

NR 149 Changes ~ (Proposed)

For Registered Base 4 Labs

DNR Certification Services

written, directed, and produced

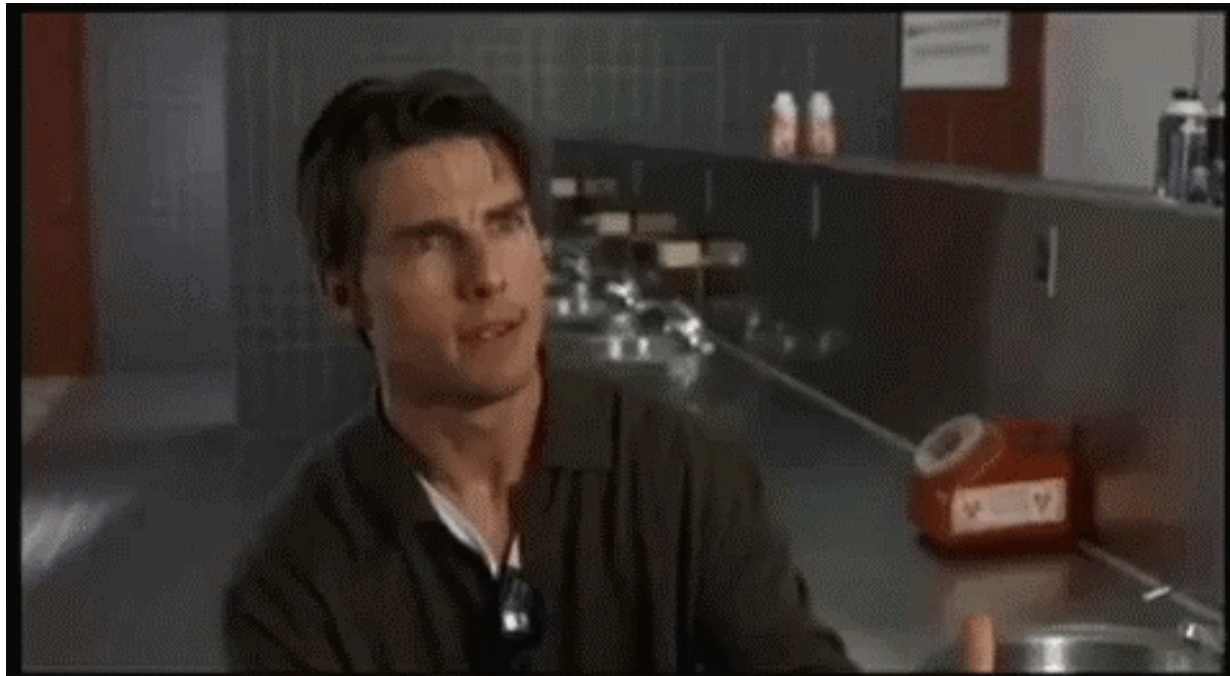
by Tom Trainor



Courtesy Wake-up Song



☺ **WE ARE HERE TO HELP** ☺



What is NR 149?

Rules

that govern lab accreditation



Why do we need to muck with it?





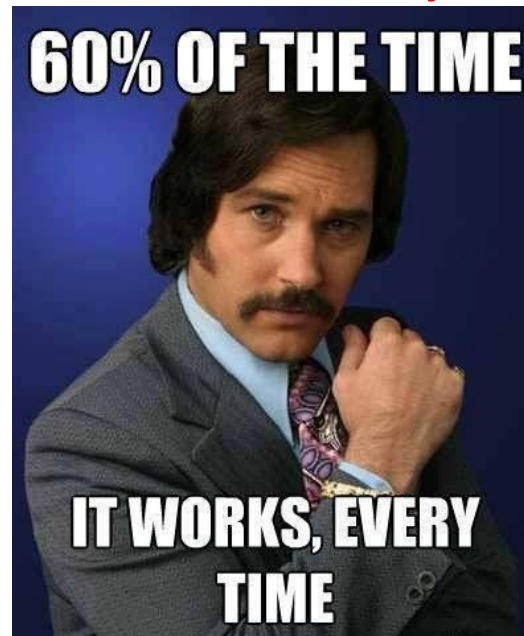
- Errors
- Housekeeping
- Clarifying language
- Some things needed to be changed
- Some things needed to be removed
- Some things need to be added

One of the errors applies to this presentation

Currently, two non-zero standards are required for ISE initial calibration (curve).

New Code

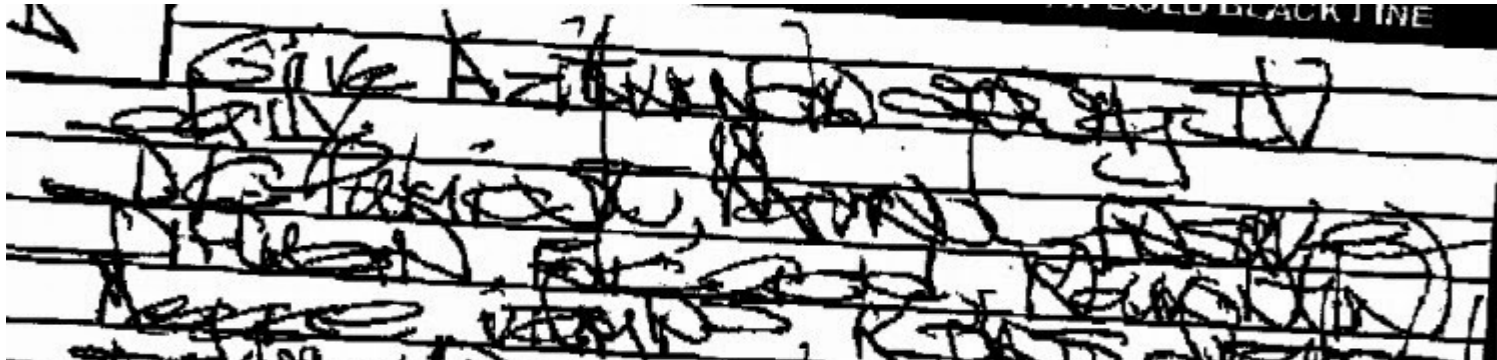
Three non-zero standards are required for ISE initial calibration.





- MOVED OR COMBINED PARTS OF CODE TO MAKE SENSE
- ELIMINATED REDUNDANT LANGUAGE
- SOME “NOTES” WERE CODIFIED
- SAID GOODBYE TO UNIMPORTANT “NOTES”
- REMOVED INTERWEB LINKS

- If 2 PTs fail in a row, submit a corrective action plan to the Department. We clarified this to the intent which was not “submit” but just “prepare”.
- We clarified in a “Note” that we understand the analyst may not always be able to identify the exact cause of isolated QC failures.
- Legible documentation includes the quality and permanence of the record – can a person decipher the numbers and letters written.



- Weights must be “suitable” – \geq class 2 equivalent
- When specific temperatures are required, the operating temperature shall be checked and documented (TSS, BOD).
- When do we need to do a new curve (just once)?
 - Instrument undergoes non-routine maintenance
 - The expected behavior of the instrument changes
 - A CCV (known standard) fails – AND
 - Immediate analysis of a CCV under the same conditions fails.
 - CA taken does not result in a passing CCV

- CCV (known standards) analyzed before analyzing any environmental samples or batch QC samples (i.e. method blank)
- LOD and LOQs must be adjusted for:
 - Sample dilutions performed just before analysis
 - Sample dilutions performed by using a smaller amount of sample at the beginning of the test

Proficiency Testing samples due date was changed

OLD DATE = 8.15 EACH YEAR

NEW DATE = 8.31 EACH YEAR

That means 16 more days to get your PT samples to the
Department



MORE HELP?

What else did we do to make your lives better



- Certificates shall be displayed CONSPICUOUSLY
- Cap on technology fees is removed

Average Municipal WWTP: \$61 savings

Average Industrial WWTP: \$46 savings

- Requirement to include a standard in the curve near the LOQ
- Quality Manual
 - Organization and management structure
 - List of major analytical instruments and support equipment
 - Procedures for reviewing analytical data and reporting results

- If immediate reanalysis resolves a QC failure corrective action documentation is not needed
- No need to document the date of receipt for standards and reagents anymore
- Expiration dates are not required when one is not provided by the vendor
- Removed references to the “analytical methods manual” which was confusing – it is just your SOPs
- Allowance to extend the expiration date of standards and reagents if you can prove it – only use for qualitative reason once expired

- SOPs
 - analytes
 - Applicable matrices
 - Method sensitivity
- All references to COC
- Requirement to check balances with a 1 mg and 1 g weight monthly
 - just one weight in the expected range of use
- Requirement to demonstrate that the bottles used and carboys are free of contamination – NO CARBOY BLANKS

- No need to perform a new curve annually for total phosphorus or ammonia using colorimetric technology



- Requirement to perform a sample dilution at the lowest required dilution to obtain a response within an a curve
- Removed all references to matrix spikes, matrix spike duplicates, replicates, and QCS (method)

MORE THAN 15 REQUIREMENTS REMOVED TO HELP YOU



Now let's talk about what we added

Laboratory Ethics – these things are prohibited

- Fabrication, falsification, misrepresentation of data
- Time traveling i.e. recording of dates improperly
- Unwarranted manipulation of samples, software, or analytical conditions
- Concealing or failing to report a known improper or unethical behavior or action associated with sample analysis



Method modifications

- When a lab incorporates a procedure that is neither expressly permitted or prohibited by the method, the Department will assess the scientific validity of the procedure to determine if the procedure is within the scope of the method. The underlying chemistry and both the ruggedness and robustness of the method shall remain unchanged. The Department may seek the advice of council in making this determination.



PT requirements when 3 WP PTs fail in a row

- The laboratory will need to perform 2 consecutive passing PTs from 2 unique WP PT studies received at least 10 business days apart and perform them in separate batch's.

PT requirements

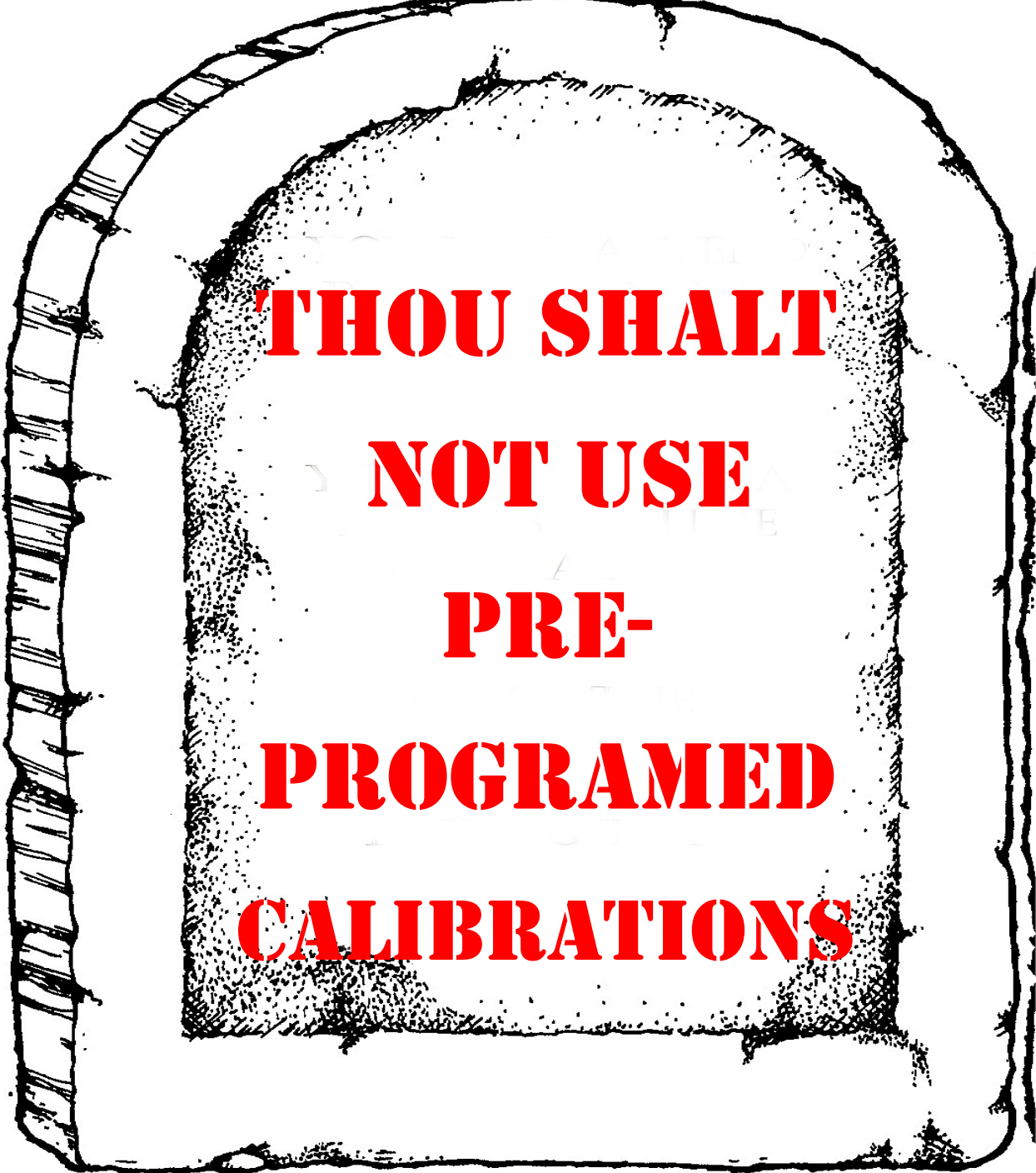
- Labs must report a proper method code for each PT result or the PT fails and a new PT is required

Corrective Action

- Root cause analysis shall be performed when there is recurrence

Standard and reagent containers shall be labeled with an expiration date, chemical name and concentration.

- **SOPs**
 - Need to include both preparation and analysis.
 - How are interferences treated
- **Currently NIST traceable weights can be used to verify other expired weights**
- **A method blank may not be used to zero the instrument for colorimetric technologies – zero with reagent water**
- **When required by method, the lab shall process each calibration standard, CCV, and ICV in the same manner as samples**
- **If an LCS is also used as a CCV, the CCV limits are to be used**



**THOU SHALT
NOT USE
PRE-
PROGRAMMED
CALIBRATIONS**

- **Excluding calibration points**
 - The rationale for the exclusion is documented.
 - Regulatory limits can still be met
 - The linear range is limited to the highest standard.
- **Initial Calibration (your curve)**
 - If the laboratory decides (this is optional) to utilize evaluation of the x-intercept into determining the acceptability of a new curve, **then the absolute value of the x-intercept must be less than the LOD**
 - If the laboratory decides (this is optional) to utilize evaluation of calibration point residuals (read backs) then **the recovery for each point is 90-110% except for the lowest point which is 80-120%.**

$$Y = mx + b$$

x = concentration

y = response (absorbance)

m = slope

b = y-intercept

We want to know what the instrument will read when the absorbance = 0

When the absorbance = 0 (no instrument response), $Y = 0$

$$Y = mx + b$$

$$0 = mx + b$$

Solving for x we have

$$-b = mx$$

$$-b/m = x$$

$$x = - (y\text{-intercept})/\text{slope}$$

$$Y = mx + b$$

x = concentration

y = response (absorbance)

m = slope

b = y-intercept

$$x = - (y\text{-intercept})/\text{slope} = x\text{-intercept}$$

Don't worry about the y-intercept (x = 0)

We care about the x-intercept because the x-intercept needs to be lower than the MDL or you already have a concentration greater than your MDL without any instrument response. Chances are your method blank will not pass if this is the case.

$$Y = mx + b$$

x = concentration

y = response (absorbance)

m = slope

b = y-intercept

$$x = - (y\text{-intercept})/\text{slope} = x\text{-intercept}$$

What can I do if my x-intercept is greater than 0?

- *Assess your initial calibration (curve)*
 - ✓ Are my high standards over weighing the low end
 - ✓ Is the low end of my curve properly defined
 - ✓ Is the lowest standard in my curve too high
- *Is my LOD unrealistically low*

- **Limit of Detection (LOD)**

- The new procedure is required.
- Regulatory limits shall be met
- The LOD shall be adjusted when the sample amounts differ from those used for the LOD determination.



- **Limit of Quantitation (LOQ)**

- Regulatory limits shall be met
- The LOQ shall be equal to $10/3 \times \text{LOD}$ or equal to the lowest concentration standard in your curve

- **Reporting Limits**

- Applies to BOD and is equal to 2 mg/L for 300 mL
- Applies to TSS and is equal to $1000 / (\text{sample volume in mL})$

- **Method Blanks**

- One per batch up to 20 samples, 21 samples require 2
- The absolute value of the blank must be less than the LOD, or 5% of the regulatory limit, or 10% of the sample concentration
- Blanks should not be “too negative”

- **Lab Control Sample (LCS)**

- One per batch up to 20 samples, 21 samples require 2
- The lab may use the CCV limits for the LCS instead of generating their own in-house control limits

- **ICV is required for ISE**

Technology: BOD, cBOD

- Maintain room at 17 to 23 °C during BOD analysis
- Use the theoretical saturation point, based on temperature and barometric pressure, on each day of analysis, to assess supersaturation
- [note – when barometric pressure and temperature measurements are available on the DO meter take these measurements from the DO meter]
- Assess (and treat) supersaturation each day of analysis
- Seed samples that have been disinfected or inhibited
- Do not add nitrogenous demand inhibitor to GGA, method blanks, or seed material

BOD (continued)

- Dilutions to expect 2 mg/L depletion
- Equipment with multiple D.O. probes – calibrate each and sample records shall be traceable to each
- Optical D.O. probes – calibrate each day
- Barometric pressure – local, not adjusted to sea level
- Chlorine strips must test down to 0.1 mg/L
- Wide bore pipet tips
- GGA – no averaging, each one must pass or qualify the data
- Method blanks – no averaging, each one must pass or qualify the data

Technology: Colorimetry

- Initial calibration (curve) – use calibration blanks, be sure to assign the measured response **(not 0 automatically because it is a blank)**
- TP Digestion, closed vial – $150 \pm 2^{\circ}\text{C}$ for at least 30 min
- Do not dilute samples after adding color reagent

Technology: ISE

- Initial calibration (curve) each analysis day
- Use an Initial Calibration Verification (ICV) to verify each initial calibration (curve), except for pH

Technology: Gravimetry Assays – Residue (Solids, TSS)

- The laboratory may not use Buchner funnels or Gooch crucibles for determination of TSS

Buchner



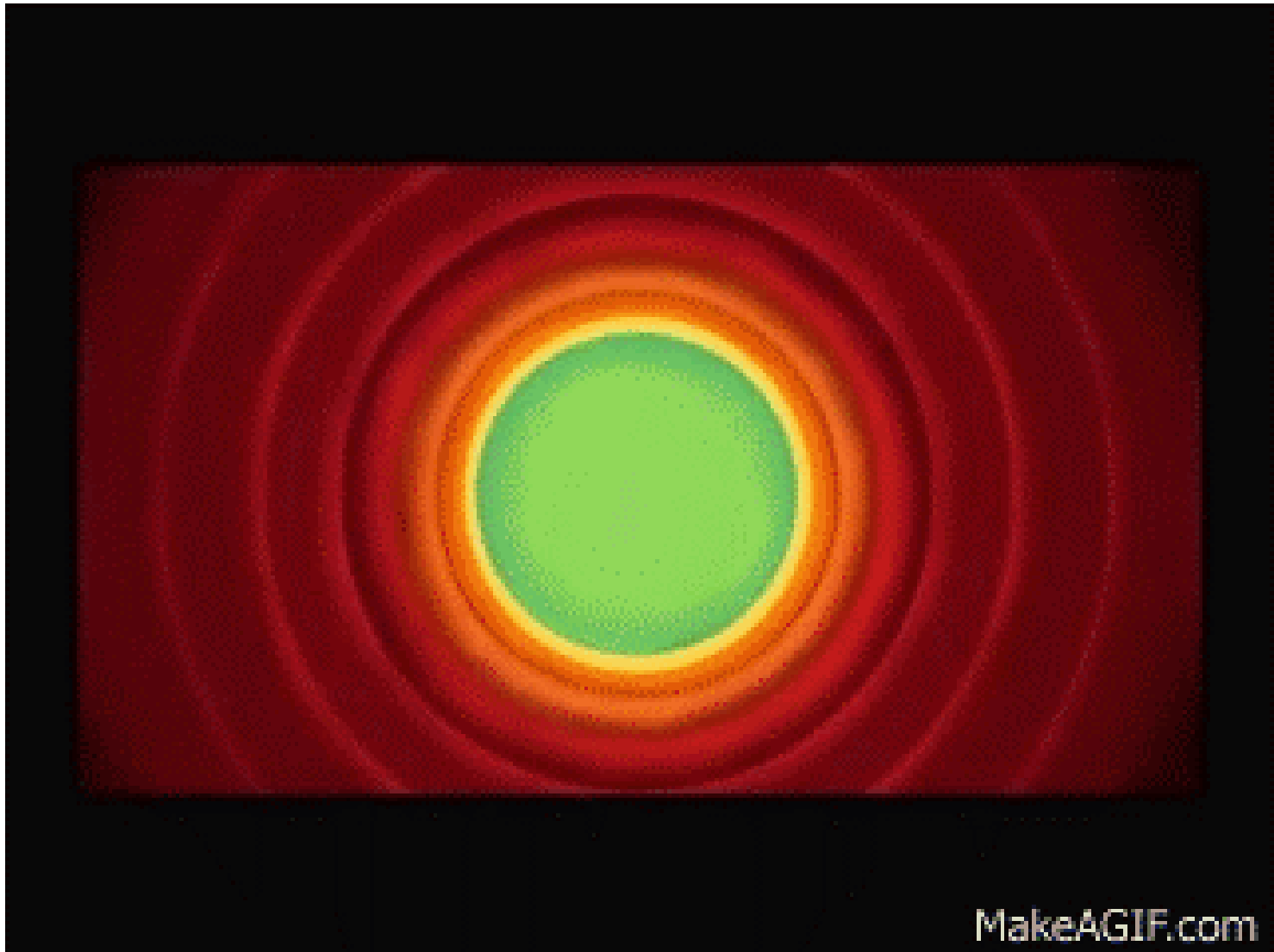
Gooch



- Use wide-bore pipets

- December 12, 2018 - To NRB Board for authorization to adopt the revised rule
- **January - February 2019 – Obtain Legislative approval**
- September 1, 2019 – Target effective date





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