.631	111.648	119.665	3.11880	*
37	103.683	111.952	120.221	3.21681	*
38	103.735	112.256	120.777	3.31487	*
39	103.787	112.560	121.333	3.41296	*
40	103.838	112.864	121.889	3.51111	*

41	103.890	113.167	122.445	3.60928	*
42	103.941	113.471	123.002	3.70750	*
43	103.992	113.775	123.558	3.80574	*
44	104.043	114.079	124.115	3.90402	*
45	104.094	114.383	124.671	4.00232	*
46	104.146	114.687	125.228	4.10064	*

Stability Analysis

Batch 1

Fitted Line : $Y = 100.71052632 + 0.3038277512 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	104.197	114.990	125.784	4.19900	*
48	104.248	115.294	126.341	4.29737	*

Stability Analysis

Batch 2

Fitted Line : $Y = 99.657894737 + -0.26076555 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	97.6157	99.6579	101.700	0.79444	
1	97.5872	99.3971	101.207	0.70408	
2	97.5260	99.1364	100.747	0.62646	
3	97.4185	98.8756	100.333	0.56684	
4	97.2491	98.6148	99.981	0.53131	
5	97.0050	98.3541	99.703	0.52479	
6	96.6838	98.0933	99.503	0.54832	
7	96.2944	97.8325	99.371	0.59835	
8	95.8521	97.5718	99.291	0.66898	
9	95.3717	97.3110	99.250	0.75443	
10	94.8646	97.0502	99.236	0.85026	
11	94.3389	96.7895	99.240	0.95333	
12	93.7999	96.5287	99.257	1.06154	
13	93.2514	96.2679	99.284	1.17347	
14	92.6959	96.0072	99.318	1.28815	
15	92.1350	95.7464	99.358	1.40490	
16	91.5700	95.4856	99.401	1.52325	
17	91.0018	95.2249	99.448	1.64286	
18	90.4310	94.9641	99.497	1.76346	
19	89.8581	94.7033	99.549	1.88487	*
20	89.2836	94.4426	99.602	2.00694	*
21	88.7076	94.1818	99.656	2.12956	*
22	88.1305	93.9211	99.712	2.25263	*
23	87.5523	93.6603	99.768	2.37609	*
24	86.9734	93.3995	99.826	2.49988	*
25	86.3937	93.1388	99.884	2.62396	*
26	85.8133	92.8780	99.943	2.74827	*
27	85.2325	92.6172	100.002	2.87280	*
28	84.6511	92.3565	100.062	2.99752	*
29	84.0693	92.0957	100.122	3.12240	*
30	83.4872	91.8349	100.183	3.24742	*
31	82.9047	91.5742	100.244	3.37257	*
32	82.3219	91.3134	100.305	3.49784	*
33	81.7389	91.0526	100.366	3.62321	*
34	81.1556	90.7919	100.428	3.74868	*
35	80.5721	90.5311	100.490	3.87422	*
36	79.9884	90.2703	100.552	3.99985	*
37	79.4045	90.0096	100.615	4.12554	*
38	78.8205	89.7488	100.677	4.25129	*
39	78.2363	89.4880	100.740	4.37711	*
40	77.6520	89.2273	100.803	4.50297	*

41	77.0676	88.9665	100.865	4.62888	*
42	76.4830	88.7057	100.928	4.75484	*
43	75.8984	88.4450	100.992	4.88084	*
44	75.3136	88.1842	101.055	5.00688	*
45	74.7288	87.9234	101.118	5.13295	*
46	74.1439	87.6627	101.182	5.25905	*

Stability Analysis

Batch 2

Fitted Line : $Y = 99.657894737 + -0.26076555 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	73.5589	87.4019	101.245	5.38519	*
48	72.9738	87.1411	101.309	5.51135	*

Stability Analysis

Batch 3

Fitted Line : $Y = 98.013157895 + -0.15430622 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
0	96.0813	98.0132	99.945	0.75153	
1	96.1467	97.8589	99.571	0.66605	
2	96.1812	97.7045	99.228	0.59262	
3	96.1718	97.5502	98.929	0.53622	
4	96.1039	97.3959	98.688	0.50261	
5	95.9655	97.2416	98.518	0.49644	
6	95.7540	97.0873	98.421	0.51870	
7	95.4780	96.9330	98.388	0.56603	
8	95.1519	96.7787	98.405	0.63285	
9	94.7898	96.6244	98.459	0.71368	
10	94.4025	96.4701	98.538	0.80433	
11	93.9975	96.3158	98.634	0.90184	
12	93.5801	96.1615	98.743	1.00420	
13	93.1536	96.0072	98.861	1.11008	
14	92.7204	95.8529	98.985	1.21857	
15	92.2822	95.6986	99.115	1.32901	
16	91.8401	95.5443	99.248	1.44097	
17	91.3950	95.3900	99.385	1.55412	
18	90.9474	95.2356	99.524	1.66821	
19	90.4978	95.0813	99.665	1.78306	
20	90.0467	94.9270	99.807	1.89854	
21	89.5942	94.7727	99.951	2.01453	*
22	89.1406	94.6184	100.096	2.13096	*
23	88.6861	94.4641	100.242	2.24775	*
24	88.2308	94.3098	100.389	2.36485	*
25	87.7747	94.1555	100.536	2.48222	*
26	87.3181	94.0012	100.684	2.59983	*
27	86.8610	93.8469	100.833	2.71763	*
28	86.4034	93.6926	100.982	2.83561	*
29	85.9454	93.5383	101.131	2.95374	*
30	85.4871	93.3840	101.281	3.07201	*
31	85.0285	93.2297	101.431	3.19041	*
32	84.5695	93.0754	101.581	3.30891	*
33	84.1104	92.9211	101.732	3.42751	*
34	83.6510	92.7667	101.883	3.54619	*
35	83.1914	92.6124	102.034	3.66496	*
36	82.7316	92.4581	102.185	3.78380	*
37	82.2716	92.3038	102.336	3.90270	*
38	81.8115	92.1495	102.488	4.02166	*
39	81.3513	91.9952	102.639	4.14068	*
40	80.8909	91.8409	102.791	4.25974	*

41	80.4304	91.6866	102.943	4.37886	*
42	79.9698	91.5323	103.095	4.49801	*
43	79.5091	91.3780	103.247	4.61720	*
44	79.0483	91.2237	103.399	4.73643	*
45	78.5874	91.0694	103.551	4.85569	*
46	78.1265	90.9151	103.704	4.97498	*

Stability Analysis

Batch 3

Fitted Line : $Y = 98.013157895 + -0.15430622 X$

TIME	L_BOUND	PREDICT	U_BOUND	STD_ERR	EXP
47	77.6654	90.7608	103.856	5.09431	*
48	77.2043	90.6065	104.009	5.21365	*

Stability Analysis

95% Two-Sided Confidence Limit

Separate Intercepts and Separate Slopes

BATCH	ESTIMATED DATING PERIOD (MONTHS/WEEKS)
1	18
2	18
3	20