

Trey: Good morning, and thank you all for joining us. Today's webinar "Understanding Cosmetic Stability Testing" is being presented by Cosmetic Chemist Perry Romanowski. Perry is the author of the Chemist's Corner Blog and Training Program, and one of the founders of the Beauty Brains. He's been a Cosmetic Chemist since the early 1990s is currently the Vice President of "Brains Publishing." Most recently, Perry was part of a hair care innovation group at Alberto Culver. He's recognized as an innovator and thought leader in the cosmetics industry, and has made numerous media appearances including spots on popular TV shows including "The Dr Oz Show" and "The Rachael Ray Show," his latest book, "It's OK to Have Lead in Your Lipstick" answers common questions about cosmetic products. And Perry's articles also appear on the Prospector knowledge center on a bi-monthly basis.

My name is Trey McDonald's with you, I'll be moderating today's event. Please send us your questions by typing them in the question box located on your screen. Our presenter will answer the questions at the end of the presentation. We are recording today's event and we'll send you a link by email when the presentation has been posted to the UL Prospector knowledge center. Now I would like to turn the presentation over to Perry,

Perry: Hello, everyone, thank you, Trey, for that, and thank you, UL Prospector for setting this up. I'm really excited to talk to you about today's topic, Stability Testing. And so in this presentation, we're gonna be talking for about a half hour, and along the way, we'll be taking questions. There's a question box there so you can put in your questions for me to answer later at the end. So let's begin with stability testing. Just a moment, there we go. Actually, let's begin with a little bit about me since you may or may not know me, Trey did a nice introduction there, but currently, I'm the vice president of a company I started called Element 44, which I provide cosmetic science information, and I do a little bit of consulting. And so, you can contact me through my website chemistcorner.com. For years I was a Cosmetic Chemist, a Formulator. And the stuff that I'm gonna present to you today comes out of my experience as a formulator because I had to do these types of tests all the time. Stability testing isn't a thing that anybody learns in college or while you're studying chemistry, but it turns out to be one of the primary activities that you spend your time as a formulator doing. It's extremely important both in regards with creating new formulations, and with regards to keeping yourself legal in terms of having the proper background information for your formulations that are sold on the market.

In addition to being a Formulator for, geez, almost 17 years in hair care and skin care products, I'm an author, and you might be familiar with "Beginning Cosmetic Chemistry," which is a book I co-wrote with Randy Schuler. Which just gives you an introduction to the cosmetic industry, and you can find information about testing, like stability testing in there, but about raw materials, formulating, and that sort of thing. So that book is still available I encourage you to check that out. And also, here's my website Chemist Corner, this provides more detailed information that formulators would wanna know. And so I encourage you to check out that resource, most of things there are free. And I just wanted to give you special attention to our forum at Chemist Corner, another free resource, but this is a place where any questions you want answered about cosmetic science you can find them here. We have a number of formulators from around the world actually, who participate in discussions here. And so, this is a good free resource for anybody who has questions from anywhere about cosmetic science in the world. And then finally, there's the practical cosmetic formulator which is online training, and you can get that through there, so. All right, that is my commercial introduction, let me get into the bulk of the presentation today.

Today, we're going to be talking about Stability Testing, and I'm really going to cover it in three sections. First, we'll define stability testing, what it is, and why you would ever want to do that. Then we'll talk about when you're gonna test your formulas, and why you're gonna test them. And then finally, I'll get right into some specifics about the basics of conducting a stability test.

The objectives of this presentation, so hopefully, after listening to this webinar, going through this, you're gonna first know the basic philosophy of stability testing, you're also going to be aware of the relevant regulations and the rules that you have to follow. You're also going to know why and when you're going to conduct a stability test. It's not always obvious whether you should or not. And then finally, probably most important, you're gonna know how do you could conduct those stability tests. So we'll get into each of these along the way, hopefully, you'll come out of this knowing these things. I also want to mention that I'm a U.S. based formulator, cosmetic science, and so, my focus is really on what we do here in the U.S., and things around the world might be a little different. I know in the EU they have some different kinds of stability guidelines, in Asia they also do. And I also realize that we have a worldwide audience here. So I can provide links to some of the relevant EU and Asian sources. But I have to say that most of the things that you're

gonna learn in here are applicable anywhere around the world. And as a Formulator this certainly is practical information that would be applicable to anybody wherever they're formulating. Now, whether the specific paperwork that you got to follow or keep track of, that might be different in other places around the world, but the specific testing really applies anywhere.

All right, so let's begin with our introduction with what is stability testing? Now, as I said, when you're going through college and learning chemistry you don't really even hear about stability testing. I had never heard about stability testing when I got into the cosmetic industry, and it turns out that, you know, most of my formulating time was spent either preparing stability samples, putting them up in their conditions, or going through and testing them for PH, viscosity, appearance, and that sort of thing. And stability testing is important because what it is, is a predictive test. Essentially, you need to test whether your product is going to remain stable and remain functional over the course of the life of the product. You don't wanna put products out into the market that fall apart pretty quickly. And so, we do stability testing to ensure that the products, when the consumer buys the product it remains the same quality that it did when it left the manufacturing plant. Ideally, your products will last and maintain their integrity for at least a year after manufacturing. In the cosmetic industry that's kind of a rough guideline of what we go for, we want one year of stability.

Some of the other things that stability testing can do, it can determine the product longevity in terms of the aesthetics. So what it looks like, what it smells like, also, the functionality, whether the product can remain functional after a certain amount of time. Like I said, in the industry we target a one-year stability guide, but really, you know, people ask me all the time, whether their products will be good for longer than that. It turns out consumers often will buy a product and then they'll put it under their bathroom countertops, and then they'll forget about it and they'll pull it out a year and a half later, two years later, and wonder if it's still good. The reality is, if you have a nicely stable product it could probably be stable for two years, three years, or even longer.

Some other things that stability testing will help us tell is, the microbial quality. Now, it's really important that your products remain microbe-free, and preservative testing is extremely important in this regard. Microbes will grow wherever there's water and food, and cosmetics it turns out, are pretty good mediums to grow microbes. And so, you need to do stability testing to ensure that your products don't get contaminated over time. Another thing that stability testing will look at is whether your packaging will remain stable over time. So, most of the stuff that I'm going to talk about is focused on the actual chemicals in the formula. But stability testing isn't just for the formulas it's for the entire product, and that entire product also is the packaging. And so, you have to ensure that your package and your formula also remain compatible and stable.

And one of the things I wanna mention about stability testing is that the decision of whether something is stable or not stable is really a subjective decision. That's not a thing that you can just say there's no number, or the way you could say is, if your formula got this certain number in a test then it's stable, and if it didn't get that certain number then it's unstable. You'll find that stability testing is really a subjective decision. And often, you'll find that your marketing people will want you to reduce your standards of what's stable, more frequently than you really want to. So you have to come up with these decisions whether something is stable or not really on your own and based on what your company is comfortable with.

So let's just look at from a regulatory standpoint. Now, there are some cosmetics in the U.S. that are considered over the counter drugs, these are products that actually fight diseases, so, anti dandruff shampoos are over the counter drugs, sunscreens are, antiperspirants are, toothpaste that contain fluoride those would also be OTCs. So, in fact, if it has a monograph an OTC monograph through the FDA, and there are about 20 different monographs for products that are personal care products, then for those types of products you are supposed to follow the pharmaceutical testing standards, which is published in the CFR, Code of Federal Regulations part 211. Now, I've included a link in the slide here that's a shortened link, but that's gonna take you to a PDF that explains all of the details of OTC product stability testing that you need to know. So there you go, this webinar is filled with some links which will make it easier for you to find these things.

So that is from a regulatory standpoint, that's the stability required, if it's an OTC product you have to follow some specific rules. However, we work with cosmetics and I want to tell you that from a cosmetic standpoint

there are no specific regulations or requirements, except that you have to have this testing done. Some form of stability testing needs to be done to demonstrate that your product is safe. Because one of the laws in the United States as far as cosmetics go is it is illegal to sell unsafe products. Now, the FDA does not tell you how you prove safety, but stability testing is definitely one method of demonstrating that your product is safe. So you have to do this type of stability testing to demonstrate safety.

But like I said, there's no specific requirements, they don't tell you how to do it, they do tell you that you have to have it done and it has to be something you... So the manufacturer of the product has to do it and it has to be something that is seen as reasonable as far as the rest of the industry goes, and as far as the FDA is concerned. And it should be something that's efficient. So it doesn't take a huge number of samples to do. But most importantly, you must document what you do and that you did it. So you have to keep your documentation whenever you make a batch and you run a stability test, you have to keep documentation to demonstrate that you actually did it. Also the group in the United States which it's sort of our industry lobbying group the PCPC, or the Personal Care Products Council, they actually have a recommended guideline for doing stability tests, and so, if you'd like you can purchase through the PCPC their guidelines on stability testing, or you can just follow the stuff that I'm going to discuss right here, which follows along with what they suggest to you.

All right, we've given an introduction to what stability testing is, so let's talk about when you might run a stability test. There are a number of different moments that you might run stability testing, but the number one is probably as a formulator, whenever you're creating a new prototype, you're gonna want to run a stability test. Now, pretty early in the prototyping process, I should mention, you don't wanna run a stability test on every prototype that you do because most of your prototypes you're going to get rid of. So, when you're doing prototyping you can sort of ignore stability testing until you get to a point where you've created a product that shows some promise. So, once you have a product that's a prototype that shows some promise and as your final product, then what you wanna do is make a batch of that, put it up on a stability test to ensure that it holds together. Because the worst thing you could do is to create a great prototype or product that functions really well, and then you can't keep it stable. So if you can't keep it stable you don't have a product.

Another time to test is whenever you get a new raw material supplier. So you may have a formula that is perfectly stable and it works all the time, but you have your supplier for whatever ingredient you're using, say it's Acetyl alcohol in a conditioner, say you get a new supplier in Acetyl alcohol, just because an ingredient has exactly the same name doesn't mean it's going to work the same way in your product. And so, anytime you get a new raw material or a new source of a raw material you certainly should run a stability test to ensure that the ingredients remains the same. And whenever you, you know, your purchasing people will often find that they can get a lower price on an ingredient and they'll encourage you to accept that new supplier for a raw material. And so, when you're doing that, you'll have to do a stability test before you can actually start using that in production.

Another time to do stability testing is when you have new production equipment, often if you get a new mixer, a new tank out in your manufacturing facilities, you should do stability testing on any formula that's going to be put in there. Because any kind of production change, or equipment change, or procedural change, those can have an effect on whether your product remains stable or not. And so, whenever you have those things you should run a stability test. Whenever you have new packaging, your marketing people often love to change the shape of your bottles, or change the resin to find cost savings, so there's lots of good reasons, whenever there's new packaging, you should run stability test because whenever your formula interacts with a package there is a potential there for the formula to fall apart and require...well, then could require reformulation or different packaging.

Then some other points is when you're doing scale up, going from the beaker to the production facility, don't assume that just because something was stable in the lab that's gonna be stable in manufacturing. You should always have a first production run, stability test done. Because that's actually the product that gets out in the market, and so, you should have that stability testing information. Now, what you don't wanna have is your first production run to be your first stability test. You should always do lab stability tests to give yourself a good idea of whether your product will be stable on a larger scale. Usually, if it's stable on a small scale it will be stable on a large scale but that's not always the case. Then, of course, there's ongoing QA and QC. So you've had a product for a long time, you should just, every so often, be doing stability testing on your batches. Raw materials can change even from within suppliers that do things in slightly different

ways and they don't tell you. So you wanna just ensure, from a quality standpoint, that your products remain stable. And so, every six months I would say, of a formula you should be doing some stability testing. And then finally, as I mentioned before, there are regulations as far as stability testing goes and so, if you're working with over the counter formulations then you'll want to follow those stability tests.

All right, let's look into the general guidelines. Now, I mentioned that stability testing, it's really up to you the manufacturer to decide what your stability testing protocols look like. So I'm gonna give you some general guidelines to follow when you're putting those together. There's really three things that are important when it comes to that. First, the type of packaging that you're testing it in. Next, the storage conditions that you're going to be testing in, and then finally, the schedule at which you test your formulations. And so, we're gonna go into each of these in a little bit more detail.

All right, let's look at the basics of a stability test. So you're the formulator, this is basically what you do. First, you make the batches and you fill them into the containers, and you put them up onto the different environmental conditions that they're gonna go onto. Then, over the course of whatever your schedule is you do...you test your samples, you take them down from their storage conditions, you let them equilibrate and then you test them. And then finally, you make your decision about whether the product is stable, or the formula is stable or not.

Just a word about the batch creation, so, as a formulator this is the part where you have to do a little thinking, right? So to come up with a stability test, what you really wanna do is make one batch, so that batch has to be large enough to be able to fill however many number of samples that you have. So it's going to be calculated based on the size of the container. Often, cosmetic products are filled in eight-ounce bottles, but sometimes, the company that I used to work for we had a 15 ounce bottle, VO5, shampoo it was quite a bargain. So you had to figure out the size of each container, and the conditions that you're going to test them. And then you just multiply it out, so if you were going to test shampoo formula for example, you might need... Well, we'll go into some specifics of what you need. But when you're creating a batch you have to calculate out enough of the batch to make all the samples. And then what you also wanna do is calculate enough of the batch and add 10% to that formula, because what's going to happen is you're going to spill it or in the filling. And you also wanna have retains because your marketing people or production people will want to see what the batch was. So always keep a couple of retains. So, I always do about 10% larger batch sizes than you actually calculate out.

Also, you're going to want to do a control batch. This is almost always the case, whenever you have a formula that is currently stable you wanna make that formula that's stable and then you're gonna wanna make the test batch, which is the stability batch of, you know, whatever formula you're testing. So, for example, if you are looking at a supplier of a new raw material, what you wanna do is you'll make a batch using the current raw material supplier, and then you make a batch using the second raw material supplier. And then when you run your stability test, you can compare the stability profile of each of those different variables. And so, almost always, whenever you're running a stability test you're going to wanna...you wanna do a companion control batch versus your test batch.

Once you make that batch, you wanna take your initial readings and they should be within the specifications of your formula. And then, also, you're going to want to put those samples into the appropriate packaging. Now, ideally, you do the stability test in the final packaging of your product, but that doesn't always happen because the final packaging is often not ready. So what you wanna do is at least have packaging that has the same resin at minimum, and then it's always good to do stability testing in glass also. So like four-ounce glass jars, because that can give you a sense of, you know, whether the formula would be stable no matter what packaging it's in. So I always encourage people to do stability testing both in the final packaging and in glass samples.

Let's look at sample storage. There are a variety of temperatures that you wanna store things at. The company I worked for, we did it at 50 degrees C, which for most companies, that's too high. Because what's going to happen is that, that 50 C is above the melting point of some of your ingredients or it's close to the melting point of some of your ingredients, and so, you're gonna get reactions that you know, you normally wouldn't get. So it's not a really good predictor. But 45 C is a good number, 35 C is also good. And then you wanna have a room temperature sample or 25 C. And then you wanna have a refrigerated sample, a 4 C sample. The room temperature and the 4 C are the internal controls, so we wouldn't expect those samples



to change that much at all. And you might expect the samples at accelerated temperatures to change. Basically, there's an idea that when you increase the temperature of a sample you increase the speed of reaction, you know. And if you remember organic chemistry, I think it was the Arrhenius equation which demonstrated temperature and kinetics of a reaction are, roughly, they're linearly related. And so, the idea here is if we increase the temperature we're going to speed along reactions that would have happened at room temperature. And so, that's the idea why stability testing at accelerated temperatures can give you a better idea of what's going to happen at room temperature in the future. I should say, this is just as already soft science and we're just trying to predict, so it's not an exact science.

Also, in storage you want to put your samples at different lighting conditions. So sunlight, direct sunlight exposure and UV light exposure, because when products are put in stores sometimes they're put in the window so they get sunlight coming through the window, or they're put on store shelves and they get exposed to UV light. And so, you wanna know what happens to your formulas under those conditions. Well, this pretty storage box is what usually happens. This here is an actual stability storage closet, and so, you can see the samples get all over the place and it gets pretty crazy in stability. So, you know, if you have a lot of tests going on, you'll get a lot of samples like that.

All right, next is the evaluation schedule, here, the standard time checks. So you should have a two-week check at all of the conditions. And then, a four-week check, and we'll get into specifically what you're checking, in a moment. And at eight week point, that's when you could start to make some decisions about whether the formula is stable or not. I should say, before that, you can make a decision of whether the product is failing stability. If after two weeks your formulation is separating into different layers and it's not supposed to do that, then it's failed and you can start to take some action. But if nothing happens at two weeks and at four weeks, you wait until eight weeks to decide whether it's stable. Just because something is stable for two weeks doesn't mean that it's going to be stable, it could fall apart easily at four weeks? And just because it's stable at four weeks doesn't mean it's not gonna fall apart at eight weeks. But typically, we believe if after 8 weeks...actually after 12 weeks, it's stable after 12, then we have a pretty good confidence it'll be stable at 26 weeks, 52, and beyond. But these are some standard time checks that you might look for stability testing.

So what kind of testing do you do? Well, you check the appearance of the product, that's the number one thing that you're gonna check, it should look the same way that it looked when you first made the batch. And how do you know what it looks like when you first made the batch? Well, the refrigerated sample, the 4 C sample, and the room temperature sample should be good indications about what it looked like originally. Now, we use the 4 C sample as the ultimate control, because if something is stored in cold storage, you don't expect there to be any chemical reactions. And sometimes people will go through and they'll take a picture of what the product looked like beforehand, but it's very difficult to compare pictures to actual samples. And so, the 4 C is really your appearance control. Then we have odor evaluations, and odor evaluations just involves you smelling these things and to ensure that they smell the same over time. Again, the 4C sample is looked at as your control because we expect it to change the least over time, although the room temperature sample could also be looked at as the standard there. Odor evaluations, they should be done on a blinded basis, and you kind of have to train your nose to get good at smelling these things. But as a cosmetic chemist, this is just one of those things that you have to get good at.

For other standard tests, we have the PH testing. And, you know, PH testing is relevant for anything that is water based, certainly, but if it also is a oil and water emulsion, then a PH testing makes sense. If you're working with water in oil emulsions, it doesn't make as much sense, and anhydrous formulas, it doesn't make sense to do PH testing either, because those numbers don't really mean anything. But the reason we do PH testing is that, one, it's easy to do, and, two, a change in PH is indicative of some sort of a change in the chemical reaction. So that might indicate some stability problem. It also can be indicative of what... you might have some unreacted residual ingredients that come in with your raw materials. And the PH can also affect the functionality of the product, if you have a hair conditioner you like the PH to be, you know, around four, maybe a little lower than four, to get it to work at the maximum ability of it work. If your PH is up to six or seven, it's not going to work the same way.

Another thing that we look at is the viscosity, and the viscosity gives you a good measure of the consistency of the formula, and it can help indicate whether there's any separation going on with the product. So PH, viscosity, odor, and appearance, these are the four main things you test on every sample in a stability test. There are some other types of evaluations that you can do, the weight loss. And so, what this involves is

weighing your sample in the final package before you test it, and then putting up at both room temperatures and at 45 C, waiting some time to see if that test the integrity of your packaging. For specialty products like aerosols, what you do is you put them up on stability and then at the appropriate checkpoint, you cut open the container and you can look on the inside of the can to see if there's any sort of corrosion going on. And you can also look for spray tests. And so, the packaging formulae interaction, that can also be type of evaluation that you do. Of course, there's performance testing to ensure that your product performs the way you expect it to. And this can get in more detail, you can go to panel testing where you have consumers test your products, or you can have clinical testing, or you may send your product out for some type of clinical test to ensure that it still performs the way that you expect it to.

Highly important are microbial tests, and there are two types of these. There's the contamination test, so once you make your formula you should test it for contaminations, and this takes about two to five days. And your plate count should be zero when you first start. Then there's also the Micro Challenge Test or the Preservative Efficacy Test, PET. And this is just to ensure that your preservation system works over time. What you do is you test a sample of your formula against, you know, five problematic microbes. And your sample should be able to kill all of these microbes effectively over time. And so, when you do this in the stability test you wanna test it at your initial formula, you wanna make sure you run it through both the contamination test and the micro challenge test. And then your 8 weeks at 45 C, you should also do a microbial challenge test of that formula just to ensure that your preservative system will be effective over the course of a year.

There's also freeze-thaw testing, this essentially is where you freeze a formula and then you thaw it out. And this is going to show whether your product will remain stable as it's shipped across the United States or across the world, where it can get really hot at night, or really hot in a day, or really cold at night and sometimes it'll freeze, it'll thaw. And so, if for emulsion formulas, a freeze-thaw is an excellence indication of whether your formula is gonna stay together. The way you do that is you freeze three samples overnight, you just put them in a freezer, and then you pull them out and you thaw them. And once they hit room temperature you can test them for PH, viscosity, appearance, odor. You repeat that cycle three times, and if your emulsion can go through this, you can have pretty good faith that your product is gonna be stable over a long time.

There are some additional tests that you could do, people are always looking for ways to accelerate their testing. And let me just be honest with you, there's really no good way to accelerate your testing. Now, there are some things that people try, the centrifuge, for example, is one, freeze-thaw testing is another. And these things do have their place, they can be really good at indicating that your product is unstable, if you can't pass the centrifuge test you can be pretty confident that it's not gonna be stable over time. But just because you do pass the centrifuge test doesn't mean that it will be stable over time. So, if you wanna feel good about yourself I would run the centrifuge tests, or run freeze-thaw tests, but don't rely on that as your only indication of whether your product is stable. Really, stability testing just requires time. There's also package testing that you can do, I mentioned the weight loss test, there's also drop test. So essentially, you fill your container in a bottle and you drop it at some certain height and you see if it cracks open. Also, you can do compatibility test where you cut up a bottle, you put into your formula and you see if any of the color leeches out. So these are other tests that you can do.

Let's look at the decisions, like I said, cosmetics should be stable for at least one year. For things like sunscreens and over the counter drugs, they're actually three years on there. And so, those are specific pharmaceutical testing that you can follow up. But for cosmetics, we target a one-year stability. And just a rough guide in the cosmetic industry, 8 weeks, a sample stored at 45 C for 8 weeks is approximately equal to one year of stability stored at room temperature. And so, that's a rough guideline that we go to, so if you can be stable at 45 C for 8 weeks, you can feel pretty confident that your product will be stable at room temperature for a year. Now, this isn't a guarantee but it's a pretty good guideline. If you're just changing the fragrance on a formula, sometimes you can shortcut this, you could do a four-week stability, you can make a decision about whether it's stable after four weeks. Usually, when you're doing stability testing at eight weeks, we would give a provisional pass of the product, but we wouldn't give a complete stability test pass until a 12-week mark. So if you can pass your stability testing after 12 weeks, you can have really good confidence that it'll be stable for a year. And, of course, we also did a 52-week verification test. And so, I always enjoyed picking out that 52-week sample because that was something I made like a year ago, and often, you can't even find that sample, but it's always a challenge.

All right, some other pass fails decisions, so how do you decide whether it's passed or fail? Well, if something is out of spec, technically it fails. And now, this is...now, what the specification is that's so to be determined upfront, but if it goes out of spec, technically it fails. Now, sometimes that's not enough to fail it, for example, if you're out of the PH range by like 0.1 unit or 0.2 units, sometimes that's not gonna really matter, then that could have just been because of the way your PH meter was calibrated. But you have to decide that, also out of spec, your viscosity could be out of spec, but if it's out of spec only a little bit, then it's up to you. But, certainly, when you have color changes, or you have texture changes, or you have odor changes, those will be all indicative of failing stability.

Let's just get through this one. You might wonder, should you do the stability testing yourself or should you outsource it. Now, for all formulators I say, you should be doing some stability testing. And there are times when it really makes more sense to do it your own, whenever you're making prototypes it makes much more sense to do it your own because you don't wanna outsource all of your prototype stability, it just doesn't make sense. When you're looking for a secondary source of a raw material, it makes sense to do your own stability testing. And if you're just changing the fragrance in a formula, it also makes sense to just do your own, it's not as complicated. But there are times when it makes sense to outsource this work. Now, whenever you're doing first production runs, for example, you want some official paperwork and you want something to point to. And it's really good if you could outsource that first production run of your product. If you're having space issues, you don't have a lot of storage space for all your samples and you're running a bunch of stability testing, then it does make sense to outsource your stability testing. And, of course, if you don't have the equipment to run stability tests, if you don't have multiple ovens, and a freezer, and a light box, then it also makes sense to outsource stability testing.

UL Prospector or UL, they actually have services, and stability testing is one of the services that they do. I provided a link right here where you can contact them and see if they make sense for you. I've never used them as a stability testing facility, but if you're looking to outsource it's something that makes sense.

All right, let's just sum up this. This is the stability testing made easy, right? Step one, you make your batches. Step two, you take your initial readings. Step three, you fill your containers. Step four, you put the samples in your accelerated conditions. Step five, is you evaluate these formulas at different checkpoints. And then, step six, after 8 weeks or 12 weeks you decide, is your formula stable? And if your formula is stable, everybody's happy, if it's not, you have to go back and reformulate.

Here's just a quick example for us to finish up here. Suppose you have a new fragrance adding to a skin lotion that's sold in an eight ounce bottle, the samples that you will require, I suggest both glass and packaging. So if it's a four ounce glass bottle, or a four ounce glass jar and an eight ounce bottle, you would need 33 samples of the glass, 33 samples of the package. You put up your control tests, you test your initial readings for PH, viscosity, odor, color, the bottle weight, and your microbial samples. And just to give you an example here, here is just a handy chart that I would use about the samples you put up. So wherever there is a green box here, that means you need a sample for it. So for this sample we need 4 samples for the 45 C at 2 weeks, 8 weeks, 4 weeks, 12 weeks. Then for 37 room temperature and 4 C we could go all the way out to 52 weeks. For your light box, it's your UV light, you want to go out to 12 weeks. You need some samples for freeze-thaw, do three cycles of that. You also want a microbial test, so at room temperature and 45 C at 8 weeks. And then finally your weight loss samples at room temperature and 45 degree C. So you can use this chart here to put up your stability samples, the checkpoints and the conditions under which they are.

All right, that brings us to the end here. Just as a summary of cosmetic stability testing, remember, it's predictive of a product's shelf life, one year minimum, is what we go for, for cosmetic products. I wanna remind you that it's not an exact science, right? And so, whether something passes stability or not, it's really subjective. And it's also not something where you can just say, well, if it's stable at 45 degrees for 8 weeks, then it's stable. It may or may not be stable, but that's just, that's closely, that's, you know, that's a general guideline that we follow. For cosmetics there are no specific regulations, at least in the U.S., that you have to follow stability testing protocol. The protocol that I've given you here is certainly one that you can follow, you can just write this up. You can say this is the stability testing protocol that we follow and it's perfectly a legitimate way to do stability testing, but you could come up with different ways. You could put at different temperatures, you could do it at different testing intervals, you could also do different tests. And so, it really depends upon you, but the key is have this stuff written down and follow what you've written down. The test design really depends on the type of product that you're testing. And it also depends on the type of [inaudible] used.

00:41:51]

And there's really numerous factors involved in deciding when you test and what you test, and I've gone through a lot of those here, in this talk today. And so, now that brings us to the end and I'm ready to take questions. Now, to do questions, there is a question box on the side, and feel free to put in some questions there. I'm gonna take a short two-minute break to allow you guys to start writing in your questions, and then we'll have a question and answer session for the rest of the hour.

Trey: Perfect, thank you, Perry, for that. And again, as Perry said, we will be taking questions. So do type those questions into our question box so here [SP] on the screen. While Perry does take a look at those we will address a couple of the questions we've received so far. A lot of questions coming in, where can I download these slides? Where can I get a copy of the presentation? We will be sending a copy of the recorded presentation and these slides to your email address, so actually be looking for that, sometime tomorrow, you should see that follow up email. So as Perry does continue to take a look at these questions that is one that we have come in so far. So again, a ton of great questions, so do continue to send us those questions by typing them in the question box located on your screen there. We'll let Perry have a few more minutes to take a look at these questions. Again, a big thank you to everybody for attending and some great material so far...

[00:43:13]

[Silence]

[00:43:31]

Perry: All right, well, we certainly have a number of questions here. Thank you so much, for everyone for your questions, and thank you also, for attending. I hope you found the information really helpful and useful. It's really based on my practical experience and this is the type of stability testing protocol that is followed by big companies in the cosmetic industry in United States. And so, if you pattern yourself after the information I shared here, you should have complete confidence that you're gonna be legal, and you're gonna be confident that it should be predictive of whether your formulas will remain stable on store shelves. Okay. So let's look at some of the questions here, first question I see here is from Carol, and she asks, "If the product pass the negative 10C to 45C 5 cycle test," so that's the freeze-thaw test, "do I still need to perform the temperature variations test? If the samples fail 10 C to 45 C cycles does that mean a fail stability?"

A great question, so, the first part of that is, yes. Even if you pass your freeze-thaw cycles, that's a good sign that it's going to be stable over time. But that is not sufficient to predict whether the formula will be stable over time. And so, you should still do the primary stability testing where you're putting it up at each of the conditions and over time. Like I said, you cannot shortcut stability testing, and, well, if you do a stability freeze thaw-test and something fails, then you can have confidence that it's probably gonna fail. Although, I should say that it may or may not fail over that time, but, certainly, if it passes a freeze-thaw, that does not mean it's gonna pass full stability testing.

All right, another question here. "Is a stability test at 45 C for 4 weeks a good indication for a product that is designed to have a 6-month shelf life?" That's a good question, and it really depends on your level of risk acceptability. But I would say, if it passes 45 C for 4 weeks, I would have confidence of giving it a 6-month shelf life. Although, I would always target a one-year shelf life for your products, because people certainly keep their cosmetics for longer than a year. And I should also say that, that timing, it starts from when you make the batch, so you make it on this day, it's only going to last one year after you make it. And often products are not sold for...you know, they don't even get in consumers hands after, you know, four or five months. But if it's a made to order product, and it's six months shelf life, probably 45 C at 4 weeks would be a good indication of the stability.

All right, next question here. "Should the test samples be placed in front of south-facing sun or north-facing sun?" We always do stability testing on a north-facing sun so it doesn't get direct sunlight but it gets indirect sunlight. But it really depends on your level of risk. I would go with indirect sunlight and the north-facing. So essentially, you don't want it to have direct sunlight because that's not exactly...well, that probably gives too much light.



"Do you leave the sample in the front of the window day and night, if so, will there be temperature fluctuation?" Yes, there will be temperature fluctuation, you have to do your best to control that temperature fluctuation. And, actually, what big companies do to get around this is that they use light boxes. So instead of putting it in windows you actually have a light box. But the company that I work with, when I first started there, we had a stability window where we put samples, and immediately, samples would freeze, there was no temperature control. So they would freeze, and so, sometimes the product might be stable or unstable and we really wouldn't know it. But if all you have is an uninsulated window, that could be good enough, but, ideally, you get a stability light box. And so, that would be your ideal situation. And if you're outsourcing your stability testing, certainly, you should insist that they have a light box where they're doing the test, not in a window.

Okay, just getting through a couple more of these questions. "Thank you, so much for the testing. Can microbe counts be used to perform the contamination test? And what is considered a passing result, bacterial count of 10 power 2, 3, or 4 per mil?" It's great question, my specialty is not in microbial challenge tests. There's a lab that I work with called Cosmetic Test Labs, I'd encourage you to check them out. But as far as I understand it, when you're doing contamination tests your count on that should be zero. So you shouldn't have any contamination upfront. If you do have contamination upfront, then your product, you know...you certainly don't wanna sell a product like that, and it's also indicative that your preservative system is not strong enough. So, I'm not sure what micro count is, I think that's some sort of brand name kit so I can't really comment whether that's good enough to use or not.

Here's a question from Hade, "How important is a stability test when the production is handmade in a small volume?" Well, handmade in a small volume, right now you're under the same regulations with the FDA as the big guys. And, you know, stability testing is a matter of product safety, so I would say it's highly important. It's certainly, critically important for microbial challenge test, if it's a handmade product, it can get more easily contaminated than products made under aseptic conditions at a manufacturing facility. So certainly, from a preservatives standpoint you should do stability testing for that. Now, as far as whether the product remains stable over time and if it's handmade, actually a smaller volume...if you're making products at a smaller volume, it's more likely that they're gonna be less stable, because those are harder to control a lot of the factors. So I would say, in that case, it's even more important. But if you're selling products and your customer does not expect the product to stay together very well, then, you know, they might not expect...you might just put "shake well before using" on there. But from a stability standpoint and a microbial standpoint, it's critically important there.

I'm just going through these questions, looks like we have people from France, welcome. All right, here's a question from Nancy, "Is it necessary to stability test your final product if you are using readymade and preserve bases? They have been stability tested by the supplier and adding actives to these?" Absolutely, it's important to do a stability testing on your final product if you're using in a readymade base. Any time you change that base formula by adding actives, or adding a different fragrance, you should be doing stability testing, because any additional ingredient that you add to your formula can affect the product's stability. And ultimately, what's required for the FDA and for testing is that you demonstrate that the formula that is on the shelves that you are selling has been stability tested. They don't care that the base was stability tested, that you have the base is stability tested it's... That gives you a [inaudible 00:52:22] you're going to have a...that you're going to have success and your product is going to be stable, but it's not a guarantee. And so, if you're using a pre-made base that's already preserved, you still need to do stability testing.

All right, let's see more questions here. "Hi Perry, I am trying to make a solvent micro emulsion I'm confused about the appearance, is a solvent micro emulsion transparent?" This is from Jennifer. Yes, micro emulsions if they're made correctly, they should be transparent. The reason is that if you're micro emulsion that means you have particle sizes below 100 nanometers, and our human eyes we can't see things that are smaller than 100 nanometers. And so, it should be clear. So if you made a correct micro emulsion, then it should be a clear formula.

This question is from Raphael. "Is it possible to predict the stability test centrifuge at different speeds and times?" I had talked about centrifuge testing. And centrifuge testing can give you an indication of stability but it's not a substitute for stability testing. So if it fails a centrifuge test it may or may not be unstable, but likely, it will be unstable if it fails the centrifuge test. However, if it passes your centrifuge test that does not mean

that in the future it's going to be stable. Centrifuge test, all that does is separate the particles in your emulsion, but what can happen over time is that you can have coagulation of those particles, and that's just something that's not gonna happen in a centrifuge test.

All right, this question is from Ann Lauren. "No specific requirements for cosmetic standard products, even safety assessment by safety advisor?" This presentation today was about stability testing, and so, when I said there's no specific requirements for cosmetics it's in terms of stability testing. The only requirement is that you are required to do stability testing. And from a safety standpoint, the other rule is that you can't sell unsafe products, and so, you have to have ways to demonstrate that your product is safe and it's safe with people. And so, having a safety advisor assess your formulas, it's a great idea. It's not a requirement for cosmetics in United States.

See a question here. "Hi, Perry, thank you for this webinar," this is from Catherine, "from the literature I've gone through, you test the sample every day for seven days once a week, for four weeks, and do you do micro biological test each time? If so, what's the recommended testing of specific microorganisms? Or are they titled, microbial count?" Let's see, so...I mean, whatever stability testing protocol you follow, I mean, there's the one you described here, testing every day for seven days, through a week, and then once a week for four weeks. That's certainly one way you could do it, I think it's overkill to test every day for seven days. We found that testing at the two-week mark, the four-week mark, eight-week mark was the best. And as far as microbial testing, we did microbial testing at eight weeks, and so, that was really what we focused on there.

All right, we have a ton of questions I'm gonna go... Let's see, I'll get to as many as I can. Thank you, all for the questions and if I don't get it by the end of the hour I will also try to answer them, and if I have your email, I can send it along.

"Is using one [inaudible 00:56:36] sample for over 12 weeks temperature okay, versus having one of each?" Yeah, you can do that. You can use one jar sample at the different conditions, and then just put it back at the condition. So what you do is you put up one jar at two weeks you take it down let it equilibrate, take your reading and then put it back for the next case. So yes, that's certainly something you can do.

"Going to the centrifuge test what speed and RPMs do you recommend for this kind of test?" I don't have a lot of experience with this and I don't encourage it. So I would say, you know, you just have to test it for your own samples.

"Is there a correlation between centrifugal speed test and the time, will the emulsion remain stable?" I would say, no, there isn't. Centrifuge tests are not a good way to predict whether the product is going to be stable over time. And so, while it's a good check it's not something I would suggest you do.

"Are the U.S. regulations quite different from the EU regarding the test?" No, they're not that different at all and the stuff that I presented here could be applicable to the EU too.

"Is a sun station the only test color stability, or does it affect other characteristics of..." The light affects two things mainly, mainly it affects the color, of course, but it also can affect the odor. Fragrances are highly sensitive to light, and so, that's really what you wanna look for, color changes and odor changes.

Next we have, "What if a product passes 8 weeks but fails 12 weeks, will I still have 1 year expiration?" No, probably not. If you're failing at 12 weeks, you probably will not have 1 year of stability.

All right, next question from Rasya [SP]. "Is it necessary to test for microbial for anhydrous products? Sometimes I see anhydrous products with preservatives but I guess it's optional." You probably could get away with not testing an anhydrous product for microbial contamination. If there's no water in it, you're not going to see any growth anyway. So, from a stability standpoint it doesn't make a lot of sense to do that. It does make sense to put preservatives in anhydrous products because they can get contaminated by the user and that can lead to microbial change, but from a stability standpoint, you don't really need to do that.

All right, let's see, we're coming up over the hour but I'll keep answering until I'm told to not answer anymore. "After each freeze-thaw, like, should we perform full testing such as PH, viscosity, or the organoleptic is sufficient?" I would say, after each freeze-thaw test, you should do PH, viscosity, appearance, and odor, so do those four things. It really doesn't take that much time.

"Well, would you design your stability program to accommodate 30 months shelf life requirements?" So for 30 months I would go out to, you know, at least, I would go, you know, 26 weeks, if you can pass 26 weeks of stability then you can have a lot of confidence. You might be able to make decisions after 12 weeks, but it really is gonna depend on your formulation but that, you know, that would be your guide there.

Another question about the centrifuge test, we've already answered that. And Casey, "Intended packaging materials say probably propylene is yet to be made available, can the stability results in glass be adopted?" Yes, you can use the glass stability testing information as a guide, and helps you have confidence. But ultimately, when you're launching your product you're gonna have to do a full stability test on that first production run. What's also a good idea is you can do a package compatibility test, so even if you don't have the bottles, if you know what the bottle is gonna be made out of, get a sample of what the bottle is gonna be made out of, put that in your formula, put it up at 45 C for 8 weeks and see if there's any color leaching or any change like that.

"Perry, the micro count is not entirely straight forward but I would be covering this..." oh, yes, that's Dean, Dean Godfrey and I are working on something for a seminar on preservation of your cosmetics. And so, look for that in future, probably by the end of the month we'll have that. Okay.

Next question, "How do you determine the migration between packaging and product?" This is pretty much...you just visually look at it, so you can see if there's any color leaching into your formula then you have a problem. You know, you have to look at it, you have to be very observant.

All right, we have Margaret asked, "Are there accepted standards for how long stability testing records are kept?" I would say you should keep them for at least a year. A lot of companies do a purging of records, you know, it really depends on your company. We kept them forever, then we did a seven-year purge, so we kept things for seven years. I think companies nowadays are keeping things for three years, and so, that would that would be my guess. I would say, do things digitally and keep them, you know, as long as you want because they're not taking up space. But there are no specific rules that I know of, for how long to keep the data.

"Do you believe that weekly stability observations are necessary for accurate stability records or are the time frames mentioned in the presentation sufficient?" I believe that the time frames mentioned in the presentation are sufficient. So if you follow those you should have confidence there.

"Are there guidelines to estimate a durability of five years?" If it passes room temperature... You know what? I don't really know. But, I mean, I guess, a room temperature sample stored for a year and it passes, it should pass for another year. I don't really know, I don't have a five year number.

"Are the stability tests the same for all kinds of cosmetic: solids, liquids, etc?" No, they're not all the same, it really depends on your formula, we did a li... For example, we had a Bomb, we would do a penetration test where you drop a thing into the sample and see how far it penetrated. And you do that instead of viscosity. And so it's gonna depend, for solids there aren't a lot of different testing you can do. You can look at appearance, odor, and then, you know, granular size, that sort of things. But it really depends on the type of formula that you're making.

"Should we perform stability under light box even for products in opaque packaging?" Great question, yes, you should do that, probably you won't see anything, but there is a chance that the light gets through your packaging and it affects your formula. So, yes, you should you that.

"Initial testing, what steps are taken at what temperature and what time after?" For initial testing it should be done at 25 C, as soon as it equilibrates. So within 24 hours of you making the batch, 25 C and then, there you go.

"We'll be ISO audited, how important is to have a regulation source in the protocol?" I think the important thing in your protocol is that you have your protocol written out, you demonstrate that you are following it. And I do not know all the ISO-specific rules, and so, you might want to have a consultant specifically talk to you about helping you get through ISO auditing.

"Microbial challenge test, what are the five problematic microbes, and can you tell us more details on how to [inaudible 01:05:05]." So I have that written up on my blog, I don't have the details of those microbes now, but I'd encourage you to go to Chemist Corner and look up microbial challenge test. And I have written in there the specific microbes that are tested for. I don't encourage you to do this yourself, if you're focused on formulating, it's gonna be really hard to set up agar plates, have your ovens, keep things aseptic. You should just outsource this kind of work. You can do contaminations tests yourself, but preservative efficacy tests, I would encourage you to outsource that, a place like Cosmetic Test Labs is great for that.

All right, just a few more questions that I'll answer, and I'm sorry if I don't get to them all. Okay, let's see, we have here, "If the product has a specific color how can you test the stability of it? Is there any procedure to accelerate the possible changes of the color or the procedure, the 45 test, can we use this checkpoint?" So the color testing, what you wanna do is your 4 C sample is your color control, although you can also have a color control using like Minolta Color Meter for when you first make the batch. Or you can take a picture of it and then compare the pictures. But that's how you test the stability of specific colors, it's, you know, you use your eye and you just compare the colors. It takes a little practice to get good at that. And you might have a couple of people do it if there's significant color changes. But it's really just a subjective test.

Anna [inaudible 01:06:46], "Is the leak test...does that need to be done if you're wor...?" Yes, a leak test should be done if you're worried about your package leaking. So whenever you have new packaging, and new caps, then it's a good idea to do leak testing.

"How do we predict about decomposition process possibly in our formulas, products and cosmetics?" I'm not exactly sure what that means, but I think if you run a stability test and it remains within the specifications that you set up at the start, then the product, you can be confident that is not decomposing and it's going to remain stable. Now, as far as decomposition, that's an environmental testing, if that's what you're talking about, I don't really know about the environmental impact testing, that's a different kind of testing. And so, I guess that would be a different seminar, sorry.

"Where can you get a light box?" Look up accelerated lighting stability testing on Google and you should be able to find a variety of sources.

"How long do you run the sunlight stability?" We did light stability for 12 weeks, so you could check it at 2, 4, 8, and 12. After 12 we didn't do that.

Someone asks, "What is room temperature equivalent of a sample for 50 C for two weeks, is there a way to calculate this?" No, not really, because 50 C there's just a lot that can go different there. But if you wanted, yes, 50C at two weeks, maybe it's stable for a month. But really, it's highly dependent on the type of formula that you have.

"Do you still need to sunlight test in package formula?" I think we are already went through that and said, yes, it's a good idea to do that.

"I'm formulating a topical cream using natural ingredients. Natural ingredients usually last less than one year. Do you have a recommendation of stability test that's suitable for this kind of formulation?" The test that I just showed you here is predictive for one year, if it passes that, you're in good shape, if you're looking to shortcut that, I don't have any recommendations for that. Now, I would say, you're using natural ingredients you have to decide how do you determine that it's not stable for a year, or how long they last. You have to have some specific tests that it's not passing that you want to continue. So I'd need more details about like what is making you think that it's not passing. But really, the presentation that I gave here, the stability protocol, I'd encourage everyone whether you're doing natural formulations or not, to follow that. "For emulsions, whose face [SP] can be diluted, is dilution of various degrees an indication of instability?"



No, not really, there's no shortcut, you can't dilute the product and use that as a determination of stability, there's really no shortcut there. It could be a good indication of that, maybe predictive, but it really is dependent and I wouldn't encourage that kind of shortcutting.

"What are the checkpoints as far as packaging goes?" Well, for... If you're doing weight loss, it's room temperature, 45 C at 8 weeks, and seeing that.

Asking about over the counter formulation, "Is there research where we can get many formulations with different actives...?" Yes, you can go to the FDA. I actually, on chemistcorner.com, I wrote a post called, "Cosmetic Products That Have FDA Monographs." Look that up there. And I have links to all of the specific OTC formulation monographs, and then you can find that information there. "Do you have any recommendation for green tea color change stability, to help stability stop the instability?" Probably what's happening is that the color is changing, then you could use an antioxidant in there and use darker packaging that could help stop your stability issues.

"The presentation mainly discussed stability testing in the U.S., considered differences in temperature and humidity conditions, will it be the same if conducted in a tropical country?" Great question, yes, it'll be the same if you're doing the temperature. As far as temperature goes, yes. But you might also want to control for humidity because that can affect your packaging, but if it's in packaging, really, humidity shouldn't affect it. So this type of testing should work in tropical countries. And, in fact, we sold our products in tropical countries and this is what we used.

Another question, "Which bacteria do you use for micro stability?" We went through that, like I said, I've written something up on Chemist Corner, so I think I've answered that one already.

"How do you use an inherently unstable ingredient such as dihydroxyacetone? How does it affect the stability approach?" Well, when you're formulating DHA products they have to remain functional over time, and so, you can use the same protocol just when you're evaluating whether it's stable or not. You have to run a performance testing to make sure that it still works.

Someone asks, "If keeping 50 C for 7 days indicative of stable for 3 years?" I'm gonna go with no, it would be great if it were, but no, 50 C for 7 days will not predict 3 years.

All right, it looks like we're coming up on 10:15, which is 15 more bonus minutes than you were expecting. I'm sorry, if I didn't get to everyone's questions. Boy, there were a lot of questions. Thank you so much to everyone who has submitted questions. And I'll try to answer as many of these as I can, if I have your email I could send that. Or, if you want to send me these questions directly I'm at thejoggler@gmail.com. Thanks, again, I really appreciate you taking the time to listen to this presentation, and I hope you found it worthwhile. If you have any other questions you can go check out my website chemistcorner.com. And, of course, UL Prospector is a great resource for finding information about cosmetic raw materials, cosmetic technology, and they do a great job. And thanks, again, so much, for UL Prospector for putting this together. And thank you, everyone, for attending. I'm Perry Romanowski, now, go make the difference.

Trey: Perfect, a big thank you to Perry for a really great and informative presentation. As I did mention before, Perry is one of our expert authors. So we do encourage you to check out some of his other articles on the Prospector knowledge center. Again, we will be sending everybody a link of the recording of the presentation and the slides, so you can review the information again, and also share that with your company. So do check your inbox for that email link tomorrow. Again, a big thank you for everybody for...