HOW NOT TO TEST A PSA: LESSONS LEARNED OVER THE YEARS

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Introduction

Much of what we learn in life comes from experience in our own mistakes or learning from other people's mistakes. This is also true in the world of physical testing of pressure sensitive adhesives (PSA's). This paper covers the many valuable lessons we have learned over years of performing tests on PSA products, including peel, tack, shear, release and unwind tests. Some of these lessons were from book learning and positive experiences, but include lessons learned from mistakes we have made and ones we have witnessed.

As with most things we try to get done in life, a lot of problems occur because of lack of planning or inadequate preparation. This is also true of testing projects, where devoting time and brain power before the test will greatly increase the chances of testing objectives being met. We'll cover planning, preparation, execution and reporting of results. By presenting lessons learned, we hope to demonstrate ways to avoid mistakes and to show the proper way to plan, prepare, perform and report for a physical testing project.

There is a quote attributed to the German philosopher, Friedrich Nietzsche; *that which does not kill us, makes us stronger*. We hope this paper will strengthen the audience's ability to gain value from testing projects.

PLANNING

There are a few different sub-categories to the planning stage that we'll cover in the following sections.

Objective - Why do you want to test?

One of the most common mistakes in judgement in the early stages of a testing project is forgetting to ask yourself why you want to take the time and spend the money to do testing. No one wakes up in the morning and thinks I don't have anything better to do today, so what the heck let's test some PSA products. There is always a reason. Forgetting the reason, or jumping ahead and performing testing without thinking about the reason can lead to disappointment and wasted time/money.

It is human nature to want to get going and run ahead as fast as one can. Most of the time, you start thinking about the results you want to see before the test even starts. Here's an example of what can happen when the objective is not considered first.

We have had several instances where a customer calls and states they have one set of samples for peel adhesion testing, for example. After some initial discussions, we work out a quote, one set of samples are sent in and we perform the test. After we send the report, the customer call back and asks if the material was in spec or not. Long story short, there was a performance issue with a recent lot of material and several previous lots had performed fine. After more questions and answers, we set up some problem investigation tests on actual surfaces, including samples from recent lots and past lots. After

this was completed, some other factors in application were discovered. Time and money could have been saved with more upfront thought and discussion on the objective of the test.

There are four major reasons for testing and they have different approaches:

- Product Development – creating a new product for a current or new application area. This involves both developing the product and providing technical sales tools for product launch.
- Quality Assurance – making sure the latest lot is compared with previous lots and falls within known control limits and meets end-use performance needs.
- Product change – either a voluntary (cost reduction, second sourcing, etc.) or a forced change (vendor shut down, product pruning, etc.).
- Problem Solving – an end use performance issue that requires immediate attention and involves variables outside of the manufacturing process.

Product change and product development involves both standard methods and modified methods customized to specific substrates, exposure conditions and dwell times to provide both a standard evaluation (same language between supplier, coater and end-user) and an application specific evaluation of performance based on end-use needs. Problem solving needs to focus on application specific surfaces, to take into account the surface energy of the substrate, surface roughness, etc., and application and exposure conditions in the interest of understanding cause and potential solutions for the problem as quickly as possible. Quality assurance testing needs to incorporate standard substrates, methods and testing conditions because the primary need is to have a consistent comparison of production to specifications that is repeatable and reproducible.

Since most testing is comparative in nature, it is important to have a good performing control on which to establish a base performance level, and then compare candidates or different lots to the control. With problem solving efforts, having both "good" and "bad" performing samples is essential to confirm the problem and aid in establishing a test that can truly differentiate between samples that meet or do not meet requirements.

Details – are we sure?

There is no such thing as a stupid question – only forgetting (or not knowing) to ask an important question (see the questions in the appendix of this paper). What can happen if you forget to sweat the details?

- Customer called to have some peel adhesion tests performed. A quote was sent for peel adhesion testing according to a standard method. Samples were sent in, testing was performed and a report sent. After the customer received the report, they called and stated that they had asked for the testing to be performed on polypropylene, not stainless steel. This was found in some e-mail correspondence, but was not communicated in the quote or the lab project work order.

Sampling – how much of what and when?

When a testing program is in the planning stages, it is a good idea, and in some cases required by standards or law, to consider how many individual tests are going to be performed for a given sample. These go by various names, such as specimens or replicates, but the basic definition is how many individual tests will be performed to determine a sample average and standard deviation. While some industry standard methods, such as ASTM D3330 for peel adhesion, state a minimum number of replicates and have some precision and bias testing history, other methods leave the decision up for grabs. Either way, the person assigned to set up the testing program needs to consider this factor before trial runs are performed or samples are taken from production.

What happens if this isn't considered or a bad assumption is made? Problems can happen. Here are two examples from past experience.

1. **Only sampling from one lot** – many tests were set up and performed using samples from one lot of PSA base material. After several testing programs, an initial specification range was determined from this data. While this was happening, production was moved to another coating facility with more production lots run using new lots of raw materials. More variation was introduced and out-of-specification results were seen. There was nothing wrong with the material, there were not process issues. Problem was that the sampling and testing was not set up to include multiple lots of material made on the final production line. In our industry, this is an extremely important factor given the multiple, continuous roll processes involved in the final product (check out the figure after this section).
2. **Not considering sample location** – Issues arose in one project because the sampling and data analysis plan did not take into account cross direction (CD) and machine direction (MD) potential for variation. Production samples were sent in and tested with the resulting average and standard deviation not being what the customer expected based on development testing work. After some post-testing discussion and follow-up trial work, it was discovered that there was an uneven airflow issue in the drying/curing oven that resulted in different peel-tack-shear behavior across the web. Had the sampling plan and following data analysis been set up to look at short term MD, long term MD and CD potential variation separately, this could have been understood and addressed before commercial production was started.

Here's a graphic on the multiple sources of variation in the production of a PSA tape material (this example is based on a PSA tape used in a medical application):

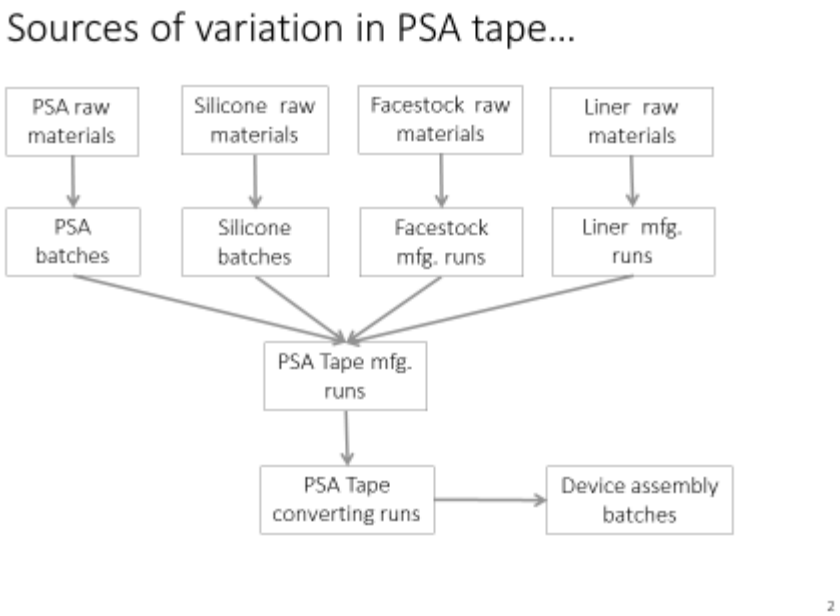

Figure 1. Sources of variation in a PSA Tape

When sampling plans are made, there is always some level of assumption to be made on expected or known variation, lot size and necessary quality level based on risk. There are published guidance document on process validation and sampling methods that can be used, including the following:

Process Validation

- Guidance for Industry - Process Validation: General Principles and Practices, January 2011
- Global Harmonized Task Force (GHTF) - Quality Management Systems – Process Validation Guidance, GHTF/SG3/N99-10:2004 (Edition 2)

Statistical Sampling Methods

- Zero Acceptance Number Sampling Plans, Fifth Edition, Nicholas L. Squeglia
- ANSI/ASQ Z1.4-2003 (R2013): Sampling Procedures and Tables for Inspection by Attributes
- ANSI/ASQ Z1.9-2003 (R2013): Sampling Procedures and Tables for Inspection by Variables for Percent Nonconforming

PREPARATION

Do you have the right equipment and is it ready?

You'll see a repeating theme in this paper – it is always a good idea, and in some cases required by standards or law, to make sure the testing/analysis equipment you plan on using is capable of producing results that you and your customer (and possibly the FDA) can have confidence in. Bad things can happen if you don't make sure of this upfront.

- **Testing performed on equipment where unknown variation occurred** – testing was performed on equipment where one parameter was not part of regular calibration checks or preventive maintenance (PM). In this case, the testing rate was not part of regular checks and had, over time, gone outside of test method tolerance. After investigation, the parameter was added to regular calibration checks.

Similar to process validation discussed earlier in this paper, there is guidance on how to qualify equipment before use in the following document:

- GHTF, Authoring Group SG3, "Quality Management Systems – Process Validation Guidance", edition 2 – January 2004.

Steps in qualification are:

- Design Qualification (DQ) – what equipment is needed? Are you sure you have the right equipment before you buy it?
- Installation qualification (IQ) – you received the equipment, now make sure you have it set up according to manufacturer instructions and that it is ready to test.
- Operational Qualification (OQ) – does the equipment operate the way it is supposed to? If there are different use parameters, this is the step to check them to determine how the equipment will be set up and used for the testing programs at your facility.
- Performance Qualification (PQ) – can you do it right more than once in a row? This step confirms whether or not the equipment can produce results with confidence over time, with different operators, under expected outside factors, etc.

Are the methods you plan on using capable and proven?

Even if you have qualified equipment, are you going to use industry standard methods, custom developed methods or significantly modified standard methods? If you are planning on using one of the latter two types, you probably will need to do some level of test method validation. Here is a guidance document on method validation:

- ICH, "VALIDATION OF ANALYTICAL PROCEDURES: TEXT AND METHODOLOGY Q2 (R1)", November 2005.

Important elements to consider in method validation:

- Specificity – can the method determine the needed result in a pile of other information?
- Linearity – if you are measuring and comparing samples over a range, is it a straight line or a roller coaster?
- Range – great if you can prove a single point is accurate, but make sure the equipment and method are reliable over the expected range of measurement.
- Accuracy – expected +/- on a single point

- Precision
    - Repeatability – same sample, same operator, different day – do you get the same result?
    - Reproducibility – different operators, different test equipment – do you get the same result?
- Detection limit – what is the minimum value that can be detected and in which you can have confidence in reporting?
- Quantitation limit – level of +/- confidence in reporting a result over the expected range
- Robustness – how well the method can perform when subjected to possible, real variations in environment, materials, etc., whether within or outside the control of the analyst.
- System suitability – combined elements of expected precision, equipment qualification, method validation, sampling and analyst training

What happens if you don't perform method validation when it is needed? This can happen.

- A need for sudden impact resistance for an adhesive was needed. A test unit was on hand that was designed for use in an industry standard method for paint chip resistance. Work was done to develop a modified method, samples were tested and some difference in performance was observed and reported. No worries until the same samples were tested again and not only were different results seen, but different trends as well. Long story short, sample preparation and manual test procedures were not repeatable or reproducible.

Solution? Be sure about what you have in equipment and method capabilities before making promises.

Are the technicians/analysts trained, capable and authorized?

Oh yeah – the human part. Human Capital. Carbon based work units. Easiest thing to forget and the easiest thing to go wrong. Just because you showed someone a method 2 years ago and they did a test about the same time doesn't mean they know how to do it correctly now. Here we go again - it is always a good idea, and in some cases required by standards or law, to make sure the testing analysts/technicians are trained, authorized and proficient in the method before performing the test. What could possibly go wrong? Read on:

- Production QA testing was performed for break tensile strength and out-of-specification (OOS) results were observed. The average test results were significantly under the minimum specification value and were about ½ of the previous test results. Long story short, turns out that the technician had not performed this test in over a year and had recorded the average value of the test rather than the peak (kind of makes sense that the values were about ½ of previous results). The technician was used to doing peel adhesion tests where the average value was reported.

ISO 17025 third party accredited testing laboratories and any manufacturing/design companies involved in FDA regulated industries are required to make sure testing technicians are proficient in the test methods they perform for design and release for sale testing. Even if you aren't involved in these areas, it is still a great idea to make sure the personnel that are performing tests know clearly how to do the tests.

Test Sample Preparation

Test results are only as good as the samples, substrates and preparation methods used for the tests.

- Since sample cutting is so easy and simple, this can be delegated to anyone. Right? Peel specimen sample cutting was performed and one week dwell samples laminated for testing. The test scans were examined and they were all either sloping up or sloping down. None were flat. After examination and discussion, the samples had been cut on a shear cut table and were not uniform along the cut length.
- OPP Packaging tape test samples were cut and laminated for one week peel adhesion tests. During testing, all samples failed by the facestock splitting at a nick in the specimens from the shear knife blade used in sample cutting.
- Lower than expected peel adhesion results were observed. A lab investigation revealed that the MEK used as one of the panel cleaning solvents was taken from an industrial grade drum rather than a reagent grade bottle. Comparison testing showed the industrial grade solvent left residue on the panels that lowered adhesion.

Moral of this story - make sure that sample cutting, lamination, mounting are considered and confirmed before investing time in testing.

EXECUTION

Now that you have clearly planned the tests and are completely prepared to do the tests, what could possibly go wrong?

Is the equipment calibrated and in good working order?

Here's an example of what can happen if assumptions are made in calibration:

90 degree liner release testing was to be performed on an adhesive tape sample. The values were expected to be low, so a decision was made to use a 1 lb. load cell. It happened that a 1 lb. load cell was in the release test machine and the calibration log indicated that the cell had already been calibrated for other testing. The analyst proceeded to test liner release and discovered that the test values were out of specification. After reviewing the procedure, sample preparation and machine setup, the analyst noticed that the load cell calibration previously done that day was performed with the cell in 180 degree angle test configuration. After re-calibrating the load cell in 90 degree configuration, sample testing was repeated and the values were found to now be in specification. Load cell calibration difference between 90 and 180 degree angles is small, but when very low force values are measured, it is a critical difference.

REPORTING

Confirming and Securing Test Results

Here we go again - it is a good idea, and in some cases required by standards or law, to ensure that test results are reviewed, approved and secured from tampering. Why?

- Test results were transferred to a spreadsheet for calculating average and standard deviation. Results were reported but further checks by the customer raised concerns on slight differences between checked average values and reported average values. Turns out that the spreadsheet cell ranges used in calculating the average were not correct due to a cut-and-paste error from a previous testing program.

The US FDA has established guidance and regulations for validating computer systems and software (including spreadsheets) used in calculating results that affect a drug or medical device's release for sale:

Guidance

- U.S. Food and Drug Administration, Science and Research, Field Science and Laboratories, Volume III - 4.5 Development and Validation of Spreadsheets for Calculation of Data, Document III-04, Version 1.4, Revised 1/31/13, Web link: https://www.fda.gov/ScienceResearch/FieldScience/ucm174286.htm.
- U.S. Food and Drug Administration, "General Principles of Software Validation; Final Guidance for Industry and FDA Staff", January 11, 2002

Regulations

- 21 CFR Part 11, "ELECTRONIC RECORDS; ELECTRONIC SIGNATURES"
- 21 CFR 820.70 (i), Production and Process Controls, Automated processes.

Are the samples different or not?

So Sample A average is 3.8 lbs,/inch peel adhesion and Sample B average is 3.5 lbs./inch peel adhesion in side-by-side tests. Does that mean Sample A has higher peel adhesion? Let's dig into that one.

- A customer received a report and responded back that they thought a 0.3 lbs./inch difference between samples meant the higher average value sample was "better" in performance. Take a look at the following table and see what you think:

Table 1. Comparison of Peel Adhesion between Samples A and B

Peel Adhesion (ASTM D 3330)
1 minute dwell

	Sample	
	A	B
Replicate	(lbs/in.)	(lbs/in)
1	3.23	3.68
2	4.21	3.11
3	4.13	3.74
4	3.24	3.32
5	4.32	3.54
Avg	3.83	3.48
σ	0.54	0.26
Failure Mode	A	A

Avg. - average of the replicates
σ - standard deviation of the replicates
A - adhesive failure - adhesive cleanly removed from substrate.
GH - ghosting - a shadow or stain remained on the substrate.

While it may be tempting to try to draw conclusions from this test, there are ways to determine if these are different populations or not. Microsoft Excel has a Student's T-Test as a formula that can be used to compare samples. For this sample, the t-test result with parameters we chose came out to be 0.24. Values > 0.05 indicate the populations are not significantly different. There are grey areas using this test, especially with limited data. It is a tool that can be used to check results rather than just guessing.

Remember to report failure mode, units, method, observations

Reporting a static shear average result of 8500 minutes is pretty much useless, without reporting the failure mode, applied area and weight used. Remember to include a full test description along with results to clearly communicate the result along with how the test was performed.

Following are some examples of information to include with a testing report:

- Title - a clear reference, usually a project number, and any other designation which may be pertinent
- a reference to a previous report, if applicable
- a description and identification of the item(s) tested
- characterization and/or condition of the test item(s)
- if appropriate, the date the item(s) were received and/or tested
- identification of the test method used, including any deviations from, additions to or exclusions
- specific test conditions.

- identification of specific test instruments used
- if relevant, the sampling procedure used
- deviations from the test method
- graphs, tables, etc., to support the results
- where relevant, a statement of compliance/non-compliance with requirements and/or specifications
- where relevant, a statement concerning the uncertainty of the data
- opinions and/or interpretations, where requested, appropriate or needed, are clearly marked as such
- any additional information which may be required by specific methods or clients
- the signature and title of the person accepting responsibility for the report

Out-of-Specification (OOS) or Out-of-Trend (OOT)

If you're testing to a specification and an OOS result is obtained, what do you do? Throw away the results and start over? No. Here we go again - it is a good idea, and in some cases required by standards or law, to first investigate for laboratory error when an OOS, or in some cases an OOT result is obtained. Following is a guidance document on this topic:

- "Guidance for Industry - Investigating Out-of-Specification (OOS)", U.S. Department of Health and Human Services, Food and Drug Administration, Center for Drug Evaluation and Research (CDER), October 2006

When testing in an FDA regulated industry, one cannot just throw out test results that are deemed suspect. A thorough laboratory investigation is required to determine if the results were affected by some type of lab error, before deciding what to do with the results. The FDA has seen too many cases of samples being tested until they are in specification, or cases where data was rounded multiple times to bring results into specification. Data security and reliability, along with proper investigating of lab error, is always a good idea and in some cases, it's the law.

Summary

Testing is like any other activity – better planning and preparation greatly increases the likelihood of success. Taking the time up front to consider testing objectives and sweating the details on the important questions will help pave the way for a trouble-free experience. Hopefully the experiences and advice in this paper will help give the reader some new insight and ideas for their next testing project.

APPENDIX

Questions to ask up-front for PSA tests

General Questions for all requests

- What is the reason for testing – problem investigation, QA, product development?
- When are results needed? Is this a shut-down issue requiring urgent attention?
- Sample type – single coated tape, double coated tape (if double coated tape, do you want both sides tested?). Is this a pressure-sensitive adhesive or some other type of adhesive/coating, such as a thermoset (structural) adhesive? For liquid adhesives, please send MSDS for review.
- Sample shapes/sizes that are available for testing.
- What is the facestock or carrier (i.e., paper or film)? Does the facestock or carrier stretch (elongate) in the test direction? You may want us to reinforce the carrier with a non-elongating tape to take this factor out of the peel test, or we could test as-is.
- Number of samples – number of separate/different samples to be tested
- Any special preparation or sample exposure conditions
- Any special testing conditions (normally we condition and test at 23° C/50% RH)
- Any test protocols or standards – industry, OEM, military?
- Will the samples arrive ready to test or is sample preparation needed (i.e., cast adhesive onto 2 mil PET, dry and test)?
- Will a standard report, including test description, results and observations, meet your needs or are there other deliverables you require (such as certificate of analysis, statistical analysis)?

Peel Adhesion

- Sample type – single coated tape, double coated tape (if double coated tape, do you want both sides tested?). Is this a pressure-sensitive adhesive or some other type of adhesive/coating, such as a thermoset (structural) adhesive? For liquid adhesives, please send MSDS for review.
- Sample shapes/sizes that are available for testing.
- Do you have a specific test method in mind (i.e., ASTM D3330)?
- Test substrate – stainless steel or something else?
- Peel angle – normally 180° or 90°.
- Dwell time before peeling
- Peel rate – normally 12 inches (or 300 mm)/minute.
- Number of replicates – normally 5.

Tack

- Type of tack test desired – probe tack, loop tack, rolling ball tack, inverted probe tack? Do you have a specific test method in mind (i.e., ASTM D6195)?
- Test Substrate – stainless steel or something else? Probe tack is stainless steel only.
- Test speed – loop tack is normally 12 inches/minute and probe tack is normally 24 inches/minute. If a different speed is desired, we may have to use a tensile tester for loop tack or the PMA-1000 for probe tack.
- Dwell time – set for loop tack and probe tack. For other dwell times we can use our PMA-1000.
- Number of replicates – normally 5 for loop tack, probe tack and rolling ball tack.

Shear

- Do you want static or dynamic shear? Do you have a specific test method in mind?
- Test Substrate – stainless steel or something else?
- If static shear – what is the weight, applied area (height x width), dwell time before hanging? Is this normal static shear or SAFT?
- If dynamic - what is the test speed, applied area (overlap x width), dwell time before testing?
- Number of replicates – normally 5

Release

- Is this liner release (release liner removed from adhesive/carrier), unwind (for self-wound tapes) or release from backing (tape applied on tape)?
- Do you have a specific test method in mind (i.e., ASTM D5375)?
- Angle of test – normally 90° or 180°
- Test speed – low speed is normally 12 inches/minute for tapes. Labels are normally 300 inches/minute and higher, and can be higher for tapes (high speed unwind).
- Width of sample – normally 2" wide for label release and 1" wide for tape release.
- For liner release – is liner pulled from adhesive/carrier or is adhesive/carrier pulled from liner?
- Number of replicates – normally 3 to 5 depending on the test method.

Tensile and Elongation (T&E)

- Do you have a specific test method in mind (i.e., ASTM D3759)?
- Do you want machine direction and cross direction or both?
- Do you only want to know break tensile and break elongation, or are you interested in other properties (i.e., force value at 3% elongation)?
- Number of replicates – normally 5.
- For pressure sensitive adhesive products, the T&E results are usually determined by the facestock. If you are interested in adhesive-only T&E properties, we could perform the T&E test on the adhesive film alone or check mechanical properties of the adhesive layer with our PMA-1000.

Coat Weight/Basis Weight/Caliper

- Do you want basis weight/coat weight (i.e., grams/meter^2) or caliper (i.e., thickness in mils)?
- Do you have a specific test method in mind (i.e., ASTM F2217)?
- Number of replicates – normally 3 to 10 depending on method. Normal for the method OK or is a different number needed?

Tear Resistance

- Do you have a specific test method in mind (i.e., ASTM D1004)?
- Do you want machine direction and cross direction or both?
- Is there a special sample shape – i.e., moustache die as in PSTC-39 (Die C in ASTM D624)?
- Number of replicates – normally 5.

T-Peel

- Do you have a specific test method in mind (i.e., ASTM D1876 or F88)?
- Will the samples arrive ready to test or do we need to laminate before testing. If so, how do we laminate and what is the dwell time before testing? Do we need to apply heat to laminate the material?
- Tail supported or not supported?
- Number of replicates – minimum of 10 is called for in ASTM D1876.

Viscosity

- Do you have a specific test method in mind (i.e., ASTM D2556)?
- Do you know the approximate viscosity range or character of the sample (i.e., is it like molasses or is it about 20,000 cps.)?
- Is the sample hot melt, solvent or water based? Is there any crosslinking/curing mechanism involved that could affect the testing?
- If hot melt, what temperatures do you desire to be tested?
- Will need to see MSDS sheet for any liquid samples prior to quoting.
- Number of replicates desired

Sutherland Rub

- Do you have a specific test method in mind (i.e., ASTM D5264)?
- Receptor to use?
- Weight (normally either 2 lb. or 4 lb.)?
- Size of samples that will be provided?
- Run dry or wet with a liquid? If wet, what liquid will be used?
- Number of cycles or what is the end point desired (i.e., cannot read printed text)?
- Number of replicates desired

WVTR.

- Do you have a specific test method in mind (i.e., ASTM E96)?
- Upright cup with water, upright cup with desiccant, inverted cup with water, or something else?
- Size of samples that will be provided?
- Number of replicates desired

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Comments and Acknowledgements

I am not an expert in statistics, but I understand the subject enough to know it is important to consider statistics in sampling and in data analysis. There are folks that know statistics and they love to help those of us challenged in this area. Seek them out and listen to them.

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