Improper Laboratory Practice:

An Ounce of Prevention …

Data of Known and Documented Quality

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Office of Superfund Remediation and Technology Innovation

Agenda

- Introduction
- Improper Practices / Examples
- Warning Signs
- Cracks in the System
- The Path Forward

Contract Laboratory Program

Highlights

- Established Infrastructure with Experienced Staff and Support Contractors
- Detailed Analytical Statements of Work
- Comprehensive Quality Assurance Program
- Automated Scheduling
- Standardized Data Reporting Formats
- Electronic Data Assessment
- Manual Data Validation Usability Review

Contract Laboratory Program

Performance Monitoring

- SOPs, QMPs, MDLs
- Key Personnel
- Periodic PT Samples
- Annual On-site Audits
- Electronic Data Package Audits
- Level 4 Data Review

Terminology?

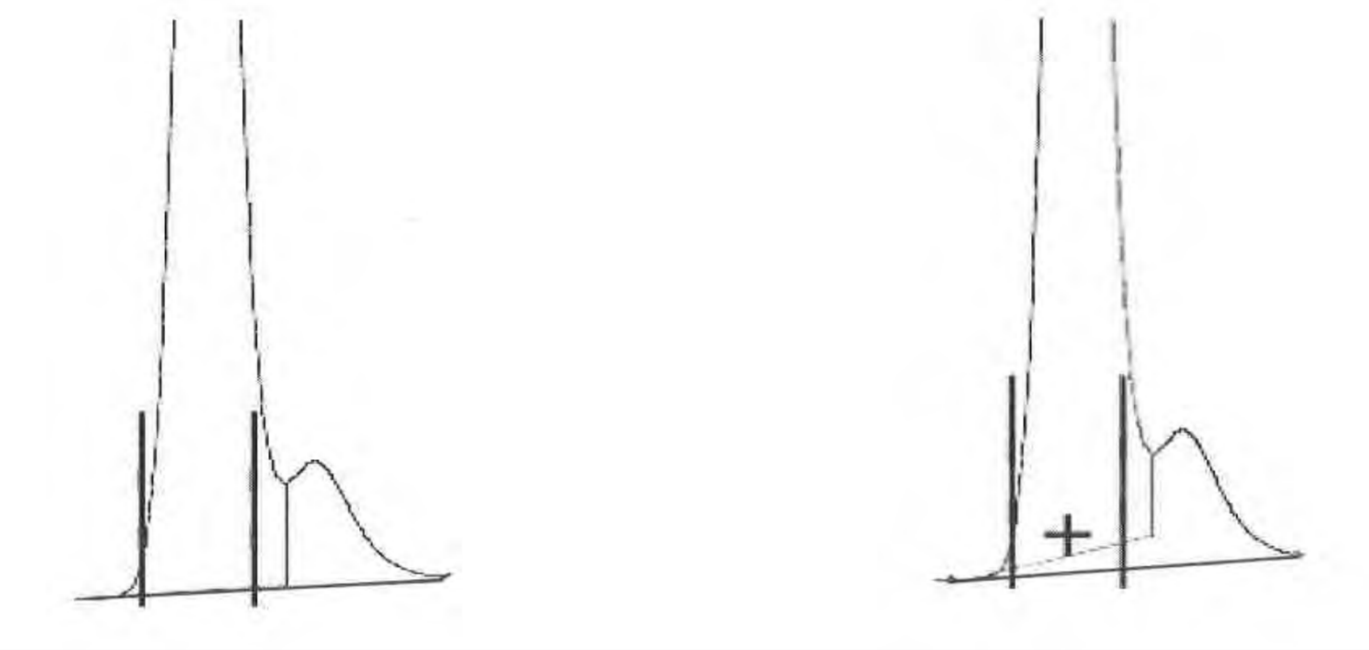
Improper Practices

Inappropriate Practices

Fraudulent Practices

Omitting points from calibration curve, Putting pH paper in sample, Leaving VOA sample open in lab, Manual Integration, Not digesting waters in metals analysis, Forgetting to add reagents, Incomplete Documentation,

Examples of Improper Manual Integration



Delta BHC

12.45 min 24.641 ng/ml

response = 45837737

%D = 23.2

Delta BHC

12.45 min 23.915 ng/ml m

response = 44486890

%D = 19.6%

Improper Manual Integration

Printed from Laboratory-provided audit trail file

23,290 22.390

Modified : Tue Feb 12 14:35:46 2013
Event : Manual Integration
Message : Changed peak amount for delta-BHC #2 from 24.6412ng/mL to 24.4597ng/mL
QuantFile: PESTC0046955.RES
Severity : 1

1

21,490

Modified : Tue Feb 12 14:35:51 2013
Event : Manual Integration
Message : Changed peak amount for delta-BHC #2 from 24.4597ng/mL to 24.275ng/mL
QuantFile: PESTC0046955.RES
Severity : 1

2

20,490

Modified : Tue Feb 12 14:35:55 2013
Event : Manual Integration
Message : Changed peak amount for delta-BHC #2 from 24.275ng/mL to 24.0887ng/mL
QuantFile: PESTC0046955.RES
Severity : 1

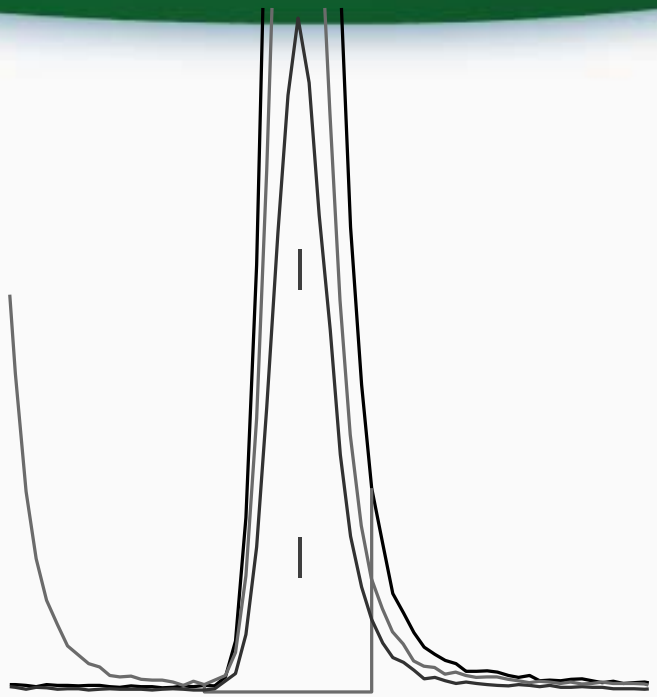
3

19,690

Modified : Tue Feb 12 14:35:58 2013
Event : Manual Integration
Message : Changed peak amount for delta-BHC #2 from 24.0887ng/mL to 23.915ng/mL
QuantFile: PESTC0046955.RES
Severity : 1

4

Examples of Improper Manual Integration



DMC-10

8.074 min 6.0 ug/L

response = 2340648

%D = 20.0

DMC-10

8.074 min 6.27 ug/L

response = 2444809

%D = 20.9%

Example of Time Travel

Obtained from hard-copy data package

```
                                G003493 epatemp
                               Quantitation Report     (QT Reviewed)

Data File : \\Inst\GCMS\G-5973.net\DATA\G003493.d          Vial: 22
Acq On    : 19 Apr 2013  14:34                         Operator: RS
Sample    : 3D18004-CCV3                               Inst    : G-5973
Misc      : SSTD0208P                                  Multiplr: 1.00
DataAcq Meth:CLPACQ.M

Quant Time: Apr 19 13:58:21 2013
Quant Results File: SOMG003493.RES
Integration File: RTEINT.P
Quant Method : \\Inst\gcms\G-5973.net\METHOD\SOMG003493.M
Quant Title  : CLP SOM1.2  BNA Calibration
QLast Update : Fri Apr 19 14:29:19 2013
Response via : Initial Calibration
```

Example of Time Travel

C010212 epatemp
Quantitation Report (QT Reviewed)

Data File : \\Inst\gcms\C-5973.net\DATA\C010212.d Vial: 8
Acq On : 2 Jun 2013 16:03 Operator: SP
Sample : 3F04004-CAL5 Inst : C-5973
Misc : VSTD10057 5GM SOIL Multiplr: 1.00
DataAcq Meth:VOAC.M

Quant Time: Jun 06 17:21:25 2013
Quant Results File: SOSC010207.RES
Integration File: Rteint.p
Quant Method : \\Inst\gcms\C-5973.net\METHOD\SOSC010207.M
Quant Title : CLP SOM1.2-VOA-SOIL- 5GM Heated Purge
QLast Update : Thu Jun 06 17:21:16 2013
Response via : Initial Calibration

Obtained from hard copy data package

C010212 audit
\\Inst\gcms\C-5973.net\DATA\C010212.d\audit.txt
Created Fri May 31 16:09:20 2013

Modified : Fri May 31 16:09:20 2013
Event : Quantitation
Message : Calculation using initial calibration
QuantFile: SOSC010207.RES
Severity : 0

Printed from the laboratory-submitted audit trail file

Modified : Fri May 31 16:09:23 2013

Improper Inorganic Practices

Analyte	True	Found	%D	True	Found	%D	True	Found	%D
Aluminum	0.0	1.2		2.0	2.0	2	20.0	19.6	-2
Antimony	0.0	0.036		0.20	0.19	-7	2.0	1.8	-8
Arsenic	0.0	0.013		0.10	0.098	-2	1.0	0.97	-3
Barium	0.0	0.039		1.0	0.89	-11	10.0	9.3	-7
Beryllium	0.0	0.0020		0.10	0.084	-16	1.0	0.96	-4
Cadmium	0.0	-0.0010		0.10	0.11	6	1.0	1.0	3
Calcium	0.0	14.2					100	99.2	-1
Chromium	0.0	0.25					2.0	2.0	-1
Cobalt	0.0	0.015		0.10	0.10	0	1.0	0.90	-11
Copper	0.0	0.0020		0.20	0.18	-13	2.0	1.9	-5
Iron	0.0	2.6		10.0	9.5	-5	100	95.7	-4
Lead	0.0	0.012		0.10	0.095	-5	1.0	0.97	-3
Magnesium	0.0	2.3		10.0	9.7	-3	100	95.7	-4
Manganese	0.0	-0.0050		0.10	0.10	2			
Nickel	0.0	0.034					1.0	1.0	0
Potassium	0.0	-16					100	102	2

Warning Signs – Organic Data

- Multiple manual integrations performed on QC samples or calibrations
- Manual Integration in lieu of instrument maintenance
  - Poor peak shapes
  - Calibration Outliers
  - GC/MS tune issues
- Inappropriate manual integrations: peak shaving and enhancement
- Unnecessary baseline adjustment

Warning Signs – Inorganic Data

- Interference Check Sample outliers
- Time gaps in calibration sequences
- Poor precision caused by dirty systems
- Running rinse blanks before QC samples
- Expired standards or gaps in traceability
- Apparent transcription errors
- Improper practices

Cracks in the Quality System

- Poor hiring decisions,
- Failure to complete or document training,
- Lack of cross-training,
- Lax documentation in sample receiving,
- Lax Quality System,
- Missed SOP updates,
- Missed internal audits

Incentivizing Good Behavior

Plan for Success

1. Robust Quality System (https://www.epa.gov/quality)
2. Well Documented Training
3. Independent QA Officer
4. Performance Testing
5. External Certifications

Incentivizing Good Behavior

Follow Through

1. Hire qualified staff
2. Train Thoroughly
3. Communicate Effectively
4. Establish Good Corporate Culture
5. Certification for Analytical Chemists and Technicians?

Thank you!

Questions

Thank you!

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Shari Myer, Analytical Services Branch Chief
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Bibliography

- http://www.epa.gov/quality
- “Leadership is an Art”, Max DePree, 1989, Dell Publishing
- “The 360 Degree Leader”, John C. Maxwell, 2005, Thomas Nelson, Inc.

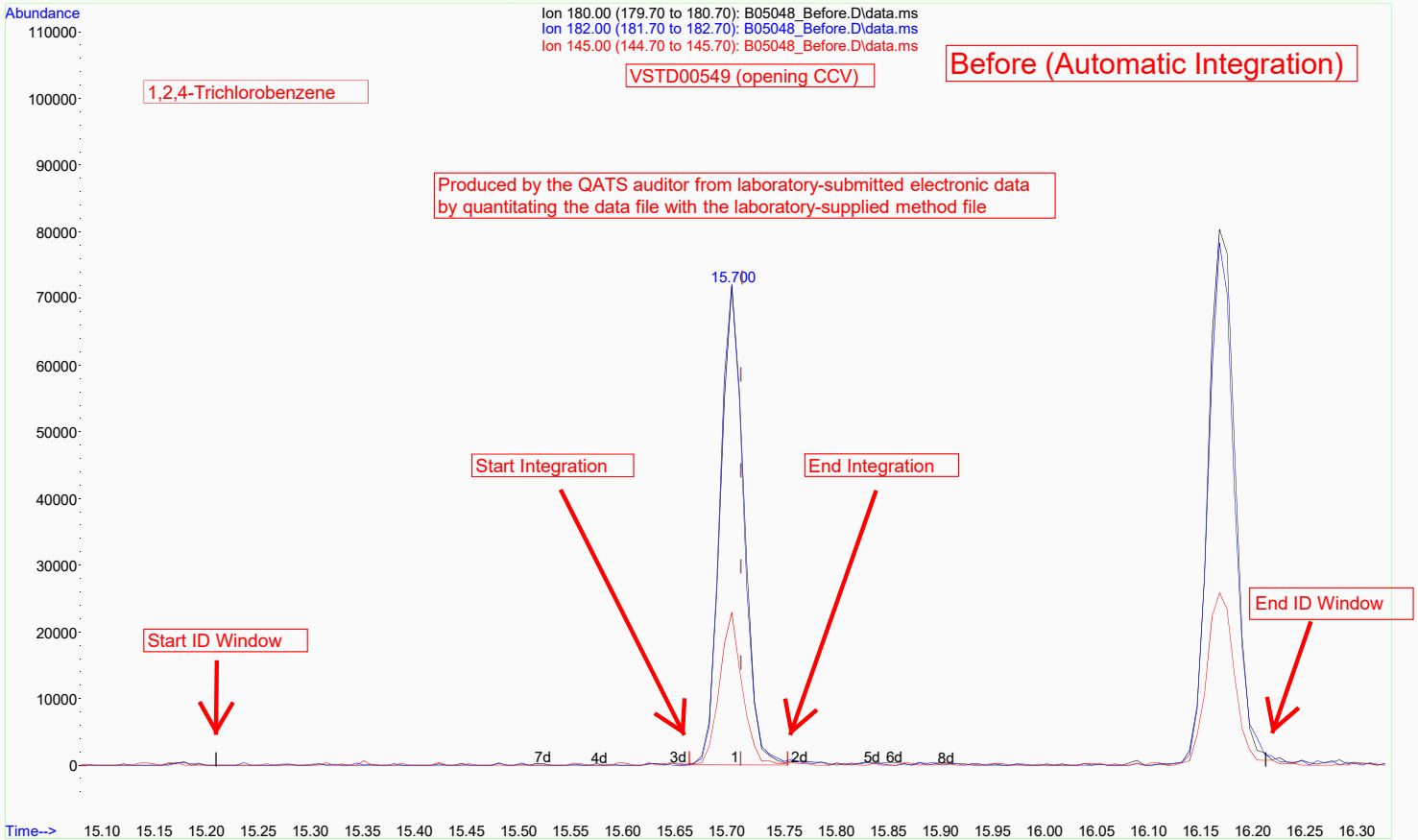
MS43

Slide 19

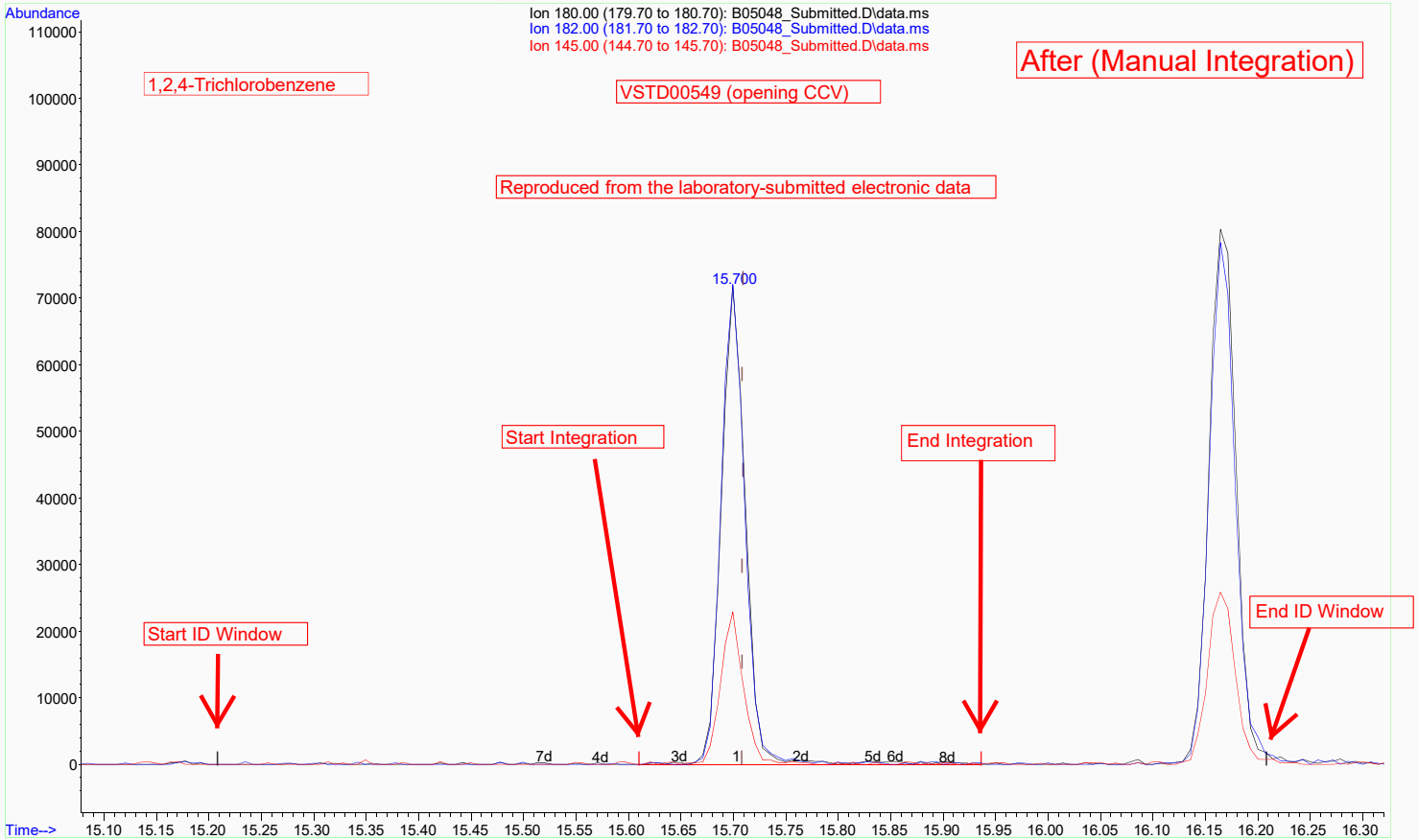
MS43 From this slide to the end, it is unclear why you need these.
Myer, Shari, 7/17/2019

FINIS

Proper Integration



Improper Manual Integration



ENCLOSURE 1B

Improper Manual Integration

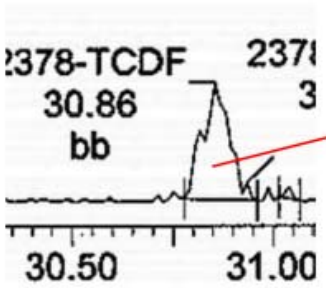
Printed from the laboratory-submitted audit trail file

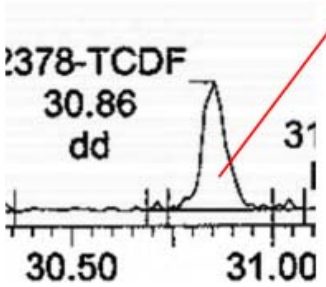
```
Modified : Sat Aug 13 12:25:55 2016 Event : Manual Integration
Message  : Changed peak amount for 1,2,4-Trichlorobenzene from 3.44525ug/L to 3.56233ug/L
QuantFile: CLPT05044.RES Severity: 1
```

```
Modified : Sat Aug 13 12:26:01 2016 Event : Manual Integration
Message  : Changed peak amount for 1,2,4-Trichlorobenzene from 3.56233ug/L to 3.50274ug/L
QuantFile: CLPT05044.RES Severity: 1
```

```
Modified : Sat Aug 13 12:26:05 2016
Event : Manual Integration
Message: Changed peak amount for 1,2,4-Trichlorobenzene from 3.50274ug/L to 3.5657ug/L
QuantFile: CLPT05044.RES Severity: 1
```

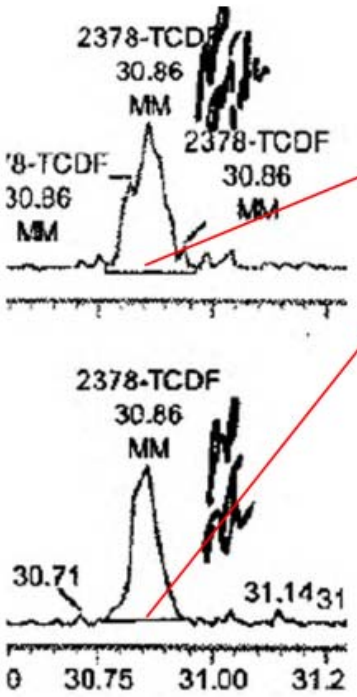
Proper Manual Integration





An enlargement of the SICPs of the manually integrated 2378-TCDF ions m/z 303.9016 and m/z 305.8987 from the ICAL standard CS12I as presented in the hardcopy data before manual integration.

Improper Manual Integration



An enlargement of the SICPs of the manually integrated 2378-TCDF ions m/z 303.9016 and m/z 305.8987 from the ICAL standard CS12I as presented in the hardcopy data after manual integration.